

## IR-4 Project Crop Library for Protocol Sampling Requirements

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<b><u>Vegetable, root and tuber, group 1</u></b> <u>Beet (Garden) or Chicory</u> <u>Beet (Sugar)</u> <u>Carrot</u> <u>Ginseng</u> <u>Horseradish</u> <u>Potato</u> <u>Radish</u> <u>Rutabaga</u> <u>Sweet potato</u> <u>Turnip</u>	<b><u>Fruit, citrus, group 10-10</u></b> <u>Grapefruit</u> <u>Lemon</u> <u>Orange or Tangerine</u>	<b><u>Herb group 25</u></b> <u>Basil</u> <u>Chives</u> <u>Cilantro or Coriander</u> <u>Dill</u> <u>Dill (Seed)</u> <u>Mint</u> <u>Parsley</u> <u>Rosemary</u> <u>Stevia</u>
<b><u>Vegetable, bulb, group 3-07</u></b> <u>Garlic</u> <u>Onion (Dry Bulb)</u> <u>Onion (Green) or Chive or Leek</u>	<b><u>Fruit, pome, group 11-10</u></b> <u>Apple</u> <u>Mayhaw</u> <u>Pear</u> <u>Apple and Pear (postharvest)</u>	<b><u>Oilseed group 20</u></b> <u>Camelina</u> <u>Canola</u> <u>Flax</u> <u>Hemp</u> <u>Safflower</u> <u>Sesame</u> <u>Sunflower</u>
<b><u>Vegetable, leafy, group 4-16</u></b> <u>Dandelion</u> <u>Endive</u> <u>Lettuce (Head &amp; Leaf)</u> <u>Lettuce (Greenhouse)</u> <u>Greens (Mustard) or Collards</u> <u>Kale</u> <u>Parsley</u> <u>Spinach</u> <u>Swiss Chard</u> <u>Watercress</u>	<b><u>Fruit, stone, group 12-12</u></b> <u>Cherry</u> <u>Peach</u> <u>Peach (postharvest)</u> <u>Plum</u>	<b><u>Fungi, edible, group 21</u></b> <u>Mushroom</u>
<b><u>Vegetable, brassica, head and stem, group 5-16</u></b> <u>Broccoli</u> <u>Cabbage or Chinese Cabbage</u> <u>Cauliflower</u>	<b><u>Berry and small fruit group 13-07</u></b> <u>Blueberry</u> <u>Caneberry or Currant</u> <u>Cranberry</u> <u>Gooseberry</u> <u>Grape</u> <u>Juneberry</u> <u>Kiwifruit</u> <u>Strawberry</u>	<b><u>Vegetable, stalk, stem, and leaf petiole, group 22</u></b> <u>Asparagus</u> <u>Celery</u> <u>Kohlrabi</u> <u>Rhubarb</u>
<b><u>Vegetable, legume, group 6</u></b> <u>Bean (Dry Shelled) with forage and hay</u> <u>Bean (Edible-Podded) with Forage</u> <u>Bean (Succulent Shelled) or Bean (Lima) or Edamame</u> <u>Bean (Succulent Shelled) with forage</u> <u>Lentil</u> <u>Pea (Dry Shelled) with forage and hay</u> <u>Pea (Edible-Podded) with Forage</u> <u>Pea (Dry Shelled) with Forage and Hay</u> <u>Pea (Succulent Shelled) with forage</u> <u>Soybean (without processing)</u>	<b><u>Nut, tree, group 14-12</u></b> <u>Almond</u> <u>Hazelnut</u> <u>Pecan</u> <u>Pistachio</u> <u>Walnut</u>	<b><u>Fruit, tropical, group 23</u></b> <u>Date</u> <u>Fig</u> <u>Guava or Carambola</u> <u>Papaya</u> <u>Olive</u>
	<b><u>Grain, cereal, group 15</u></b> <u>Barley</u> <u>Chia</u> <u>Corn (Field)</u> <u>Corn (Sweet)</u> <u>Millet</u> <u>Oat</u> <u>Quinoa</u> <u>Rice</u> <u>Sorghum (Grain)</u> <u>Sorghum (Sweet)</u> <u>Wheat</u>	<b><u>Fruit, tropical, group 24</u></b> <u>Avocado or Mango</u> <u>Banana</u> <u>Dragon fruit</u> <u>Lychee</u> <u>Passionfruit</u> <u>Persimmon</u> <u>Pineapple</u> <u>Pomegranate</u> <u>Prickly Pear Cactus</u> <u>Rambutan</u> <u>Sugar Apple</u>
<b><u>Vegetable, fruiting, group 8-10</u></b> <u>Eggplant</u> <u>Okra</u> <u>Pepper (Bell &amp; Non-Bell)</u> <u>Tomato</u> <u>Tomato (Greenhouse)</u>	<b><u>Grass, forage, fodder and hay, group 17</u></b> <u>Grasses</u>	<b><u>Miscellaneous</u></b> <u>Artichoke</u> <u>Coffee</u> <u>Hops</u> <u>Peanut</u> <u>Sugarcane</u>
<b><u>Vegetable, cucurbit, group 9</u></b> <u>Cantaloupe or Watermelon</u> <u>Cucumber</u> <u>Cucumber (Greenhouse)</u> <u>Squash (Winter) or Squash (Summer)</u>	<b><u>Animal feed, nongrass, group 18</u></b> <u>Alfalfa</u> <u>Alfalfa (grown for seed)</u> <u>Clover</u>	<u>Taro</u> <u>Ti</u> <u>Wasabi</u>



## **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot.

Each sample should be representative of the entire plot (except plot ends).

**@@** Each sample should be collected during a separate run through the entire plot. If this cannot be done due to harvesting equipment or other factors, contact the Study Director to discuss.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates.

After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**\*\*When using IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

### **18.1 All field trials except decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA		
B	01	Untreated	NA		
C	02	PPP	X (+1)		
D	02	PPP	X (+1)		

### **18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA		
B	01	Untreated	NA		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		
	02	PPP	X (+1)		

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

**Vegetable, root and tuber, group 1**

## Beet (Garden) or Chicory

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days following the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 plants per sample. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Collect a minimum of 4 lb of roots (but preferably not more than 6 lb) and 2 lb of leaves (but preferably not more than 3 lb). Roots and tops (leaves) may come from the same plants, or from different plants. Avoid sampling from plot ends. If the roots are more than about 4 inches (10 cm) in diameter, then they should be cut into 2-4 smaller pieces, and all of the pieces should be retained for the sample. If a leaf sample weighs much more than 4 lb, the sample may be reduced by cutting each plant top longitudinally to the root crown, and retaining one half for the sample. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

If excessive soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE ROOTS.**

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
RA	01	Untreated	NA	12 roots / 4 lb.	Roots
RB	01	Untreated	NA	12 roots / 4 lb.	Roots
RC	02	PPP	X ( $\pm 1$ )	12 roots / 4 lb.	Roots

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RD	02	PPP	X (+1)	12 roots / 4 lb.	Roots
TA	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
TB	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
TC	02	PPP	X (+1)	12 plants / 2 lb.	Tops (Leaves)
TD	02	PPP	X (+1)	12 plants / 2 lb.	Tops (Leaves)

## 18.2 Decline trial XX@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
RA	01	Untreated	NA	12 roots / 4 lb.	Roots
RB	01	Untreated	NA	12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
TA	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
TB	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples RC, RD, TC, and TD.

## Beet (Sugar)

### 17. RESIDUE SAMPLE COLLECTION:

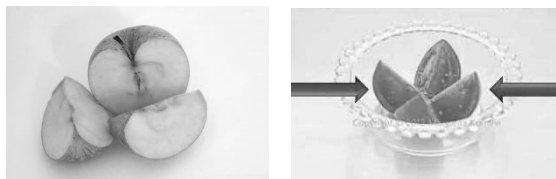
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days following the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect 12 plants per sample. Each sample should be collected during a separate run through the entire plot.

Roots and tops may come from the same plants, or from different plants. Avoid sampling from plot ends. Dead or senesced leaves should be removed from the tops samples. Top samples should include the crown, separated from the root.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Excessive soil must be removed from the roots by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE ROOTS.

The root samples should be reduced by cutting each root longitudinally into eighths and retaining opposite slices. If the retained slices are more than about 6 inches (15 cm) long, then they should each be cut into two smaller pieces, and both of these pieces should be retained for the sample. The “tail” and the “crown” of the root do not have to be retained. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.



Longitudinal cuts / Opposite slices

If needed, the sugar beet tops can be reduced in the following way:

Select 12 tops and separate them into 3 groups of 4 tops each. Divide the leaves (entire above-ground portion) of each sugar beet top into 3 approximately equal lengths. Retain top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 tops are included in each sample.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

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Samples should be collected in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID Number, Test Substance (chemical name listed in Section 15) complete sample ID (See Section 18.3), and harvest/sampling dates. See Protocol Section 19.2 for handling and shipping directions. Ship the large samples within 1 day of sample collection, if possible, as “fresh samples” to the processing facility.

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18.1 All Trials Except Decline Trial XX@@:

## 18.2 Decline trial XX@@:

[illegible]

	02	PPP		12 plants	Tops (Leaves)
	02	PPP		12 plants	Tops (Leaves)
	02	PPP		12 plants	Tops (Leaves)
	02	PPP		12 plants	Tops (Leaves)

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples RC, RD, TC, and TD.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX@@ only (Univ. of Idaho processing)

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
PA	01	Untreated	NA	350 lb.	Roots
PT	02	PPP	X (+1)	350 lb.	Roots

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX@@ only (GLP Technologies processing)

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
PA	01	Untreated	NA	150 lb.	Roots
PT	02	PPP	X (+1)	150 lb.	Roots

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT : (Samples not for processing)

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and residue sample collection utilize procedures that minimize sample degradation by keeping samples as cool as possible. It will not be necessary to freeze the samples.

Ship samples to the processing facility on the day of sampling or the day after if the samples have not been frozen. If the samples are frozen at the field site, immediate shipping will not be required (ship within 14 days of harvest). Ship by freezer truck or overnight air express. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples. Send samples for processing to: @@@**

## **19.3 PROCESSING DIRECTIONS:**

Maintain samples frozen unless processing is to be done immediately. Prior to processing, remove a minimum of 12 roots each from the treated and untreated samples. If necessary, reduce sample bulk by cutting each root into quarters and retaining opposite quarters. Label according to directions in Section 17 and store frozen.

Process the remaining roots into refined sugar, dried pulp, and molasses using simulated commercial procedure. **Divide each sample of molasses into separate containers of 50-150 grams. It is also acceptable to divide other processed samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Provide a complete description of the procedure (SOP acceptable). Send samples to: @@@

## **19.4 PROCESSED SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>MINIMUM SAMPLE SIZE OR APPROX. WEIGHT RANGE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	12 roots / 4 lb.	Roots
GT	02	PPP	X (+1)	12 roots / 4 lb.	Roots
RSA	01	Untreated	NA	2-4 lb.	Refined Sugar
RST	02	PPP	X (+1)	2-4 lb.	Refined Sugar
DPA	01	Untreated	NA	2-4 lb.	Dried Pulp
DPT	02	PPP	X (+1)	2-4 lb.	Dried Pulp
MA	01	Untreated	NA	2-4 lb.	Molasses
MT	02	PPP	X (+1)	2-4 lb.	Molasses

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

20.01- Names of all personnel conducting specific research functions

20.02- Amendments and deviations from protocol and standard operating procedures

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- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date root samples received
- 20.1.04- Storage temperatures until root samples are processed into refined sugar, dried pulp, and molasses
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into refined sugar, dried pulp, and molasses
- 20.1.07- Storage temperatures of root, refined sugar, dried pulp, and molasses samples until shipped
- 20.1.08- Date root, refined sugar, dried pulp, and molasses samples are shipped to analytical laboratory

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A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Carrot

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 plants from approximately 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove tops, retaining the roots for the sample. Each sample of carrot roots should weigh a minimum of 4 lb (but preferably not more than 6 lb). Cut the the carrots with a clean knife into at least 2 smaller segments not longer than approximately 6 inches (15 cm) and retain all of the segments for the sample. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

If loose soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse with a minimal amount of clean water, or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE ROOTS.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials Except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 roots / 4 lb.	Roots
B	01	Untreated	NA	24 roots / 4 lb.	Roots
C	02	PPP	X (+1)	24 roots / 4 lb.	Roots
D	02	PPP	X (+1)	24 roots / 4 lb.	Roots

#### 18.2 Decline trial XX@@:

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 roots / 4 lb.	Roots
B	01	Untreated	NA	24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots
	02	PPP		24 roots / 4 lb.	Roots

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Ginseng

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Each sample should be collected during a separate run through the entire plot. If this cannot be done due to harvesting equipment or some other factor, contact the Study Director to discuss. At X ( $\pm 1$ ) days after the last application collect roots from at least 12 separate areas of the plot per sample, simulating commercial practices.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Collect adequate root samples so as to yield a minimum of 2 lb. dry weight. Avoid sampling from plot ends.

Lightly rinse the roots with clean water (do not scrub), simulating commercial practices, or dip the root briefly in a bucket of water. DO NOT SCRUB OR RUB WHILE RINSING AND DRYING THE ROOTS.

After harvesting and washing, dry the roots in a drying facility to approximately 70-90% dry matter (approximately 10-30% moisture content) simulating commercial practices. (The percent dry matter may be estimated.) Document the drying process in the Field Data Book, including temperatures and drying time.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

**All trials except decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Dried Roots
B	01	Untreated	NA	2 lb.	Dried Roots
C	02	PPP	X ( $\pm 1$ )	2 lb.	Dried Roots
D	02	PPP	X ( $\pm 1$ )	2 lb.	Dried Roots

#### 18.2 Decline trial XX@@:

SAMPLE	TRT#	TREATMENT	DAYS AFTER	MINIMUM	CROP
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ID			LAST APPLIC.	SAMPLE SIZE	FRACTION
A	01	Untreated	NA	2 lb.	Dried Roots
B	01	Untreated	NA	2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots
	02	PPP		2 lb.	Dried Roots

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Shipment of frozen samples will be by freezer truck or express shipment. Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records

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- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- Data collected during sample drying, including oven temperature (may be approximate) and length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## Horseradish

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots,, collect at least 12 roots. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove tops, retaining the roots for the sample. If excessive soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method).

If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE ROOTS.

Reduce gross sample weight to a minimum of 4 lb (but preferably not more than 6 lb) by cutting each root longitudinally into halves or quarters, if necessary. Retain at least one half or quarter of each root. Cut the untreated samples first, using a clean knife on an uncontaminated surface.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
C	02	PPP	X ( $\pm 1$ )	12 roots / 4 lb.	Roots
D	02	PPP	X ( $\pm 1$ )	12 roots / 4 lb.	Roots

#### 18.2 Decline trial XX@@:

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Potato

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect 12-24 potato tubers from at least 6 plants. (If potatoes are very large, then collect 12 tubers; otherwise collect 24.) Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If excessive soil adheres to the tubers, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the tuber briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE TUBERS.

Cut the potatoes with a clean knife into at least 4 slices and retain all of the slices for the sample. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). If necessary to reduce gross sample weight, cut each tuber longitudinally into halves or quarters, retaining one half or quarter for the sample. Cut the untreated samples first, using a clean knife on an uncontaminated surface.

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

**Samples for Processing (Field trial XX only):** At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots first, collect approximately 150 lb. of fresh potatoes per sample from each plot. Each sample should be representative of the plot. Avoid sampling from plot ends. Immediately after sample collection, transport unfrozen samples in a clean burlap or similar type bag to the processing laboratory. Samples should be processed within 72 hours of harvest (see sample shipping instructions in section 19.2).

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12-24 tubers	Tubers

B	01	Untreated	NA	12-24 tubers	Tubers
C	02	PPP	X (+1)	12-24 tubers	Tubers
D	02	PPP	X (+1)	12-24 tubers	Tubers

#### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12-24 tubers	Tubers
B	01	Untreated	NA	12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers
	02	PPP		12-24 tubers	Tubers

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

#### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROXIMATE WEIGHT OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	150 lb.	Tubers
PT	02	PPP	X (+1)	150 lb.	Tubers

### 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots

and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After Harvest of processing samples maintain unfrozen and ship to the processor within 24 hours. **Insert a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples to: @@@

## **19.3 PROCESSING:**

Immediately prior to processing remove from the untreated and treated samples a “grab” sample of 12-24 potatoes. (If potatoes are very large, then collect 12 tubers; otherwise collect 24.)

As soon as possible after collection and receipt of tubers (within approximately 72 hours of harvest) process the tubers into flakes/granules, potato chips, and wet peel employing procedures that are similar in operation to commercial practices. Collect approximately 2-4 lb. per sample each of potato flakes or granules and chips, and approximately 4-5 lb. per sample of wet peel.

Place samples in appropriate containers and label. **Processed samples may be divided into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Freeze processed samples as soon as possible after processing. Send processed samples to: @@@

## **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. SIZE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	12-24 tubers	Tubers
GT	02	PPP	X (+1)	12-24 tubers	Tubers
FGA	01	Untreated	NA	2 – 4 lb.	Potato Flakes or Granules
FGT	02	PPP	X (+1)	2 – 4 lb.	Potato Flakes or Granules
PCA	10	Untreated	NA	2 – 4 lb.	Potato Chips
PCT	02	PPP	X (+1)	2 – 4 lb.	Potato Chips
WPA	01	Untreated	NA	4 – 6 lb.	Wet Peel
WPT	02	PPP	X (+1)	4 – 6 lb.	Wet Peel

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

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The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date tuber samples received
- 20.1.04- Storage temperatures until tuber samples are processed into flakes/granules, potato chips, and wet peel

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20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing of samples into flakes/granules, potato chips, and wet peel

20.1.07- Storage temperatures of tuber, flakes/granules, potato chips, and wet peel samples until shipped

20.1.08-Date tuber, flakes/granules, potato chips, and wet peel samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a “true copy” will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A “true copy” of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Radish

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Roots and tops may come from the same plants, or from different plants. At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots,, collect at least 24 marketable plants (tops and roots). Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Root samples should weigh a minimum of 4 lb (but preferably not more than 6 lb) and each top sample should weigh a minimum of 1 lb (but preferably not more than 2 lb). Avoid sampling from plot ends.

If excessive soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE TOPS OR ROOTS.

Remove tops and package separately from the roots. Roots should be cut into halves or quarters, unless they are very small. Retain all portions of the cut roots for the sample.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
RA	01	Untreated	NA	24 roots / 4 lb.	Roots
RB	01	Untreated	NA	24 roots / 4 lb.	Roots
RC	02	PPP	X ( $\pm 1$ )	24 roots / 4 lb.	Roots
RD	02	PPP	X ( $\pm 1$ )	24 roots / 4 lb.	Roots
TA	01	Untreated	NA	24 plants / 1 lb.	Tops (Leaves)
TB	01	Untreated	NA	24 plants / 1 lb.	Tops (Leaves)

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TC	02	PPP	X (+1)	24 plants / 1 lb.	Tops (Leaves)
TD	02	PPP	X (+1)	24 plants / 1 lb.	Tops (Leaves)

## 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
RA	01	Untreated	NA	12 roots / 4 lb.	Roots
RB	01	Untreated	NA	12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
TA	01	Untreated	NA	24 plants / 1 lb.	Tops (Leaves)
TB	01	Untreated	NA	24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)
	02	PPP		24 plants / 1 lb.	Tops (Leaves)

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples RC, RD, TC, and TD.

## Rutabaga

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots,, collect at least 12 roots. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Root samples should weigh a minimum of 4 lb (but preferably not more than 6 lb). Avoid sampling from plot ends.

If excessive soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE ROOTS.

Cut each of the rutabaga roots into at least 4 slices, and retain all of the slices for the sample. If any of the slices are more than about 6 inches (15 cm) long, then they should be cut into two smaller slices and both should be retained for the sample.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
C	02	PPP	X ( $\pm 1$ )	12 roots / 4 lb.	Roots
D	02	PPP	X ( $\pm 1$ )	12 roots / 4 lb.	Roots

#### 18.2 Decline trial XX@@:

SAMPLE	TRT#	TREATMENT	DAYS AFTER	MINIMUM SAMPLE	CROP
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ID			LAST APPLIC.	SIZE	FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Sweet potato

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect 12 sweet potato roots per sample. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Root samples should weigh a minimum of 4 lb (but preferably not more than 6 lb). Avoid sampling from plot ends.

If excessive soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE ROOTS.**

Cut the sweet potatoes with a clean knife into at least 4 slices and retain all of the slices for the sample. If the slices are more than approximately 6 inches (15 cm) in length, then cut each of those slices into two shorter pieces, and retain all of the pieces for the sample. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

Each sample should be representative of the entire plot (except plot ends).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
C	02	PPP	X ( $\pm 1$ )	12 roots / 4 lb.	Roots

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D	02	PPP	X (+1)	12 roots / 4 lb.	Roots
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**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Turnip

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Roots and tops may come from the same plants, or from different plants. At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 marketable plants (tops and roots). Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Root samples should weigh a minimum of 4 lb (but preferably not more than 6 lb) and each top sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). Avoid sampling from plot ends.

If excessive soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE TOPS OR ROOTS.

Remove tops and package separately from the roots.

Cut each of the roots into at least 4 slices, and retain all of the slices for the sample. If any of the slices are more than about 6 inches (15 cm) long, then they should be cut into two smaller slices and both should be retained for the sample.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

**All trials:** Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
RA	01	Untreated	NA	12 roots / 4 lb.	Roots
RB	01	Untreated	NA	12 roots / 4 lb.	Roots
RC	02	PPP	X ( $\pm 1$ )	12 roots / 4 lb.	Roots

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RD	02	PPP	X (+1)	12 roots / 4 lb.	Roots
TA	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
TB	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
TC	02	PPP	X (+1)	12 plants / 2 lb.	Tops (Leaves)
TD	02	PPP	X (+1)	12 plants / 2 lb.	Tops (Leaves)

### 18.2 Decline trial XX@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
RA	01	Untreated	NA	12 roots / 4 lb.	Roots
RB	01	Untreated	NA	12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
	02	PPP		12 roots / 4 lb.	Roots
TA	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
TB	01	Untreated	NA	12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)
	02	PPP		12 plants / 2 lb.	Tops (Leaves)

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples RC, RD, TC, and TD.

**Vegetable, bulb, group 3**

## Garlic

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest at least 24 garlic plants as done commercially. (The garlic may be dried before sampling. If so, describe the drying procedure in the Field Data Book. The preharvest interval is the time between the last application and pulling the garlic from the soil. If removal of tops prior to drying and removal from the field is a local commercial practice, then that should be done at this time. Sampling should not occur until the garlic has dried according to local commercial practices. It is acceptable to move the harvested garlic from the field plots to a protected area for drying if environmental conditions will not permit drying in the field.)

At the appropriate time for garlic removal from the field following any drying time, collect a minimum of 24 cloves per sample. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends. (If the garlic cloves been moved for drying, it is acceptable to collect 24 cloves per sample at that time as described above and place them in the drying area, keeping the samples separate.) Remove the papery membrane from the garlic head, but DO NOT separate the cloves from the heads or peel the cloves.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If excessive soil adheres to the cloves, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE BULBS.

If not done at harvest, then remove tops on the sampling date. Retain trimmed cloves.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 cloves	Cloves
B	01	Untreated	NA	24 cloves	Cloves

C	02	PPP	X (+1)	24 cloves	Cloves
D	02	PPP	X (+1)	24 cloves	Cloves

**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 cloves	Cloves
B	01	Untreated	NA	24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves
	02	PPP		24 cloves	Cloves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Onion (Dry Bulb)

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the plots as done commercially. (The onions should be dried before sampling, if drying is a local commercial practice. If so, describe the drying procedure in the Field Data Book. The preharvest interval is the time between the last application and pulling the bulbs from the soil. If removal of roots, tops, and outer sheaths prior to drying and removal from the field is a local commercial practice, then that should be done at this time. It is acceptable to move the harvested onions from the field plots to a protected area for drying if environmental conditions will not permit drying in the field.)

At the appropriate time for onion collection following any drying time, collect a minimum of 24 onion bulbs per sample. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends. (If the onions have been moved for drying, it is acceptable to collect 24 onions per sample at that time as described above and place them in the drying area, keeping the samples separate.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If excessive soil adheres to the bulbs, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE BULBS.

Samples should weigh a minimum of 4 lb (but preferably not more than 6 lb). If not done at harvest, cut off roots and tops and remove outer sheaths (or husks) on the sampling date. Retain trimmed bulbs. Cut onion bulbs into quarters with a clean knife (unless they are very small) and retain all quarters for the sample. If the sample weight will exceed 8 lb, then two quarters from each onion may be discarded, retaining opposite quarters. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### **18. FIELD RESIDUE SAMPLE INVENTORY:**

#### **18.1 All trials except decline trial XX@@:**

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 bulbs / 4 lb.	Bulbs
B	01	Untreated	NA	24 bulbs / 4 lb.	Bulbs
C	02	PPP	X (+1)	24 bulbs / 4 lb.	Bulbs
D	02	PPP	X (+1)	24 bulbs / 4 lb.	Bulbs

**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 bulbs / 4 lb.	Bulbs
B	01	Untreated	NA	24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs
	02	PPP		24 bulbs / 4 lb.	Bulbs

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Onion (Green) or Chive or Leek

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, followed by treated collect at least 24 whole plants as done commercially. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). Remove roots and dead leaves.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If excessive soil adheres to the plants, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE PLANTS.**

**Decline trial XX@@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 plants / 4 lb.	Plants
B	01	Untreated	NA	24 plants / 4 lb.	Plants
C	02	PPP	X (+1)	24 plants / 4 lb.	Plants
D	02	PPP	X (+1)	24 plants / 4 lb.	Plants

#### 18.2 Decline trial XX@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 plants / 4 lb.	Plants
B	01	Untreated	NA	24 plants / 4 lb.	Plants

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	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants
	02	PPP		24 plants / 4 lb.	Plants

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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**Vegetable, leafy, group 4**

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## Dandelion

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 plants per sample as done commercially. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample must weigh at least 1 lb (but preferably not more than 2 lb). Avoid sampling from the plot ends. Remove dead and senesced leaves only. DO NOT TRIM.

If excessive soil adheres to the leaves, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE LEAVES.

Process untreated samples first.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 1 lb.	Plant
B	01	Untreated	NA	12 plants / 1 lb.	Plant
C	02	PPP	X ( $\pm 1$ )	12 plants / 1 lb.	Plant
D	02	PPP	X ( $\pm 1$ )	12 plants / 1 lb.	Plant

#### 18.2 Decline trial XX@@:

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 1 lb.	Plant
B	01	Untreated	NA	12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant
	02	PPP		12 plants / 1 lb.	Plant

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Endive

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 heads per sample. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove dead and/or senesced leaves. DO NOT TRIM.

If excessive soil adheres to the heads, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE HEADS.

**Decline trial XX@@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Heads
B	01	Untreated	NA	12 heads	Heads
C	02	PPP	X ( $\pm 1$ )	12 heads	Heads
D	02	PPP	X ( $\pm 1$ )	12 heads	Heads

#### 18.2 Decline trial XX@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Heads
B	01	Untreated	NA	12 heads	Heads
	02	PPP		12 heads	Heads

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	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads
	02	PPP		12 heads	Heads

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Lettuce (Head & Leaf)

### **10. TEST SYSTEM/CROP:**

LETTUCE (HEAD AND LEAF) - Use a commercial variety. Report: variety, source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments, including whether to use head or leaf lettuce**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher. **If the same Field Research Director has been assigned one head lettuce trial and one leaf lettuce trial, it is not required that other means of differentiation listed in Section 11.4 be used(except that independently prepared tank mixes must be used), but adhering to these means of differentiation is strongly preferred.**

### **17. RESIDUE SAMPLE COLLECTION:**

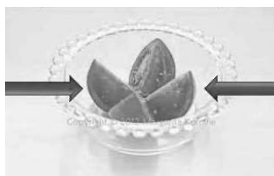
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect 12 plants (with the roots cut off) per sample. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove dead and/or senesced leaves. DO NOT TRIM.

If excessive soil adheres to the foliage, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE LEAVES.

Cut each lettuce plant (head or leaf) longitudinally into quarters with a clean knife on an uncontaminated surface. Retain all quarters of each lettuce plant (head or leaf). If the sample weight will exceed 4 lb, then two quarters may be discarded, retaining opposite quarters. Process the untreated samples first. For samples with wrapper leaves be sure to retain these leaves. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.



Opposite quarters

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23).

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Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Sections 18.1 and 18.2) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 HEAD LETTUCE—All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
HA	01	Untreated	NA	12 plants / 2 lb.	Head with Wrapper Leaves
HB	01	Untreated	NA	12 plants / 2 lb.	Head with Wrapper Leaves
HC	02	PPP	X (+1)	12 plants / 2 lb.	Head with Wrapper Leaves
HD	02	PPP	X (+1)	12 plants / 2 lb.	Head with Wrapper Leaves

### 18.2 HEAD LETTUCE—Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
HA	01	Untreated	NA	12 plants / 2 lb.	Head with Wrapper Leaves
HB	01	Untreated	NA	12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves
	02	PPP		12 plants / 2 lb.	Head with Wrapper Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples HC and HD.

### 18.3 LEAF LETTUCE SAMPLES—All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
LB	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
LC	02	PPP	X (+1)	12 plants / 2 lb.	Plant (without roots)
LD	02	PPP	X (+1)	12 plants / 2 lb.	Plant (without roots)

### 18.4 LEAF LETTUCE SAMPLES—Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
LB	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)

	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)

\*\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples LC and LD.

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## Lettuce (Greenhouse)

### 10. TEST SYSTEM/CROP:

LETTUCE (GREENHOUSE) - Use a commercial variety suitable for greenhouse production. Report: variety, source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the registration of greenhouse applications; see Section 23 for these assignments. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### 17. RESIDUE SAMPLE COLLECTION:

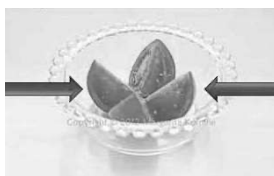
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect 12 plants (with the roots cut off) per sample. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove dead and/or senesced leaves. DO NOT TRIM.

If excessive soil adheres to the foliage, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE LEAVES.

Cut each head longitudinally into quarters with a clean knife on an uncontaminated surface. Retain all quarters of each head. If the sample weight will exceed 4 lb, then two quarters may be discarded, retaining opposite quarters. Process the untreated samples first. For samples with wrapper leaves be sure to retain these leaves. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.



Opposite quarters

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Sections 18.1 and 18.2) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at

the facility where the freezers are located.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:**

**18.3 All trials except decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
B	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
C	02	PPP	X (+1)	12 plants / 2 lb.	Plant (without roots)
D	02	PPP	X (+1)	12 plants / 2 lb.	Plant (without roots)

**18.4 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
B	01	Untreated	NA	12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)
	02	PPP		12 plants / 2 lb.	Plant (without roots)

**\*\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples LC and LD.**

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## Greens (Mustard) or Collards

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, beginning with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect greens (above ground portion, leaves) from at least 12 separate areas of the plot. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). If necessary, remove any senescent or decomposed leaves.

If excessive soil adheres to the greens, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE GREENS.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Greens (leaves)
B	01	Untreated	NA	4 lb.	Greens (leaves)
C	02	PPP	X (+1)	4 lb.	Greens (leaves)
D	02	PPP	X (+1)	4 lb.	Greens (leaves)

#### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Greens (leaves)
B	01	Untreated	NA	4 lb.	Greens (leaves)

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	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)
	02	PPP		4 lb.	Greens (leaves)

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Kale

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, beginning with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect leaves from at least 12 plants. Leaves must be sampled from at least two levels on each plant. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). If necessary, remove any senescent or decomposed leaves.

If excessive soil adheres to the leaves, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE LEAVES.**

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Leaves
B	01	Untreated	NA	4 lb.	Leaves
C	02	PPP	X (+1)	4 lb.	Leaves
D	02	PPP	X (+1)	4 lb.	Leaves

#### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Leaves
B	01	Untreated	NA	4 lb.	Leaves
	02	PPP		4 lb.	Leaves

	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves
	02	PPP		4 lb.	Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Spinach

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$  days) after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect spinach (above ground portion, leaves) from at least 12 plants per sample. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 2 lb. (but preferably not more than 4 lb.). If necessary, remove any senescent or decomposed leaves.

If excessive soil adheres to the leaves, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE LEAVES.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Greens (leaves)
B	01	Untreated	NA	12 plants / 2 lb.	Greens (leaves)
C	02	PPP	X (+1)	12 plants / 2 lb.	Greens (leaves)
D	02	PPP	X (+1)	12 plants / 2 lb.	Greens (leaves)

#### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Greens (leaves)
B	01	Untreated	NA	12 plants / 2 lb.	Greens (leaves)

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	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)
	02	PPP		12 plants / 2 lb.	Greens (leaves)

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Swiss Chard

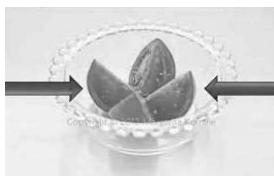
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 plants per sample as done commercially. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends. Remove dead and senesced leaves and roots only. DO NOT TRIM.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If excessive soil adheres to the plants, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE PLANTS.

Cut each head longitudinally into quarters with a clean knife on an uncontaminated surface. Retain all quarters of each head. If the sample weight will exceed 4 lb, then two quarters may be discarded, retaining opposite quarters. Process the untreated samples first. For samples with wrapper leaves be sure to retain these leaves. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.



Opposite quarters

Process untreated samples first.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### **18. FIELD RESIDUE SAMPLE INVENTORY:**

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**18.1 All trials except decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Plant
B	01	Untreated	NA	12 plants / 2 lb.	Plant
C	02	PPP	X (+1)	12 plants / 2 lb.	Plant
D	02	PPP	X (+1)	12 plants / 2 lb.	Plant

**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Plant
B	01	Untreated	NA	12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant
	02	PPP		12 plants / 2 lb.	Plant

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Watercress

### **17. RESIDUE SAMPLE COLLECTION:**

#### **First harvest:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X( $\pm$ 1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect leaves and stems from at least 12 sites within each plot per sample. Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 1 lb. (but preferably not more than 3 lb.).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If necessary, remove any senescent or decomposed leaves.

If excessive soil adheres to the leaves, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE LEAVES.

**After the first harvest / sampling, the entire plot should be cut and allowed to re-grow prior to making a second set of application(s) to the same treated plot.**

**For the second harvest**, follow the procedures described above for the first harvest.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### **18. FIELD RESIDUE SAMPLE INVENTORY:**

#### **18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
1A	01	Untreated	NA	1 lb.	Leaves and stems
1B	01	Untreated	NA	1 lb.	Leaves and stems
1C	02	PPP	X( $\pm$ 1)	1 lb.	Leaves and stems

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1D	02	PPP	X(+1)	1 lb.	Leaves and stems
Second Cutting / Harvest					
2A	01	Untreated	NA	1 lb.	Leaves and stems
2B	01	Untreated	NA	1 lb.	Leaves and stems
2C	02	PPP	X(+1)	1 lb.	Leaves and stems
2D	02	PPP	X(+1)	1 lb.	Leaves and stems

**18.2 Decline Trial XX@@:** *(It is probably only necessary to do the decline for one cutting or the other.)*

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
1A	01	Untreated	NA	1 lb.	Leaves and stems
1B	01	Untreated	NA	1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
Second Cutting / Harvest					
2A	01	Untreated	NA	1 lb.	Leaves and stems
2B	01	Untreated	NA	1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems
	02	PPP		1 lb.	Leaves and stems

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples 1C and 1D.

**Vegetable, brassica, head and stem, group 5**

## Broccoli

### 17. RESIDUE SAMPLE COLLECTION:

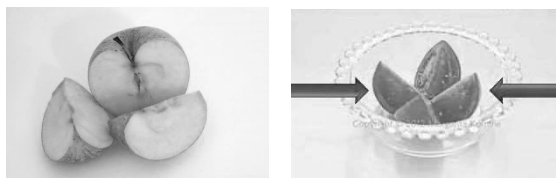
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect flower heads from a minimum of 12 plants as done commercially. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove dead and/or senesced leaves.

If excessive soil adheres to the plants, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE PLANTS.

Cut each flower head (including stem (stalk)) longitudinally into quarters with a clean knife on an uncontaminated surface. Retain all quarters of each flower head. If the sample weight will exceed 4 lb, then two quarters may be discarded and opposite quarters retained for the sample. Each retained section of the head should contain some florets and stem (stalk). Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.



Longitudinal cuts / Opposite quarters

Process untreated samples first.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

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## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Flower Head**
B	01	Untreated	NA	12 plants / 2 lb.	Flower Head**
C	02	PPP	X (+1)	12 plants / 2 lb.	Flower Head**
D	02	PPP	X (+1)	12 plants / 2 lb.	Flower Head**

\*\*Includes Florets, Stem (Stalk) and Jacket Leaves

### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Flower Head**
B	01	Untreated	NA	12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**
	02	PPP		12 plants / 2 lb.	Flower Head**

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

\*\*Includes Florets, Stem (Stalk) and Jacket Leaves

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## 19 Cabbage or Chinese Cabbage

### 17. RESIDUE SAMPLE COLLECTION:

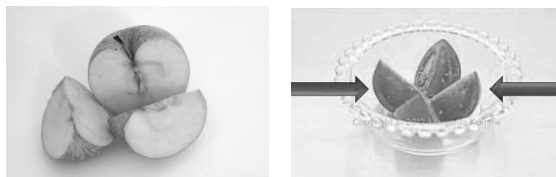
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect 12 heads from each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove only dead or senesced leaves.

If excessive soil adheres to the heads, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE HEADS.

Cut each head longitudinally into quarters with a clean knife on an uncontaminated surface. Retain all quarters of each head. If the sample weight will exceed 8 lb, then two quarters may be discarded and opposite quarters retained for the sample. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.



Longitudinal cuts / Opposite quarters

Process the untreated samples first. Retain the wrapper leaves on the heads.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

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## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Head with Wrapper Leaves
B	01	Untreated	NA	12 heads	Head with Wrapper Leaves
C	02	PPP	X (+1)	12 heads	Head with Wrapper Leaves
D	02	PPP	X (+1)	12 heads	Head with Wrapper Leaves

### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Head with Wrapper Leaves
B	01	Untreated	NA	12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves
	02	PPP		12 heads	Head with Wrapper Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Cauliflower

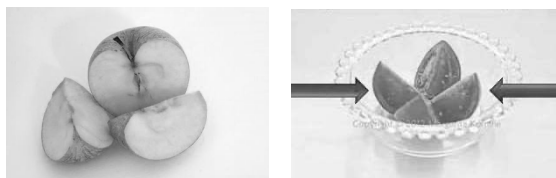
### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect 12 flower heads (with stem attached) from each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If excessive soil adheres to the heads, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE HEADS.

Cut each head longitudinally into quarters with a clean knife on an uncontaminated surface. Retain all quarters of each head. If the sample weight will exceed 8 lb, then two quarters may be discarded and opposite quarters retained for the sample. Each retained section of the head should contain some florets, stem (stalk) and jacket leaf foliage. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.



Longitudinal cuts / Opposite quarters

Process the untreated samples first.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

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**18.1 All trials except decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Flower Head with Stem
B	01	Untreated	NA	12 heads	Flower Head with Stem
C	02	PPP	X (+1)	12 heads	Flower Head with Stem
D	02	PPP	X (+1)	12 heads	Flower Head with Stem

**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Flower Head with Stem
B	01	Untreated	NA	12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem
	02	PPP		12 heads	Flower Head with Stem

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

**Vegetable, legume, group 6**

## Bean (Dry Shelled) or Pea (Southern) or Cowpea with Forage and Hay

### **10. TEST SYSTEM/CROP:**

BEAN (Dry Shelled) - Use a commercial variety. Report: variety, source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for the assignment of succulent Southern pea trials and dried shelled Southern pea trials**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated plot and two treated plots.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. See Parts 17 & 18 for requirements for residue sampling.

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

**17. RESIDUE SAMPLE COLLECTION:** See below for residue sample collection instructions for edible-podded, succulent-shelled and dry pea field trials.

### **17.2 RESIDUE SAMPLE COLLECTION:**

#### **Forage Samples (TRT 03):**

**For forage samples, cut the sample at 6-inch to prebloom stage, at approximately 30% dry matter.** Harvest and sample forage from at least 12 separate areas of each plot. Begin with the untreated plot first or utilize separate personnel for the treated and untreated plots, and then sample the treated plot. Each sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). Each sample should be collected during a separate run through the entire plot. Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

#### **Hay Samples (TRT 03):**

**For hay samples, cut the succulent plant when pods are one-half to fully mature. The hay should be field-dried to a moisture content of 10 to 20 percent.** Begin with the untreated plot or utilize separate personnel for the treated and untreated plots. Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the hay in a manner simulating commercial practices. For each sample, collect a minimum of 1 lb of hay from at least 12 separate areas of the plot. Crops which are harvested mechanically can be sampled from the harvester as it proceeds through the crop.

Allow the hay to dry in the field to a moisture content of approximately 10-20% (80-90% dry matter). (Percent dry matter may be estimated.) If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. When the hay has dried, collect samples from the untreated plot first, followed by the treated plot. Hay samples should

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weigh a minimum of 1 lb (but preferably not more than 3 lb). Determine (or estimate) and report the moisture content of the hay upon sampling (i.e. placing the samples in the sample bags). **Please note that the harvest date is the date that hay is cut. This is followed by drying (if needed) and then sampling.**

**Seed Samples (TRT 02):** The harvest should begin **X (± 1)** days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots,. Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Each sample should be collected during a separate run through the entire plot. Avoid harvesting from plot ends.

If hand harvested, take beans/peas from high and low areas and peas exposed and sheltered by foliage in proportion to pea distribution from at least twelve separate areas of each plot. The pods may be shelled by hand or mechanically simulating commercial practices.

Alternatively, harvest entire pea plants and run the plants through a thresher to obtain the dry pea seeds. Crops which are harvested mechanically can be sampled from the harvester as it proceeds through the crop. If needed or customary, dry the peas in pods following local commercial practices before shelling and sampling.

The pre-harvest interval is the length of time between the last application and pulling the pods from the living plant. Record in the Field Data Book all drying details including the method, length of drying time, and temperatures. [Harvest should occur at **X (± 1)** days after the last application, followed by drying (if needed), then sampling].

The seed samples should be free of any foliage or pod fragments. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

**All samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

#### 18. FIELD RESIDUE SAMPLE INVENTORY:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
DSA	01	Untreated	NA	2 lb.	Dry seed
DSB	01	Untreated	NA	2 lb.	Dry seed
DSC	02	PPP	X (±1)	2 lb.	Dry seed
DSD	02	PPP	X (±1)	2 lb.	Dry seed
FA	01	Untreated	NA*	2 lb.	Forage
FB	01	Untreated	NA*	2 lb.	Forage
FC	03	PPP	NA*	2 lb.	Forage
FD	03	PPP	NA*	2 lb.	Forage

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HA	01	Untreated	NA**	1 lb.	Hay
HB	01	Untreated	NA**	1 lb.	Hay
HC	03	PPP	NA**	1 lb.	Hay
HD	03	PPP	NA**	1 lb.	Hay

\* For forage samples, cut the sample at 6-inch to prebloom stage, at approximately 30% dry matter.

\*\* For hay samples, cut the succulent plant when pods are one-half to fully mature. The hay should be field-dried to a moisture content of 10 to 20 percent.

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## Bean (Edible Podded) with forage

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** At  $X(\pm 1)$  days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect two samples of forage by cutting plants at soil level. Select 12 plants from separate areas of the plot and separate them into 4 groups. Divide each plant into 3 approximately equal lengths. Take a top, middle, and bottom portion from each group of 3 plants ensuring that parts of all 12 plants are included in each sample. If 12 plants (above-ground portion) weigh less than 3 lb, do not cut them into 3 portions; retain the entire above-ground portion of each plant for the sample. If 12 whole plants (above-ground portion) weigh less than 1 lb, then collect additional plants (up to 48 total) to attain 1-lb samples. **Note to Study Director: Forage samples should be collected before bloom. This may mean that some applications are made after forage collection. Delete this note.**

At  $X(\pm 1)$  days after the last application, collect two samples of beans from each plot. Each sample should be representative of the entire plot (except plot ends). Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots first, collect pods with seed from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample of pods with seed should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each sample should be representative of the entire plot (except plot ends). Avoid sampling from plot ends.

Take pods from high and low areas and pods exposed and sheltered by foliage in proportion to pod distribution.

If necessary, shake off or brush off loose soil and describe in the Field Data Book the method used.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Sections 18.1 and 18.2) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Pods w/seed
SB	01	Untreated	NA	2 lb.	Pods w/seed
SC	02	PPP	$X(\pm 1)$	2 lb.	Pods w/seed



SD	02	PPP	X( $\pm 1$ )	2 lb.	Pods w/seed
FA	01	Untreated	NA	12 plant fractions	Bean forage
FB	01	Untreated	NA	12 plant fractions	Bean forage
FC	02	PPP	X( $\pm 1$ )	12 plant fractions	Bean forage
FD	02	PPP	X( $\pm 1$ )	12 plant fractions	Bean forage

## 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Pods w/seed
SB	01	Untreated	NA	2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
FA	01	Untreated	NA	12 plant fractions	Bean forage
FB	01	Untreated	NA	12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC, SD, FC, and FD.

**Bean (Succulent Shelled) or Bean (Lima) or Edamame**

**17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trials:** Collect two samples of beans from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect beans in pods from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

**EDAMAME TRIALS ONLY:**

Edamame are ready to pick when the seeds are about full size and when pods are bright green without yellowing.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Harvest pods from high and low areas and pods exposed and sheltered by foliage in proportion to pod distribution. Shell the succulent beans and remove the pods, retaining the seeds for the sample. Ensure that the bean samples are free of any foliage or pod fragments. Record in the Field Data Book the method used to shell the beans. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb) of seed.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**EDAMAME TRIALS ONLY:** In addition to the collection of bean seeds described above, collect two samples of pods with seed from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect pods with seed from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample of pods with seed should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each sample should be representative of the entire plot (except plot ends). Avoid sampling from plot ends.

Take pods from high and low areas and pods exposed and sheltered by foliage in proportion to pod distribution.

If necessary, shake off or brush off loose soil and describe in the Field Data Book the method used.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

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**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**  
**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

# **18. FIELD RESIDUE SAMPLE INVENTORY:**

## **18.1 All trials except decline trial XX@@:**

SAMPLE ID	TRT #	TREAT- MENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
B	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
C	02	PPP	X (+1)	2 lb.	Succulent Bean Seeds Without Pods
D	02	PPP	X (+1)	2 lb.	Succulent Bean Seeds Without Pods

## **18.2 All trials except decline trial XX@@: EDAMAME ONLY**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Pods w/seed
B	01	Untreated	NA	2 lb.	Pods w/seed
C	02	PPP	X (+1)	2 lb.	Pods w/seed
D	02	PPP	X (+1)	2 lb.	Pods w/seed

## **18.2 or 18.3 Decline trial XX@@:**

SAMPLE ID	TRT #	TREAT- MENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
B	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Bean (Succulent Shelled) with forage

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** At  $X(\pm 1)$  days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect two samples of foliage by cutting plants at soil level from at least 12 separate areas of each plot. Select 12 plants and separate them into 4 groups. Divide each plant into 3 approximately equal lengths. Take a top, middle, and bottom portion from each group of 3 plants ensuring that parts of all 12 plants are included in each sample. If 12 plants (above-ground portion) weigh less than 3 lb, do not cut them into 3 portions; retain the entire above-ground portion of each plant for the sample. If 12 whole plants (above-ground portion) weigh less than 1 lb, then collect additional plants (up to 48 total) to attain 1-lb samples. **Note to Study Director: Forage samples should be collected before bloom. This may mean that some applications are made after forage collection. Delete this note.**

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Collect two samples of beans from each plot. Each sample should be representative of the entire plot (except plot ends). At  $X(\pm 1)$  days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect beans in pods from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

Harvest pods from high and low areas and pods exposed and sheltered by foliage in proportion to pod distribution. Shell the succulent beans and remove the pods, retaining the seeds for the sample. Ensure that the bean samples are free of any foliage or pod fragments. Record in the Field Data Book the method used to shell the beans. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb) of seed.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;** **Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
SB	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
SC	02	PPP	$X(\pm 1)$	2 lb.	Succulent Bean Seeds Without Pods

SD	02	PPP	X( $\pm$ 1)	2 lb.	Succulent Bean Seeds Without Pods
FA	01	Untreated	NA	12 plant fractions	Bean forage
FB	01	Untreated	NA	12 plant fractions	Bean forage
FC	02	PPP	X( $\pm$ 1)	12 plant fractions	Bean forage
FD	02	PPP	X( $\pm$ 1)	12 plant fractions	Bean forage

## 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
SB	01	Untreated	NA	2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
	02	PPP		2 lb.	Succulent Bean Seeds Without Pods
FA	01	Untreated	NA	12 plant fractions	Bean forage
FB	01	Untreated	NA	12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage
	02	PPP		12 plant fractions	Bean forage

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC, SD, FC, and FD.

## Lentil

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest dry lentils in pods from at least twelve separate areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid harvesting from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If appropriate, take lentils from high and low areas and beans exposed and sheltered by foliage in proportion to lentil distribution. Alternatively, harvest entire lentil plants and run them through a thresher to obtain lentil pods.

If needed or customary, dry the lentils in pods following local commercial practices before shelling and sampling. The preharvest interval is the length of time between the last application and pulling the bean plants from the soil. Record in the Field Data Book all drying details including the method, length of drying time, and temperatures. [Harvest should occur at X ( $\pm 1$ ) days, followed by drying, then sampling].

After harvest and drying (if needed), shell lentils and remove pods, retaining the seeds for the sample. The lentil samples should be free of any foliage or pod fragments. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

**Decline trial XX@@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Dry Lentil Seed
B	01	Untreated	NA	2 lb.	Dry Lentil Seed
C	02	PPP	X ( $\pm 1$ )	2 lb.	Dry Lentil Seed
D	02	PPP	X ( $\pm 1$ )	2 lb.	Dry Lentil Seed

#### 18.2 Decline trial XX@@@:

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Dry Lentil Seed
B	01	Untreated	NA	2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed
	02	PPP		2 lb.	Dry Lentil Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Shipment of frozen samples will be by freezer truck or express shipment. Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)

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- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- Data collected during sample drying, including oven temperature (may be approximate) and length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## Pea (Dry Shelled) with Forage and Hay

### **17. RESIDUE SAMPLE COLLECTION:**

#### **All trials except decline trial:**

**Seed Samples:** The harvest should begin X ( $\pm 1$ ) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots. The pre-harvest interval is the length of time between the last application and pulling the pea plants from the soil or the pods from the living plant. Record in the Field Data Book all drying details including the method, length of drying time, and temperatures. [Harvest should occur at X ( $\pm 1$ ) days after the last application, followed by drying (if needed), then sampling].

Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Each sample should be collected during a separate run through the entire plot. Avoid collecting samples from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If hand harvested (sampled), take peas from high and low areas and peas exposed and sheltered by foliage in proportion to pea distribution from at least twelve separate areas of each plot. The pods may be shelled by hand or mechanically simulating commercial practices.

Alternatively, harvest entire pea plants and run the plants through a thresher to obtain the dry pea seeds. If needed or customary, dry the peas in pods following local commercial practices before shelling and sampling.

The pea samples should be free of any foliage or pod fragments. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

#### **Forage Samples:**

At X ( $\pm 1$ ) days after the last application, harvest and sample forage from at least 12 separate areas of each plot. Pea plants should be cut when pods have begun to form, at approximately 25% dry matter. Begin with the untreated plot first or utilize separate personnel for the treated and untreated plots, and then sample the treated plot. Each sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). Each sample should be collected during a separate run through the entire plot. All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

#### **Hay Samples:**

At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the hay in each plot. Pea vines should be between full bloom and pod formation. Allow the hay to dry in the field to a moisture content of approximately 10-20% (80-90% dry matter). (Percent dry matter may be estimated.) If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area. When the hay has dried, collect samples from the untreated plot first, followed by the treated plot. Hay samples should weigh a minimum of 1 lb (but preferably not more than 3 lb). Determine (or estimate) and report the moisture content of the hay upon

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sampling (i.e. placing the samples in the sample bags).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Dry Pea seed
SB	01	Untreated	NA	2 lb.	Dry Pea seed
SC	02	PPP	X (+1)	2 lb.	Dry Pea seed
SD	02	PPP	X (+1)	2 lb.	Dry Pea seed
VA	01	Untreated	NA	2 lb.	Pea forage
VB	01	Untreated	NA	2 lb.	Pea forage
VC	02	PPP	X (+1)	2 lb.	Pea forage
VD	02	PPP	X (+1)	2 lb.	Pea forage
HA	01	Untreated	NA	1 lb.	Pea hay
HB	01	Untreated	NA	1 lb.	Pea hay
HC	02	PPP	X (+1)	1 lb.	Pea hay
HD	02	PPP	X (+1)	1 lb.	Pea hay

### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Dry Pea seed
SB	01	Untreated	NA	2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed
	02	PPP		2 lb.	Dry Pea seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC and SD.

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Shipment of frozen samples will be by freezer truck or express shipment. Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first

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application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.

20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots

20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

20.17- Data collected during sample drying, including oven temperature (may be approximate) and length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## Pea (Edible-Podded) with forage

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** At one day after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots,, collect two samples of forage by cutting plants at soil level. Select 12 plants and separate them into 4 groups. Divide each plant into 3 approximately equal lengths. Take a top, middle, and bottom portion from each group of 3 plants ensuring that parts of all 12 plants are included in each sample. If 12 plants (above-ground portion) weigh less than 3 lb, do not cut them into 3 portions; retain the entire above-ground portion of each plant for the sample. If 12 whole plants (above-ground portion) weigh less than 1 lb, then collect additional plants (up to 48 total) to attain 1-lb samples. **Note to Study Director: Forage samples should be collected after pods begin to form, at approximately 25% dry matter (may be estimated). This may mean that some applications are made after forage collection. Delete this note.**

At X( $\pm$ 1) days after the last application, collect two samples of peas in pods from each plot. Each sample should be representative of the entire plot (except plot ends). When the peas are commercially mature, starting with the untreated samples, collect peas from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples will include succulent pea seed in pod. Avoid sampling from plot ends. If appropriate, take peas from high and low areas and peas exposed and sheltered by foliage in proportion to pea distribution.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

#### All trials:

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Pods w/seed
SB	01	Untreated	NA	2 lb.	Pods w/seed
SC	02	PPP	X( $\pm$ 1)	2 lb.	Pods w/seed
SD	02	PPP	X( $\pm$ 1)	2 lb.	Pods w/seed
FA	01	Untreated	NA	12 plant fractions	Pea forage
FB	01	Untreated	NA	12 plant fractions	Pea forage

FC	02	PPP	X( $\pm 1$ )	12 plant fractions	Pea forage
FD	02	PPP	X( $\pm 1$ )	12 plant fractions	Pea forage

## 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Pods w/seed
SB	01	Untreated	NA	2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
	02	PPP		2 lb.	Pods w/seed
FA	01	Untreated	NA	12 plant fractions	Pea forage
FB	01	Untreated	NA	12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC, SD, FC, and FD.

**Pea (Succulent Shelled) with forage**

**17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** At  $X(\pm 1)$  days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect two samples of forage by cutting plants at soil level from at least 12 separate areas of each plot. Select 12 plants and separate them into 4 groups. Divide each plant into 3 approximately equal lengths. Take a top, middle, and bottom portion from each group of 3 plants ensuring that parts of all 12 plants are included in each sample. If 12 plants (above-ground portion) weigh less than 3 lb, do not cut them into 3 portions; retain the entire above-ground portion of each plant for the sample. If 12 whole plants (above-ground portion) weigh less than 1 lb, then collect additional plants (up to 48 total) to attain 1-lb samples. **Note to Study Director: Forage samples should be collected before bloom. This may mean that some applications are made after forage collection. Delete this note.**

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Collect two samples of peas from each plot. Each sample should be representative of the entire plot (except plot ends). At  $X(\pm 1)$  days after the last application, starting with the untreated samples, collect peas from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot.

Shell peas and remove pods, retaining the seeds for the sample. Ensure that the samples are free of any foliage or pod fragments. Avoid sampling from plot ends. If appropriate, take peas from high and low areas and peas exposed and sheltered by foliage in proportion to pea distribution.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Sections 18.1 and 18.2) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:**

**18.1 All trials except decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Pea Seed
SB	01	Untreated	NA	2 lb.	Pea Seed
SC	02	PPP	$X(\pm 1)$	2 lb.	Pea Seed
SD	02	PPP	$X(\pm 1)$	2 lb.	Pea Seed

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FA	01	Untreated	NA	12 plant fractions	Pea forage
FB	01	Untreated	NA	12 plant fractions	Pea forage
FC	02	PPP	X( $\pm$ 1)	12 plant fractions	Pea forage
FD	02	PPP	X( $\pm$ 1)	12 plant fractions	Pea forage

## 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Pea Seed
SB	01	Untreated	NA	2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
	02	PPP		2 lb.	Pea Seed
FA	01	Untreated	NA	12 plant fractions	Pea forage
FB	01	Untreated	NA	12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage
	02	PPP		12 plant fractions	Pea forage

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC, SD, FC, and FD.



## Soybean (without processing)

### **17. RESIDUE SAMPLE COLLECTION:**

#### **Seed Samples:**

**All trials except decline trial:** Collect two samples of seed from each plot. Each sample should be representative of the entire plot (except plot ends). Each sample should be collected during a separate run through the entire plot. If this can not be done due to harvesting equipment or other factor, contact the Study Director to discuss.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

At X ( $\pm 1$ ) days after the last application, starting with the untreated samples or utilizing separate personnel for the treated and untreated plots, collect seeds in pods from at least 12 separate areas of each plot. Shell the seeds and remove the pods, retaining the seeds for the sample. Ensure that the samples are free of any foliage or pod fragments. Avoid sampling from plot ends.

If appropriate, take samples from high and low areas and soybeans exposed and sheltered by foliage in proportion to soybean distribution. Each sample should weigh at least 2 lb (but preferably not more than 3 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

#### **Forage Samples:**

**All trials except decline trial:** Collect soybean forage (entire above-ground portion of plant) when the plants are 6-8 inches tall (15-20 cm) from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot. If this can not be done due to harvesting equipment or other factor, contact the Study Director to discuss.

**Forage samples should be collected at the appropriate growth stage (no later than beginning pod formation), regardless of how many applications have been made.** Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each sample should be representative of the entire plot (except plot ends). Avoid sampling from plot ends. If necessary, shake off or brush off loose soil and describe in the Field Data Book the method used.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

#### **Hay Samples:**

**All trials except decline trial:** Cut soybeans to be sampled for hay at mid-to-full bloom stage, before bottom leaves begin to fall, or when pods are approximately 50% developed, from at least 12 separate areas of each plot. Allow the plants to field-dry to a moisture content of approximately 10-20% (moisture content may be estimated). Each sample should be collected during a separate run through the entire plot. If this can not be done due to harvesting equipment or other factor, contact the Study Director to discuss. If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area.

**Hay samples should be collected at the appropriate growth stage (no later than beginning pod formation), regardless of how many applications have been made.** Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each sample should be representative of the entire plot (except plot ends). Avoid sampling from plot ends. If necessary, shake off or brush off loose soil and describe in the Field Data Book the method used.

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In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Soybean Seed
SB	01	Untreated	NA	2 lb.	Soybean Seed
SC	02	PPP	X (±1)	2 lb.	Soybean Seed
SD	02	PPP	X (±1)	2 lb.	Soybean Seed
FA	01	Untreated	NA	2 lb.	Soybean Forage
FB	01	Untreated	NA	2 lb.	Soybean Forage
FC	02	PPP	NA	2 lb.	Soybean Forage
FD	02	PPP	NA	2 lb.	Soybean Forage
HA	01	Untreated	NA	1 lb.	Soybean Hay
HB	01	Untreated	NA	1 lb.	Soybean Hay
HC	02	PPP	NA	1 lb.	Soybean Hay
HD	02	PPP	NA	1 lb.	Soybean Hay

### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	2 lb.	Soybean Seed
SB	01	Untreated	NA	2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed
	02	PPP		2 lb.	Soybean Seed

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	02	PPP		2 lb.	Soybean Seed
FA	01	Untreated	NA	2 lb.	Soybean Forage
FB	01	Untreated	NA	2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
	02	PPP		2 lb.	Soybean Forage
HA	01	Untreated	NA	1 lb.	Soybean Hay
HB	01	Untreated	NA	1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay
	02	PPP		1 lb.	Soybean Hay

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC, SD, FC, FD, HC, and HD.

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**Vegetable, fruiting, group 8**

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## Eggplant

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 fruit from at least 12 plants per sample. Each sample should be collected during a separate run through the entire plot. If fruit are small, harvest a minimum of 24 fruit from at least 12 plants per sample.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take fruit from both sides of row, high and low areas, fruit exposed and sheltered by foliage. Avoid sampling from plot ends. If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruit	Fruit
B	01	Untreated	NA	12 fruit	Fruit
C	02	PPP	X (+1)	12 fruit	Fruit
D	02	PPP	X (+1)	12 fruit	Fruit

#### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruit	Fruit
B	01	Untreated	NA	12 fruit	Fruit
	02	PPP		12 fruit	Fruit

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	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit
	02	PPP		12 fruit	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Okra

### 12. TEST SITE PREPARATION:

Prepare or select a test site that has been maintained following good local agricultural practices for the production of okra including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Harvest the crop if needed on a regular basis as per standard commercial practice to prevent fruit from reaching a size too large or in such poor condition as to preclude use as samples.**

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 24 fruit from at least 12 plants per sample. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take fruit from both sides of row, high and low areas, fruit exposed and sheltered by foliage. Avoid sampling from plot ends.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Pods
B	01	Untreated	NA	4 lb.	Pods

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C	02	PPP	X (+1)	4 lb.	Pods
D	02	PPP	X (+1)	4 lb.	Pods

**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLICATION	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Pods
B	01	Untreated	NA	4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods
	02	PPP		4 lb.	Pods

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Pepper (Bell) & Pepper (Non-Bell)

### 10. TEST SYSTEM/CROP:

PEPPER (BELL & NON-BELL) - Use a commercial variety. Report: variety, source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments. See below for assignment of bell or non-bell pepper trials.** Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher. **If the same Field Research Director has been assigned one bell pepper trial and one non-bell pepper trial, this meets criterion 1C in Section 11.4. It is not required that other means of differentiation listed in Section 11.4 be used (except that independently prepared tank mixes must be used), but using additional means of differentiation is strongly preferred.**

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 fruit from at least 12 plants per sample. If fruit are small (much less than 4 lb per 12 fruit), harvest a minimum of 24 fruit from at least 12 plants per sample. Each bell pepper sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). Each non-bell pepper sample should weigh a minimum of 2 lb (but preferably not more than 6 lb). Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take fruit from both sides of row, high and low areas, fruit exposed and sheltered by foliage. Avoid sampling from plot ends. If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Bell Pepper Trials except Decline Trial XX@@:

SAMPLE	TRT#	TREATMENT	DAYS AFTER	MINIMUM SAMPLE SIZE	CROP
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ID			LAST APPLIC.		FRACTION
BA	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
BB	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
BC	02	PPP	X (+1)	12 fruits from at least 12 plants / 4 lb.	Fruit
BD	02	PPP	X (+1)	12 fruits from at least 12 plants / 4 lb.	Fruit

#### 18.2 Bell Pepper Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
BA	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
BB	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples BC and BD.

#### 18.3 All Non-Bell Pepper Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
NA	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
NB	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
NC	02	PPP	X (+1)	24 fruits from at least 12 plants / 2 lb.	Fruit
ND	02	PPP	X (+1)	24 fruits from at least 12 plants / 2 lb.	Fruit

#### 18.4 Non-Bell Pepper Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
NA	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
NB	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit

\*\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples NC and ND.

## Tomato

### **10. TEST SYSTEM/CROP:**

Tomato - Use a commercial variety. Report: variety (**indicate whether it is a small-fruited or large-fruited variety**), source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments. See below for assignment of small-fruited (cherry or grape tomato, generally less than 2 9/32 inches in diameter) or large-fruited variety (including processing tomatoes). Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.**

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last test substance application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 fruit from at least 12 different plants per sample. If tomatoes are small-fruited (grape or cherry tomatoes), harvest a minimum of 24 fruit from at least 12 plants per sample. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Harvest in a manner that assures a representative sample from the plot (except plot ends). Harvested fruit should be commercially acceptable. Large-fruited varieties do not need to be ripe or red in color, but must be mature in size. Small-fruited varieties should be red at harvest. Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb) for large-fruited tomatoes or a minimum of 2 lb (but preferably not more than 3 lb) for small-fruited tomatoes. Harvest additional fruit to meet minimum weight if necessary. Harvest fruit from both sides of row, fruit exposed and sheltered by foliage. Avoid harvesting from the plot ends.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before proceeding to the treated plot(s). Harvest and sampling methods can be outlined generally in SOP's, but describe methods fully in the Field Data Book.

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (See Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Fresh Tomato Fruit of a Processing Variety to be Processed into Paste and Puree (Field Trial XX):** Collect

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one sample each from the Trt 01 and Trt 03 plots. Harvest 100-120 lb of tomato fruit per plot at X ( $\pm$ 1) days after the last test substance application.

For processing tomatoes, red fruit should be harvested as per commercial practice. Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest fruit from high and low areas, both sides of row, fruit exposed and sheltered by foliage. Avoid harvesting from the plot ends.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before proceeding to the treated plot(s).

Samples should be collected in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID Number, Test Substance (chemical name listed in Section 15) complete sample ID (See Section 18.3), and harvest/sampling dates. See Protocol Section 19.2 for handling and shipping directions. Ship the large samples within 1 day of sample collection, if possible, as “fresh samples” to the processing facility.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Large-Fruited Tomato Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
LB	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
LC	02	PPP	X ( $\pm$ 1)	12 fruits from at least 12 plants / 4 lb.	Fruit
LD	02	PPP	X ( $\pm$ 1)	12 fruits from at least 12 plants / 4 lb.	Fruit

### 18.2 Large-Fruited Tomato Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
LB	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples LC and LD.

### 18.3 All Small-Fruited Tomato Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
SB	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
SC	02	PPP	X ( $\pm$ 1)	24 fruits from at least 12 plants / 2 lb.	Fruit
SD	02	PPP	X ( $\pm$ 1)	24 fruits from at least 12 plants / 2 lb.	Fruit

### 18.4 Small-Fruited Tomato Decline Trial XX@@:

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
SB	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC and SD.

#### 18.5 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	100-120 lb.	Fresh Fruit
PT	02	PPP	X (+1)	100-120 lb.	Fresh Fruit

#### 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

##### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in a refrigerator at approximately 4°C until they are shipped. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

#### **19.3 PROCESSING:**

Immediately prior to processing tomatoes, remove representative “grab” samples of untreated and treated fruit from the larger samples (4-6 lb. for each sample). Place the “grab” samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc.

Using simulated commercial processing (provide detailed description of equipment and procedures), produce tomato paste and puree from the untreated and treated samples. Process untreated fruit first, followed by treated fruit. All trials: Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of puree and one sample of paste each from both the untreated and treated fruit samples. Each tomato puree and tomato paste sample should weigh approximately 2-4 lb.

Place samples in appropriate containers and label. **Divide each sample of paste and puree into separate containers of 50-150 grams. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Processing waste and excess puree and paste may be discarded. If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical tomato storage conditions to prevent test substance residue degradation.

Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or “express” shipment. Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, communication note, etc. For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. RANGE OF SAMPLE	CROP FRACTION
GA	01	Untreated	NA	4-6 lb.	Fruit
GT	02	PPP	X (+1)	4-6 lb.	Fruit
PAA	01	Untreated	NA	2-4 lb.	Paste
PAT	02	PPP	X (+1)	2-4 lb.	Paste

PUA	01	Untreated	NA	2-4 lb.	Puree
PUT	02	PPP	X (+1)	2-4 lb.	Puree

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

### **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions

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20.1.02- Deviations from protocol and standard operating procedures

20.1.03- Date fresh tomato fruit samples received

20.1.04- Storage temperatures until fresh tomato fruit samples are processed into paste and puree

20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing of samples into paste and puree

20.1.07- Storage temperatures of tomato fruit, paste, and puree samples until shipped

20.1.08- Date tomato fruit, paste, and puree samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Tomato (Greenhouse)

### 10. TEST SYSTEM/CROP:

Tomato - Use a commercial variety. Report: variety (**indicate whether it is a small-fruited or large-fruited variety**), source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. **Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher. See below for assignment of small-fruited (cherry or grape tomato, generally less than 2 9/32 inches in diameter) or large-fruited variety.**

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last test substance application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect at least 12 fruit from at least 12 different plants per sample. If tomatoes are small-fruited (grape or cherry tomatoes), harvest a minimum of 24 fruit from at least 12 plants per sample. Each sample should be collected during a separate run through the entire plot. Take fruit from high and low areas of each plant. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb) for large-fruited tomatoes or a minimum of 2 lb (but preferably not more than 3 lb) for small-fruited tomatoes.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Large-Fruited Tomato Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit

LB	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
LC	02	PPP	X (+1)	12 fruits from at least 12 plants / 4 lb.	Fruit
LD	02	PPP	X (+1)	12 fruits from at least 12 plants / 4 lb.	Fruit

### 18.2 Large-Fruited Tomato Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
LB	01	Untreated	NA	12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit
	02	PPP		12 fruits from at least 12 plants / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples LC and LD.

### 18.3 All Small-Fruited Tomato Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
SB	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
SC	02	PPP	X (+1)	24 fruits from at least 12 plants / 2 lb.	Fruit
SD	02	PPP	X (+1)	24 fruits from at least 12 plants / 2 lb.	Fruit

### 18.4 Small-Fruited Tomato Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
SB	01	Untreated	NA	24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit
	02	PPP		24 fruits from at least 12 plants / 2 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC and SD.

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**Vegetable, cucurbit, group 9**

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## Cantaloupe (watermelon)

### **12. TEST SITE PREPARATION:**

Prepare or select a test site that has been maintained following good local agricultural practices for the production of melons including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Harvest the crop if needed on a regular basis as per standard commercial practice to prevent fruit from reaching a size too large or in such poor condition as to preclude use as samples. Document such harvests in the Field Data Book.**

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

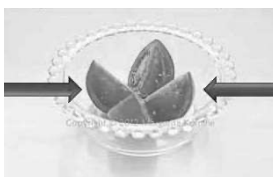
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect a minimum of 12 marketable sized fruit per sample from 12 separate plants. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse with clean water using a minimal of clean water. Pat lightly with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE FRUIT.**

Cut each fruit into quarters or eighths from stem end to blossom end. Gross sample weight should be a minimum of 4 lb (but preferably not more than 8 lb). If the sample weight does not exceed 8 lb before cutting, then all sections should be retained for the sample. If the sample weight needs to be reduced, retain two sections from the opposite sides of the fruit, including the center portion with seeds. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a

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freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 4 lb.	Fruit
B	01	Untreated	NA	12 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	12 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	12 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 4 lb.	Fruit
B	01	Untreated	NA	12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Cucumber

### 10. TEST SYSTEM/CROP:

Cucumber - Use a commercial variety. Report: variety (**indicate whether it is a pickling, slicing, or English variety, and whether it is a miniature or large-fruited variety**), source, lot number, date received, and other descriptive information if available. **Do not use "Armenian cucumber", which is a type of melon (*Cucumis melo* var. *flexuosus*)**.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments. See below for assignment of pickling, slicing, English, or miniature variety. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.**

### 12. TEST SITE PREPARATION:

Prepare or select a test site that has been maintained following good local agricultural practices for the production of cucumbers including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Harvest the crop if needed on a regular basis as per standard commercial practice to prevent fruit from reaching a size too large or in such poor condition as to preclude use as samples. Document such harvests in the Field Data Book.**

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### 17. RESIDUE SAMPLE COLLECTION:

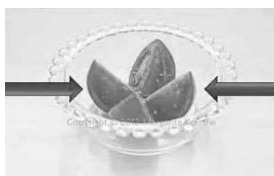
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect samples. For slicing varieties and English varieties collect a minimum of 12 fruits from 12 separate plants per sample. Pickling and miniature varieties require at least 24 fruits from at least 12 separate plants weighing a minimum of 4 lb (but preferably not more than 8 lb). Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse with clean water using a minimal of clean water. Pat lightly with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE FRUIT.**

For all types, cut each fruit into quarters from the stem end to blossom end. If the sample weight does not exceed 8 lb before cutting, then all quarters should be retained for the sample. If needed, reduce gross sample weight to a minimum of 4 lb (but preferably not more than 8 lb) by retaining two sections from the opposite sides of the fruit, including the center portion with seeds. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

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Opposite sections

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Sections 18.1 and 18.2) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:****18.1 All Field Trials with Small Varieties (Pickling and Miniature) except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
SB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
SC	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
SD	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
SB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC and SD.

**18.3 All Field Trials with Large Varieties (English and Slicing) except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits	Fruit
LB	01	Untreated	NA	12 fruits	Fruit
LC	02	PPP	X (+1)	12 fruits	Fruit
LD	02	PPP	X (+1)	12 fruits	Fruit

**18.4 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits	Fruit
LB	01	Untreated	NA	12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples LC and LD.

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## Cucumber (Greenhouse)

### 10. TEST SYSTEM/CROP:

Cucumber - Use a commercial variety of cucumber adapted to greenhouse production systems, either a large variety (e.g. an English type) or a miniature variety. Report the variety, source, lot number, date received, and other descriptive information, if available. **Do not use "Armenian cucumber", which is a type of melon (*Cucumis melo* var. *flexuosus*).**

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. The following table lists the type of cucumber to test in each greenhouse trial.

Large Greenhouse Variety (e.g. English Type)	Miniature Greenhouse Variety

### 12. TEST SITE PREPARATION:

Prepare or select a test site that has been maintained following good local agricultural practices for the production of cucumbers including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Harvest the crop if needed on a regular basis as per standard commercial practice to prevent fruit from reaching a size too large or in such poor condition as to preclude use as samples. Document such harvests in the Field Data Book.**

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### 17. RESIDUE SAMPLE COLLECTION:

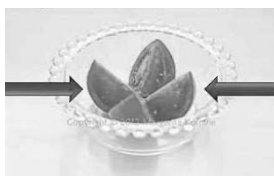
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect samples. For large varieties (e.g. English varieties) collect a minimum of 12 fruits from 12 separate plants per sample. Miniature varieties (harvested at 6-8 inches in length) require at least 24 fruits from at least 12 separate plants weighing a minimum of 4 lb (but preferably not more than 8 lb). Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse with clean water using a minimal of clean water. Pat lightly with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE FRUIT.**

For all types, cut each fruit into quarters from the stem end to blossom end. If the sample weight does not exceed 8 lb before cutting, then all quarters should be retained for the sample. If needed, reduce gross sample weight to a minimum of 4 lb (but preferably not more than 8 lb) by retaining two sections from the opposite sides of the fruit, including the center portion with seeds. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

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Opposite sections

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Sections 18.1 and 18.2) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:****18.1 All Field Trials with Miniature Varieties:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
SB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
SC	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
SD	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
SB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC and SD.

**18.3 All Field Trials with Large Varieties (English) except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits	Fruit
LB	01	Untreated	NA	12 fruits	Fruit
LC	02	PPP	X (+1)	12 fruits	Fruit
LD	02	PPP	X (+1)	12 fruits	Fruit

**18.4 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 fruits	Fruit
LB	01	Untreated	NA	12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit
	02	PPP		12 fruits	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples LC and LD.

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## Squash (Winter) or Squash (Summer)

### **12. TEST SITE PREPARATION:**

Prepare or select a test site that has been maintained following good local agricultural practices for the production of squash including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Harvest the crop if needed on a regular basis as per standard commercial practice to prevent fruit from reaching a size too large or in such poor condition as to preclude use as samples. Document such harvests in the Field Data Book.**

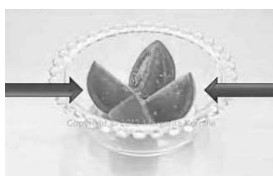
The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 12 marketable size fruit per sample from 12 separate plants. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 4 lb (but preferably not more than 8 lb). Cut each fruit into quarters or eighths from the stem end to the blossom end. If needed, reduce gross sample weight to a minimum of 4 lb (but preferably not more than 8 lb) by retaining two sections from the opposite sides of the fruit, including the center portion with seeds. If the sample weight does not exceed 8 lb before cutting, then all quarters or eighths should be retained for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Opposite sections

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse with clean water using a minimal of clean water. Pat lightly with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE FRUIT.**

**Decline trial XX@@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23).

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Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 4 lb.	Fruit
B	01	Untreated	NA	12 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	12 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	12 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 4 lb.	Fruit
B	01	Untreated	NA	12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit
	02	PPP		12 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

**Fruit, citrus, group 10**

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## Grapefruit

### **10. TEST SYSTEM/CROP:**

Grapefruit - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of grapefruit including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

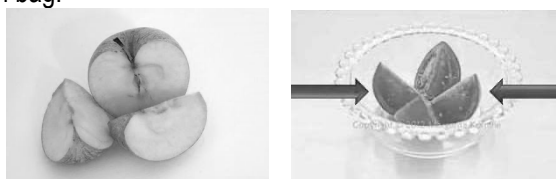
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last treatment, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 24 fruit per sample from at least 4 trees. Each sample should be collected during a separate run through the entire plot. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from the plot end trees.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Each sample should weigh a minimum of 4 lb (but preferably not more than 8 lb). Cut each fruit longitudinally into quarters with a clean knife on an uncontaminated surface. If the sample weighs less than 8 lb, then retain all four quarters. Otherwise, reduce gross sample weight by retaining only opposite quarters from each fruit for the sample. If further weight reduction is necessary, both of those opposite quarters may be cut longitudinally into two slices, and one slice from each quarter should be retained for the sample. If the retained slices are more than approximately 6 inches (15 cm) long, then they should each be cut into two smaller pieces, and both of the pieces should be retained for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

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Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:  
**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All field trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Lemon

### **10. TEST SYSTEM/CROP:**

LEMON - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of lemons including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

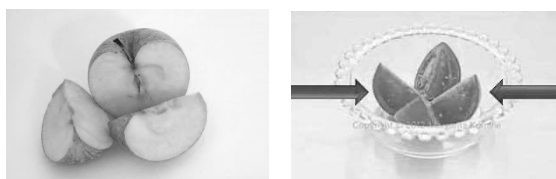
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last treatment, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 24 fruit per sample from at least 4 trees. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot end trees. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Each sample should weigh a minimum of 4 lb (but preferably not more than 8 lb). Cut each fruit longitudinally into quarters with a clean knife on an uncontaminated surface. If the sample weighs less than 8 lb, then retain all four quarters. Otherwise, reduce gross sample weight by retaining only opposite quarters from each fruit for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue

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from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All field trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Orange or Tangerine

### **10. TEST SYSTEM/CROP:**

ORANGE - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of oranges including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

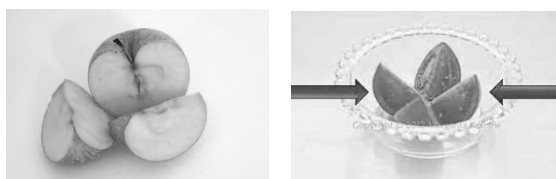
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 fruit per sample from at least 4 trees. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Each sample should weigh a minimum of 4 lb (but preferably not more than 8 lb). Cut each fruit longitudinally into quarters with a clean knife on an uncontaminated surface. If the sample weighs less than 8 lb, then retain all four quarters. Otherwise, reduce gross sample weight by retaining only opposite quarters from each fruit for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before

proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (See Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Fresh Orange Fruit to be Processed into Dried pulp, Juice and Oil (Field Trial XX):** Harvest approximately 450-500 lb of oranges from each plot at X ( $\pm 1$ ) days after the last test substance application. These samples will be processed into dried pulp, juice, and oil. For processing, oranges should be harvested as per commercial practice. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from plot ends.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before proceeding to the treated plot(s).

Samples should be collected in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID Number, Test Substance (chemical name listed in Section 15) complete sample ID (See Section 18.3), and harvest/sampling dates. See Protocol Section 19.2 for handling and shipping directions. Ship the large samples as "fresh" samples, within 1 day of collection, to the processing facility.

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

### **18.1 All field trials except Decline Trial XX@@:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>MINIMUM SAMPLE SIZE</b>	<b>CROP FRACTION</b>
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X ( $\pm 1$ )	24 fruits / 4 lb.	Fruit
D	02	PPP	X ( $\pm 1$ )	24 fruits / 4 lb.	Fruit

### **18.2 Decline Trial XX@@@:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>MINIMUM SAMPLE SIZE</b>	<b>CROP FRACTION</b>
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	450-500 lb.	Fresh Fruit
PT	02	PPP	X (+1)	450-500 lb.	Fresh Fruit

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

### 19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:

Contact the processing lab as soon as you know the date you expect to ship the large, fresh samples for processing, so that the lab will be ready to receive them and begin the processing part of the study as needed. If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in a refrigerator at approximately 4°C until they are shipped. At the time of shipping large samples, contact the processing lab (document this communication in the field data book). **Insert a true copy of Field Data Book Part 8B and a blank copy of Field**

**Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

### **19.3 PROCESSING:**

Immediately prior to processing oranges, remove a representative 24-fruit sample (approximately 4-6 lb. for each sample) of oranges from the untreated and treated samples, and immediately place in the freezer.

Using simulated commercial processing (provide detailed description of equipment and procedures), produce dried pulp, juice and oil from the untreated and treated samples. Process untreated fruit first, followed by treated fruit. From the untreated fruit collect one sample each of juice, dried pulp, and oil, using all available fruit after removing the 24-fruit sample. From the treated fruit collect one sample each of juice, dried pulp, and oil, using all available fruit after removing the 24 fruit sample.

Place samples in appropriate containers and label. **Divide each sample of juice and oil into separate containers of 50-150 grams. It is also acceptable to divide dried pulp samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Processed samples should be frozen as soon as possible after processing is completed. For these samples, the processing lab should follow Protocol Section 19 procedures required for freezing/shipping samples to the analytical lab.

If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical orange storage conditions to prevent test substance residue degradation.

Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. For analysis of processed fractions, send samples to: @@@

### **19.4 PROCESSED SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>APPROX. WGT. OR VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	4-6 lb	Whole fruit, just prior to processing
GT	02	PPP	4-6 lb	Whole fruit, just prior to processing
DPA	01	Untreated	All available	Dried pulp
DPT	02	PPP	All available	Dried pulp
JA	01	Untreated	All available	Juice
JT	02	PPP	All available	Juice
OA	01	Untreated	All available	Oil
OT	02	PPP	All available	Oil

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

### **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions

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20.1.02- Deviations from protocol and standard operating procedures

20.1.03- Date fresh orange fruit samples received

20.1.04- Storage temperatures until fresh orange fruit samples are processed into dried pulp, juice, and oil

20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing of samples into dried pulp, juice, and oil

20.1.07- Storage temperatures of orange fruit, dried pulp, juice, and oil until shipped

20.1.08- Date orange fruit, dried pulp, juice, and oil are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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**Fruit, pome, group 11**

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## Apple

### **10. TEST SYSTEM/CROP:**

APPLE - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of apples including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

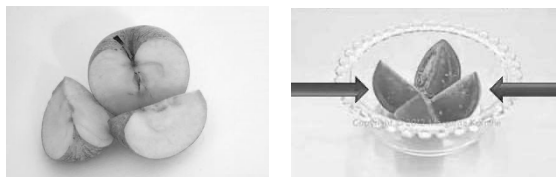
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 fruit per sample from at least 4 trees. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial

**All trials:** Fruit samples should each weigh a minimum of 4 lb (but preferably not more than 8 lb). **All apples, regardless of size, should be cut into halves or quarters (cut from stem end to opposite end into four or eight pieces).** If the sample size before cutting is greater than 8 lb, then only the opposite quarters or eighths should be retained for the sample. (If sample weight reduction is not needed, then all portions of the cut fruit should be retained.) Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue

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Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (See Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before proceeding to the treated plot(s).

Samples should be collected in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID Number, Test Substance (chemical name listed in Section 15) complete sample ID (See Section 18.3), and harvest/sampling dates. Do not freeze these samples. Ship the large samples as “fresh” samples, within 1 day of collection, to the processing facility. See Protocol Section 19.2 for residue sample handling and shipping directions.

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

[illegible]

	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### **18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT. OF SAMPLE</b>	<b>CROP FRACTION</b>
PA	01	Untreated	NA	70-100 lb.	Fruit
PT	02	PPP	X (+1)	70-100 lb.	Fruit

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### **19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT): (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

Contact the processing lab as soon as you know the date you expect to ship the large, fresh samples for processing, so that the lab will be ready to receive them and begin the processing part of the study as needed. If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in a refrigerator at approximately 4°C until they are shipped. At the time of shipping large samples, contact the processing lab (document

this communication in the field data book). **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

### **19.3 PROCESSING:**

Immediately prior to processing apples, remove representative “grab” samples of untreated and treated fruit from the larger samples (approximately 4-6 lb. for each sample). Place the “grab” samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc.

Using simulated commercial processing (provide detailed description of equipment and procedures), produce unpasteurized juice and wet pomace from the untreated and treated samples. Process untreated fruit first, followed by treated fruit. From the untreated fruit collect one sample each of a minimum of 1000 ml of unpasteurized juice and 1000 ml of wet pomace. From the treated fruit collect one sample each of a minimum of 1000 ml of unpasteurized juice and 1000 ml of wet pomace.

Place samples in appropriate containers and label. **Divide each sample of juice into separate containers of 50-150 grams. It is also acceptable to divide wet pomace samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Processed samples should be frozen as soon as possible after processing is completed. For these samples, the processing lab should follow Protocol Section 19 procedures required for freezing/shipping samples to the analytical lab.

If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical apple storage conditions to prevent test substance residue degradation. Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or “express” shipment. Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. For analysis of processed fractions, send samples to: @@@

### **19.4 PROCESSED SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>APPROX. WGT. OR VOL. OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	4-6 lb	Fruit
GT	02	PPP	4-6 lb	Fruit
PJA	01	Untreated	1000-2000 ml	Unpasteurized Juice
PJT	02	PPP	1000-2000 ml	Unpasteurized Juice
WPA	01	Untreated	1000-2000 ml	Wet Pomace
WPT	02	PPP	1000-2000 ml	Wet Pomace

### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

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The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
  - 20.1.02- Deviations from protocol and standard operating procedures
  - 20.1.03- Date fresh apple samples received
  - 20.1.04- Storage temperatures until fresh apple samples are processed into juice and wet pomace
  - 20.1.05- Processing Methodology (SOPs are acceptable)
- .....

20.1.06- Data collected and observations made during processing of samples into juice and wet pomace

20.1.07- Storage temperatures of fruit, juice, and wet pomace until shipped

20.1.08- Date fruit, juice, and wet pomace are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Mayhaw

### **10. TEST SYSTEM/CROP:**

MAYHAW - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of mayhaws including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 80 fruits per sample from at least 4 trees. Each sample should be collected during a separate run through the entire plot. Samples should weigh a minimum of 4 lb (but preferably not more than 6 lb). The fruits do not have to be ripe in color. Avoid sampling from end trees.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take fruits from all four quarters of each tree, high and low areas, fruit exposed and sheltered by foliage in proportion to fruit load distribution.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

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**18. FIELD RESIDUE SAMPLE INVENTORY:****18.1 All Field Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	80 fruits / 4 lb.	Fruit
B	01	Untreated	NA	80 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	80 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	80 fruits / 4 lb.	Fruit

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	80 fruits / 4 lb.	Fruit
B	01	Untreated	NA	80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Pear

### **10. TEST SYSTEM/CROP:**

PEAR - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of pears including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

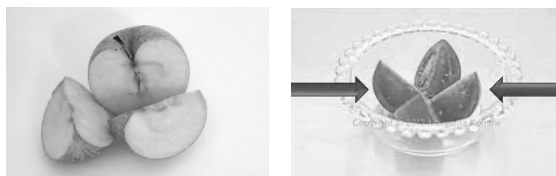
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect 24 fruit per sample from at least 4 trees. Each sample should be collected during a separate run through the entire plot. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from row ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Each sample should weigh a minimum of 4 lb. (but preferably not more than 8 lb.). **All pears, regardless of size, should be cut into halves or quarters (cut from stem end to opposite end into four or eight pieces).** If the sample size is greater than 8 lb, then **only the opposite quarters or eighths should be retained for the sample.** (If sample weight reduction is not needed, then all portions of the cut fruit should be retained.) Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another.

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If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Apple and Pear (Postharvest)

### **10. TEST SYSTEM/CROP:**

Apple and Pear (Post-Harvest) - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of: one untreated and one or more treated lots harvested crop fraction. See Part 15 for a list of assigned treatments.

The individual harvested lots shall be of adequate size to ensure that no more than 80% of the treated lot will be needed to provide the necessary plant material for the sample. Requirements for residue sampling are outlined in Parts 17 & 18.

**11.2** Starting with the untreated samples, collect/harvest, treat and sample one treatment before initiating the application of the next treatment. If another treatment must be made before the previous sample has been collected due to drying times, be sure that the different lots are placed far enough away from each other to insure no cross contamination can occur – separate tables at a minimum.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

**11.4** An independently prepared tank-mix must be used in each trial if a Field Research Director is assigned more than one trial in this study. Multiple trials at the same site must be conducted using at least 1) different application dates (at least 30 days) or 2) different varieties (confirm with the study director if this option is chosen.)

**11.5** Mark plots with identifiable markers containing at minimum the Field ID number and treatment number or treatment name that will persist for the duration of the field research trial or that can be readily replaced.

**11.6** This study is not designed for statistical evaluation of field data.

### **12. TEST SITE PREPARATION:**

Select fruit from a site that has been maintained following good local agricultural practices for the production of the crop, including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. The crop fraction to be treated will come from a site with a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years. Avoid sources of the commodity that have been treated with test substance, if the product is registered for field use.

Select a test site for the post-harvest treatment that has been maintained following good local agricultural practices for post-harvest applications. The post-harvest treatment test site should have a known pesticide history of a minimum of 1 month and preferably 3 months.

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## **14. TEST SUBSTANCE APPLICATION:**

**14. 1 Simulate commercial application practices** by applying the test substance in a manner that represents a major application technique that is used by area commercial growers, while following the directions specified in Section 15.

- For spray applications, use equipment that will provide uniform application of the test substance and result in adequate penetration and coverage of the mass of the commodity flowing in a packing line.
- For drench and dip applications use equipment that will provide adequate penetration and coverage of the commodity lots.
- The test substance, if applied in a mixture, must be applied to the test system within 2 hours of mixing.
- Each field trial requires a unique spray mixture; i.e., do not use the spray mixture from one field trial on another field trial.
- Agitate the test substance during the application, if practical, to ensure that it is well mixed.

**14. 2 Full Calibrations** for output and speed must be performed to ensure accurate delivery for the spray applications. A calibration consists of a minimum of three consecutive, documented checks for nozzle or hopper output and speed (equipment or walking speed). Individual nozzle outputs must not differ from the mean output by greater than 5% . Calibrations are not possible for the dip and drench applications, but care must be taken to meet directions and times specified in Part 15.

### **Discharge/Output Calibrations must be performed:**

Just prior to the first application of test substance<sup>1</sup>, completely calibrate the spray equipment used in the simulated packing lines. If additional treatments are to be made at later dates, another complete calibration must be performed and documented when application parameters or equipment components have changed between applications. Recalibration is required after any of the following have changed: application type; intended nozzle or hopper output; application equipment including nozzle tips, etc. The recalibration is required even if the equipment has been changed back to the parameters of the initial calibration. Use equipment logs to document changes in the equipment parameters.

**Rechecking the output**, at a minimum, is necessary for multiple applications, as long as parameters have not changed. A single output check may be conducted to confirm consistent delivery ( $\pm 5\%$  of the last complete calibration) just prior to subsequent applications.

### **The equipment must be completely re-calibrated if:**

- the recheck results in an output that differs from the mean of the complete calibration by greater than 5%
- the variation of any nozzle's output from the mean output is greater than 5% .

**Calculations for the amount of test substance to be applied will always be based upon mean output calculated from the most recent complete calibration data, not on the recheck results.**

Speed Calibrations of simulated packing lines must also be performed prior to the first test substance.

Complete calibration data from another trial (performed on the day of or day prior to the application in this trial) may be used. However, a recheck (single output check) must be performed just prior to the application in this trial, but subsequent to any other applications with the application equipment.

**14.3 Actual Application Rate:** Record actual application pass-times for the simulated packing line or in the Field Data Book and verify the accuracy of the application against the expected (calculated) results. **The application is considered acceptable if the accuracy is within -5% and +10% of the target rate specified in Section 15. If the**

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<sup>1</sup>"Just prior" includes the day prior to the application, but calibration on the day of use is preferred.

application did not meet this range, the Study Director must be notified of this deviation before proceeding with this trial. Record the actual dip times.

The submitted Field Data Book shall contain the original calibration data or a true copy of all complete calibrations referenced, along with the original data from the rechecks performed for this trial.

#### 15. APPLICATION TREATMENTS AND TIMING:

Trt#	Treatment	Target Rate of active ingredient	Target Rate Of formulated product*	Application Type	Spray Volume Range
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	PPP	lbs ai/100 gallons of water	lbs /100 gallons water	Drench	NA
03	PPP	lbs ai/100 gallons of water	lbs /100 gallons water	Dip	NA
04	PPP plus wax	lbs ai/100 gallons of water plus wax	lbs /100 gallons water plus wax	High Volume spray	95 to 105 gallons water or water plus wax per 200,000 lbs fruit
05	PPP plus wax	lbs ai/200,000 lbs fruit	0 lbs /200,000 lbs fruit	Low Volume spray	10 to 25 gallons per 200,000 lbs fruit
06	Trt #02 followed by Trt #05	lbs ai/100 gallons of water  Followed by  lbs ai/200,000 lbs fruit plus wax	lbs /100 gallons water  Followed by  lbs /200,000 lbs fruit plus wax	Drench  Followed by  Low Volume spray	NA   10 to 25 gallons per 200,000 lbs fruit

\*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).

**Make 1 application to fruit following harvest using the methods of application described below. The treatments for each trial are listed in the table below. Use a high Gloss Carnauba wax such as CARNAUBA PREMIUM from Decco. Mix the test substance with only wax in the Low-Volume spray. Mix with water plus wax in the High-Volume spray. Mix wax with water in a 1:4 ratio for the High-Volume spray e.g. If mixing 100 gallons of solution use 20 gallons of wax and 80 gallons of water for the High Volume spray.**

#### TRIAL XX ONLY

Fruit that are to receive two post-harvest treatments should be washed between applications after the fungicide solution had dried on the fruit surface following the first application. To wash fruit, run them through a simulated commercial washing facility that utilizes water, chlorine, Decco Neutral Cleaner 241 or Decco Alkaline Cleaner 125. After the wash water has drained from the fruit and/or they have been run across roller sponges, they should be treated with the second application as outlined in the table above. Fruit should be treated with the second application within 2 days of the first application. If fruit must be held longer than 16 hours but less than 2 days after the first application, then they should be placed in typical pome fruit storage conditions until they are treated.

**Treatment 02 – Drench treatment:** Mix a solution of PPP in water at the above ratio. Place apples or pears in a container such as a plastic basin or box with holes to allow the solution to drain. Pour the fungicide solution over the apples or pears. Let the solution drain and then lay out the fruit on a clean surface to dry. Do not let the fruit sit in a puddle of fungicide solution as they are drying, i.e. provide drainage from the fruit. If the lot needs to be drenched in more than one batch, finish application to the complete lot, before taking the samples.

**Treatment 03 – Dip Treatment:** Mix a solution of PPP in water at the ratio above. Dip the apples or pears in this solution for a period of 30 (+3) seconds and then remove and allow draining. Agitate the solution while dipping by moving the fruit up, down and all around in the dipping solution. After excess fungicide solution has drained from the fruit, lay the fruit out on a clean surface to dry. Do not let the fruit sit in a puddle of fungicide solution as they are drying, i.e. provide drainage from the fruit. If the lot needs to be dipped in more than one batch, finish application to the complete lot, before taking the samples.

**Treatment 04 – High Volume Spray:** For APPLES mix a solution of PPP in only Carnauba wax, do not add water. For PEARS mix a solution of PPP in water plus Carnauba wax at the ratio of one part Carnauba wax to 4.5 parts of soft water, e.g. for 10 gal. of mix, use 1 gal. of Carnauba wax and 9 gal. of soft water. Spray the apples or pears with the appropriate solution as they pass along a commercial packing line or simulated packing line. Apply the fungicide solution to apples or pears at the rate of 95 to 105 gallons of fungicide solution per 200,000 lbs of fruit. Lay out the fruit on a clean surface to dry. Do not let the fruit sit in a puddle of fungicide solution as they are drying, i.e. provide drainage from the fruit.

**Treatment 05 – Low Volume Spray:** For APPLES mix a solution of PPP in only Carnauba wax, do not add water. For PEARS mix a solution of PPP in water plus Carnauba wax at the ratio of one part Carnauba wax to 4.5 parts of soft water, e.g. for 10 gal. of mix, use 1 gal. of Carnauba wax and 9 gal. of soft water. Spray the apples or pears with the appropriate solution as they pass along a commercial packing line or simulated packing line. Apply the fungicide solution to apples or pears at the rate of 10 to 25 gallons of fungicide solution per 200,000 lbs of fruit. Lay out the fruit on a clean surface to dry. Do not let the fruit sit in a puddle of fungicide solution as they are drying, i.e. provide drainage from the fruit.

**Treatment 06 = Treatment 02 followed by drying followed by washing, followed by Treatment 05.**

In the field trials indicated below, phytotoxicity data must be collected at 7-14 days after each application using a 0-4 scale and entered into Field Data Book 6K2. If the samples are collected within 14 days of the last application, then the assessment should be made on the day of sample collection. If a rating of 2 or higher is given to a lot, then a follow-up rating is needed 7-14 days after that, even if there is no additional test substance application in the interim, unless this rating is given to the crop at sample collection.

Phytotoxicity Data is Required in the Field Trials Listed Below:			

## 16. SUPPLEMENTAL CROP TREATMENTS:

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop and selecting commercially acceptable crop at harvest. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

Note: some people do not grow their own post-harvest commodities. Does this need to be reworded?

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director.**

## 17. RESIDUE SAMPLE COLLECTION:

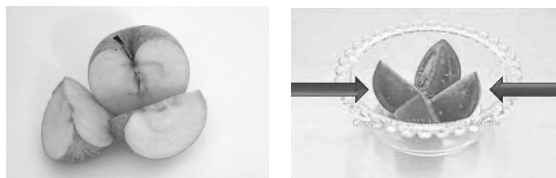
**All Trials (RAC samples):** Collect two samples from each lot. Each sample should be representative of the entire lot. Collect samples on the day of treatment after the fungicide solution has dried on the fruit. Start with the untreated lot. Collect a minimum of 24 fruit per sample. Each sample should be collected during a separate run

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through the entire lot.

Fruit samples should each weigh a minimum of 4 lb (but preferably not more than 8 lb). **All fruits, regardless of size, should be cut into quarters or eighths (cut from stem end to opposite end into four or eight pieces).** If the sample size is greater than 8 lb, then only the opposite quarters should be retained for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

During sampling, All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before proceeding to the treated plot(s). **If another treatment must be made before the previous sample has been collected, due to drying times, be sure that the different lots are placed far enough away from each other to insure no cross contamination can occur – separate tables at a minimum**

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (See Section 24). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Fresh Apples to be Processed into Juice and Wet Pomace (Field Trial NY25):** Collect 70 to 100 lb of apples from each lot on the day of treatment after the fungicide solution has dried on the fruit.

During sampling, All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated lot(s) before proceeding to the treated lot(s).

Samples should be collected in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID Number, Test Substance (chemical name listed in Section 15) complete sample ID (See Section 18.3), and harvest/sampling dates. Do not freeze these samples. Ship the large samples as “fresh” samples, within 1 day of collection, to the processing facility. See Protocol Section 19.2 for residue sample handling and shipping directions.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Apple Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
AA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
AB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
AC	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
AD	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

**18.2 Apple Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
AA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
AB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples AC and AD.

**18.3 All Pear Field Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
PA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
PB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
PC	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
PD	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

**18.4 Pear Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
PA	01	Untreated	NA	24 fruits / 4 lb.	Fruit
PB	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples PC and PD.

## **Fruit, stone, group 12**

## Cherry

### **10. TEST SYSTEM/CROP:**

CHERRY - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of cherries including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect cherries from a minimum of 4 trees. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 2 lb. of fruit (but preferably not more than 3 lb., weighed after removing pits and stems). Fruit does not need to be entirely ripe in color. Some fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from trees on row ends. Remove pits (seeds) and stems. If pit removal is done at a different location than the plots, then the harvested fruit should be protected from any excessively high temperatures during transport between the two places and appropriately documented. Process untreated sample first. Record the length of time from completion of the pit removal to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

.....

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Cherries without pits & stems
B	01	Untreated	NA	2 lb.	Cherries without pits & stems
C	02	PPP	X (+1)	2 lb.	Cherries without pits & stems
D	02	PPP	X (+1)	2 lb.	Cherries without pits & stems

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Cherries without pits & stems
B	01	Untreated	NA	2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems
	02	PPP		2 lb.	Cherries without pits & stems

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Peach

### **10. TEST SYSTEM/CROP:**

PEACH - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of peaches including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

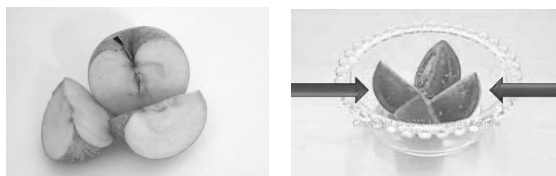
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 24 fruits from at least 4 trees. Each sample should be collected during a separate run through the entire plot. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from row ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Remove pits from fruit and sample at least 4 lb of fruit (but preferably not more than 8 lb). **If the sample size before pit removal is greater than 8 lb, then the fruits should be quartered (cut from stem end to opposite end into four pieces) and the opposite quarters retained for the sample.** If pit removal is done at a different location than the plots, then the harvested fruit should be protected from any excessively high temperatures during transport between the two places and appropriately documented. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the pit removal and/or sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Peach (Postharvest)

### **10. TEST SYSTEM/CROP:**

Peach (Postharvest) - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one or more treated lots harvested crop fraction. See Part 15 for a list of assigned treatments.

The individual harvested lots shall be of adequate size to ensure that no more than 80% of the treated lot will be needed to provide the necessary plant material for the sample. Requirements for residue sampling are outlined in Parts 17 & 18.

**11.2** Starting with the untreated samples first, collect/harvest, treat and sample one treatment before initiating the application of the next treatment. If another treatment must be made before the previous sample has been collected due to drying times, be sure that the different lots are placed far enough away from each other to insure no cross contamination can occur – separate tables at a minimum.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

**11.4** An independently prepared tank-mix must be used in each trial if a Field Research Director is assigned more than one trial in this study. Multiple trials at the same site must be conducted using at least 1) different application dates (at least 30 days) or 2) different varieties (confirm with the study director if this option is chosen.)

**11.5** Mark plots with identifiable markers containing at minimum the Field ID number and treatment number or treatment name that will persist for the duration of the field research trial or that can be readily replaced.

**11.6** This study is not designed for statistical evaluation of field data.

### **12. TEST SITE PREPARATION:**

Select fruit from a site that has been maintained following good local agricultural practices for the production of the crop, including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. The crop fraction to be treated will come from a site with a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years. Avoid sources of the commodity that have been treated with test substance, if the product is registered for field use.

Select a test site for the post-harvest treatment that has been maintained following good local agricultural practices for postharvest applications. The postharvest treatment test site should have a known pesticide history of a minimum of 1 month and preferably 3 months.



## **14. TEST SUBSTANCE APPLICATION:**

**14. 1 Simulate commercial application practices** by applying the test substance in a manner that represents a major application technique that is used by area commercial growers, while following the directions specified in Section 15.

- For spray applications, use equipment that will provide uniform application of the test substance and result in adequate penetration and coverage of the mass of the commodity flowing in a packing line.
- For drench and dip applications use equipment that will provide adequate penetration and coverage of the commodity lots.
- The test substance, if applied in a mixture, must be applied to the test system within 2 hours of mixing.
- Each field trial requires a unique spray mixture; i.e., do not use the spray mixture from one field trial on another field trial.
- Agitate the test substance during the application, if practical, to ensure that it is well mixed.

**14. 2 Full Calibrations** for output and speed must be performed to ensure accurate delivery for the spray applications.

A calibration consists of a minimum of three consecutive, documented checks for nozzle or hopper output and speed (equipment or walking speed). Individual nozzle outputs must not differ from the mean output by greater than 5%. Calibrations are not possible for the dip and drench applications, but care must be taken to meet directions and times specified in Part 15.

### **Discharge/Output Calibrations must be performed:**

Just prior to the first application of test substance<sup>2</sup>, completely calibrate the spray equipment used in the simulated packing lines. If additional treatments are to be made at later dates, another complete calibration must be performed and documented when application parameters or equipment components have changed between applications. Recalibration is required after any of the following have changed: application type; intended nozzle or hopper output; application equipment including nozzle tips, etc. The recalibration is required even if the equipment has been changed back to the parameters of the initial calibration. Use equipment logs to document changes in the equipment parameters.

**Rechecking the output**, at a minimum, is necessary for multiple applications, as long as parameters have not changed. A single output check may be conducted to confirm consistent delivery ( $\pm 5\%$  of the last complete calibration) just prior to subsequent applications.

### **The equipment must be completely re-calibrated if:**

- the recheck results in an output that differs from the mean of the complete calibration by greater than 5%
- the variation of any nozzle's output from the mean output is greater than 5% .

**Calculations for the amount of test substance to be applied will always be based upon mean output calculated from the most recent complete calibration data, not on the recheck results.**

Speed Calibrations of simulated packing lines must also be performed prior to the first test substance.

Complete calibration data from another trial (performed on the day of or day prior to the application in this trial) may be used. However, a recheck (single output check) must be performed just prior to the application in this trial, but subsequent to any other applications with the application equipment.

**14.3 Actual Application Rate:** Record actual application pass-times for the simulated packing line or in the Field Data Book and verify the accuracy of the application against the expected (calculated) results. **The application is considered acceptable if the accuracy is within -5% and +10% of the target rate specified in Section 15. If the application did not meet this range, the Study Director must be notified of this deviation before proceeding with this trial. Record the actual dip times.**

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<sup>2</sup>"Just prior" includes the day prior to the application, but calibration on the day of use is preferred.

The submitted Field Data Book shall contain the original calibration data or a true copy of all complete calibrations referenced, along with the original data from the rechecks performed for this trial.

## **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

## **17. RESIDUE SAMPLE COLLECTION:**

**All Trials (RAC samples):** Collect two samples from each lot. Each sample should be representative of the entire lot. Collect samples on the day of treatment after the fungicide solution has dried on the fruit. Start with the untreated lot. Collect a minimum of 24 fruit per sample. Each sample should be collected during a separate run through the entire lot. Up to 100% of the fruit treated with each post-harvest treatment may be collected for the two samples.

**All trials:** Remove pits from fruit and sample at least 4 lb of fruit (but preferably not more than 8 lb). **If the sample size before pit removal is greater than 8 lb, then the fruits should be quartered (cut from stem end to opposite end into four pieces) and the opposite quarters retained for the sample.** If pit removal is done at a different location than the plots, then the harvested fruit should be protected from any excessively high temperatures during transport between the two places and appropriately documented. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the pit removal and/or sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

During sampling, All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before proceeding to the treated plot(s). **If another treatment must be made before the previous sample has been collected, due to drying times, be sure that the different lots are placed far enough away from each other to insure no cross contamination can occur – separate tables at a minimum**

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (See Section 24). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

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**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

# 18. FIELD RESIDUE SAMPLE INVENTORY:

## 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Plum

### **10. TEST SYSTEM/CROP:**

PLUM - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of plums including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 fruits from a minimum of 4 trees. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Sampled plums do not have to be fully ripe in color. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove pits. If pit removal is done at a different location than the plots, then the harvested fruit should be protected from any excessively high temperatures during transport between the two places and appropriately documented. Fruit samples should each weigh a minimum of 4 lb. (but preferably not more than 6 lb.). Process untreated sample first. Record the length of time from completion of the pit removal to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of

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pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1), and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

**Trial XX only:** On the same day as the harvest of fresh plums, following the above sample collection procedures, collect from each plot one sample weighing a minimum of 30 lb. for processing into dried plums. Remove the pits, then immediately chill the pitted samples and transport the chilled (but not frozen) samples to the processing location within 36 hours of harvest. (If the drying process is begun on the same day as harvest, it is not necessary to pre-chill the plums.) If pit removal is done at a different location than the plots, then the harvested fruit should be protected from any excessively high temperatures during transport between the two places.

A 4-6 lb (minimum 4 lb) sub-sample shall be removed from the fresh plums just prior to processing into dried plums, labeled as Sample GA (untreated) and Sample GT (treated), and placed in a freezer without processing. Dry the remainder of the samples following Standard Operating Procedures in a manner that simulates commercial practices. Dry the treated and untreated fruit separately to a moisture content of 25% ( $\pm 5\%$ ). Record the exact procedures followed including drying time and temperatures. Indicate the SOP that has been followed to determine the moisture content, or provide a description of the procedure.

When drying is complete, bag one dried fruit sample from each treatment, weighing a minimum of 4 lb. per sample (but preferably not more than 6 lb. per sample). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Plums without pits
B	01	Untreated	NA	24 fruits / 4 lb.	Plums without pits
C	02	PPP	X (+1)	24 fruits / 4 lb.	Plums without pits
D	02	PPP	X (+1)	24 fruits / 4 lb.	Plums without pits

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Plums without pits
B	01	Untreated	NA	24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits

	02	PPP		24 fruits / 4 lb.	Plums without pits
	02	PPP		24 fruits / 4 lb.	Plums without pits

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 DRIED PLUM RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	4 lb.	Plums without pits
GT	02	PPP	X (+1)	4 lb.	Plums without pits
DA	01	Untreated	NA	4 lb.	Dried plums without pits
DT	02	PPP	X (+1)	4 lb.	Dried plums without pits

### 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. Shipment of frozen samples will be by freezer truck or express shipment. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

### 20. FIELD DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)

- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- In the dried plum trial only, include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **Berry group 13 and other small fruit**



## Blueberry

### **10. TEST SYSTEM/CROP:**

BLUEBERRY - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of blueberries including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect marketable-sized berries. Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Berries should be mature in size and mostly mature in color.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

In highbush or rabbiteye blueberry plots, harvest from at least 4 bushes, excluding the bushes at the ends of the plot. Take the fruit from at least 12 areas of the plot, and from high and low areas. Collect fruit exposed and sheltered by foliage.

In lowbush blueberry plots, collect the fruit from a swath diagonally across the plot. Harvest berries exposed and sheltered by foliage.

Try to collect fruit free of stems. Remove leaves and other trash.

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**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
C	02	PPP	X (+1)	2 lb.	Berries
D	02	PPP	X (+1)	2 lb.	Berries

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Caneberry or Currant

### **10. TEST SYSTEM/CROP:**

CANEBERRY - Use a commercial variety of raspberry. (Blackberry may be used in one or two of the Oregon trials and in the California trial.) Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of caneberries including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect marketable-sized berries. Each sample should be collected during a separate run through the entire plot.

For each sample, take the fruit from at least 12 separate areas of the plot, from high and low areas, fruit exposed and sheltered by foliage. Avoid sampling from the end crowns (canes). Try to collect fruit free of stems. Remove leaves and other trash.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Berries should be mature in size and mostly mature in color.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

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Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
C	02	PPP	X (+1)	2 lb.	Berries
D	02	PPP	X (+1)	2 lb.	Berries

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Cranberry

### **10. TEST SYSTEM/CROP:**

CRANBERRY - Use a commercial variety. Report: variety, age of vines, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of cranberries including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect marketable-sized cranberries (berries do not have to be ripe in color) from 12 separate areas in the plot. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Avoid sampling from plot ends.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### **18. FIELD RESIDUE SAMPLE INVENTORY:**

#### **18.1 All Field Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Fruit
B	01	Untreated	NA	2 lb.	Fruit

C	02	PPP	X (+1)	2 lb.	Fruit
D	02	PPP	X (+1)	2 lb.	Fruit

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Fruit
B	01	Untreated	NA	2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit
	02	PPP		2 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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#### **10. TEST SYSTEM/CROP:**

GOOSEBERRY - Use a commercial variety. Report: variety, age of shrubs, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

#### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of gooseberries including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

#### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

#### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). (Ripeness suitable for the fresh market is preferred, but ripeness suitable only for processing is acceptable. If the berries are at a level of ripeness that is suitable for processing but not for fresh market then this should be documented in the Field Data Book.) At X ( $\pm 1$ ) days after last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect berry samples that weigh a minimum of 2 lb (but preferably not more than 3 lb). Each sample should be collected during a separate run through the entire plot. Take berries from at least 12 separate areas of the plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse with clean water using a minimal of clean water. Pat lightly with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE FRUIT.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
C	02	PPP	X (+1)	2 lb.	Berries
D	02	PPP	X (+1)	2 lb.	Berries

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.



## Grape

### **10. TEST SYSTEM/CROP:**

GRAPE - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of grapes including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Samples for each treatment must be collected from 12 separate vines, so the test plots must be chosen accordingly. For the purposes of this study, a “vine” is defined as a whole plant.**

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated samples, collect at least 12 bunches of grapes (or portions of 12 bunches) from (at least 12) separate vines (separate plants). Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each treated and untreated sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Avoid sampling from end vines. Take grapes from both treated sides of each vine, from high and low areas, fruit exposed and sheltered by foliage.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag

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prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**Trial XX1 only:** On the same day as the harvest of fresh grapes, also collect one untreated sample and one treated sample of approximately 30 to 45 lb each to be dried into raisins. A 2-3 lb (minimum 2 lb) sub-sample shall be removed from the fresh grapes just prior to processing into raisins, labeled as Sample RGA (untreated) and Sample RGT (treated), and placed in a freezer without processing.

The processing into raisins is the responsibility of the Field Research Director of this trial, and should be done in a manner similar to commercial practices. The raisins should be dried outdoors on a clean, flat surface, to a moisture content of 15% ( $\pm 3\%$ ). The raisins may be moved to a covered area if precipitation is imminent. Record the exact procedures followed including drying time and temperatures. Indicate the SOP that has been followed to determine the moisture content, or provide a description of the procedure.

When the raisins have sufficiently dried, collect one untreated sample (Sample RA) and one treated sample (Sample RT) of at least 3 lb each. Stems should be removed from the collected samples.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**\*\*When using IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**Samples for Processing (Trial XX2 only):** Collect one untreated (sample PA) and one treated sample (PT) of approximately 50-60 lb each to be processed into juice.

During harvest and sampling, All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation from the untreated plot(s) before proceeding to the treated plot(s).

Samples should be collected in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID Number, Test Substance (chemical name listed in Section 15) complete sample ID (See Section 18.3), and harvest/sampling dates. Do not freeze these samples. Ship the large samples as "fresh" samples, within 1 day of collection, to the processing facility. See Protocol Section 19.2 for residue sample handling and shipping directions.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 bunches / 2 lb.	Fruit
B	01	Untreated	NA	12 bunches / 2 lb.	Fruit
C	02	PPP	X (+1)	12 bunches / 2 lb.	Fruit
D	02	PPP	X (+1)	12 bunches / 2 lb.	Fruit

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**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 bunches / 2 lb.	Fruit
B	01	Untreated	NA	12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit
	02	PPP		12 bunches / 2 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

**18.3 RAISIN RESIDUE SAMPLE INVENTORY: Trial XX1 only**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
RGA	01	Untreated	NA	2 lb.	Fruit (sub-sample)
RGT	02	PPP	X (+1)	2 lb.	Fruit (sub-sample)
RA	01	Untreated	NA	3 lb.	Raisins
RT	02	PPP	X (+1)	3 lb.	Raisins

**18.4 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX2 only**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX.WGT. OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	50-60 lb.	Fruit (for juice)
PT	02	PPP	X (+1)	50-60 lb.	Fruit (for juice)

**19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

See below for instructions for handling residue samples (raisins and fruit not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

**19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing and Raisin Samples)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the

laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

Immediately place samples PA and PT in containers with a means of maintaining a cool temperature and transport them to the processing facility. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

## **19.3 PROCESSING:**

Monitor sample-storage temperature at the processing facility prior to processing. As soon as possible after receiving samples PA and PT they should be processed into juice following simulated commercial practice after first removing a 2-3 lb "grab" sample of fruit from each (these "grab" samples should be labeled as samples PGA and PGT, respectively).

Place samples in appropriate containers and label with sample ID, crop fraction, field ID number, treatment number, treated/untreated, name of Field Research Director, date and initials of the person making these entries. Freeze samples at temperatures generally less than -18 °C (0 °F) until shipped. If possible, ship samples within 14 days of processing. **Divide each sample of juice into separate containers of 50-150 grams. Each portion of the divided sample should be representative of the whole sample.**

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for specific instructions. Ship by freezer truck (such as ACDS), overnight air express, or by any other carrier that maintains frozen sample integrity. When shipping by a means other than a freezer truck, pack all samples in dry ice (approximately 3 lb. dry ice per 1 lb.). All storage temperatures are to be monitored and documented. Send samples to: @@@

## **19.4 PROCESSED RESIDUE SAMPLES INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT. RANGE OR VOLUME OF SAMPLE</b>	<b>CROP FRACTION</b>
PGA	01	Untreated	NA	2-3 lb.	Fruit
PGT	01	PPP	X (+1)	2-3 lb.	Fruit
JA	02	Untreated	NA	1000-2000 ml	Juice
JT	02	PPP	X (+1)	1000-2000 ml	Juice

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

20.01- Names of all personnel conducting specific research functions

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- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- In the raisin trial only, include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date fresh grape samples received
- 20.1.04- Storage temperatures until fresh grape samples are processed into juice
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into juice
- 20.1.07- Storage temperatures of fruit and juice until shipped
- 20.1.08- Date fruit and juice are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in

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Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Juneberry

### **10. TEST SYSTEM/CROP:**

JUNEBERRY - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of juneberries including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect marketable-sized berries. Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Berries should be mature in size and mostly mature in color.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Harvest from at least 4 bushes, excluding the bushes at the ends of the plot. Take the fruit from at least 12 areas of the plot, and from high and low areas. Collect fruit exposed and sheltered by foliage.

Try to collect fruit free of stems. Remove leaves and other trash.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the

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untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
C	02	PPP	X (+1)	2 lb.	Berries
D	02	PPP	X (+1)	2 lb.	Berries

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Berries
B	01	Untreated	NA	2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries
	02	PPP		2 lb.	Berries

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.



## Kiwifruit

### 10. TEST SYSTEM/CROP:

KIWIFRUIT - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### 12. TEST SITE PREPARATION:

Select a test site that has been maintained following good local agricultural practices for the production of kiwifruit including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 marketable-sized fruits from a minimum of 4 vines. Each sample should be collected during a separate run through the entire plot. Take fruits from high and low areas, exposed and sheltered by foliage. Avoid sampling from plot ends. Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit

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B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Strawberry

### **10. TEST SYSTEM/CROP:**

STRAWBERRY - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of strawberries including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). (Ripeness suitable for the fresh market is preferred, but ripeness suitable only for processing is acceptable. If the berries are at a level of ripeness that is suitable for processing but not for fresh market then this should be documented in the Field Data Book.) At X (+1) days after last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect berry samples that weigh a minimum of 2 lb (but preferably not more than 3 lb). Each sample should be collected during a separate run through the entire plot. Take berries from at least 12 separate areas of the plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove caps, retaining the berries for the sample.

If loose soil or other debris adheres to fruit, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse using a minimal amount of clean water. Pat lightly with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE FRUIT.

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**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

### **18.1 All Field Trials except Decline Trial XX@@:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>MINIMUM SAMPLE SIZE</b>	<b>CROP FRACTION</b>
A	01	Untreated	NA	2 lb.	Berries without caps
B	01	Untreated	NA	2 lb.	Berries without caps
C	02	PPP	X (+1)	2 lb.	Berries without caps
D	02	PPP	X (+1)	2 lb.	Berries without caps

### **18.2 Decline Trial XX@@:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>MINIMUM SAMPLE SIZE</b>	<b>CROP FRACTION</b>
A	01	Untreated	NA	2 lb.	Berries without caps
B	01	Untreated	NA	2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps
	02	PPP		2 lb.	Berries without caps

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

**Nut, tree, group 14**

## Almond

### **10. TEST SYSTEM/CROP:**

ALMOND - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of almonds including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the almonds by shaking the trees or hitting the branches to induce the nuts to drop onto tarps spread on the ground. Allow the nuts to dry on the ground for 5-10 days before collecting samples. If wet weather is possible during the drying period, the nuts may be moved to a protected area for drying.

When the nuts have dried, collect approximately 5-15 lb of nuts for each sample. Each sample should be collected during a separate run through the entire plot. Collect the nuts from at least 4 trees, avoiding end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.) Nuts should be collected from various areas beneath the trees. (If the nuts have previously been moved to a protected area, then they should be collected from various areas in the location in which they have been drying.) Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Remove hulls and retain at least 2 lb of hulls per sample for samples E-H. Impartially select a minimum of 2 lb (but preferably not more than 4 lb) of nutmeat per sample. Avoid sampling from the plot ends. Hulls should be removed on the same day as harvest of the nuts. Alternatively, the nuts may be stored at approximately 40 °F for up to two days prior to removal of the hulls.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

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## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
NA	01	Untreated	NA	2 lb.	Nutmeat
NB	01	Untreated	NA	2 lb.	Nutmeat
NC	02	PPP	X (+1)	2 lb.	Nutmeat
ND	02	PPP	X (+1)	2 lb.	Nutmeat
HA	01	Untreated	NA	2 lb.	Hulls
HB	01	Untreated	NA	2 lb.	Hulls
HC	02	PPP	X (+1)	2 lb.	Hulls
HD	02	PPP	X (+1)	2 lb.	Hulls

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
NA	01	Untreated	NA	2 lb.	Nutmeat
NB	01	Untreated	NA	2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
HA	01	Untreated	NA	2 lb.	Hulls
HB	01	Untreated	NA	2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls
	02	PPP		2 lb.	Hulls

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples NC, ND, HC, and HD.

## Hazelnut

### **10. TEST SYSTEM/CROP:**

HAZELNUT - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of hazelnuts including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, spread tarps on the ground for nuts to fall on. If necessary, tarps can be laid out and samples collected from one tree at a time. Induce nuts to fall by shaking and/or hitting tree limbs. For each sample, collect approximately 8 - 12 lb of hazelnuts (nuts with shells that meet commercial standards) from tarps in each plot. If husks are present, remove husks before collecting the sample. Collect samples from a minimum of 4 trees. Nuts should be collected from various areas beneath the trees. Avoid sampling end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.) Each sample should be collected during a separate run through the entire plot.

Allow the nuts to dry on the tarps for 2-10 days or approximately 6% moisture. (Samples may be removed from the orchard and taken back to the research station to dry.) If wet weather is possible during the drying period, the nuts may be moved to a protected area for drying. It is acceptable to place the nuts in a dryer set at 95-104° F (35-40° C) for up to 4 days. If dryer capacity is limited, untreated samples may be collected for drying up to four days before treated samples.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove shells to yield at least 2 lb (but preferably not more than 4 lb) of nutmeats per plot sample. Dried nuts may be stored at approximately 40 °F for up to 4 days prior to removal of the shells. Shells should be removed within 4 days of drying.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.



**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
C	02	PPP	X (+1)	2 lb.	Nutmeat
D	02	PPP	X (+1)	2 lb.	Nutmeat

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## **10. TEST SYSTEM/CROP:**

PECAN - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

## **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of pecans including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

## **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the pecans by shaking the trees or hitting the branches to induce the nuts to drop onto tarps spread on the ground. Allow the nuts to dry on the ground for 5-10 days before collecting samples. If wet weather is possible during the drying period, the nuts may be moved to a protected area for drying. When the nuts have dried, collect approximately 8 - 12 lb of pecans (nuts with shells that meet commercial standards) from each plot. Each sample should be collected during a separate run through the entire plot. Collect samples from a minimum of 4 trees. Nuts should be collected from various areas beneath the trees. Avoid sampling end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove shells to yield at least 2 lb (but preferably not more than 4 lb) of nutmeats per plot sample. Shells should be removed on the same day as harvest of the nuts. Alternatively, the nuts may be stored at approximately 40 °F for up to two days prior to removal of the shells.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

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**18.1 All Field Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
C	02	PPP	X (+1)	2 lb.	Nutmeat
D	02	PPP	X (+1)	2 lb.	Nutmeat

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Pistachio

### **10. TEST SYSTEM/CROP:**

PISTACHIO - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of pistachios including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the pistachios by shaking the trees or hitting the branches to induce the nuts to drop into a mechanical harvester or onto tarps spread on the ground. Collect approximately 5-15 lb of nuts for each sample. Each sample should be collected during a separate run through the entire plot. Collect the nuts from at least 4 trees. If collecting from tarps, the nuts should be collected from various areas beneath the tree after they have fallen onto the ground. Avoid sampling from end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove shells. Impartially select a minimum of 2 lb (but preferably not more than 3 lb) of nutmeat per sample. Shells should be removed on the same day as harvest of the nuts. Alternatively, the nuts may be stored at approximately 40 °F for up to two days prior to removal of the shells.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### **18. FIELD RESIDUE SAMPLE INVENTORY:**

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**18.1 All Field Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
C	02	PPP	X (+1)	2 lb.	Nutmeat
D	02	PPP	X (+1)	2 lb.	Nutmeat

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Walnut

### 10. TEST SYSTEM/CROP:

WALNUT - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### 12. TEST SITE PREPARATION:

Select a test site that has been maintained following good local agricultural practices for the production of walnuts including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the walnuts by shaking the trees to induce the nuts to drop onto tarps spread on the ground. Collect approximately 5-15 lb of nuts for each sample. Each sample should be collected during a separate run through the entire plot. Collect the nuts from at least 4 trees. The nuts should be collected from various areas beneath the tree after they have fallen onto the ground. Avoid sampling from end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove shells. Impartially select a minimum of 2 lb (but preferably not more than 3 lb) of nutmeat per sample. Shells should be removed on the same day as harvest of the nuts. Alternatively, the nuts may be stored at approximately 40 °F for up to two days prior to removal of the shells.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Field Trials except Decline Trial XX@@:

SAMPLE	TRT#	TREATMENT	DAYS AFTER	MINIMUM	CROP
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ID			LAST APPLIC.	SAMPLE SIZE	FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
C	02	PPP	X ( $\pm$ 1)	2 lb.	Nutmeat
D	02	PPP	X ( $\pm$ 1)	2 lb.	Nutmeat

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Nutmeat
B	01	Untreated	NA	2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Grain, cereal, group 15



## **17. RESIDUE SAMPLE COLLECTION:**

### **Hay Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). When the grain is at the milk to soft dough stage, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the hay in a manner simulating commercial practices. Do not separate the grain from the stalks for the hay sample (hay sample should be the entire stalk including grain).

The hay sample must be collected when it has a moisture content of approximately 10-20% (approximately 80% to 90% dry matter). If samples are greater than approximately 20% moisture content, then additional field drying may be necessary. Each sample should weigh a minimum of 1 lb (but preferably not more than 2 lb). If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

### **Grain and Straw Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest barley in a manner simulating commercial practices. Separate the barley into grain and straw and collect samples of each. Each grain sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each straw sample should weigh a minimum of 1 lb (but preferably not more than 2 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

### **All Samples:**

At the stages discussed in the paragraphs above: Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the barley using an experimental plot harvester. Collect duplicate samples of each fraction from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from plot ends. Impartially collect two samples of each fraction by taking at least 12 grab samples of grain and straw at uniform intervals over the plot.

Alternatively, the barley can be harvested and collected in the following manner. For each sample, collect the fraction from at least 12 separate areas of the plot. **For grain and straw samples only:** Place the harvested stalks in uncontaminated plastic bags. Thresh the heads and collect duplicate grain samples a minimum of 2 lb (but preferably not more than 3 lb). Retain the straw and collect duplicate straw samples weighing a minimum of 1 lb (but preferably not more than 2 lb). **For hay samples only:** take duplicate samples of the hay (sampled as the whole cut stalk with the grain still attached) having a moisture content of approximately 10% to 20% (approximately 80% to 90% dry matter). (Percent dry matter may be estimated.) Hay samples should weigh a minimum of 1 lb (but preferably not more than 2 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**For Samples to be Processed into Bran, Flour, and Pearled Barley (Trial XX only):** Collect one sample from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest barley in a manner simulating commercial practices using an experimental plot harvester. Separate the barley into grain and straw and retain only the grain. Each

grain sample should weigh approximately 100-120 lb.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
GC	02	PPP	X (+1)	2 lb.	Grain
GD	02	PPP	X (+1)	2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
SC	02	PPP	X (+1)	1 lb.	Straw
SD	02	PPP	X (+1)	1 lb.	Straw
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
HC	02	PPP	X (+1)	1 lb.	Hay
HD	02	PPP	X (+1)	1 lb.	Hay

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	GA	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain

	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		2 lb.	Grain
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples GC, GD, SC, SD, HC, and HD.

### 18.3 PROCESSING RESIDUE SAMPLE FOR PROCESSING INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	100-120 lb.	Grain
PT	02	PPP	X (+1)	100-120 lb.	Grain

### 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in raw data.

Prior to shipment, hold samples frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck..** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in a freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

#### **19.3 PROCESSING:**

Store the barley grain at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

Immediately prior to processing barley, remove representative "grab" samples of untreated and treated grain from the larger samples (approximately 2-4 lb for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce bran, flour, and pearled barley (minimum 2 lb per sample). Process untreated sample first, followed by treated sample. All trials: Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of each matrix from both the untreated and treated barley samples. Process each sample separately.

Place samples in appropriate containers and label. **Processed samples may be divided into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. RANGE OF SAMPLE	CROP FRACTION
GA	01	Untreated	NA	2-4 lb.	Grain
GT	02	PPP	X (+1)	2-4 lb.	Grain
BA	01	Untreated	NA	2-4 lb.	Bran
BT	02	PPP	X (+1)	2-4 lb.	Bran
FA	01	Untreated	NA	2-4 lb.	Flour
FT	02	PPP	X (+1)	2-4 lb.	Flour

PBA	01	Untreated	NA	2-4 lb.	Pearled Barley
PBT	02	PPP	X (+1)	2-4 lb.	Pearled Barley

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

### **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date barley samples received

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20.1.04- Storage temperatures until unprocessed barley samples are processed into bran, flour, and pearled barley

20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing of samples into bran, flour, and pearled barley

20.1.07- Storage temperatures of unprocessed grain and processed samples until shipped

20.1.08- Date unprocessed grain and processed samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Chia

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, collect chia seed as done commercially from at least 12 areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends. (Chia seed is mature after the flower petals have begun to drop off.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 1 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

Avoid sampling from the plot ends.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	1 lb.	Seed
B	01	Untreated	NA	1 lb.	Seed
C	02	PPP	X (+1)	1 lb.	Seed
D	02	PPP	X (+1)	1 lb.	Seed

#### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	1 lb.	Seed
B	01	Untreated	NA	1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed

	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Corn (Field)

### **17. RESIDUE SAMPLE COLLECTION:**

#### **Kernels plus Cob with Husks Removed Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At milk stage (approximately 17-24 days after silking initiation), starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest stalks of corn from at least 12 separate areas of the plot in a manner simulating commercial practices. Remove the ears from the stalks, and then remove the husks from the ears. Remove the husks. Collect at least 2 lb of kernels plus cob per sample (but preferably not more than 5 lb). Cut each corn cob with a clean knife into at least 2 smaller segments not longer than approximately 6 inches (15 cm) and retain all of the segments for the sample. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

#### **Forage Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At milk stage (approximately 17-24 days after silking initiation) starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest corn forage in a manner simulating commercial practices from at least 12 plants. Remove the ears from the stalks (stems). Select 12 stems and separate them into 3 groups of 4 stems each. Divide each stem with leaves attached into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 stems are included in each sample. Collect at least 2 lb per sample (but preferably not more than 5 lb).

#### **Grain Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest ears of corn in a manner simulating commercial practices from at least 12 plants. Collect at least 2 lb (but preferably not more than 4 lb) of grain per sample.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

#### **Stover Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest at least 12 stalks of corn in a manner simulating commercial practices. Remove the ears from the stalks and discard. Separate the stalks into 3 groups of at least 4 stalks each. Divide each stalk into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 stalks are included in each sample. Allow the stalks to dry. When the stalks have reached a moisture content of approximately 15-20% (80-85% dry matter), then collect stover samples. (Percent dry matter may be estimated.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for

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the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
KA	01	Untreated	NA	2 lb.	Kernels + Cob with Husks Removed
KB	01	Untreated	NA	2 lb.	Kernels + Cob with Husks Removed
KC	02	PPP	NA	2 lb.	Kernels + Cob with Husks Removed
KD	02	PPP	NA	2 lb.	Kernels + Cob with Husks Removed
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP	NA	2 lb.	Forage
FD	02	PPP	NA	2 lb.	Forage
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
GC	02	PPP	X (±1)	2 lb.	Grain
GD	02	PPP	X (±1)	2 lb.	Grain
SA	01	Untreated	NA	12 stalks	Stover
SB	01	Untreated	NA	12 stalks	Stover
SC	02	PPP	X (±1)	12 stalks	Stover
SD	02	PPP	X (±1)	12 stalks	Stover

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
KA	01	Untreated	NA	2 lb.	Kernels + Cob with Husks Removed
KB	01	Untreated	NA	2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
	02	PPP		2 lb.	Kernels + Cob with Husks Removed
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage

	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
SA	01	Untreated	NA	12 stalks	Stover
SB	01	Untreated	NA	12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples KC, KD, FC, FD, GC, GD, SC, and SD.

## **17. RESIDUE SAMPLE COLLECTION:**

### **Forage Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest corn in a manner simulating commercial practices from at least 12 separate areas of the plot. Separate the corn into forage samples (fresh-cut stalks with ears removed) and samples of kernels + cob with the husks removed (see below for instructions for kernels + cob with husks removed). Each forage sample should weigh a minimum of 2 lb (but preferably not more than 5 lb). For forage samples: Select 12 stalks and separate them into 3 groups of 4 stalks each. Divide each stalk with leaves attached into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 stalks are included in each sample.

### **Stover Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest stalks of corn from at least 12 separate areas of the plot in a manner simulating commercial practices and allow the stalks to dry. Remove the ears from the stalks. When the stalks have reached a moisture content of approximately 15-20% (80-85% dry matter), then collect stover samples. (Percent dry matter may be estimated.) If local weather conditions will not permit adequate drying in the field, the stalks may be moved to a protected area in order to achieve the required moisture content.

### **Kernels + Cob with Husks Removed:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest stalks of corn from at least 12 separate areas of the plot in a manner simulating commercial practices. Remove the ears from the stalks, and then remove the husks from the ears. Remove the husks. Collect at least 4 lb per sample (but preferably not more than 8 lb) of kernels + cob with husks removed. Cut each corn cob with a clean knife into at least 2 smaller segments not longer than approximately 6 inches (15 cm) and retain all of the segments for the sample. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

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### 18.1 All trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
KA	01	Untreated	NA	2 lb.	Kernels + Cob with Husks Removed
KB	01	Untreated	NA	2 lb.	Kernels + Cob with Husks Removed
KC	02	PPP	NA	2 lb.	Kernels + Cob with Husks Removed
KD	02	PPP	NA	2 lb.	Kernels + Cob with Husks Removed
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP	NA	2 lb.	Forage
FD	02	PPP	NA	2 lb.	Forage
SA	01	Untreated	NA	12 stalks	Stover
SB	01	Untreated	NA	12 stalks	Stover
SC	02	PPP	X ( $\pm 1$ )	12 stalks	Stover
SD	02	PPP	X ( $\pm 1$ )	12 stalks	Stover

## 18.2 Decline Trial XX@@@:

[illegible]

	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples KC, KD, FC, FD, SC, and SD.

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## **17. RESIDUE SAMPLE COLLECTION:**

### **Hay Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). When the grain is at the early boot stage or approximately 40 inches (1 meter) tall (whichever comes first), starting with the untreated plot or utilizing separate personnel for the treated and untreated plots starting with the untreated plot or utilizing separate personnel for the treated and untreated plots or utilizing separate personnel for the treated and untreated plots, harvest the hay in a manner simulating commercial practices. Do not separate the grain from the stalks for the hay sample (hay sample should be the entire stalk including grain).

The hay sample must be collected when it has a moisture content of approximately 10-20% (approximately 80% to 90% dry matter). (Percent dry matter may be estimated.) If samples are greater than approximately 20% moisture content, then additional field drying may be necessary. Each sample should weigh a minimum of 1 lb (but preferably not more than 2 lb). If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area.

### **Forage Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). When the millet is at the 10 inch stage to early boot stage, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the forage in a manner simulating commercial practices. The forage sample must be collected when it has a moisture content of approximately 65-75% (approximately 25-35% dry matter). (Percent dry matter may be estimated.) Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

### **Grain and Straw Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest millet in a manner simulating commercial practices. Separate the millet into grain and straw and collect samples of each. Each grain sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each straw sample should weigh a minimum of 1 lb (but preferably not more than 2 lb). [Millet grain = kernel + hull, *except* pearl millet grain = kernel with hull removed.]

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

### **All Samples:**

At the stages discussed in the paragraphs above: Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the millet using an experimental plot harvester. Collect duplicate samples of each fraction from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from plot ends. Impartially collect two samples of each fraction by taking at least 12 grab samples of grain and straw at uniform intervals over the plot.

Alternatively, the millet can be harvested and collected in the following manner. For each sample, collect the fraction from at least 12 separate areas of the plot (cut at approximately 15 cm above the ground).

**For grain and straw samples only:** Place the harvested stalks in uncontaminated plastic bags. Thresh the heads and collect duplicate grain samples a minimum of 2 lb (but preferably not more than 3 lb). Retain the straw and collect duplicate straw samples weighing a minimum of 1 lb (but preferably not more than 2 lb).

**For hay samples only:** take duplicate samples of the hay (sampled as the whole cut stalk with the grain still attached) having a moisture content of approximately 10% to 20% (approximately 80% to 90% dry matter). (Percent dry matter may be estimated.) Hay samples should weigh a minimum of 1 lb (but preferably not more than 2 lb).

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**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
GC	02	PPP	X (+1)	2 lb.	Grain
GD	02	PPP	X (+1)	2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
SC	02	PPP	X (+1)	1 lb.	Straw
SD	02	PPP	X (+1)	1 lb.	Straw
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
HC	02	PPP	X (+1)	1 lb.	Hay
HD	02	PPP	X (+1)	1 lb.	Hay
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP	X (+1)	2 lb.	Forage
FD	02	PPP	X (+1)	2 lb.	Forage

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
	02	PPP		1 lb.	Straw



	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples GC, GD, SC, SD, HC, HD, FC, and FD.

## **17. RESIDUE SAMPLE COLLECTION:**

### **Hay Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). When the grain is at the milk to soft dough stage, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the hay in a manner simulating commercial practices. Do not separate the grain from the stalks for the hay sample (hay sample should be the entire stalk including grain).

The hay sample must be collected when it has a moisture content of approximately 10-20% (approximately 80% to 90% dry matter). (Percent dry matter may be estimated.) If samples are greater than approximately 20% moisture content, then additional field drying may be necessary. Each sample should weigh a minimum of 1 lb (but preferably not more than 2 lb). If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area.

### **Forage Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). When the oats are at the 6-8 inch stage to stem elongation (jointing) stage, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the forage in a manner simulating commercial practices. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

### **Grain and Straw Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest oats in a manner simulating commercial practices. Separate the oats into grain and straw and collect samples of each. Each grain sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each straw sample should weigh a minimum of 1 lb (but preferably not more than 2 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

### **All Samples:**

At the stages discussed in the paragraphs above: Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the oats using an experimental plot harvester. Collect duplicate samples of each fraction from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from plot ends. Impartially collect two samples of each fraction by taking at least 12 grab samples of grain and straw at uniform intervals over the plot.

Alternatively, the oats can be harvested and collected in the following manner. For each sample, collect the fraction from at least 12 separate areas of the plot (cut at approximately 15 cm above the ground).

**For grain and straw samples only:** Place the harvested stalks in uncontaminated plastic bags. Thresh the heads and collect duplicate grain samples a minimum of 2 lb (but preferably not more than 3 lb). Retain the straw and collect duplicate straw samples weighing a minimum of 1 lb (but preferably not more than 2 lb).

**For hay samples only:** Take duplicate samples of the hay (sampled as the whole cut stalk with the grain still attached) having a moisture content of approximately 10% to 20% (approximately 80% to 90% dry matter). (Percent dry matter may be estimated.) Hay samples should weigh a minimum of 1 lb (but preferably not more than 2 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator/Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**For Samples to be Processed into Flour and Rolled Oats (Trial XX only):**

Collect one sample from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest oats in a manner simulating commercial practices. Separate the oats into grain and straw and retain only the grain. Each grain sample should weigh approximately 30-50 lb.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator/Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:**

**18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
GC	02	PPP	X (+1)	2 lb.	Grain
GD	02	PPP	X (+1)	2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
SC	02	PPP	X (+1)	1 lb.	Straw
SD	02	PPP	X (+1)	1 lb.	Straw
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
HC	02	PPP	X (+1)	1 lb.	Hay
HD	02	PPP	X (+1)	1 lb.	Hay
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP	X (+1)	2 lb.	Forage
FD	02	PPP	X (+1)	2 lb.	Forage

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain

	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples GC, GD, SC, SD, HC, HD, FC, and FD.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	30-50 lb.	Grain
PT	02	PPP	X (+1)	30-50 lb.	Grain

### 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

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See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### **19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for Processing)**

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in raw data.

Prior to shipment, hold samples frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures.

If possible, ship samples within 14 days of harvest. Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

#### **19.3 PROCESSING:**

Store the oat grain at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

Immediately prior to processing oats, remove representative "grab" samples of untreated and treated grain from the larger samples (approximately 2-4 lb for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce flour and rolled oats (minimum 2 lb per sample). Process untreated sample first, followed by treated sample. All trials: Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of each matrix from both the untreated and treated oat samples. Process each sample separately.

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Place samples in appropriate containers and label. **Processed samples may be divided into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen samples at temperatures generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2-4 lb.	Grain
GT	02	PPP	X (+1)	2-4 lb.	Grain
OFA	01	Untreated	NA	2-4 lb.	Flour
OFT	02	PPP	X (+1)	2-4 lb.	Flour
ROA	01	Untreated	NA	2-4 lb.	Rolled Oats
ROT	02	PPP	X (+1)	2-4 lb.	Rolled Oats

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information

- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

#### **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date oat samples received
- 20.1.04- Storage temperatures until unprocessed oat samples are processed into flour and rolled oats
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into flour and rolled oats
- 20.1.07- Storage temperatures of unprocessed grain and processed samples until shipped
- 20.1.08- Date unprocessed grain and processed samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

**17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect quinoa seed as done commercially from at least 12 areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends. (Quinoa seed is mature after most of the leaves have fallen and the dried seed head remains on the stalk.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 2 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

Avoid sampling from the plot ends.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:****18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Seed
B	01	Untreated	NA	2 lb.	Seed
C	02	PPP	X (+1)	2 lb.	Seed
D	02	PPP	X (+1)	2 lb.	Seed

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Seed
B	01	Untreated	NA	2 lb.	Seed
	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed



	02	PPP		2 lb.	Seed
	02	PPP		2 lb.	Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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**17. RESIDUE SAMPLE COLLECTION:****Grain and Straw Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest rice in a manner simulating commercial practices. Separate the rice into grain (kernel and hull) and straw and collect samples of each. Each grain sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each straw sample should weigh a minimum of 1 lb (but preferably not more than 2 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**If using an experimental plot harvester:** Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots and then the treated plot, harvest the rice using an experimental plot harvester. Collect duplicate samples of each fraction from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from plot ends by starting harvest when equipment is within the plot. Impartially collect two samples of each fraction by taking at least 12 grab samples of grain and straw at uniform intervals over the plot.

Only use equipment that harvests panicles and does not dispose of rice straw. After panicles are harvested for grain samples, cut straw samples from the same area where grain has been harvested.

Alternatively, the rice can be harvested and collected in the following manner. For each sample, collect panicles and straw from at least 12 separate areas of the plot (cut at approximately 15 cm above the ground). (Each sample should be collected by making separate runs through the entire plot.) Place the harvested stalks in uncontaminated plastic bags. Thresh the rice and collect duplicate grain samples a minimum of 2 lb (but preferably not more than 3 lb). Retain the straw and collect duplicate straw samples weighing a minimum of 1 lb (but preferably not more than 2 lb). Do not use threshing equipment that could contaminate the grain sample with the straw sample.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**For Samples to be Processed into Polished Rice, Hulls, and Bran (Trial XX only):**

Collect one sample from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest rice in a manner simulating commercial practices. Separate the rice into grain and straw and retain only the grain. Each grain sample should weigh approximately 30-50 lb.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**  
**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
GC	02	PPP	X (+1)	2 lb.	Grain
GD	02	PPP	X (+1)	2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
SC	02	PPP	X (+1)	1 lb.	Straw
SD	02	PPP	X (+1)	1 lb.	Straw

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples GC, GD, SC, and SD.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	30-50 lb.	Grain
PT	02	PPP	X (+1)	30-50 lb.	Grain

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

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See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### **19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in raw data.

Prior to shipment, hold samples frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in a freezer within one hour after collection, samples should be placed in a cooler immediately after collection. Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

#### **19.3 PROCESSING:**

Store the rice grain at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

Immediately prior to processing rice, remove representative "grab" samples of untreated and treated grain from the larger samples (approximately 2-4 lb for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce polished rice, hulls, and bran (minimum 2 lb per sample).

Process untreated sample first, followed by treated sample. Place samples in appropriate containers and label. **Processed samples may be divided into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen samples at temperatures generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GGA	01	Untreated	NA	2-4 lb.	Grain
GGT	02	PPP	X (+1)	2-4 lb.	Grain
PRA	01	Untreated	NA	2-4 lb.	Polished Rice
PRT	02	PPP	X (+1)	2-4 lb.	Polished Rice
HA	01	Untreated	NA	2-4 lb.	Hulls
HT	02	PPP	X (+1)	2-4 lb.	Hulls
BA	01	Untreated	NA	2-4 lb.	Bran
BT	02	PPP	X (+1)	2-4 lb.	Bran

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)

- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date rice samples received
- 20.1.04- Storage temperatures until unprocessed rice samples are processed into polished rice, hulls, and bran
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into polished rice, hulls, and bran
- 20.1.07- Storage temperatures of unprocessed grain and processed samples until shipped
- 20.1.08- Date unprocessed grain and processed samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Sorghum (Grain)

### 17. RESIDUE SAMPLE COLLECTION:

#### **Forage Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At soft dough to hard dough stage, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest sorghum forage in a manner simulating commercial practices from at least 12 plants. Select 12 stalks and separate them into 3 groups of 4 stalks each. Divide each stalk with leaves attached into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 stalks are included in each sample.

#### **Grain Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest grain in a manner simulating commercial practices from at least 12 plants. Collect at least 2 lb (but preferably not more than 4 lb) of grain per sample.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

#### **Stover Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest at least 12 stalks of sorghum in a manner simulating commercial practices and allow the stalks to dry. Remove the grain from the stalks. When the stalks have reached a moisture content of approximately 15-20% (80-85% dry matter), then collect stover samples. (Percent dry matter may be estimated.) To reduce the sample size, separate the 12 stalks into 3 groups of 4 stalks each. Divide each stalk with leaves attached into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 stalks are included in each sample.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
.....					

FA	01	Untreated	NA	12 stalks	Forage
FB	01	Untreated	NA	12 stalks	Forage
FC	02	PPP	NA	12 stalks	Forage
FD	02	PPP	NA	12 stalks	Forage
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
GC	02	PPP	X ( $\pm 1$ )	2 lb.	Grain
GD	02	PPP	X ( $\pm 1$ )	2 lb.	Grain
SA	01	Untreated	NA	12 stalks	Stover
SB	01	Untreated	NA	12 stalks	Stover
SC	02	PPP	X ( $\pm 1$ )	12 stalks	Stover
SD	02	PPP	X ( $\pm 1$ )	12 stalks	Stover

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	12 stalks	Forage
FB	01	Untreated	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
	02	PPP	NA	12 stalks	Forage
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
SA	01	Untreated	NA	12 stalks	Stover
SB	01	Untreated	NA	12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover
	02	PPP		12 stalks	Stover

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC, FD, GC, GD, SC, and SD.



**17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, harvest and sample stalks from at least 12 plants. Each sample should be collected during a separate run through the entire plot. Select 12 stalks and separate them into 3 groups of 4 stalks each. Divide each stalk with leaves attached into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 stalks are included in each sample.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**Processing samples (Field trial XX only):** At X ( $\pm 1$ ) days after the last application, collect one additional untreated sample and one additional treated sample of approximately 90-120 lb each with heads removed. Harvest and sample stalks from at least 12 separate areas of each plot. Begin with the untreated plot first, and then sample the treated plot. Heads should be cut off or stripped off the stalks and then discarded. Divide each stalk into 3 approximately equal lengths and retain all the pieces (the division is needed to make the samples more compact).

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags (as described above) or other containers which will maintain the integrity of the sample. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample container with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**18. FIELD RESIDUE SAMPLE INVENTORY:****18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 stalks	Stalks
B	01	Untreated	NA	12 stalks	Stalks
C	02	PPP	X ( $\pm 1$ )	12 stalks	Stalks
D	02	PPP	X ( $\pm 1$ )	12 stalks	Stalks

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 stalks	Stalks
B	01	Untreated	NA	12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks
	02	PPP		12 stalks	Stalks

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX@@ only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROXIMATE WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	90-120 lb.	Stalks
PT	02	PPP	X ( $\pm 1$ )	90-120 lb.	Stalks

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in a refrigerator at approximately 4°C until they are shipped. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

#### **19.3 PROCESSING:**

Immediately prior to processing sorghum, remove representative “grab” samples of untreated and treated stalks from the larger samples (approximately 2-4 lb. for each sample). Place the “grab” samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Using simulated commercial practices (provide detailed description of equipment and procedures) produce syrup. Collect one sample of syrup from both untreated and treated samples. Syrup samples should have a volume of approximately 1000-2000 ml each.

Place samples in appropriate containers and label. **Divide each sample of syrup into separate containers of 50-150 grams. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen processed samples at temperatures generally less than -18 °C until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. Shipment of frozen samples will be by freezer truck or “express” shipment. Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT./VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2-4 lb.	Stalks
GT	02	PPP	X (+1)	2-4 lb.	Stalks
SA	01	Untreated	NA	1000-2000 ml	Syrup
ST	02	PPP	X (+1)	1000-2000 ml	Syrup

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial.** At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps

.....

- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date sorghum samples received
- 20.1.04- Storage temperatures until samples are processed into syrup
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into syrup
- 20.1.07- Storage temperatures of stalk and syrup samples until shipped
- 20.1.08- Date stalk and syrup samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Wheat

### **17. RESIDUE SAMPLE COLLECTION:**

#### **Forage Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). When the wheat is at the 6-8 inch stage to stem elongation (jointing) stage, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the forage with a scythe, a “weed whacker”, or a similar tool. For each sample, collect the fraction from at least 12 separate areas of the plot. The forage sample must be collected when it has a moisture content of approximately 70-80% (approximately 20-30% dry matter). (Percent dry matter may be estimated.) Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

#### **Hay Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). When the grain is at the milk to soft dough stage, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the hay in a manner simulating commercial practices. Harvest the wheat with a scythe, a “weed whacker”, or a similar tool. For each sample, collect the fraction from at least 12 separate areas of the plot (cut at approximately 15 cm above the ground). Do not separate the grain from the stalks for the hay sample (hay sample should be the entire stalk including grain).

The hay sample must be collected when it has a moisture content of approximately 10-20% (approximately 80% to 90% dry matter). (Percent dry matter may be estimated.) If samples are greater than approximately 20% moisture content, then additional field drying may be necessary. Each sample should weigh a minimum of 1 lb (but preferably not more than 2 lb). If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area.

#### **Grain and Straw Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest wheat using an experimental plot harvester. Collect duplicate samples of each fraction from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from plot ends. (Alternatively, the wheat may be harvested using a scythe, a “weed whacker”, or a similar tool.) Impartially collect two samples of each fraction by taking at least 12 grab samples of grain and straw at uniform intervals over the plot. Each grain sample should weigh a minimum of 2 lb (but preferably not more than 3 lb). Each straw sample should weigh a minimum of 1 lb (but preferably not more than 2 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Place the harvested stalks in uncontaminated plastic bags. Thresh the heads and collect duplicate grain samples a minimum of 2 lb (but preferably not more than 3 lb). Retain the straw and collect duplicate straw samples weighing a minimum of 1 lb (but preferably not more than 2 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

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**For Samples to be Processed into Bran, Flour, Middlings, Shorts, and Germ (Trial XX only):** Collect one sample from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest wheat in a manner simulating commercial practices. Separate the wheat into grain and straw and retain only the grain. Each grain sample should weigh approximately 400-500 lb.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
GC	02	PPP	X (+1)	2 lb.	Grain
GD	02	PPP	X (+1)	2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw
SB	01	Untreated	NA	1 lb.	Straw
SC	02	PPP	X (+1)	1 lb.	Straw
SD	02	PPP	X (+1)	1 lb.	Straw
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
HC	02	PPP	X (+1)	1 lb.	Hay
HD	02	PPP	X (+1)	1 lb.	Hay
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP	X (+1)	2 lb.	Forage
FD	02	PPP	X (+1)	2 lb.	Forage

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Grain
GB	01	Untreated	NA	2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
	02	PPP		2 lb.	Grain
SA	01	Untreated	NA	1 lb.	Straw

SB	01	Untreated	NA	1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
	02	PPP		1 lb.	Straw
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples GC, GD, SC, SD, HC, HD, FC, and FD.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX@@ only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	400-500 lb.	Grain
PT	02	PPP	X (+1)	400-500 lb.	Grain

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in raw data.

Prior to shipment, hold samples frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures. If

the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck. For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in a freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

## **19.3 PROCESSING:**

Store the wheat grain at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

Immediately prior to processing wheat, remove representative "grab" samples of untreated and treated grain from the larger samples (approximately 2-4 lb for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce bran, flour, middlings, shorts, and germ (minimum 2 lb per sample). Process untreated sample first, followed by treated sample. All trials: Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of each matrix from both the untreated and treated wheat samples. Process each sample separately.

Place samples in appropriate containers and label. **Processed samples may be divided into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).



Maintain all frozen samples at temperatures generally less than –18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GGA	01	Untreated	NA	2-4 lb.	Grain
GGT	02	PPP	X (+1)	2-4 lb.	Grain
WBA	01	Untreated	NA	2-4 lb.	Bran
WBT	02	PPP	X (+1)	2-4 lb.	Bran
WFA	01	Untreated	NA	2-4 lb.	Flour
WFT	02	PPP	X (+1)	2-4 lb.	Flour
WMA	01	Untreated	NA	2-4 lb.	Middlings
WMT	02	PPP	X (+1)	2-4 lb.	Middlings
WSA	01	Untreated	NA	2-4 lb.	Shorts
WST	02	PPP	X (+1)	2-4 lb.	Shorts
WGA	01	Untreated	NA	2-4 lb.	Germ
WGT	02	PPP	X (+1)	2-4 lb.	Germ

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information

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- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date wheat samples received
- 20.1.04- Storage temperatures until unprocessed wheat samples are processed into bran, flour, middlings, shorts, and germ
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into bran, flour, middlings, shorts, and germ
- 20.1.07- Storage temperatures of unprocessed grain and processed samples until shipped
- 20.1.08- Date unprocessed grain and processed samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Grass, forage, fodder and hay, group 17

## Grasses

### **10. TEST SYSTEM/CROP:**

GRASSES - NATIONAL STUDY: Use a commercial variety of bluegrass, Bermuda grass, bromegrass, or fescue. Report: variety, source, lot number, date received, and other descriptive information if available.

GRASSES - REGIONAL STUDY IN THE PACIFIC NORTHWEST: Use a commercial variety of bluegrass, ryegrass, bromegrass, or fescue. Report: variety, source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher and the assignment of grass variety to each trial.**

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends).

#### **Forage Samples:**

At X ( $\pm 1$ ) days after the last application, harvest and sample forage from at least 12 separate areas of each plot. (The grass maturity should be from 6-8 inches tall to boot stage, at approximately 15-30% dry matter. The dry matter may be estimated.) Begin with the untreated plot first, and then sample the treated plot. Each sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). Each sample should be collected during a separate run through the entire plot. All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

#### **Hay Samples:**

At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the hay in each plot. (The grass should be in the boot to early head stage.) Allow the hay to dry in the field to a moisture content of approximately 10-20% (80-90% dry matter). (Percent dry matter may be estimated.) If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area. When the hay has dried, collect samples from the untreated plot first, followed by the treated plot. Hay samples should weigh a minimum of 1 lb (but preferably not more than 3 lb). Each sample should be collected during a separate run through the entire plot. Determine (or estimate) and report the moisture content of the hay upon sampling (i.e. placing the samples in the sample bags).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of

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cooling and temperature-monitoring samples in order to maintain integrity.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP		2 lb.	Forage
FD	02	PPP		2 lb.	Forage
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
HC	02	PPP		1 lb.	Hay
HD	02	PPP		1 lb.	Hay

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC, FD, HC, and HD.

**Animal feed, nongrass, group 18**

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## Alfalfa

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends).

#### Forage Samples:

At X ( $\pm 1$ ) days after the last application, harvest and sample forage from at least 12 separate areas of each plot. Begin with the untreated plot first, and then sample the treated plot. Each sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). Each sample should be collected during a separate run through the entire plot. All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). If hay samples will be collected from the same plot as forage samples, mark the areas where forage samples have been collected so that hay samples are not taken from regrowth in these same areas.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

#### Hay Samples:

At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the hay in each plot. Allow the hay to dry in the field to a moisture content of approximately 10-20% (80-90% dry matter). (Percent dry matter may be estimated.) If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area. When the hay has dried, collect samples from the untreated plot first, followed by the treated plot. Hay samples should weigh a minimum of 1 lb (but preferably not more than 3 lb). Determine (or estimate) and report the moisture content of the hay upon sampling (i.e. placing the samples in the sample bags).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except Decline Trial XX@@:

SAMPLE			DAYS AFTER	MINIMUM SAMPLE	CROP
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ID	TRT#	TREATMENT	APPLIC.	SIZE	FRACTION
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP		2 lb.	Forage
FD	02	PPP		2 lb.	Forage
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
HC	02	PPP		1 lb.	Hay
HD	02	PPP		1 lb.	Hay

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC, FD, HC, and HD.



**Alfalfa (grown for seed)**

**17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the seed in each plot. Harvest the untreated plot first, followed by the treated plot. Collect two samples per plot of seed from at least 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot. **For mechanical harvesting of alfalfa seed, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the alfalfa seed using an experimental plot harvester. Collect duplicate samples of seed from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from plot ends. Impartially collect two samples of each fraction by taking at least 12 grab samples of seed at uniform intervals over the plot.**

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Seed samples should each weigh a minimum of ½ lb (but preferably not more than 2 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:**

**18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	½ lb.	Seed
B	01	Untreated	NA	½ lb.	Seed
C	02	PPP	X ( $\pm 1$ )	½ lb.	Seed
D	02	PPP	X ( $\pm 1$ )	½ lb.	Seed

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	½ lb.	Seed
B	01	Untreated	NA	½ lb.	Seed
	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed

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	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed
	02	PPP		½ lb.	Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Clover

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends).

#### **Forage Samples:**

At X ( $\pm 1$ ) days after the last application, harvest and sample forage from at least 12 separate areas of each plot. Clover should be at the 4-8 inch to prebloom stage, at approximately 25-35% dry matter. Begin with the untreated plot first, and then sample the treated plot. Each sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). Each sample should be collected during a separate run through the entire plot. All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). If hay samples will be collected from the same plot as forage samples, mark the areas where forage samples have been collected so that hay samples are not taken from regrowth in these same areas.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

#### **Hay Samples:**

At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the hay in each plot. Clover should be in the early to full bloom stage. Allow the hay to dry in the field to a moisture content of approximately 10-20% (80-90% dry matter). (Percent dry matter may be estimated.) If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area. When the hay has dried, collect samples from the untreated plot first, followed by the treated plot. Hay samples should weigh a minimum of 1 lb (but preferably not more than 3 lb). Determine (or estimate) and report the moisture content of the hay upon sampling (i.e. placing the samples in the sample bags).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### **18. FIELD RESIDUE SAMPLE INVENTORY:**

#### **18.1 All trials except Decline Trial XX@@:**

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
FC	02	PPP		2 lb.	Forage
FD	02	PPP		2 lb.	Forage
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
HC	02	PPP		1 lb.	Hay
HD	02	PPP		1 lb.	Hay

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Forage
FB	01	Untreated	NA	2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
	02	PPP		2 lb.	Forage
HA	01	Untreated	NA	1 lb.	Hay
HB	01	Untreated	NA	1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay
	02	PPP		1 lb.	Hay

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC, FD, HC, and HD.

## Herb group 25

## Basil

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot OR two treated plots.

The individual plots shall be of adequate size to ensure that no more than 50% of the sampled area will be needed to provide the necessary plant material. Requirements for residue sampling are outlined in Parts 17 & 18.

**Please note that dried samples will be required as well and therefore plan plot sizes to yield sufficient sample size for both fresh and dried samples.**

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

For greenhouse trials, use separate greenhouses or compartments/rooms/barriers within a greenhouse for the treated and untreated plots. Alternatively, the treated plants may be sprayed in a different greenhouse or other enclosed area than the one housing the untreated plants and then moved into the greenhouse with the untreated plants after the spray solution has dried.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect plants (above ground portion, stems and leaves) as done commercially. Alternatively, the plants may be clipped to collect just the upper portion of the stems with leaves, as this is also a common commercial practice. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends. If sampling requires more than 50% of the plots to be harvested, contact the study director.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take plants from a minimum of 12 areas of each plot. Each sample should weigh a minimum of 2 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

If loose soil adheres to the stems and leaflets, shake off. DO NOT TRIM OR WASH.

Remove dead and senesced leaflets only, except for the following circumstance: To reduce sample weight, woody portions of the stems may be removed, retaining the leaves for the samples. If the basil plants are very large (greater than 8 oz. each, on average, after removal of woody portions), then further reduce the sample weight by sub-sampling whole branches with foliage from high and low, all quarters of the plants.

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**Dried Samples:** Collect additional basil samples following the above procedures, for processing into dried basil. In all trials, untreated samples for drying may be collected up to 3 days prior to the treated samples for drying. Collect two untreated and two treated samples and deliver to the drying facility within 48 hours of harvest. Collect enough fresh material to provide a minimum 1 lb of dried basil per sample. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb dried, but this is not a requirement. Storage prior to drying should be at ambient temperatures (do not freeze) and the temperatures should be monitored and recorded. Dry according to local commercial practices (document procedures). The recommended drying practice is in a forced-air drier at 90-100 °F for approximately 24 hours, but the drying temperature may be as high as 130 °F if the outside environment is humid. The plants should be spaced in the dryer such that all sides receive sufficient air flow to permit even drying.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Stems & Leaves
FB	01	Untreated	NA	2 lb.	Fresh Stems & Leaves
FC	02	PPP	X (+1)	2 lb.	Fresh Stems & Leaves
FD	02	PPP	X (+1)	2 lb.	Fresh Stems & Leaves
DA	01	Untreated	NA	1 lb.	Dried Stems & Leaves
DB	01	Untreated	NA	1 lb.	Dried Stems & Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Stems & Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Stems & Leaves

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Stems & Leaves
FB	01	Untreated	NA	2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves

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	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
	02	PPP		2 lb.	Fresh Stems & Leaves
DA	01	Untreated	NA	1 lb.	Dried Stems & Leaves
DB	01	Untreated	NA	1 lb.	Dried Stems & Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Stems & Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Stems & Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

20.01- Names of all personnel conducting specific research functions

20.02- Amendments and deviations from protocol and standard operating procedures

20.03- Test site information

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- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- Include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## Chives

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot OR two treated plots.

The individual plots shall be of adequate size to ensure that no more than 50% of the sampled area will be needed to provide the necessary plant material. Requirements for residue sampling are outlined in Parts 17 & 18.

**Please note that dried samples will be required as well and therefore plan plot sizes to yield sufficient sample size for both fresh and dried samples.**

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect plants (above ground portion, leaves) as done commercially. Each sample should be collected during a separate run through the entire plot. Take plants from a minimum of 12 areas of each plot. If sampling requires more than 50% of the plots to be harvested, contact the study director.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take plants from a minimum of 12 areas of each plot. Each sample should weigh a minimum of 2 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

If loose soil adheres to the leaves, shake off. DO NOT TRIM OR WASH. Remove dead and senesced leaves portions only. Avoid sampling from the plot ends. If needed to reduce sample weight, hardy portions of the chives (peduncle) and blooms may be removed, retaining the leaves for the samples.

**Dried Samples:** Collect additional samples following the above procedures, for processing into dried chives. Collect two untreated and two treated samples and deliver to the drying facility within 48 hours of harvest. In all trials, untreated samples for drying may be collected up to 3 days prior to the treated samples for drying. Collect enough fresh material to provide a minimum 1 lb of dried chives per sample. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb dried, but this is not a requirement. Storage prior to drying should be at ambient temperatures (do not freeze) and the temperatures should be monitored and recorded. Dry according to local commercial practices (document procedures). The recommended drying practice is in a forced-air drier at 90-100 °F for approximately 24 hours, but the drying temperature may be as high as 130 °F if the outside

environment is humid. The plants should be spaced in the dryer such that all sides receive sufficient air flow to permit even drying.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves
FB	01	Untreated	NA	2 lb.	Fresh Leaves
FC	02	PPP	X (+1)	2 lb.	Fresh Leaves
FD	02	PPP	X (+1)	2 lb.	Fresh Leaves
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

~~NOTE: Do not~~ Greenhouse trials do not collect dried leaves.

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves
FB	01	Untreated	NA	2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
	02	PPP		2 lb.	Fresh Leaves
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves

DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment

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20.08- Treatment application data

20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)

20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)

20.11- Residue sample shipping information

20.12- Description of crop destruction, or explanation for lack of destruction

20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.

20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots

20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

20.17- Include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## Cilantro or Coriander

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot OR two treated plots.

The individual plots shall be of adequate size to ensure that no more than 50% of the sampled area will be needed to provide the necessary plant material. Requirements for residue sampling are outlined in Parts 17 & 18.

**Trial CAXX:** Please note that dried samples will be required as well and therefore plan plot sizes to yield sufficient sample size for both fresh and dried samples.

**Trial CAYY:** Please note that seed samples will be required as well and therefore plan plot sizes to yield sufficient sample size for both fresh samples and seed samples. Alternatively, separate plots may be established for the collection of fresh leaves and stems, and seeds, respectively.

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect plants (above ground portion, stems and leaves) as done commercially. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take plants from a minimum of 12 areas of each plot. Each sample should weigh a minimum of 2 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

If loose soil adheres to the stems and leaflets, shake off. DO NOT TRIM OR WASH. Remove dead and senesced leaflets only. Avoid sampling from the plot ends. To reduce sample weight, woody portions of the stems may be removed, retaining the leaves for the samples. If sampling requires more than 50% of the plots to be harvested, contact the study director.

**Dried Samples:** Collect additional samples following the above procedures, for processing into dried cilantro. Collect two untreated and two treated samples and deliver to the drying facility within 48 hours of harvest. In all trials, untreated samples for drying may be collected up to 3 days prior to the treated samples for drying. Collect enough fresh material to provide a minimum 1 lb of dried cilantro per sample. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb dried, but this is not a requirement. Storage prior to drying should be at ambient temperatures (do not freeze) and the temperatures should be monitored and recorded. Dry according to local commercial practices (document procedures). The recommended drying practice is in a forced-air drier at 90-100 °F for approximately 24 hours, but the drying temperature may be as high as 130 °F if the

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outside environment is humid. The plants should be spaced in the dryer such that all sides receive sufficient air flow to permit even drying.

**Seed Samples (Field trial CAYY only):** Collect two seed samples from each plot. At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect enough seeds to provide at least 1 lb per sample. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb, but this is not a requirement. If sampling requires more than 50% of the plots to be harvested, contact the study director.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FC	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
FD	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems

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	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

### 18.3 FIELD RESIDUE SAMPLE INVENTORY: Trial CA@@@ only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	1 lb.	Seeds
SB	01	Untreated	NA	1 lb.	Seeds
SC	02	PPP	X (+1)	1 lb.	Seeds
SD	02	PPP	X (+1)	1 lb.	Seeds

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## 20. FIELD DOCUMENTATION AND RECORD KEEPING:

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All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- Include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## Dill

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1 Each test site will consist of two untreated plots and two treated plots.** The additional plots are necessary because of the sample types required in this study. Fresh dill samples and dried dill samples will require immature plants with immature seed heads. Dill seed samples will require mature plants with fully developed dill seed heads.

The individual plots shall be of adequate size to ensure that no more than 50% of the sampled area will be needed to provide the necessary plant material. Requirements for residue sampling are outlined in Parts 17 & 18.

**Please note that dried samples will be required as well and therefore plan plot sizes to yield sufficient sample size for both fresh and dried samples.**

**Trial XX:** Please note that oil samples will be required as well and therefore plan plot sizes to yield sufficient sample sizes for the production of dill oil.

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

**11.4** Mark plots with identifiable markers containing at minimum the Field ID number and treatment number or treatment name that will persist for the duration of the field research trial or that can be readily replaced.

**11.5** This study is not designed for statistical evaluation of field data.

### **17. RESIDUE SAMPLE COLLECTION:**

**Fresh leaves and stems samples; all trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application collect plants (leaves and stems) as done commercially. Start with the untreated plot first. Each sample should be collected during a separate run through the entire plot. Take plants (leaves and stems with immature seeds only) from at least 12 areas of each plot. Each fresh sample should weigh a minimum of 2 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Avoid sampling from the plot ends. If sampling requires more than 50% of the plots to be harvested, contact the study director.

**Dried Samples:** Collect additional samples following the above procedures, for processing into dried dill. Collect two untreated and two treated samples and deliver to the drying facility within 48 hours of harvest. Collect enough

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fresh material to provide a minimum 1 lb per dried sample. In all trials, untreated samples for drying may be collected up to 3 days prior to the treated samples for drying. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb dried, but this is not a requirement. Storage prior to drying should be at ambient temperatures (do not freeze) and the temperatures should be monitored and recorded. The recommended drying practice is in a forced-air drier at 90-100 °F for approximately 24 hours, but the drying temperature may be as high as 130 °F if the outside environment is humid. The plants should be spaced in the dryer such that all sides receive sufficient air flow to permit even drying.

**Fresh leaves and stems samples; decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**Seed samples; all trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect dill seed as done commercially from at least 12 areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 1 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

Avoid sampling from the plot ends.

**Seed samples; decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** If loose soil adheres to the stems and leaflets, shake off. DO NOT TRIM OR WASH. Remove dead and senesced leaflets only. Avoid sampling from the plot ends. Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**Samples being processed into oil (Trial XX only):** Collect one sample from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application collect fresh plants (leaves and stems with immature green seeds) as done commercially. Start with the untreated plot first. Take plants (leaves and stems only) from approximately 12 areas of each plot. The untreated fresh sample should weigh approximately 300-500 lb. and the treated sample should weigh 100-200 lb.

If loose soil adheres to the stems and leaflets, shake off. DO NOT TRIM OR WASH. Remove dead and senesced leaflets only. Avoid sampling from the plot ends. Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

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Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FC	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
FD	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves
SA	01	Untreated	NA	1 lb.	Seed
SB	01	Untreated	NA	1 lb.	Seed
SC	02	PPP	X (+1)	1 lb.	Seed
SD	02	PPP	X (+1)	1 lb.	Seed

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves
SA	01	Untreated	NA	1 lb.	Seed
SB	01	Untreated	NA	1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed

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	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX.WGT. RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	300-500 lb.	Fresh Leaves & Stems
PT	02	PPP	X (+1)	100-200 lb.	Fresh Leaves & Stems

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be taken to a processing facility prior to shipment to an analytical laboratory.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in raw data.

Prior to shipment, hold samples frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

### 19.2 PROCESSING SAMPLE HANDLING:

If samples for processing are not taken to the processing facility within 24 hours of sample collection, they should be stored in a refrigerator at approximately 4°C.

Immediately prior to processing dill, remove representative “grab” samples of untreated and treated dill from the larger samples (approximately 2 lb for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce oil (minimum 1 liter). Process untreated sample first, followed by treated sample.

Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of oil from both the untreated and treated samples, **but each sample should be split into at least two containers. Each portion of the divided sample should be representative of the whole sample.** Process each sample separately. Place oil samples in appropriate containers and label. Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical dill storage conditions to prevent test substance residue degradation. Processing on the day of sample collection is preferred. If processing is not done on the day of sample collection, then temperatures in the storage area should be monitored and recorded.

Maintain all frozen samples at temperatures generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ) until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or “express” shipment. If using “express shipment” (overnight carriers such as Federal Express or Airborne) add quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. For analysis, send samples to: @@@

### **19.3 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT./VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
GT	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
OA	01	Untreated	NA	300-500 ml	Oil
OT	02	PPP	X (+1)	100-200 ml	Oil

### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records

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- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- Include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations relevant to dill processing should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook. At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Storage temperatures until unprocessed dill samples are processed into oil
- 20.1.04- Processing Methodology (SOPs are acceptable)
- 20.1.05- Data collected and observations made during processing of samples into oil
- 20.1.06- Storage temperatures of unprocessed dill and oil samples until shipped
- 20.1.07- Date unprocessed dill and oil samples are shipped to analytical laboratory

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## Dill (Seed)

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect dill seed as done commercially from at least 12 areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 1 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

Avoid sampling from the plot ends.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	1 lb.	Seed
B	01	Untreated	NA	1 lb.	Seed
C	02	PPP	X ( $\pm 1$ )	1 lb.	Seed
D	02	PPP	X ( $\pm 1$ )	1 lb.	Seed

#### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	1 lb.	Seed
B	01	Untreated	NA	1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed

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	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed
	02	PPP		1 lb.	Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Mint

### **10. TEST SYSTEM/CROP:**

MINT - Use a commercial variety of peppermint or spearmint. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

NOTE to be removed by SD: Be aware that double cutting in PNW is an acceptable practice, but in other geographies (i.e. Wisconsin) it can cause the loss of commercial crops in the following year. Be cautious about this practice in some geographies.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot **OR** two treated plots.

The individual plots shall be of adequate size to ensure that no more than 50% of the sampled area will be needed to provide the necessary plant material. Requirements for residue sampling are outlined in Parts 17 & 18.

**All trials: Please note that dried samples will be required as well and therefore plan plot sizes to yield sufficient sample size for both fresh and dried samples.**

**Trial XX:** Please note that oil samples will be required as well; plan plot sizes to yield sufficient sample sizes for the production of mint oil.

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of mint including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**Fresh Foliage Samples: All trials except decline trial:** Collect two samples of fresh mint from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect plants as

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done commercially. Each sample should be collected during a separate run through the entire plot. Take samples of a minimum of 2 lb of mint leaves and stems per sample. Take plants from a minimum of 12 areas of each plot. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If loose soil adheres to the plants, shake off. DO NOT TRIM OR WASH. Remove dead and/or senesced leaves.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**Dried Samples:** Collect additional samples following the above procedures, for processing into dried mint leaves. Collect two untreated and two treated samples and deliver to the drying facility within 48 hours of harvest. Collect enough fresh material to provide a minimum 1 lb per dried sample. In all trials, untreated samples for drying may be collected up to 3 days prior to the treated samples for drying. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb dried, but this is not a requirement. Storage prior to drying should be at ambient temperatures (do not freeze) and the temperatures should be monitored and recorded. The recommended drying practice is in a forced-air dryer at 90-100 °F for approximately 24 hours, but the drying temperature may be as high as 130 °F if the outside environment is humid. The plants should be spaced in the dryer such that all sides receive sufficient air flow to permit even drying.

**All samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Mint Tops to be Processed into Oil (Trial XX@@ only):** At X (+1) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest (cut) the mint and allow the mint to dry in the field for 1-5 days. Let the mint dry to an estimated moisture level of 50-60% to facilitate oil extraction. (It is not necessary to determine precise moisture levels in the mint for this study.) If wet weather is expected, the harvested mint may be moved to a sheltered area for drying. When the mint is deemed sufficiently dry, collect one sample each from the Trt 01 plot and from the Trt 03 plot weighing approximately 30-100 lb untreated hay (sufficient to yield approximately 100-200 ml of oil) and 15-50 lb. treated hay (sufficient to yield approximately 40-60 ml of oil). Take plants from a minimum of 12 areas of each plot. Each sample should be representative of the entire plot (except plot ends). If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place these samples in clean, open mesh bags or other suitable containers for transport to the processing site. Identify each sample container with correct Field ID number, complete sample ID (See Section 18.3) and harvest/sampling dates. Within 24 hours of sample collection, transport the foliage samples to the processing site.

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

### **18.1 All Trials except Decline Trial XX@@:**

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Mint Tops (Leaves & Stems)
FB	01	Untreated	NA	2 lb.	Fresh Mint Tops (Leaves & Stems)
FC	02	PPP	X (+1)	2 lb.	Fresh Mint Tops (Leaves & Stems)
FD	02	PPP	X (+1)	2 lb.	Fresh Mint Tops (Leaves & Stems)
DA	01	Untreated	NA	1 lb.	Dried Mint Tops (Leaves & Stems)
DB	01	Untreated	NA	1 lb.	Dried Mint Tops (Leaves & Stems)
DC	02	PPP	X (+1)	1 lb.	Dried Mint Tops (Leaves & Stems)
DD	02	PPP	X (+1)	1 lb.	Dried Mint Tops (Leaves & Stems)

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Mint Tops (Leaves & Stems)
FB	01	Untreated	NA	2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
	02	PPP		2 lb.	Fresh Mint Tops (Leaves & Stems)
DA	01	Untreated	NA	1 lb.	Dried Mint Tops (Leaves & Stems)
DB	01	Untreated	NA	1 lb.	Dried Mint Tops (Leaves & Stems)
DC	02	PPP	X (+1)	1 lb.	Dried Mint Tops (Leaves & Stems)
DD	02	PPP	X (+1)	1 lb.	Dried Mint Tops (Leaves & Stems)

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX@@ only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	30-100 lb.	Mint Hay
PT	03	PPP	X (+1)	15-50 lb.	Mint Hay

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be taken to a processing facility prior to shipment to an analytical laboratory.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to

permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

If samples for processing are not taken to the processing facility within 24 hours of sample collection, they should be stored in a refrigerator at approximately 4°C.

Immediately prior to processing, remove representative "grab" samples of untreated and treated mint hay from the larger samples (approximately 2 lb for each sample). Place the "grab" samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc.

Using simulated commercial processing method (provide detailed description of equipment and procedures), produce mint oil from the untreated and treated samples. Process untreated mint first, followed by treated mint. The mint oil samples should be approximately 100-200 ml (untreated) and 40-50 ml (treated).

Place samples in appropriate containers, label, and freeze. **Each sample should be split into at least two containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Processing waste and excess oil may be discarded. The spent mint hay samples do not need to be retained after processing. If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical mint storage conditions to prevent test substance residue degradation. Processing on the day of sample collection is preferred. If processing is not done on the day of sample collection, then temperatures in the storage area should be monitored and recorded.

Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or "express" shipment.

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Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples to: @@@

### **19.3 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. SAMPLE SIZE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2 lb	Mint Hay
GT	03	PPP	X (+1)	2 lb	Mint Hay
OA	01	Untreated	NA	100-200 ml	Mint Oil
OT	03	PPP	X (+1)	40 – 60 ml	Mint Oil

### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the

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test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.

- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol
- 20.17-Data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

#### **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations relevant to mint processing should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook. At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Storage temperatures until unprocessed mint samples are processed into oil
- 20.1.04- Processing Methodology (SOPs are acceptable)
- 20.1.05- Data collected and observations made during processing of samples into oil
- 20.1.06- Storage temperatures of unprocessed mint and oil samples until shipped
- 20.1.07- Date unprocessed mint and oil samples are shipped to analytical laboratory

## Parsley

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot OR two treated plots.

The individual plots shall be of adequate size to ensure that no more than 50% of the sampled area will be needed to provide the necessary plant material. Requirements for residue sampling are outlined in Parts 17 & 18.

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect plants (above ground portion, stems and leaves) as done commercially. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take plants from a minimum of 12 areas of each plot. Each sample should weigh a minimum of 2 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

If loose soil adheres to the stems and leaflets, shake off. DO NOT TRIM OR WASH. Remove dead and senesced leaflets only. Avoid sampling from the plot ends. To reduce sample weight, woody portions of the stems may be removed, retaining the leaves for the samples.

**Dried Samples:** Collect additional samples following the above procedures, for processing into dried parsley. Collect two untreated and two treated samples and deliver to the drying facility within 48 hours of harvest. In all trials, untreated samples for drying may be collected up to 3 days prior to the treated samples for drying. Collect enough fresh material to provide a minimum 1 lb per sample of dried parsley. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb dried, but this is not a requirement. Storage prior to drying should be at ambient temperatures (do not freeze) and the temperatures should be monitored and recorded. Dry according to local commercial practices (document procedures). The recommended drying practice is in a forced-air drier at 90-100 °F for approximately 24 hours, but the drying temperature may be as high as 130 °F if the outside environment is humid. The plants should be spaced in the dryer such that all sides receive sufficient air flow to permit even drying.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

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**All trials for samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FC	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
FD	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in raw data.

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Prior to shipment, hold samples frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. Send samples to: @@@**

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction

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20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.

20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots

20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

20.17- Include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## Rosemary

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot OR two treated plots.

The individual plots shall be of adequate size to ensure that no more than 50% of the sampled area will be needed to provide the necessary plant material. Requirements for residue sampling are outlined in Parts 17 & 18.

**Please note that dried samples will be required as well and therefore plan plot sizes to yield sufficient sample size for both fresh and dried samples.**

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect plants (above ground portion, stems and leaves) as done commercially. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take plants from a minimum of 12 areas of each plot. Each sample should weigh a minimum of 2 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

If loose soil adheres to the stems and leaflets, shake off. DO NOT TRIM OR WASH.

Remove dead and senesced leaflets only, except for the following circumstance: To reduce sample weight, woody portions of the stems may be removed, retaining the leaves for the samples. If the rosemary plants are very large (greater than 8 oz. each, on average, after removal of woody portions), then further reduce the sample weight by sub-sampling whole branches with foliage from high and low, all quarters of the plants.

**Dried Samples:** Collect additional rosemary samples following the above procedures, for processing into dried rosemary. Collect two untreated and two treated samples and deliver to the drying facility within 48 hours of harvest. In all trials, untreated samples for drying may be collected up to 3 days prior to the treated samples for drying. Collect enough fresh material to provide a minimum 1 lb per sample of dried rosemary. It is preferable to collect more than the minimum weight of untreated samples, up to 2 lb dried, but this is not a requirement. Storage prior to drying should be at ambient temperatures (do not freeze) and the temperatures should be monitored and recorded. Dry according to local commercial practices (document procedures). The recommended drying practice

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is in a forced-air drier at 90-100 °F for approximately 24 hours, but the drying temperature may be as high as 130° F if the outside environment is humid. The plants should be spaced in the dryer such that all sides receive sufficient air flow to permit even drying.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**For Rosemary to be Processed into Oil (Trial XX only):** Follow above procedure and collect one sample from each plot weighing approximately 150-200 lb untreated (sufficient to yield approximately 100-120 ml of oil) and 80-120 lb treated (sufficient to yield approximately 40-50 ml of oil). Each sample should be representative of the entire plot (except plot ends). If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place these samples in clean, open mesh bags or other suitable containers for transport to the processing site. Identify each sample container with correct Field ID number, complete sample ID (See Section 18.3) and harvest/sampling dates. Transport the foliage sample to the processing site for processing into mint oil within 24 hours.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FC	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
FD	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
FB	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems

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	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
	02	PPP		2 lb.	Fresh Leaves & Stems
DA	01	Untreated	NA	1 lb.	Dried Leaves
DB	01	Untreated	NA	1 lb.	Dried Leaves
DC	02	PPP	X (+1)	1 lb.	Dried Leaves
DD	02	PPP	X (+1)	1 lb.	Dried Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT. RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	150-200 lb.	Fresh Leaves & Stems
PT	02	PPP	60 (+3)	80-120 lb.	Fresh Leaves & Stems

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be taken to a processing facility prior to shipment to an analytical laboratory.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

If samples for processing are not taken to the processing facility within 24 hours of sample collection, they should be stored in a refrigerator at approximately 4°C. Using simulated commercial processing method (provide detailed description of equipment and procedures), produce rosemary oil from the untreated and treated samples. Process untreated mint first, followed by treated mint. The rosemary oil samples should be approximately 100-200 ml (untreated) and 40-50 ml (treated).

Immediately prior to processing, remove representative “grab” samples of untreated and treated rosemary from the larger samples (approximately 2 lb for each sample). Place the “grab” samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc.

Place samples in appropriate containers, label, and freeze. **Each sample should be split into at least two containers.** Processing waste and excess oil may be discarded. The spent rosemary hay samples (Processed mint foliage samples E and F) do not need to be retained after processing. If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical rosemary storage conditions to prevent test substance residue degradation. Processing on the day of sample collection is preferred. If processing is not done on the day of sample collection, then temperatures in the storage area should be monitored and recorded.

Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Shipment of frozen samples will be by freezer truck or “express” shipment. Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples. Send samples to: @@@**

## **19.3 PROCESSED RESIDUE SAMPLE INVENTORY:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. SAMPLE VOLUME	CROP FRACTION
GA	01	Untreated	NA	2 lb.	Fresh Leaves & Stems
GT	02	PPP	X (+1)	2 lb.	Fresh Leaves & Stems
OA	01	Untreated	NA	100-120 ml	Rosemary Oil
OT	02	PPP	X (+1)	40 – 50 ml	Rosemary Oil

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

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All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- Include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## Stevia

### **10. TEST SYSTEM/CROP:**

STEVIA - Use a commercial variety of stevia. Report: variety, planting date, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except Decline Trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Each sample should be collected during a separate run through the entire plot. If this cannot be done due to harvesting equipment or other factors, contact the Study Director to discuss.

**Fresh Samples:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect plants (above ground portion, stems and leaves) as done commercially. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Take plants from a minimum of 12 areas of each plot. Each sample should weigh a minimum of 1 lb. It is preferable to collect more than the minimum weight of untreated samples, up to 5 lb, but this is not a requirement.

If loose soil adheres to the stems and leaflets, shake off. DO NOT TRIM OR WASH.

Remove and discard dead and senesced leaflets. Remove the woody portions of the stems, retaining the leaves for the samples. If the stevia plants are very large (greater than 4 oz. each, on average, after removal of woody portions), then further reduce the sample weight by sub-sampling leaves from whole branches with foliage from high and low, all quarters of the plants.

**Dried Samples:** At X ( $\pm 1$ ) days after the last application, starting with the control plot, collect whole plants and then dry to approximately 8-12% moisture and then separate leaves from stems. Moisture content may be estimated. Document procedures for drying and estimating moisture in the Field Data Book. Discard the stems and collect dried leaves as the sample.

Alternatively, at X ( $\pm 1$ ) days after the last application, starting with the control plot, collect leaves (cut or pull from the stems) and then dry to approximately 8-12% moisture. Moisture content may be estimated. Document procedures for drying and estimating moisture in the Field Data Book.

**Decline trial:** Insert instructions (fresh samples only for decline)

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for one plot before proceeding to the next.

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (common chemical name and formulation), complete sample ID (see Section 18) and harvest/sampling dates.

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See Section 19 for residue sample handling directions.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID #; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; TRT #; Harvest Date; Sample Date; Field Research Director:** enter name and telephone number.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All trials except decline trial:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM WGT. OF SAMPLE	CROP FRACTION
FA	01	Untreated	NA	1 lb	Fresh leaf
FB	01	Untreated	NA	1 lb	Fresh leaf
FC	02	PPP	X (+1)	1 lb	Fresh leaf
FD	02	PPP	X (+1)	1 lb	Fresh leaf
DA	01	Untreated	NA	0.5 lb	Dried leaf
DB	01	Untreated	NA	0.5 lb	Dried leaf
DC	02	PPP	X (+1)	0.5 lb	Dried leaf
DD	02	PPP	X (+1)	0.5 lb	Dried leaf

### 18.2 Decline trial:

SAMPLE ID*	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM WGT. OF SAMPLE	CROP FRACTION
FA	01	Untreated	NA	1 lb	Fresh leaf
FB	01	Untreated	NA	1 lb	Fresh leaf
FE	02	PPP	X (+1)	1 lb	Fresh leaf
FF	02	PPP	X (+1)	1 lb	Fresh leaf
FG	02	PPP	X (+1)	1 lb	Fresh leaf
FH	02	PPP	X (+1)	1 lb	Fresh leaf
FC	02	PPP	X (+1)	1 lb	Fresh leaf
FD	02	PPP	X (+1)	1 lb	Fresh leaf
FI	02	PPP	X (+1)	1 lb	Fresh leaf
FJ	02	PPP	X (+1)	1 lb	Fresh leaf
FK	02	PPP	X (+1)	1 lb	Fresh leaf
FL	02	PPP	X (+1)	1 lb	Fresh leaf
DA	01	Untreated	NA	0.5 lb	Dried leaf
DB	01	Untreated	NA	0.5 lb	Dried leaf
DC	02	PPP	X (+1)	0.5 lb	Dried leaf
DD	02	PPP	X (+1)	0.5 lb	Dried leaf

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

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## Oil seed group 20

## Camelina

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot edges).

**For Fresh Seed Sample:** At X ( $\pm$  1) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect the camelina seed. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples may be harvested (cut) and threshed using a small plot combine. Collect at least 2 lb of the seed per sample.

**Mechanical swather:** If necessary, a mechanical swather may be used to harvest (cut) the camelina. Allow the camelina to air-dry if needed, on the stubble surface for no longer than 5 days. The seed may then be threshed using a small plot combine or threshed by hand. If hand threshed, for each sample, collect the fraction from at least 12 separate areas of the plot.

**Hand-harvesting:** Alternatively, the camelina may be cut using a sickle mower. Swath the camelina (cut at approximately 6 inches above soil level) and allow to air-dry if needed, on the stubble surface for no longer than 5 days. Pick up the swath and mechanically thresh the seed using a small plot combine or thresh by hand. If hand threshed, for each sample, collect the fraction in a separate run from at least 12 separate areas of the plot.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

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**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**  
**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Camelina Seed to be Processed into Refined Oil (Field trial XX only):** Following one of the above procedures, collect one untreated and one treated 50-60 lb (approximate) whole seed samples for processing into refined oil. During harvest and sampling, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place samples in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
C	02	PPP	X (+1)	2 lb.	Whole Seed
D	02	PPP	X (+1)	2 lb.	Whole Seed

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	50-60 lb.	Whole Seed
PT	02	PPP	X (+1)	50-60 lb.	Whole Seed

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

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## **19.1 FRESH SEED RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in a freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples. Send samples to: @@@**

## **19.3 PROCESSING:**

Store the seed frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

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Immediately prior to processing seed, remove representative “grab” samples of untreated and treated seed from the larger samples (approximately 2-4 lb. for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce refined oil. Process untreated seed first, followed by treated seed. Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of refined oil from both the untreated and treated seed samples. Process each sample separately.

Place refined oil samples in appropriate containers and label. **Processed samples may be divided into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Maintain all frozen samples at temperatures generally less than –18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or “express” shipment. If using “express shipment” (overnight carriers such as Federal Express or Airborne) add quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT./VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2–4 lb.	Whole Seed
GT	02	PPP	X ( <u>±</u> 1)	2–4 lb.	Whole Seed
OA	01	Untreated	NA	1000-2000 ml	Refined Oil
OT	02	PPP	X ( <u>±</u> 1)	1000-2000 ml	Refined Oil

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data

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- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date seed samples received
- 20.1.04- Storage temperatures until seed samples are processed into refined oil
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into refined oil
- 20.1.07- Storage temperatures of seed and refined oil samples until shipped
- 20.1.08- Date seed and refined oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.



## Canola

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect mature canola seed. Each sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples may be collected by using an experimental plot harvester or by hand-harvesting.

**If a harvester is used:** Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the canola seed from each plot. Harvest the seed from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from the plot ends. Impartially collect samples of whole seed that weigh a minimum of 2 lb but preferably not more than 4 lb.

**Hand-harvesting:** Alternatively, canola seed may be harvested and sampled canola seed by hand. Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest bundles of plants from at least 12 separate areas of the plot to ensure representative, impartial sample that represents the entire plot (except plot ends), in a separate run for each sample. Carry plants to threshing location, using uncontaminated containers if needed. Thresh the canola plants and collect whole seed samples that weigh a minimum of 2 lb but preferably not more than 4 lb. Thresh the untreated plants first.

Whatever the method of harvesting, All trials: Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

If the canola seed need additional drying prior to sampling, the plants may be cut when the seed is commercially mature and dried in the field or greenhouse/similar enclosed facility prior to sampling the seed as per local commercial practices.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical

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name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Canola Seed to be Processed into Refined Oil and Meal (Field trial XX only):** Following one of the above procedures, collect one untreated and one treated 50-60 lb (approximate) whole seed samples for processing into refined oil and meal. During harvest and sampling, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place samples in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

See below for inventories of field residue samples (raw agricultural commodities) and processing residue samples (commodities to be sent to a processing facility prior to residue analysis).

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
C	02	PPP	X (+1)	2 lb.	Whole Seed
D	02	PPP	X (+1)	2 lb.	Whole Seed

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	50-60 lb.	Whole Seed
PT	02	PPP	X (+1)	50-60 lb.	Whole Seed

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### **19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in raw data.

Prior to shipment, hold samples frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in a freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for

more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

### 19.3 PROCESSING:

Store the canola seed frozen at temperatures generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

Immediately prior to processing canola seed, remove representative "grab" samples of untreated and treated canola seed from the larger samples (approximately 2-4 lb. for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce canola meal and refined oil. Process untreated seed first, followed by treated seed. All trials: Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of canola meal and one sample of refined oil from both the untreated and treated canola seed samples. Process each sample separately.

Place meal and refined oil samples in appropriate containers and label. **Divide each sample of oil into separate containers of 150-300 ml. It is also acceptable to divide meal samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen samples at temperatures generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

### 19.4 PROCESSED RESIDUE SAMPLE INVENTORY:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT./VOL. RANGE OF SAMPLE	CROP FRACTION
GA	01	Untreated	NA	2-4 lb.	Whole Seed
GT	02	PPP	X (+1)	2-4 lb.	Whole Seed
MA	01	Untreated	NA	5-7 lb.	Meal
MT	02	PPP	X (+1)	5-7 lb.	Meal
OA	01	Untreated	NA	1000-2000 ml	Refined Oil
OT	02	PPP	X (+1)	1000-2000 ml	Refined Oil

### 20. FIELD DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial.** At a minimum, collect and maintain the following raw data:

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- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date canola seed samples received
- 20.1.04- Storage temperatures until canola seed samples are processed into meal and refined oil
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into refined oil
- 20.1.07- Storage temperatures of canola seed, meal, and refined oil samples until shipped

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20.1.08- Date canola seed, meal, and refined oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Flax

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot edges).

**For Fresh Seed Sample:** At X ( $\pm$  1) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect the flax seed. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples may be harvested (cut) and threshed using a small plot combine. Collect at least 2 lb of the seed per sample.

**Mechanical swather:** If necessary, a mechanical swather may be used to harvest (cut) the flax. Allow the flax to air-dry if needed, on the stubble surface for no longer than 5 days. The seed may then be threshed using a small plot combine or threshed by hand. If hand threshed, for each sample, collect the fraction from at least 12 separate areas of the plot.

**Hand-harvesting:** Alternatively, the flax may be cut using a sickle mower. Swath the flax (cut at approximately 6 inches above soil level) and allow to air-dry if needed, on the stubble surface for no longer than 5 days. Pick up the swath and mechanically thresh the seed using a small plot combine or thresh by hand. If hand threshed, for each sample, collect the fraction in a separate run from at least 12 separate areas of the plot.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

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**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Flax Seed to be Processed into Refined Oil and Meal (Field trial XX only):** Following one of the above procedures, collect one untreated and one treated 400-500 lb (approximate) whole seed samples for processing into refined oil and meal. During harvest and sampling, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place samples in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
C	02	PPP	X (+1)	2 lb.	Whole Seed
D	02	PPP	X (+1)	2 lb.	Whole Seed

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	400-500 lb.	Whole Seed
PT	02	PPP	X (+1)	400-500 lb.	Whole Seed

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

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## **19.1 FRESH SEED RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in a freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples. Send samples to: @@@**

## **19.3 PROCESSING:**

Store the seed frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

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Immediately prior to processing seed, remove representative “grab” samples of untreated and treated seed from the larger samples (approximately 2-4 lb. for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce meal and refined oil. Process untreated seed first, followed by treated seed. Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of meal and one sample of refined oil from both the untreated and treated seed samples. Process each sample separately.

Place meal and refined oil samples in appropriate containers and label. **Divide each sample of oil into separate containers of 150-300 ml. It is also acceptable to divide meal samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Maintain all frozen samples at temperatures generally less than –18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or “express” shipment. If using “express shipment” (overnight carriers such as Federal Express or Airborne) add of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### 19.4 PROCESSED RESIDUE SAMPLE INVENTORY:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT./VOL. RANGE OF SAMPLE	CROP FRACTION
GA	01	Untreated	NA	2-4 lb.	Whole Seed
GT	02	PPP	X (+1)	2-4 lb.	Whole Seed
MA	01	Untreated	NA	2-4 lb.	Meal
MT	02	PPP	X (+1)	2-4 lb.	Meal
OA	01	Untreated	NA	1000-2000 ml	Refined Oil
OT	02	PPP	X (+1)	1000-2000 ml	Refined Oil

#### 20. FIELD DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial.** At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records

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- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date flax seed samples received
- 20.1.04- Storage temperatures until flax seed samples are processed into meal and refined oil
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into meal and refined oil
- 20.1.07- Storage temperatures of flax seed, meal, and refined oil samples until shipped
- 20.1.08- Date flax seed, meal, and refined oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Hemp

### 10. TEST SYSTEM/CROP:

Hemp - Use a commercial variety of industrial hemp (a variety of *Cannabis sativa* with a THC content not greater than 0.3%). Report: variety (**indicate whether it is a CBG/CBD-oil, or a fiber/seed variety**), source, lot number, date received, and other descriptive information if available.

Fiber/seed trials:					
CBD-oil trials:					

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments, and for assignment of CBG/CBD-oil or fiber/seed variety. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.**

### 16. SUPPLEMENTAL CROP TREATMENTS:

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### 17. RESIDUE SAMPLE COLLECTION:

**IMPORTANT:** Hemp plants that will be sampled must undergo THC testing of the foliage and involucreal leaves of the hemp inflorescences prior to harvest, not more than 30 days before sample collection (in accordance with state/provincial and USDA regulations) to confirm that THC content is not greater than 0.3%. All samples must be collected from the flowering tops of the plant by cutting the top 5-8 inches from the main stem (that includes the leaves and flowers), terminal bud (that occurs at the end of a stem), or central kola (cut stem that could develop into a bud) of the flowering top of the plant. Samples may not be sent to the analytical laboratory until after THC content has been confirmed. If the result is that THC is greater than 0.3%, contact the Study Director as soon as possible. **INCLUDE A COPY OF THE THC TEST RESULTS WITH THE SAMPLE SHIPMENT.**

**All Fiber/seed trials:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot edges).

**For Seed Sample:** At X ( $\pm 1$ ) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect the hemp seed from at least 12 areas of the plot. The hemp plants should still be green, and the seeds beginning to shatter.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples may be harvested (cut) and threshed using a small plot combine. The seed may be collected for samples as the combine proceeds through the crop. Collect at least 1 lb of the seed per sample. Alternatively, plants may

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be cut by hand and then run through a stationary, mechanical thresher to obtain the seed.

After harvest, seed should be dried to 7-10% moisture before collection of samples. This may be done by placing the seed in a protected area for drying, or by the use of a dryer. The dryer temperature should not exceed 95° F (35° C); the drying time may vary depending on the plants' initial moisture content and the outside humidity. Document the drying procedure and the estimation of moisture content in the Field Data Book. Each sample should weigh a minimum of 1 lb (but preferably not more than 3 lb).

**For Fiber Sample:** At X ( $\pm 1$ ) days after the last application, harvest hemp plants from at least 12 separate areas of each plot. Begin with the untreated plot first, and then harvest the treated plot. The harvested plant should be left to dry in the plot to a moisture content of less than 15%. Document the estimation of moisture content in the Field Data Book. If necessary, the harvested plants may be moved to a protected area for drying. Once the plants have sufficiently dried, then fiber samples may be collected. Each sample should be collected during a separate run through the entire plot. The fiber sample may contain stems, leaves, flowers, and/or seeds. Each sample should weigh a minimum of 2 lb (but preferably not more than 4 lb). If sample reduction is necessary to reduce the sample weight, select at least 12 plants and separate them into 3 groups of at least 4 plants each. Divide each plant into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all of the plants are included in each sample. If sample reduction is not needed, the plants may be cut to fit into sample bags and include all of the cut plant parts in the sample.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**All CBG/CBD trials:** At X ( $\pm 1$ ) days after the last application, harvest hemp plants from at least 12 separate areas of each plot. Collect two samples from each plot. Begin with the untreated plot first, and then harvest the treated plot. Each sample should be representative of the entire plot (except plot edges). Remove the floral biomass (kolas or flower buds) from the harvested plants and transport to a dryer (or to a drying room). (Alternatively, floral biomass may be harvested directly from the living plants.) Collect enough floral biomass to produce 1 lb. per sample after drying. (Approximately 3-4 lb. of fresh flower buds is recommended to produce 1 lb. after drying.) The floral biomass that is collected may include some foliage that is among the flower buds. The floral biomass should be dried to approximately 8-12% moisture. The dryer temperature should not exceed 95° F (35° C); the drying time may vary depending on the plants' initial moisture content and the outside humidity. Air-drying is also acceptable in low-humidity environments. Document the drying procedure and the estimation of moisture content in the Field Data Book. After the flower buds have sufficiently dried, collect two samples from each plot. Each sample should weigh a minimum of 1 lb.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

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**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Hemp Seed to be Processed into Oil, Seed Meal (Presscake), Flour, and Hemp Hearts (Field trial XX only):** Following one of the above procedures, collect one untreated and one treated 10-20 lb (approximate) whole seed samples for processing into refined oil and meal. During harvest and sampling, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

**For Flower Buds to be Processed into CBD Oil (Field Trial XX only):** At X ( $\pm 1$ ) days after the last application, harvest hemp plants from at least 12 separate areas of each plot. Begin with the untreated plot first, and then harvest the treated plot. Each sample should be representative of the entire plot (except plot edges). Remove the floral biomass (colas or flower buds) from the harvested plants and transport to a dryer. (Alternatively, floral biomass may be harvested directly from the living plants.) Collect enough floral biomass to produce 4 lb. after drying. (Approximately 10-15 lb. of fresh flower buds is recommended to produce 4 lb. after drying.) The floral biomass should be dried to approximately 8-12% moisture. Document the drying procedure and the estimation of moisture content in the Field Data Book. After the flower buds have sufficiently dried, collect one sample from each plot. Each sample should weigh a minimum of 4 lb.

Place samples in a container or containers suitable for transport to the processing laboratory. Identify each sample with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Fiber/seed Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	1 lb.	Whole Seed
SB	01	Untreated	NA	1 lb.	Whole Seed
SC	02	PPP	X ( $\pm 1$ )	1 lb.	Whole Seed
SD	02	PPP	X ( $\pm 1$ )	1 lb.	Whole Seed
FA	01	Untreated	NA	2-4 lb.	Fiber
FB	01	Untreated	NA	2-4 lb.	Fiber
FC	02	PPP	X ( $\pm 1$ )	2-4 lb.	Fiber
FD	02	PPP	X ( $\pm 1$ )	2-4 lb.	Fiber

### 18.2 All CBG/CBD Trials:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FBA	01	Untreated	NA	4 lb.	Flower Buds
FBB	01	Untreated	NA	4 lb.	Flower Buds
FBT	02	PPP	X ( $\pm 1$ )	4 lb.	Flower Buds
FBD	02	PPP	X ( $\pm 1$ )	4 lb.	Flower Buds

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.4 SEED PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	10-20 lb.	Whole Seed
PT	02	PPP	X (+1)	10-20 lb.	Whole Seed

#### 18.4 CBD PROCESSING RESIDUE SAMPLE INVENTORY: (Field trial XX only)

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FBU1	01	Untreated	NA	4 lb.	Flower Buds
FBT1	02	PPP	X (+1)	4 lb.	Flower Buds

### 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

#### 19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in



a freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. CBD flower buds may be hand delivered chilled or at ambient temperature. Shipment of frozen samples will be by freezer truck or "express" shipment, or hand delivery by field personnel. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.**

Send seed processing samples to: @@@

**Send CBD flower buds processing samples to:** Bronson Hung, IR-4 Western Region Laboratory, Center for Health and the Environment (CHE), Univ of California, Davis, Building 3792, 1250 Old Davis Road, Room 129, Davis, CA 95616-8615, Cell#: (530) 752-4742, e-mail: bkhung@ucdavis.edu

### **19.3 PROCESSING:**

Store the seed frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

**Seed Processing:** Immediately prior to processing seed, remove representative "grab" samples of untreated and treated seed from the larger samples (approximately 1-3 lb. for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce meal (presscake), oil, flour, and hemp hearts (dehulled hemp seed). Process untreated seed first, followed by treated seed. Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of each processed fraction from both the untreated and treated seed samples. Process each sample separately.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

### **Flower Bud Processing:**

Prior to processing flower bud samples, remove representative "grab" samples of untreated and treated buds from the larger samples (approximately 1 lb. for each sample). Remove grab samples prior to or after homogenizing the flower bud samples with dry ice. Using simulated commercial processing (provide detailed description of equipment and procedures) produce CBD oil. Process untreated buds first, followed by treated buds. Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of each processed fraction from both the untreated and treated seed samples. Process each sample separately. **One untreated sample and one treated sample should be processed using ethanol, and one untreated sample and one treated sample should be processed using super-critical CO<sub>2</sub>.** CBD oil will be processed in the same facility as the analytical laboratory. Processed samples will not be shipped or transferred prior to sample analysis.

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**All Processed Samples:** Place processed samples in appropriate containers and label. **Divide each sample of hemp seed oil into separate containers of 150-300 ml. It is also acceptable to divide other processed samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Maintain all frozen samples at temperatures generally less than –18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY (Seed Processing):**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT./VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GSA	01	Untreated	NA	1-3 lb.	Whole Seed
GST	02	PPP	X (+1)	1-3 lb.	Whole Seed
PFA	01	Untreated	NA	1-3 lb.	Flour
PFT	02	PPP	X (+1)	1-3 lb.	Flour
HHa	01	Untreated	NA	1-3 lb.	Hemp Hearts
HHT	02	PPP	X (+1)	1-3 lb.	Hemp Hearts
HMA	01	Untreated	NA	1-3 lb.	Meal
HMT	02	PPP	X (+1)	1-3 lb.	Meal
OA	01	Untreated	NA	500-1000 ml	Oil
OT	02	PPP	X (+1)	500-1000 ml	Oil

#### **19.5 PROCESSED RESIDUE SAMPLE INVENTORY (CBD Processing):**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GFA	01	Untreated	NA	1 lb.	Flower buds
GFB	02	PPP	X (+1)	1 lb.	Flower buds
ECBDA	01	Untreated	NA	1.0 – 50 g.	CBD Oil (ethanol)
ECBDT	02	PPP	X (+1)	1.0 – 50 g.	CBD Oil (ethanol)
SCBDA	01	Untreated	NA	1.0 – 50 g.	CBD Oil (CO <sub>2</sub> )
SCBDT	02	PPP	X (+1)	1.0 – 50 g.	CBD Oil (CO <sub>2</sub> )

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment

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20.08- Treatment application data

20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)

20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)

20.11- Residue sample shipping information

20.12- Description of crop destruction, or explanation for lack of destruction

20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.

20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots

20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

20.1.01- Names of all personnel conducting specific research functions

20.1.02- Deviations from protocol and standard operating procedures

20.1.03- Date flower bud samples received

20.1.04- Storage temperatures until flower bud samples are processed into commodities listed in Part 19

20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing into commodities listed in Part 19

20.1.07- Storage temperatures of flower bud and processed samples until shipped

20.1.08- Date flower bud and processed samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility. starting with the untreated plot or utilizing separate personnel for the treated and untreated plotsRegional Field Coordinator

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## Safflower

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the safflower seed from each plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples may be collected by using an experimental plot harvester or by hand-harvesting.

**If a harvester is used:** Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the safflower seed from each plot. Harvest the seed from a sufficiently wide and long swath that is representative of the entire plot. Avoid sampling from the plot ends. Impartially collect samples of whole seed that weigh a minimum of 2 lb but preferably not more than 4 lb.

**Hand-harvesting:** Alternatively, seed may be harvested and sampled by hand. Collect heads in a separate run for each sample from at least 12 plants. Thresh the heads and collect the whole seed samples (minimum 2 lb, but preferably not more than 4 lb). Thresh the untreated head from untreated plants first.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Whatever the method of harvesting used, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Safflower Seed to be Processed into Oil and Meal (Field trial XX only):** Following one of the above

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procedures, collect one untreated and one treated 60-70 lb (approximate) whole seed samples for processing into refined oil and meal. During harvest and sampling, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place samples in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

### **18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
C	02	PPP	X (+1)	2 lb.	Whole Seed
D	02	PPP	X (+1)	2 lb.	Whole Seed

### **18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### **18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	60-70 lb.	Whole Seed
PT	02	PPP	X (+1)	60-70 lb.	Whole Seed

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### **19.1 SEED RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

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For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLES:**

After residue sample collection, the samples should be placed into a freezer. If the samples cannot be placed into a freezer within one hour, an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity will be used. The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented.

Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

## **19.3 PROCESSING:**

As soon as possible after receiving the safflower seed for processing, process them as done commercially, into safflower oil and meal. Remove a "grab" sample (approximately 2-4 lb per sample) from the treated and untreated seed prior to processing. Start with the untreated samples first. Process each sample separately. Process the seeds into oil and meal.

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Place samples in appropriate containers and label. **Divide each sample of oil into separate containers of 150-300 ml. It is also acceptable to divide meal samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s). Immediately freeze the processed samples and hold at least 0 °F (-18 °C) until shipped to the analytical lab.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT. /VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2-4 lb.	Whole Seed
GT	02	PPP	X (+1)	2-4 lb.	Whole Seed
MA	01	Untreated	NA	2-4 lb.	Meal
MT	02	PPP	X (+1)	2-4 lb.	Meal
OA	01	Untreated	NA	1000-2000 ml	Refined Oil
OT	02	PPP	X (+1)	1000-2000 ml	Refined Oil

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)

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- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

#### **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date safflower seed samples received
- 20.1.04- Storage temperatures until safflower seed samples are processed into meal and oil
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into
- 20.1.07- Storage temperatures of safflower seed, meal, and oil samples until shipped
- 20.1.08- Date safflower seed, meal, and oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.



## Sesame

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot edges).

**For Fresh Seed Sample:** At X ( $\pm$  1) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect the sesame seed. Each sample should weigh a minimum of 2 lb (but preferably not more than 3 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples may be harvested (cut) and threshed using a small plot combine. Collect at least 2 lb of the seed per sample.

If necessary, a mechanical swather may be used to harvest (cut) the sesame. Allow the sesame to air-dry if needed, on the stubble surface for no longer than 5 days. The seed may then be threshed using a small plot combine or threshed by hand. If hand threshed, for each sample, collect the fraction in a separate run from at least 12 separate areas of the plot.

Alternatively, if hand harvested (cut), swath the sesame (cut at approximately 6 inches above soil level) and allow to air-dry if needed, on the stubble surface for no longer than 5 days. Pick up the swath and mechanically thresh the seed using a small plot combine or thresh by hand. If hand threshed, for each sample, collect the fraction from at least 12 separate areas of the plot.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

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**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Sesame Seed to be Processed into Oil (Field trial XX only):** Following one of the above procedures, collect one untreated and one treated 50-60 lb (approximate) whole seed samples for processing into refined oil. During harvest and sampling, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place samples in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
C	02	PPP	X (+1)	2 lb.	Whole Seed
D	02	PPP	X (+1)	2 lb.	Whole Seed

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	50-60 lb.	Whole Seed
PT	02	PPP	X (+1)	50-60 lb.	Whole Seed

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an

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analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### **19.1 FRESH SEED RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and collection of samples for processing, utilize procedures that minimize sample degradation. Place samples in a freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in a freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0 °F) until shipped. Monitor and document all on-site storage temperatures. If possible, ship samples within 14 days of harvest.

Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples. Send samples for processing to: @@@**

#### **19.3 PROCESSING:**

Store the seed frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than

24 hours' duration due to freezer cycling, sample movement, etc., until processing.

Immediately prior to processing seed, remove representative "grab" samples of untreated and treated seed from the larger samples (approximately 2-4 lb. for each sample). Using simulated commercial processing (provide detailed description of equipment and procedures) produce sesame oil. Process untreated seed first, followed by treated seed. Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Collect one sample of oil from both the untreated and treated seed samples. Process each sample separately.

Place oil samples in appropriate containers and label. **Divide each sample of oil into separate containers of 150-300 ml. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen samples at temperatures generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification by e-mail, telephone log, communication note, etc. Shipment of frozen samples will be by freezer truck or "express" shipment. If using "express shipment" (overnight carriers such as Federal Express or Airborne) add of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT./VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2 – 4 lb.	Whole Seed
GT	02	PPP	X (+1)	2 – 4 lb.	Whole Seed
OA	01	Untreated	NA	1000-2000 ml	Oil
OT	02	PPP	X (+1)	1000-2000 ml	Oil

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)

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- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date sesame seed samples received
- 20.1.04- Storage temperatures until sesame seed samples are processed into oil
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into oil
- 20.1.07- Storage temperatures of sesame seed and oil samples until shipped
- 20.1.08- Date sesame seed and oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.



## Sunflower

### **10. TEST SYSTEM/CROP:**

CCC - Use a commercial variety that is suitable for seed production. A variety suitable for oil production should be used in a trial producing samples for processing. **Indicate in 8B of the Field Data Book whether the samples are from an oil variety or a confectionary variety.** Do not use a variety that is primarily ornamental. Report: variety, source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; **see Section 23 for these assignments.** Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director.**

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the sunflower seed from each plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Samples may be collected by using an experimental plot harvester or by hand-harvesting.

**If a harvester is used:** Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest the seed from each plot. Avoid sampling from the plot ends. Impartially collect samples of whole seed weighing a minimum of 2 lb but preferably not more than 4 lb.

**Hand-harvesting:** Alternatively, seed may be harvested and sampled by hand. Starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest at least 12 heads from at least 12 separate areas of the plot in a separate run for each sample to ensure representative, impartial sample that represents the entire plot (except plot ends). Carry sunflower heads to threshing location, using uncontaminated containers if needed. Starting with the untreated heads first, thresh the sunflower heads and collect the whole seed samples that weigh a minimum of 2 lb (but preferably not more than 4 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

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**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**For Sunflower Seed to be Processed into Refined Oil and Meal (Field trial XX only):** Following one of the above procedures, collect one untreated and one treated 50-60 lb (approximate) whole seed samples for processing into refined oil and meal. During harvest and sampling, follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place samples in a container or containers suitable for shipment to the processing laboratory. Identify each sample with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
C	02	PPP	X (+1)	2 lb.	Whole Seed
D	02	PPP	X (+1)	2 lb.	Whole Seed

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Whole Seed
B	01	Untreated	NA	2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed
	02	PPP		2 lb.	Whole Seed

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.



### **18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WEIGHT RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
PA	01	Untreated	NA	50-60 lb.	Whole Seed
PT	02	PPP	X (+1)	50-60 lb.	Whole Seed

### **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### **19.1 SEED RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After harvest and residue sample collection, utilize procedures that minimize sample degradation. Place samples in freezer as soon as possible after collection, preferably within 4 hours. If samples cannot be placed in the freezer within one hour after collection, samples should be placed in a cooler immediately after collection.

Maintain samples at temperatures generally less than -18 °C (0°F) until shipped. If possible, ship samples within 14 days of harvest.

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Contact the designated person (noted below) from the processing facility prior to shipment of samples for specific instructions. Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

### 19.3 PROCESSING:

The processing facility will store the sunflower seed in frozen storage at temperatures generally less than -18°C (0°F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc., until processing.

Immediately prior to processing, remove representative "grab" samples of untreated and treated seed from the larger samples (approximately 2-4 lb per "grab" sample). Using simulated commercial processing (provide detailed description of equipment and procedures), produce sunflower meal and refined oil. Process the untreated samples first. Collect one sample of sunflower meal and one sample of refined oil from the untreated and from the treated seed samples. Do each sample separately.

Place samples in appropriate containers and label. **Divide each sample of oil into separate containers of 150-300 ml. It is also acceptable to divide meal samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Immediately (as soon as possible after processing) freeze the processed samples. Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Document all sample additions to and removals from storage in freezer logs. Monitor and document all on-site storage temperatures.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Ship by freezer truck (such as ACDS), overnight air express, or by any other carrier that maintains frozen sample integrity. When shipping by a means other than a freezer truck, pack all samples in dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). All storage temperatures are to be monitored and documented. For analysis, send samples to: @@@

### 19.4 PROCESSED SAMPLE INVENTORY:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT/VOL. RANGE OF SAMPLE	CROP FRACTION
GA	01	Untreated	NA	2-4 lb.	Whole Seed
GT	02	PPP	X (+1)	2-4 lb.	Whole Seed
MA	01	Untreated	NA	5-7 lb.	Meal
MT	02	PPP	X (+1)	5-7 lb.	Meal
OA	01	Untreated	NA	1-2 liters	Refined Oil
OT	02	PPP	X (+1)	1-2 liters	Refined Oil

### 20. FIELD DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

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The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
  - 20.1.02- Deviations from protocol and standard operating procedures
  - 20.1.03- Date sunflower seed samples received
  - 20.1.04- Storage temperatures until sunflower seed samples are processed into meal and refined oil
  - 20.1.05- Processing Methodology (SOPs are acceptable)
- .....

20.1.06- Data collected and observations made during processing of samples into

20.1.07- Storage temperatures of sunflower seed, meal, and refined oil samples until shipped

20.1.08- Date sunflower seed, meal, and refined oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Fungi, edible, group 21

## Mushroom

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 12 marketable-sized mushrooms per sample. Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 1 lb (but preferably not more than 2 lb). Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove loose adhering media by either lightly brushing with a soft dry clean brush or lightly rinsing with a minimum of clean water (document what is used to remove the debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE MUSHROOMS.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 mushrooms / 1 lb.	Mushroom
B	01	Untreated	NA	12 mushrooms / 1 lb.	Mushroom
C	02	PPP	X ( $\pm 1$ )	12 mushrooms / 1 lb.	Mushroom
D	02	PPP	X ( $\pm 1$ )	12 mushrooms / 1 lb.	Mushroom

#### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 mushrooms / 1 lb.	Mushroom
B	01	Untreated	NA	12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom

\*\*\*\*\*

	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom
	02	PPP		12 mushrooms / 1 lb.	Mushroom

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

**Vegetable, stalk, stem, and leaf petiole, group 22**



## Asparagus

### 12. TEST SITE PREPARATION:

Prepare or select a test site that has been maintained following good local agricultural practices for the production of asparagus including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Harvest the crop if needed on a regular basis as per standard commercial practice to prevent spears from reaching a size too large or in such poor condition as to preclude use as samples.**

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). . At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, cut or snap at least 24 spears per sample from 24 separate plants between soil level and 1-2 inches below soil level as done commercially. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh at least 4 lb. (but preferably not more than 6 lb). Harvest more spears if necessary.

If loose soil or other debris adheres to cut spears, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse with a minimal amount of clean water. Lightly pat to dry with clean paper towels. **DO NOT RUB WHILE RINSING AND DRYING THE SPEARS.**

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 spears / 4 lb.	Spears
B	01	Untreated	NA	24 spears / 4 lb.	Spears

\*\*\*\*\*

C	02	PPP	X ( <u>±</u> 1)	24 spears / 4 lb.	Spears
D	02	PPP	X ( <u>±</u> 1)	24 spears / 4 lb.	Spears

**18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 spears / 4 lb.	Spears
B	01	Untreated	NA	24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears
	02	PPP		24 spears / 4 lb.	Spears

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Celery

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 12 plants (above-ground portion only). Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). Remove dead and/or senesced leaves. DO NOT TRIM.

If excessive soil adheres to the foliage and stems, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE STALKS AND LEAVES.**

If necessary, reduce gross sample weight to a minimum of 4 lb (but preferably not more than 6 lb) by cutting each whole plant longitudinally into quarters with a clean knife on an uncontaminated surface. Retain at least one quarter of each plant. Each retained portion may be cut horizontally in order to fit the samples into the bags. Plants may be cut horizontally into halves or quarters even if weight reduction is unnecessary, in order to fit the samples into the sample bags. Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Process the untreated samples first. Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 4 lb.	Untrimmed leaf stalk
B	01	Untreated	NA	12 plants / 4 lb.	Untrimmed leaf stalk
C	02	PPP	X ( $\pm 1$ )	12 plants / 4 lb.	Untrimmed leaf stalk
D	02	PPP	X ( $\pm 1$ )	12 plants / 4 lb.	Untrimmed leaf stalk

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**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 4 lb.	Untrimmed leaf stalk
B	01	Untreated	NA	12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk
	02	PPP		12 plants / 4 lb.	Untrimmed leaf stalk

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Kohlrabi

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect the bulbous stem and leaves from a minimum of 12 plants as done commercially. Each sample should be collected during a separate run through the entire plot. Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove dead and/or senesced leaves.

If excessive soil adheres to the plants, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE PLANTS.**

Reduce gross sample weight to a minimum of 2 lb (but preferably not more than 3 lb) by cutting each sampled plant longitudinally into quarters with a clean knife on an uncontaminated surface. Retain at least one quarter of each plant. Process untreated samples first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Bulbous Stem and Leaves
B	01	Untreated	NA	12 plants / 2 lb.	Bulbous Stem and Leaves
C	02	PPP	X ( $\pm 1$ )	12 plants / 2 lb.	Bulbous Stem and Leaves
D	02	PPP	X ( $\pm 1$ )	12 plants / 2 lb.	Bulbous Stem and Leaves

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**18.2 Decline trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 plants / 2 lb.	Bulbous Stem and Leaves
B	01	Untreated	NA	12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves
	02	PPP		12 plants / 2 lb.	Bulbous Stem and Leaves

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Rhubarb

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 12 rhubarb stalks (petioles) per sample from separate plants. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Cut petioles at the soil line. Remove leaves, retaining the petioles for the sample. Avoid sampling from the plot ends.

If necessary, the petioles may be cut into smaller segments, retaining all of the segments. Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

If excessive soil adheres to the petioles, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE PETIOLES.

Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 petioles / 4 lb.	Petioles
B	01	Untreated	NA	12 petioles / 4 lb.	Petioles
C	02	PPP	X ( $\pm 1$ )	12 petioles / 4 lb.	Petioles
D	02	PPP	X ( $\pm 1$ )	12 petioles / 4 lb.	Petioles

#### 18.2 Decline trial XX@@:

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SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 petioles / 4 lb.	Petioles
B	01	Untreated	NA	12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles
	02	PPP		12 petioles / 4 lb.	Petioles

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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**Fruit, tropical, group 23**

Date

#### **10. TEST SYSTEM/CROP:**

DATE - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

#### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, unless each tree is treated individually by an applicator who encircles the tree during the application, in which case the treated plot may have a minimum of 4 trees. See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

#### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of dates including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable date production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

#### **15. APPLICATION TREATMENTS AND TIMING:**

Trt#	Treatment	Target Rate of active ingredient	Target Rate of formulated product*	Application Type	Spray Volume Range**
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	PPP	@@ lbs ai/acre (@@ grams ai/hectare)	@@ grams ml/acre + adjuvant *** (@@ grams ml/hectare)	@@	@@ GPA (@@ L/Ha)

\*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).

\*\*GPA=gallons per acre, L/Ha=liters per hectare

**DELETE ALL OR PART AS NECESSARY: \*\*\*All applications shall include an adjuvant at a rate recommended by the adjuvant label unless the absence of an adjuvant has been chosen to differentiate two trials conducted by the same Field Research Director (see Part 11.4). Include a copy of the adjuvant label in the Field Data Book.**

**If it appears that phytotoxicity has resulted from applications made in this trial, contact the Study Director. If possible, take one or more photographs and send them to the Study Director via email to facilitate the evaluation of crop/ test substance effects.**

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All trials except Decline Trial XX@@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest. The applicator will need to raise the paper wraps (Deglet Noor variety) or remove the mesh bag (Medjool variety) prior to application. These processes are necessary to expose the date fruit to the material. The wraps/bags should be put back in place to protect the fruit after each application.

Decline Trial XX@@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest. The applicator will need to raise the paper wraps (Deglet Noor variety) or remove the mesh bag (Medjool variety) prior to application. These processes are necessary to expose the date fruit to the material. The wraps/bags should be put back in place to protect the fruit after each application.

In the field trials indicated below, phytotoxicity data must be collected at 7-14 days after each application using a 0-4 scale and entered into Field Data Book 6K2. If the crop is harvested within 14 days of the last application, then the assessment should be made on the day of harvest. If a rating of 2 or higher is given to a plot, then a follow-up rating is needed 7-14 days after that, even if there is no additional test substance application in the interim, unless this rating is given to the crop at harvest.

Phytotoxicity Data is Required in the Field Trials Listed Below:			

**DELETE EITHER the table and instructions above OR 15.1 and 15.2 (below), depending on the complexity of the study.**

15.1 These treatments shall be applied in all trials except:

Trt#	Treatment	Target Rate of active ingredient	Target Rate of formulated product*	Application Type	Spray Volume Range**
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	PPP	@@ lbs ai/acre (@@ grams ai/hectare)	@@ grams ml/acre + adjuvant *** (@@ grams ml/hectare)	@@	@@ GPA (@@ L/Ha)

\*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).

\*\*GPA=gallons per acre, L/Ha=liters per hectare

**DELETE ALL OR PART AS NECESSARY: \*\*\*All applications shall include an adjuvant at a rate recommended by the adjuvant label unless the absence of an adjuvant has been chosen to differentiate two trials conducted by the same Field Research Director (see Part 11.4). Include a copy of the adjuvant label in the Field Data Book.**

If it appears that phytotoxicity has resulted from applications made in this trial, contact the Study Director. If possible, take one or more photographs and send them to the Study Director via email to facilitate the evaluation of crop/ test substance effects.

All trials except Decline Trial XX@@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest. The applicator will need to raise the paper wraps (Deglet Noor variety) or remove the mesh bag (Medjool variety) prior to application. These processes are necessary to expose the date fruit to the material.

Decline Trial XX@@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest. The applicator will need to raise the paper wraps (Deglet Noor variety) or remove the mesh bag (Medjool variety) prior to application. These processes are necessary to expose the date fruit to the material.

In the field trials indicated below, phytotoxicity data must be collected at 7-14 days after each application using a 0-4 scale and entered into Field Data Book 6K2. If the crop is harvested within 14 days of the last application, then the assessment should be made on the day of harvest. If a rating of 2 or higher is given to a plot, then a follow-up rating is needed 7-14 days after that, even if there is no additional test substance application in the interim, unless this rating is given to the crop at harvest.

Phytotoxicity Data is Required in the Field Trials Listed Below:			

15.2 These treatments shall be applied in the following trials only:

Trt#	Treatment	Target Rate of active ingredient	Target Rate of formulated product*	Application Type	Spray Volume Range**
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	PPP	@@ lbs ai/acre (@@ grams ai/hectare)	@@ grams ml/acre + adjuvant *** (@@ grams ml/hectare)	@@	@@ GPA (@@ L/Ha)
03	PPP	@@ lbs ai/acre (@@ grams ai/hectare)	@@ grams ml/acre + adjuvant *** (@@ grams ml/hectare)	@@	@@ GPA (@@ L/Ha)
04	PPP	@@ lbs ai/acre (@@ grams ai/hectare)	@@ grams ml/acre + adjuvant *** (@@ grams ml/hectare)	@@	@@ GPA (@@ L/Ha)

\*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).

\*\*GPA=gallons per acre, L/Ha=liters per hectare

**DELETE ALL OR PART AS NECESSARY: \*\*\*All applications shall include an adjuvant at a rate recommended by the adjuvant label unless the absence of an adjuvant has been chosen to differentiate two trials conducted by the same Field Research Director (see Part 11.4). Include a copy of the adjuvant label in the Field Data Book.**

If it appears that phytotoxicity has resulted from applications made in this trial, contact the Study Director. If possible, take one or more photographs and send them to the Study Director via email to facilitate the evaluation of crop/ test substance effects.

All trials except Decline Trial XX@@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest. The applicator will need to raise the paper wraps (Deglet Noor variety) or remove the mesh bag (Medjool variety) prior to application. These processes are necessary to expose the date fruit to the material.

Decline Trial XX@@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest. The applicator will need to raise the paper wraps (Deglet Noor variety) or remove the mesh bag (Medjool variety) prior to application. These processes are necessary to expose the date fruit to the material.

In the field trials indicated below, phytotoxicity data must be collected at 7-14 days after each application using a 0-4 scale and entered into Field Data Book 6K2. If the crop is harvested within 14 days of the last application, then the assessment should be made on the day of harvest. If a rating of 2 or higher is given to a plot, then a follow-up rating is needed 7-14 days after that, even if there is no additional test substance application in the interim, unless this rating is given to the crop at harvest.

Phytotoxicity Data is Required in the Field Trials Listed Below:			

#### **16. SUPPLEMENTAL CROP TREATMENTS:**

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

#### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect mature (dried) dates from a minimum of 4 trees. Fruits should be collected from at least one bunch per tree. The bunches should be collected from different sides of the trees. Use one of the following sample collection methods: After removing a bunch from the tree, shake it to cause ripe dates to fall off and collect fruits from among the fallen dates. In trials with the Medjool variety, ripe Medjool dates may be collected by hand directly from the bunch. Alternatively, for any date variety, dates may be collected by shaking the tree (without removing bunches) and collecting fruits from among the fallen dates. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.)

Remove stems and pits, retaining the fruit for the sample. Each sample should weigh a minimum of 2 lb. Process untreated sample first. Record the length of time from completion of the pit and stem removal to placement in a cooler for each sample in Field Data Book Part 7.A.2. **For each sample, record the weight of the pits that have been removed and record the weight per sample in the Field Data Book (e.g., Sample A pits—1.5 lbs.). The pits may be discarded after being weighed.**

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

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Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1), and harvest/sampling dates. Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Dried fruit
B	01	Untreated	NA	2 lb.	Dried fruit
C	02	PPP	X (+1)	2 lb.	Dried fruit
D	02	PPP	X (+1)	2 lb.	Dried fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Dried fruit
B	01	Untreated	NA	2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit
	02	PPP		2 lb.	Dried fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. Shipment of frozen samples will be by freezer truck or express shipment. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then

pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

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20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

20.17- Data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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Fig

## **10. TEST SYSTEM/CROP:**

FIG - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

## **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, unless each tree is treated individually by an applicator who encircles the tree during the application, in which case the treated plot may have a minimum of 4 trees. See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

## **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of figs including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

## **15. APPLICATION TREATMENTS AND TIMING:**

Trt#	Treatment	Target Rate of active ingredient	Target Rate of formulated product*	Application Type	Spray Volume Range**
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	PPP	@@ lbs ai/acre (@@ grams ai/hectare)	@@ grams ml/acre + adjuvant *** (@@ grams ml/hectare)	@@	@@ GPA (@@ L/Ha)

\*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).

\*\*GPA=gallons per acre and L/Ha=liters per hectare

\*\*\*All applications shall include an adjuvant at a rate recommended by the adjuvant label unless the absence of an adjuvant has been chosen to differentiate two trials conducted by the same Field Research Director (see Part 11.4). Include a copy of the adjuvant label in the Field Data Book.

If it appears that phytotoxicity has resulted from applications made in this trial, contact the Study Director. If possible, take one or more photographs and send them to the Study Director via email to facilitate the evaluation of crop/ test substance effects.

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All trials except Decline Trial @@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest. Just before making the last application, remove all fallen figs from the ground in the plots.

Decline Trial @@: Make @@ applications at intervals of @@ days with the last application @@ days before harvest.

In the field trials indicated below, phytotoxicity data must be collected at 7-14 days after each application using a 0-4 scale and entered into Field Data Book 6K2. If the crop is harvested within 14 days of the last application, then the assessment should be made on the day of harvest. If a rating of 2 or higher is given to a plot, then a follow-up rating is needed 7-14 days after that, even if there is no additional test substance application in the interim, unless this rating is given to the crop at harvest.

Phytotoxicity Data is Required in the Field Trials Listed Below:			

## 16. SUPPLEMENTAL CROP TREATMENTS:

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Use only maintenance pesticides that have been registered on this commodity by EPA or the corresponding agency in the country in which the trial is located. Apply according to labeled directions. Make identical applications to the untreated and treated plots.

**Consult with Study Director** if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Bird netting is an acceptable means of protecting the test system against birds and other vertebrate pests. Contact the Study Director if netting is needed during the period that applications will be made. When bird netting is used, be sure to document use and details (type, when covered, removed etc.) in the Field Data Book.

## 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples of fresh figs from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect figs from a minimum of 4 trees. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.) Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

Alternatively, it is acceptable to collect figs that have ripened and fallen from a minimum of 4 trees onto the ground, if the fallen figs are determined to still be fresh. The collection date for fresh figs is defined as the harvest date, regardless of whether figs were collected directly from the trees or from the ground.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Fig samples should each weigh a minimum of 2 lb. (but preferably not more than 4 lb.).

**Decline trial CA@@ only:** Insert instructions here or delete if there is no decline trial. Fruits should be impartially picked from high, low, sheltered and exposed areas throughout the treated plot excluding the end trees.

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**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1), and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**Fresh and Dried Fig Trial CA@@ only:** After the completion of fresh fig sample collection, collect all remaining figs from the ground in the respective plots. Figs that are moist because they are in wet soil should be discarded as they will mold and may not have good quality. If the remaining number of figs on the ground in any plot seems insufficient for sample requirements, the trees may be shaken to induce more ripe fruit to fall onto the ground for collection. Move the collected figs to a protected area for drying, keeping the treated and untreated figs separate. Wooden trays or cement surfaces are acceptable locations for spreading figs to dry. After a suitable period of drying (typically 7-14 days), collect one sample of dried figs from the control plot and two samples of dried figs from the treated plot, each weighing a minimum of 2 lb. (but preferably not more than 4 lb.). A minimum of 12 figs per sample should be collected. Dried figs should have a moisture content of approximately 12-18%. (Document a quantifiable procedure in the Field Data Book describing your methodology for estimating the moisture content.) It can be determined if the fig is not yet dry by picking it up and squeezing it between your thumb and finger. If the skin breaks and it is soft to touch, then the fruit is too fresh and the moisture will be higher than desired. Wrinkled fruit is also an indication that the fruit may be dry enough to pick with the desired moisture of 12-18%, but the moisture content must be verified.

Gently remove extraneous matter such as twigs. Dirt may be shaken off, but the figs should not be washed.

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial CA@@ and Fresh and Dried Fig Trial CA@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh figs
FB	01	Untreated	NA	2 lb.	Fresh figs
FC	02	PPP	X (+1)	2 lb.	Fresh figs
FD	02	PPP	X (+1)	2 lb.	Fresh figs

### 18.2 Decline Trial CA@@:

SAMPLE ID			DAYS AFTER	MINIMUM SAMPLE SIZE	CROP FRACTION
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	TRT#	TREATMENT	LAST APPLIC.		
FA	01	Untreated	NA	2 lb.	Fresh figs
FB	01	Untreated	NA	2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs
	02	PPP		2 lb.	Fresh figs

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC and FD.

### 18.3 Fresh and Dried Fig Trial CA@@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	2 lb.	Fresh figs
FB	01	Untreated	NA	2 lb.	Fresh figs
FC	02	PPP	X (+1)	2 lb.	Fresh figs
FD	02	PPP	X (+1)	2 lb.	Fresh figs
DA	01	Untreated	NA	2 lb.	Dried figs
FE	03	PPP	X (+1)	2 lb.	Fresh figs
FF	03	PPP	X (+1)	2 lb.	Fresh figs
DT1	03	PPP	X (+1)	2 lb.	Dried figs
DT2	03	PPP	X (+1)	2 lb.	Dried figs

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. Shipment of frozen samples will be by freezer truck or express shipment. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

## 20. FIELD DOCUMENTATION AND RECORD KEEPING:

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All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- In the dried fig trial only, include data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## Guava or Carambola

### **10. TEST SYSTEM/CROP:**

CCC - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, if the test substance is to be applied while moving straight down the row. If each tree is to be treated while encircling the tree during the application, then only 4 trees are necessary in the treated plot (because there are no "end trees"). See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of ccc including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. Harvest the crop as needed on a regular basis in accordance with standard commercial practice to prevent fruit from becoming over-sized or degrading to unmarketable condition.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

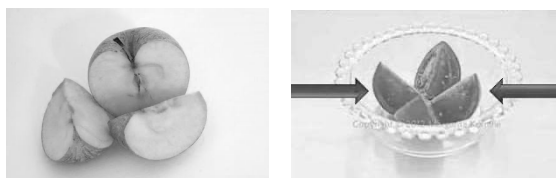
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 marketable-sized fruit per sample from at least 4 trees. Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from row ends. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Cut each fruit into quarters longitudinally with a clean knife on an uncontaminated surface. If the sample would weigh more than 8 lb before cutting, discard two quarters and retain opposite quarters of each fruit for the sample. (If sample weight reduction is not needed, then all portions of the cut fruit should be retained.) Do not peel the fruit. Include a proportional amount of the seeds. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

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Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

Fruits should be impartially picked from high, low, sheltered and exposed areas throughout the treated plot excluding the end trees.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Papaya

### **10. TEST SYSTEM/CROP:**

PAPAYA - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 8 trees, if the test substance is to be applied while moving straight down the row. If each tree is to be treated while encircling the tree during the application, then only 6 trees are necessary in each plot (because there are no “end trees”). See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of papaya including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

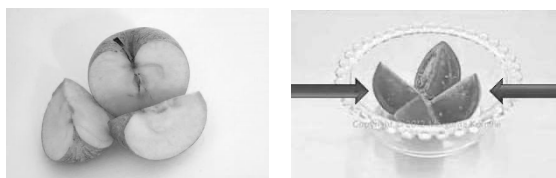
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 12 fruits (mature green to color break) per sample from the bottom of the fruit column from a minimum of 6 plants. Each sample should be collected during a separate run through the entire plot. No more than three fruits from each plant should be taken. Avoid taking fruits from end plants. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end plants to avoid.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Cut each fruit into quarters or eighths longitudinally with a clean knife on an uncontaminated surface. If the sample would weigh more than 8 lb before cutting, retain all slices. If necessary to reduce gross sample weight to a minimum of 4 lb, retain opposite quarters (or eighths) of each fruit. If the retained slices are more than about 6 inches (15 cm) long, then they should each be cut into two smaller pieces, and both of the pieces should be retained for the sample. Do not peel the fruit. Include a proportional amount of the seeds. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

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Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 4 lb.	Non-peeled fruit
B	01	Untreated	NA	12 fruits / 4 lb.	Non-peeled fruit
C	02	PPP	X (+1)	12 fruits / 4 lb.	Non-peeled fruit
D	02	PPP	X (+1)	12 fruits / 4 lb.	Non-peeled fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 4 lb.	Non-peeled fruit
B	01	Untreated	NA	12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit
	02	PPP		12 fruits / 4 lb.	Non-peeled fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.



## Olive

### **10. TEST SYSTEM/CROP:**

OLIVE - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of olives including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect fruit from several places on at least 4 trees. Each sample should be collected during a separate run through the entire plot. Olives should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from row ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove pits from fruit and collect at least 2 lb of fruit (but preferably not more than 3 lb) per sample. Process untreated sample first. Record the length of time from completion of the pit removal to placement in a cooler for each sample in Field Data Book Part 7.A.2. **For each sample, record the weight of the pits that have been removed and record the weight per sample in the Field Data Book (e.g., Sample A pits—1.5 lbs.). The pits may be discarded after being weighed.**

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**Samples for processing (Trial CAXX only):** Collect one additional untreated sample and one additional treated

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sample for processing. **Olives should be sufficiently mature for processing into oil.** Each sample should weigh approximately 50-60 lb. **Do not remove pits. Do not freeze these samples.** Samples should be stored in secure, clean containers for transportation to the processing facility. Identify each sample container with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. See Section 19.2 for shipping/transportation instructions.

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

### **18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Fruit without pits
B	01	Untreated	NA	2 lb.	Fruit without pits
C	02	PPP	X (+1)	2 lb.	Fruit without pits
D	02	PPP	X (+1)	2 lb.	Fruit without pits

### **18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Fruit without pits
B	01	Untreated	NA	2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits
	02	PPP		2 lb.	Fruit without pits

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### **18.2 PROCESSING RESIDUE SAMPLE INVENTORY: Trial CAXX only**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	50-60 lb.	Fruit with pits
PT	02	PPP	X (+1)	50-60 lb.	Fruit with pits

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### **19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to

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permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

#### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

Processing samples should be sent fresh (NOT FROZEN) in secure, clean containers to the processing facility. Samples should be shipped within 30 hours of harvest. Two options are available for transportation of these samples. They may be sent via “express shipment” (overnight carrier such as Federal Express or Airborne). Alternatively, if the processing facility is within the same state as the field trial, the samples may be transported in a vehicle driven by the Field Research Director or by a person under the management of the Field Research Director. Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

#### **19.3 PROCESSING:**

Immediately prior to processing, remove representative “grab” samples of untreated and treated olives from the larger samples (approximately 2-4 lb per “grab” sample after pit removal). **Remove the pits from the olives in these grab samples, and then place them in frozen storage.**

As soon as possible after receiving the olives for processing, process them as done commercially into oil. Start with the untreated samples first. Process each sample separately.

Place samples in appropriate containers and label. **Divide each sample of oil into separate containers of 150-300 ml. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Immediately freeze the processed samples and hold at least 0 °F (-18 °C) until shipped to the analytical lab. For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will

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be monitored and documented.

Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information).

Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT./VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2-4 lb.	Fruit without pits
GT	02	PPP	X (+1)	2-4 lb.	Fruit without pits
OA	01	Untreated	NA	200-400 mls	Oil
OT	02	PPP	X (+1)	200-400 mls	Oil

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction

20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.

20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots

20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

#### **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

20.1.01- Names of all personnel conducting specific research functions

20.1.02- Deviations from protocol and standard operating procedures

20.1.03- Date olive samples received

20.1.04- Storage temperatures until olive samples are processed into oil

20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing of samples into oil

20.1.07- Storage temperatures of olive fruit and oil samples until shipped

20.1.08- Date olive fruit and oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.



**Fruit, tropical, group 24**

## Avocado or Mango

### **10. TEST SYSTEM/CROP:**

CCC - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, if the test substance is to be applied while moving straight down the row. If each tree is to be treated while encircling the tree during the application, then only 4 trees are necessary in the treated plot (because there are no "end trees"). See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of ccc including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 24 fruits per sample from a minimum of 4 trees. Each sample should be collected during a separate run through the entire plot. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from treated end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.)

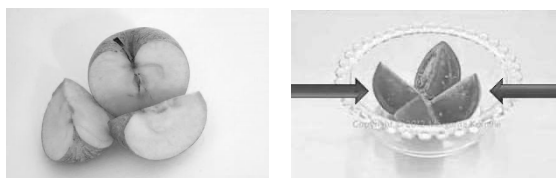
In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.  
Fruits should be impartially picked from high, low, sheltered and exposed areas throughout the treated plot excluding the end trees.

**All trials:** Cut each fruit longitudinally into quarters with a clean knife on an uncontaminated surface. Start with untreated samples first. Remove the pit and stem. If the sample would weigh more than 8 lb (after pit and stem removal), then reduce gross sample weight to a minimum of 4 lb by discarding two quarters and retaining opposite

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quarters from each fruit for the sample. This needs to be determined prior to reducing the fruit size to insure that opposite quarters are retained. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit without stem and pit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit without stem and pit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit without stem and pit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit without stem and pit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit without stem and pit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit

	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit
	02	PPP		24 fruits / 4 lb.	Fruit without stem and pit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

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## Banana

### **10. TEST SYSTEM/CROP:**

BANANA - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

**Trials XX and YY will have bagged bananas. Bag the fruit on the trees at the appropriate stage of maturity as done commercially. Do not remove the bags during test substance applications. Document in the notebook the date and maturity at bagging, type of bag used and any other pertinent information regarding this cultural practice.**

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. If individual banana plants within a trial shall be treated by encircling the plants during the application (rather than passing along the side of the plants while going down the rows), then it is acceptable for the treated plot to include plants that will remain untreated. In this circumstance at least 6 of the plants within the plot should be treated, and none of the 6 shall be considered "end plants". See Parts 17 & 18 for requirements for residue sampling.

**Do not delete** 11.2-11.6 from the draft.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of bananas including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 marketable-sized fruits per sample from at least 6 bunches from separate plants and from separate places on each of the bunches. **(Remove the bags from bagged bananas.)** Each sample should be collected during a separate run through the entire plot. Do not peel the fruit. Avoid sampling from row ends (unless individual plants have been treated as described in 11.1). The retained bananas should be placed in plastic bags before placing the sample in the cloth sample bags.

After residue sample collection, cut bananas into separate pieces before placing in freezer storage. Each banana should be cut once lengthwise; then cut each banana half into multiple cross sections. All banana pieces will be retained from each banana sampled. Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

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**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits	Fruit w/peel
B	01	Untreated	NA	24 fruits	Fruit w/peel
C	02	PPP	X (+1)	24 fruits	Fruit w/peel
D	02	PPP	X (+1)	24 fruits	Fruit w/peel

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits	Fruit w/peel
B	01	Untreated	NA	24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel
	02	PPP		24 fruits	Fruit w/peel

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Dragon fruit

### **10. TEST SYSTEM/CROP:**

DRAGON FRUIT - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of dragon fruit including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

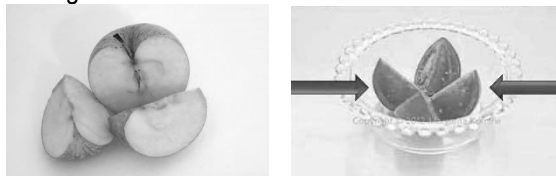
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 24 fruit from at least 4 cacti. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Collect enough fruit to yield a minimum of 4 lb (but preferably not more than 6 lb) per sample. The fruit do not need to be mature in color, and should be collected from high and low areas of the cacti.

**All trials: All dragon fruits, regardless of size, should be cut into quarters (cut from stem end to opposite end into four pieces).** If the sample size is greater than 8 lb, then only the opposite quarters should be retained for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

Fruits should be impartially picked from high, low, sheltered and exposed areas throughout the treated plot excluding the end trees.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

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Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:  
**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Fruit
B	01	Untreated	NA	4 lb.	Fruit
C	02	PPP	X (+1)	4 lb.	Fruit
D	02	PPP	X (+1)	4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	4 lb.	Fruit
B	01	Untreated	NA	4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit
	02	PPP		4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.



## Lychee

### **10. TEST SYSTEM/CROP:**

LYCHEE - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, if the test substance is to be applied while moving straight down the row. If each tree is to be treated while encircling the tree during the application, then only 4 trees are necessary in the treated plot (because there are no "end trees"). See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of lychees including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 80 fruit per sample from a minimum of 4 trees. Each sample should be collected during a separate run through the entire plot. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.) Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Gently remove any twigs or foreign matter from the fruit. Remove the pit before placing samples into labelled sample bags.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of

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pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags, and then place them in frozen storage immediately. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:  
**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;**  
**Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	80 fruits / 4 lb.	Fruit
B	01	Untreated	NA	80 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	80 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	80 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	80 fruits / 4 lb.	Fruit
B	01	Untreated	NA	80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Passionfruit

### **10. TEST SYSTEM/CROP:**

PASSIONFRUIT - Use a commercial variety. Report: variety, age of vines, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 vines, if the test substance is to be applied while moving straight down the row. If each vine is to be treated while encircling it during the application, then only 4 vines are necessary in each plot (because there are no "end vines"). See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of passionfruit including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. Harvest the crop as needed on a regular basis in accordance with standard commercial practice to prevent fruit from becoming over-sized or degrading to unmarketable condition.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 marketable-sized fruit per sample from at least 4 vines. Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). At least one fruit from each vine should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end vines. Avoid sampling from row ends. (If each vine was treated individually by an applicator who encircled it during the application, then there are no end vines to avoid.)

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Do not peel or cut the fruit.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

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Fruits should be impartially picked from high, low, sheltered and exposed areas throughout the treated plot excluding the end vines.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Persimmon

### **10. TEST SYSTEM/CROP:**

PERSIMMON - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of persimmons including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect 24 fruits per sample from at least 4 trees. Each sample should be collected during a separate run through the entire plot. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from row ends. Each sample should weigh a minimum of 4 lb. (but preferably not more than 6 lb.).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

Fruits should be impartially picked from high, low, sheltered and exposed areas throughout the treated plot excluding the end trees.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### **18. FIELD RESIDUE SAMPLE INVENTORY:**

#### **18.1 All Trials except Decline Trial XX@@:**

SAMPLE	TRT#	TREATMENT	DAYS AFTER	MINIMUM	CROP
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ID			LAST APPLIC.	SAMPLE SIZE	FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Whole Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Whole Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Whole Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Whole Fruit

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Whole Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit
	02	PPP		24 fruits / 4 lb.	Whole Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Pineapple

### **10. TEST SYSTEM/CROP:**

PINEAPPLE - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of pineapples including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

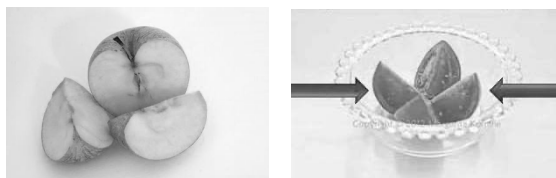
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect 12 fruits per sample. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove the crown of each fruit by twisting it off. DO NOT remove the crown by cutting. Remove the crowns, retaining the fruit for the sample. Each sample should weigh a minimum of 3 lb (but preferably not more than 15 lb).

Each fruit should be cut longitudinally into quarters (or eighths) with a clean knife on a clean surface, retaining the skin and pulp from opposite quarters (or eighths) of the fruit for the sample. (If the sample prior to cutting does not weigh more than 6 lb, then all quarters (or eighths) should be retained for the sample.) Immediately freeze the samples. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section

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23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

**Processing samples (Field trial XX only):** At X ( $\pm 1$ ) days after the last application, collect one additional untreated sample and one additional treated sample (24 fruits per sample).

Remove the crown of each fruit by twisting it off. DO NOT remove the crown by cutting. Remove the crowns, retaining the fruit for the sample. DO NOT reduce the gross sample weight.

DO NOT freeze the samples.

Place all samples in plastic-lined cloth bags. Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#;** **Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 3 lb.	Fruit without Crown
B	01	Untreated	NA	12 fruits / 3 lb.	Fruit without Crown
C	02	PPP	X ( $\pm 1$ )	12 fruits / 3 lb.	Fruit without Crown
D	02	PPP	X ( $\pm 1$ )	12 fruits / 3 lb.	Fruit without Crown

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 fruits / 3 lb.	Fruit without Crown
B	01	Untreated	NA	12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown
	02	PPP		12 fruits / 3 lb.	Fruit without Crown

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 PROCESSING RESIDUE SAMPLES INVENTORY: Trial XX only

SAMPLE	TRT#	TREATMENT	DAYS AFTER	MINIMUM	CROP FRACTION
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ID			LAST APPLIC.	SAMPLE SIZE	
PA	01	Untreated	NA	24 fruits	Fruit without Crown
PT	03	PPP	X ( $\pm 1$ )	24 fruits	Fruit without Crown

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility prior to shipment to an analytical laboratory.

### **19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

After sample collection in the field, if samples PA and PT are not shipped to the processing facility on the day of collection, they should be stored in a refrigerator at about 4 °C until they are shipped. Refrigerator logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples. Send samples for processing to: @@@**

### **19.3 PROCESSING:**

Just prior to processing, remove one grab sample of 12 fruits from the untreated pineapples (Sample GA) and one

grab sample of 12 fruits from the treated pineapples (Sample GT). Each fruit should be cut longitudinally into quarters with a clean knife on a clean surface, retaining the skin and pulp from opposite quarters of the fruit for the sample (as described in Section 17). (If the sample prior to cutting does not weigh more than 6 lb, then all quarters should be retained for the sample.) Immediately freeze these samples.

Using simulated commercial processing (provide detailed description of equipment and procedures), produce juice and process residue from the remaining fruit in samples PA and PT. [Process residue consists of tops (minus crowns), bottoms, peels, any trimmings with peels cut up, and pulp left after squeezing for juice.] Process untreated fruit first (sample PA) followed by treated fruit (sample PT). Collect one sample of juice from both untreated and treated fruit (samples JA and JT) and one sample of process residue from both untreated and treated fruit (samples PRA and PRT). Juice samples should be approximately 1000-2000 ml each. Process residue samples should each weigh approximately 2-4 lb.

Place samples in appropriate containers and label. **Divide each sample of juice into separate containers of 150-300 ml. It is also acceptable to divide process residue samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical pineapple storage conditions to prevent test substance residue degradation. Maintain all frozen processed samples at temperatures generally less than  $-18^{\circ}\text{C}$  until shipped, allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

**For express shipments (overnight carriers such as Federal Express or Airborne)**, contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. VOL/WGT. RANGE OF SAMPLE	CROP FRACTION
GA	01	Untreated	NA	12 fruits	Fruit without Crown
GT	03	PPP	X ( $\pm 1$ )	12 fruits	Fruit without Crown
JA	01	Untreated	NA	1000-2000 ml	Juice
JT	03	PPP	X ( $\pm 1$ )	1000-2000 ml	Juice
PRA	01	Untreated	NA	2-4 lb.	Process Residue
PRT	03	PPP	X ( $\pm 1$ )	2-4 lb.	Process Residue

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

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- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date pineapple samples received
- 20.1.04- Storage temperatures until pineapple samples are processed into juice and process residue
- 20.1.05- Processing Methodology (SOPs are acceptable)

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20.1.06- Data collected and observations made during processing of samples into juice and process residue

20.1.07- Storage temperatures of pineapple fruit, juice, and process residue samples until shipped

20.1.08- Date pineapple fruit, juice, and process residue samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative through the Western Region office. When the processing summary report is completed the report and all original raw data will be sent to the Western Region Field Coordinator (when an original document cannot be provided a “true copy” will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A “true copy” of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Pomegranate

### **10. TEST SYSTEM/CROP:**

POMEGRANATE - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, unless each tree is treated individually by an applicator who encircles the tree during the application, in which case the treated plot may have a minimum of 4 trees. See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of pomegranates including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

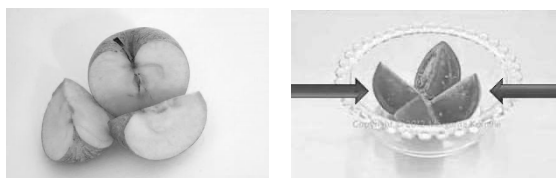
### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 marketable sized fruit (before reaching full maturity) per sample from a minimum of 4 trees. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.) Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

**Decline trial @@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Each fruit should be cut into quarters or eighths longitudinally and all portions retained. If the sample weight exceeds 8 lb, then opposite portions only should be retained for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

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Longitudinal cuts / Opposite quarters

Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**NOTE TO STUDY DIRECTOR: Not all pomegranate studies will need processing; check with IR-4 Mgt. if this is unclear. Processing data may be needed in studies in which residues are expected.**

**For Fresh Pomegranates to be Processed into Juice (Field Trial @@):** Harvest 35-50 lb of pomegranates from each plot at X ( $\pm 1$ ) days after the last test substance application. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from plot ends.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial @@ and Processing Trial @@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial @@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D

### 18.3 PROCESSING Trial @@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
P-UTC	01	Untreated	NA	35-50 lb.	Fruit
P-TRT	02	PPP	X (+1)	35-50 lb.	Fruit

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT (Samples not for processing):

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**Freezer logs** will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented.

**For express shipments (overnight carriers such as Federal Express or Airborne)**, contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

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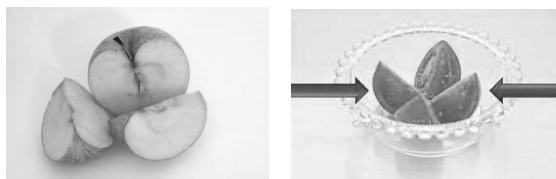
Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. For analysis, send samples to: @@@

### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

Contact the processing lab as soon as you know the date you expect to ship the large, fresh samples for processing, so that the lab will be ready to receive them and begin the processing part of the study as needed. If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in a refrigerator at approximately 4°C until they are shipped. At the time of shipping large samples, contact the processing lab (document this communication in the field data book). **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

### **19.3 PROCESSING:**

Immediately prior to processing pomegranates, remove representative “grab” samples of untreated and treated fruit from the larger samples (approximately 4-6 lb. for each sample). Place the “grab” samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Each fruit should be cut into quarters longitudinally and all quarters retained. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.



Longitudinal cuts / Opposite quarters

Process untreated sample first.

Using simulated commercial processing (provide detailed description of equipment and procedures), produce juice from the untreated and treated samples. Process untreated fruit first, followed by treated fruit. From the untreated fruit collect one sample of a minimum of 1000 ml of unpasteurized juice. From the treated fruit collect one sample of a minimum of 1000 ml of unpasteurized juice.

Place all samples into suitable clean containers and label them as indicated in the PROCESSED SAMPLE INVENTORY below. **Divide each sample of juice into separate containers of 50-150 grams. Each portion of the divided sample should be representative of the whole sample.** Processed samples should be frozen as soon as possible after processing is completed. For these samples, the processing lab should follow Protocol Section 19 procedures required for freezing/shipping samples to the analytical lab.

If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical pomegranate storage conditions to prevent test substance residue degradation. Maintain all frozen samples at temperatures generally less than -18 °C (0 °F) until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or “express” shipment. Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample

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integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. For analysis of processed fractions, send samples to: @@@

**19.4 PROCESSED SAMPLE INVENTORY:** Trial XX only

SAMPLE ID	TRT#	TREATMENT	APPROX. WGT. OR VOL. OFSAMPLE	CROP FRACTION
GA	01	Untreated	4-6 lb	Fruit
GT	02	PPP	4-6 lb	Fruit
JA	01	Untreated	1000-2000 ml	Unpasteurized Juice
JT	02	PPP	1000-2000 ml	Unpasteurized Juice

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## Prickly Pear Cactus

### **10. TEST SYSTEM/CROP:**

PRICKLY PEAR CACTUS - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1 Each test site will consist of two untreated and two treated plots (one each for the production of fruit and the other for the production of pads).**

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. See Parts 17 & 18 for requirements for residue sampling.

**11.2** Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

**11.3** If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of prickly pear cactus including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production. **Within each trial, one untreated and one treated plot must be managed for the production of fruit, and one untreated and one treated plot must be managed for the production of pads.**

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **15. APPLICATION TREATMENTS AND TIMING:**

Trt#	Treatment	Target Rate of active ingredient	Target Rate of formulated product*	Application Type	Spray Volume Range**
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	PPP	@@ lbs ai/acre	@@ grams or ml/acre + adjuvant ***	@@	@@ GPA

\*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).

\*\*GPA=gallons per acre

\*\*\*All applications shall include an adjuvant at a rate recommended by the adjuvant label unless the absence of an adjuvant has been chosen to differentiate two trials conducted by the same Field Research Director (see Part 11.4). Include a copy of the adjuvant label in the Field Data Book.

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If it appears that phytotoxicity has resulted from applications made in this trial, contact the Study Director. If possible, take one or more photographs and send them to the Study Director via email to facilitate the evaluation of crop/ test substance effects.

**Make @@ applications at intervals of @@ days with the last application @@ days before harvest. If necessary, the applications may be made on different days on the fruit sample plots than on the pad sample plots.**

## **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect samples from at least 4 cacti. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Fruit Samples:** Collect a minimum of 24 fruit per sample for Samples FA-FD to yield a minimum of 3 lb (but preferably not more than 6 lb) per sample. The fruit should be full size, but do not need to be mature in color, and should be collected from high and low areas of the cacti. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

**Pad Samples:** Collect enough young, succulent pads for Samples PA-PD to yield a minimum of 3 lb (but preferably not more than 6 lb) per sample. To reduce gross sample weight of the pads each pad may be cut longitudinally with a clean knife on an uncontaminated surface, retaining approximately 25% of each.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## **18. FIELD RESIDUE SAMPLE INVENTORY:**

### **18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	3 lb.	Fruit
FB	01	Untreated	NA	3 lb.	Fruit
FC	02	PPP	X (+1)	3 lb.	Fruit

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FD	02	PPP	X (+1)	3 lb.	Fruit
PA	01	Untreated	NA	3 lb.	Pads
PB	01	Untreated	NA	3 lb.	Pads
PC	02	PPP	X (+1)	3 lb.	Pads
PD	02	PPP	X (+1)	3 lb.	Pads

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	3 lb.	Fruit
FB	01	Untreated	NA	3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
	02	PPP		3 lb.	Fruit
PA	01	Untreated	NA	3 lb.	Pads
PB	01	Untreated	NA	3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads
	02	PPP		3 lb.	Pads

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC, FD, PC, and PD.

## Rambutan

### **10. TEST SYSTEM/CROP:**

RAMBUTAN - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, if the test substance is to be applied while moving straight down the row. If each tree is to be treated while encircling the tree during the application, then only 4 trees are necessary in the treated plot (because there are no "end trees"). See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for processing.** The plots must be large enough to provide enough sample weight to meet processing requirements.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of rambutans including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 80 fruit per sample from a minimum of 4 trees. Each sample should be collected during a separate run through the entire plot. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.) Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Do not peel or cut the fruit.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of

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pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags, and then place them in frozen storage immediately. Fruit collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	80 fruits / 4 lb.	Fruit
B	01	Untreated	NA	80 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	80 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	80 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	80 fruits / 4 lb.	Fruit
B	01	Untreated	NA	80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit
	02	PPP		80 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Sugar apple

### **10. TEST SYSTEM/CROP:**

SUGAR APPLE - Use a commercial variety. Report: variety, age of trees, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **11. TEST SYSTEM DESIGN and STATISTICAL METHOD:**

**11.1** Each test site will consist of one untreated and one treated plot.

The individual plots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled area will be needed to provide the necessary plant material. Each plot shall consist of at least 6 trees, if the test substance is to be applied while moving straight down the row. If each tree is to be treated while encircling the tree during the application, then only 4 trees are necessary in the treated plot (because there are no "end trees"). See Parts 17 & 18 for requirements for residue sampling.

**Field trial @@ will provide samples for a decline trial (multiple sampling dates).** The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of sugar apples including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

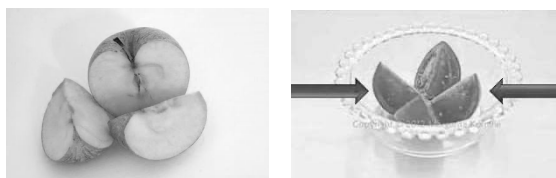
**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect a minimum of 24 marketable sized fruit (before reaching full maturity) per sample from a minimum of 4 trees. At least one fruit from each tree should be impartially picked from high, low, sheltered and exposed throughout the treated plot excluding the end trees. Avoid sampling from row ends. (If each tree was treated individually by an applicator who encircled the tree during the application, then there are no end trees to avoid.) Each sample should be collected during a separate run through the entire plot. Each sample should weigh a minimum of 4 lb (but preferably not more than 6 lb).

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials: All sugar apples, regardless of size, should be cut into quarters (cut from stem end to opposite end into four pieces).** If the sample size is greater than 8 lb, then only the opposite quarters should be retained for the sample. Retained sections for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

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Longitudinal cuts / Opposite quarters

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity. If samples are cut, and the cut samples cannot be placed in a freezer within one hour, then it may be best to do the cutting at the facility where the freezers are located.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
C	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit
D	02	PPP	X (+1)	24 fruits / 4 lb.	Fruit

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 fruits / 4 lb.	Fruit
B	01	Untreated	NA	24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit
	02	PPP		24 fruits / 4 lb.	Fruit

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.



## Miscellaneous

## Artichoke

### **10. TEST SYSTEM/CROP:**

ARTICHOKE - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of artichokes including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect 12 artichoke flower heads per sample from at least 6 plants. Each sample should be collected during a separate run through the entire plot. Avoid sampling from row ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Perennial artichokes:** Take flower heads from different quarters of the bushes, from high and low areas and flower heads exposed and sheltered by foliage.

**Annual artichokes:** Take mature flower heads.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

**All artichoke flower heads, regardless of size, should be cut into quarters with a clean knife, and all quarters should be retained for the sample.**

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

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## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Flower heads
B	01	Untreated	NA	12 heads	Flower heads
C	02	PPP	X (+1)	12 heads	Flower heads
D	02	PPP	X (+1)	12 heads	Flower heads

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 heads	Flower heads
B	01	Untreated	NA	12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads
	02	PPP		12 heads	Flower heads

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## Coffee

### **10. TEST SYSTEM/CROP:**

COFFEE - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### **12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of coffee including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

### **17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect “cherry” coffee beans in a manner that simulates commercial practices. Each sample should be collected during a separate run through the entire plot.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Immediately process the “cherries” into green beans following prominent local procedures. Completely describe the processing in the Field Data Book and provide the SOP used. Each sample should produce green beans that weigh a minimum of 2 lb (but preferably not more than 3 lb). Green beans collected for a sample may be placed in a resealable plastic bag prior to placement in a plastic-lined cloth bag.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**Samples for processing (Trial HIXX or PRXX only):** Collect one additional untreated cherry sample and one additional treated cherry sample, sufficient to yield a weight of approximately 15 lb green beans each, for processing into roasted beans and freeze-dried coffee. These samples should be collected and processed into green beans (minimum sample weight of 15 lb) as described above prior to transport to the processing facility.

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. After residue sample collection, store

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samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:**

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Green Bean
B	01	Untreated	NA	2 lb.	Green Bean
C	02	PPP	X (+1)	2 lb.	Green Bean
D	02	PPP	X (+1)	2 lb.	Green Bean

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	2 lb.	Green Bean
B	01	Untreated	NA	2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean
	02	PPP		2 lb.	Green Bean

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial HIXX or PRXX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
PA	01	Untreated	NA	15 lb.	Green Bean
PT	02	PPP	X (+1)	15 lb.	Green Bean

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, processing into green beans, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to

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permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions.

**For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

***Coughlin processing:*** After sample collection in the field, and processing to green bean, if samples PA and PT are not transported to the processing facility on the day of collection, they should be stored in a freezer at temperatures generally less than -18°C (allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc.) until transported to the facility for processing to roasted bean and freeze-dried coffee sample fractions. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.**

Send samples for processing to: Julie Coughlin, University of Hawaii at Manoa, Dept. of Plant and Env. Protection Sciences, 3190 Maile Way, St. John Rm 307, Honolulu, HI 96822, PHONE#: 808-956-2003, Cell: 808-542-3933, e-mail: jcoughli@hawaii.edu

***Other processor:*** After sample collection in the field, if samples PA and PT are not shipped to the processing facility on the day of collection, they should be stored in a refrigerator at about 4 °C or in a freezer until they are shipped. Refrigerator or freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples. Send samples for processing to: @@@**

## **19.3 PROCESSING:**

Immediately prior to processing, remove representative "grab" samples of untreated (one sample) and treated (two samples) green beans from the larger samples (approximately 2 lb per "grab" sample). Using simulated commercial practices (provide detailed description of equipment and procedures) roast the remaining green beans. Collect one sample of roasted beans from both untreated and treated samples. Roasted bean samples should weigh approximately 2-4 lb each. From the remaining roasted beans, process a sufficient volume to produce freeze-dried coffee samples (one untreated and one treated) that weigh approximately 1-2 lb each.

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**Puerto Rico processing trials only:** Use “wet processing” on the samples from trials PRXX and PRYY, and “dry processing” on the samples from the other field trials. Completely describe the processing in the Field Data Book and provide the SOP used.

Place samples in appropriate containers and label. **Processed samples may be divided into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen processed samples at temperatures generally less than –18 °C until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. Shipment of frozen samples will be by “express” shipment. Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

#### **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WEIGHT RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2 lb.	Green Bean
GC	02	PPP	X (+1)	2 lb.	Green Bean
GD	02	PPP	X (+1)	2 lb.	Green Bean
RBA	01	Untreated	NA	2-4 lb.	Roasted Bean
RBT	02	PPP	X (+1)	2-4 lb.	Roasted Bean
FDA	01	Untreated	NA	1-2 lb.	Freeze-Dried Coffee
FDT	02	PPP	X (+1)	1-2 lb.	Freeze-Dried Coffee

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial.** At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)

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- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date coffee green bean samples received
- 20.1.04- Storage temperatures until coffee green bean samples are processed into roasted and freeze-dried coffee
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into roasted coffee and freeze-dried coffee
- 20.1.07- Storage temperatures of coffee green bean, roasted coffee, and freeze-dried coffee samples until shipped
- 20.1.08- Date coffee green bean, roasted coffee, and freeze-dried coffee samples are shipped to analytical laboratory

**Coughlin processing:** A processing summary report should be prepared and submitted to the sponsor representative via the Western Region office. When the processing summary report is completed the report and all original raw data will be sent to to the Regional Regional Field Coordinator, and subsequently forwarded to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All

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original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A “true copy” of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

***Other processor:*** A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a “true copy” will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A “true copy” of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Hops

### 10. TEST SYSTEM/CROP:

HOPS - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect sufficient fresh cones from at least 5 separate hills to yield a minimum of 1 lb (but preferably not more than 2 lb) of dry cones. Take cones from both sides of the hills, high and low areas, cones exposed and sheltered by foliage. Each sample (for the sampling event) may be collected from the same set of cut/downed vines as long as they are collected during a separate run through the entire set of cut /downed vines.

Alternatively, if the hop cones are mechanically harvested, then two duplicate samples should be collected in an impartial manner from the harvested cones from a representative collection of vines from each plot (as appropriate). Avoid sampling from the plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Dry the samples at 140 °F ( $\pm 12$  °F) to a commercially acceptable moisture level of approximately 8-12%. Document drying methodology, conditions, and times in the Field Data Book. If hops are warm coming out of the dryers, then mix and allow them to equilibrate to ambient air temperature before bagging and placing in the freezer (generally not more than 1 hour). If the dryer cools samples to room temperature in the drying cycle, then additional equilibration is not necessary or required.

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	1 lb.	Dry Cones

B	01	Untreated	NA	1 lb.	Dry Cones
C	02	PPP	X (+1)	1 lb.	Dry Cones
D	02	PPP	X (+1)	1 lb.	Dry Cones

## 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	1 lb.	Dry Cones
B	01	Untreated	NA	1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones
	02	PPP		1 lb.	Dry Cones

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

## 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

After harvest and residue sample collection, samples should be placed into a freezer, if not shipped immediately after sampling. Shipments sent via overnight carriers (e.g., Federal Express, Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage; all on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Shipment of frozen samples will be by freezer truck or express shipment. Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck. Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. Send samples to: @@@**

## 20. FIELD DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

20.01- Names of all personnel conducting specific research functions

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- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.
- 20.17- Data collected during sample drying, including a description of the drying method and the length of drying time

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## Peanut

### 17. RESIDUE SAMPLE COLLECTION:

#### **Nutmeat Samples:**

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest peanut plants. If the peanut seeds (nutmeats) are commercially mature, then they may be collected on the day of harvest from at least 24 plants. Otherwise they may be left to dry in the field until they mature, or they may be dried in a protected area or in a forced air dryer. Each sample should be collected during a separate run through the harvested plants from the entire plot. Collect enough peanuts to yield a minimum of 2 lb (but preferably not more than 4 lb) nutmeat per sample. **Note that harvest is when the peanut plants are uprooted. Sampling date is when nutmeats are placed in sample bags.**

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Shell peanuts and remove the hulls, retaining the nutmeat for the sample.

#### **Hay Samples:**

**All trials:** Peanut hay consists of the dried vines and leaves left after the harvesting of peanuts from vines that have been air-dried to a moisture level of 10 to 20%. (Percent moisture level may be estimated.) The hay samples should be cut (harvested) on the same day as peanut samples are collected and placed into sample bags (sampled) as soon as possible after a sufficient period of drying. If rainy weather is expected, the hay samples may be removed to a sheltered area for drying. (Do not use forced hot air to accelerate drying.) Document all drying procedures, whether or not the samples have been moved to a sheltered area. Determine (or estimate) and report moisture content of the hay samples. Hay samples should weigh a minimum of 3 lb for the untreated sample and 2 lb for the treated sample. Two samples per plot are required. **Note that harvest date is when the peanut hay is cut. Sampling date is when peanut hay is placed into sample bags.**

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18.1) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**Samples for Processing (Trial XX only): (University of Idaho processing)** In addition to the samples escribed above, collect one untreated and one treated nutmeat sample weighing approximately 100-120 lb each for processing into meal and refined oil. These samples may be placed in plastic-lined cloth bags (as described above) or in some other secure, clean containers. Identify each sample container with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and

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harvest/sampling dates. See Section 19.2 for shipping instructions.

**Samples for Processing (Trial XX only): (GLP Technologies processing)** In addition to the samples described above, collect one untreated and one treated nutmeat sample weighing approximately 60-70 lb each for processing into meal and refined oil. These samples may be placed in plastic-lined cloth bags (as described above) or in some other secure, clean containers. Identify each sample container with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. See Section 19.2 for shipping instructions.

## 18. FIELD RESIDUE SAMPLE INVENTORY:

### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
NA	01	Untreated	NA	2 lb.	Nutmeat
NB	01	Untreated	NA	2 lb.	Nutmeat
NC	02	PPP	X (+1)	2 lb.	Nutmeat
ND	02	PPP	X (+1)	2 lb.	Nutmeat
HA	01	Untreated	NA	3 lb.	Hay
HB	01	Untreated	NA	3 lb.	Hay
HC	02	PPP	X (+1)	2 lb.	Hay
HD	02	PPP	X (+1)	2 lb.	Hay

### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
NA	01	Untreated	NA	2 lb.	Nutmeat
NB	01	Untreated	NA	2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
	02	PPP		2 lb.	Nutmeat
HA	01	Untreated	NA	3 lb.	Hay
HB	01	Untreated	NA	3 lb.	Hay
	02	PPP		2 lb.	Hay
	02	PPP		2 lb.	Hay

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples NC, ND, HC, and HD.

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial @@ only (Univ. of Idaho processing)

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	100 - 120 lb.	Nutmeat
PT	02	PPP	X (+1)	100 - 120 lb.	Nutmeat

### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial @@ only (GLP Technologies processing)

SAMPLE	TRT#	TREATMENT	DAYS AFTER	APPROX. WEIGHT	CROP FRACTION
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ID			LAST APPLIC.	RANGE OF SAMPLE	
PA	01	Untreated	NA	60-70 lb.	Nutmeat
PT	02	PPP	X (+1)	60-70 lb.	Nutmeat

## **19. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

### **19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)**

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

### **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

Ship samples for processing immediately to the processing laboratory via overnight "express shipment" such as Federal Express or Airborne. **Ship samples unfrozen.** Contact the designated person (noted below from the processing laboratory prior to shipment of samples for specific instructions. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

### **19.3 PROCESSING:**

As soon as possible after receiving the nutmeats, the processing laboratory should remove approximately 2-4 lb each of treated and untreated nutmeats and store them frozen while the remainder of the nutmeats is processed.

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Process the remaining nutmeat as done commercially into meal and refined oil. Start with the untreated sample first. Do each sample separately.

Place samples in appropriate containers and label. **Divide each sample of oil into separate containers of 150-300 ml. It is also acceptable to divide meal samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Follow proper handling practices with clean hands and tools to prevent transfer of pesticide residue from one sample to another. Immediately freeze the samples and hold at generally less than 0 °F (-18 °C) until shipment to the analytical lab.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or "express" shipment. Shipments sent via "express shipment" (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags.** Send samples to: @@@

#### **19.4 PROCESSED SAMPLE INVENTORY:**

<b>SAMPLE ID</b>	<b>TRT#</b>	<b>TREATMENT</b>	<b>DAYS AFTER LAST APPLIC.</b>	<b>APPROX. WGT./VOL. RANGE OF SAMPLE</b>	<b>CROP FRACTION</b>
GA	01	Untreated	NA	2 – 4 lb	Nutmeats
GT	02	PPP	X (+1)	2 – 4 lb.	Nutmeats
MA	01	Untreated	NA	3 – 5 lb.	Meal
MT	02	PPP	X (+1)	3 – 5 lb.	Meal
OA	01	Untreated	NA	1000-2000 ml	Refined Oil
OT	02	PPP	X (+1)	1000-2000 ml	Refined Oil

#### **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial.** At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment

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20.08- Treatment application data

20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)

20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)

20.11- Residue sample shipping information

20.12- Description of crop destruction, or explanation for lack of destruction

20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.

20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots

20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

20.1.01- Names of all personnel conducting specific research functions

20.1.02- Deviations from protocol and standard operating procedures

20.1.03- Date nutmeat samples received

20.1.04- Storage temperatures until nutmeat samples are processed into meal and refined oil

20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing of samples into meal and refined oil

20.1.07- Storage temperatures of nutmeat, meal and refined oil samples until shipped

20.1.08- Date nutmeat, meal and refined oil samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

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## Sugarcane

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest stalks (cane) in a manner simulating commercial practices from at least 12 areas of the plot. Stalks should be cut at ground level, topped at six inches below the leaf whirl, and the leaves should be removed. Select 12 stalks and separate them into 3 groups. Divide each stalk into 3 approximately equal lengths. Take top portions from one group, middle portions from the second group, and bottom portions from the third group, to ensure that parts of all 12 stalks are included in each sample.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s). Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

**Processing samples (Field trial XX only):** At X ( $\pm 1$ ) days after the last application, collect one additional untreated sample and one additional treated sample of approximately 600-700 lb each. Harvest and sample stalks from at least 12 separate areas of each plot. Begin with the untreated plot first, and then sample the treated plot.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample item to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags (as described above) or other containers which will maintain the integrity of the sample. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample container with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (See Section 18.3) and harvest/sampling dates. If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in an environment with controlled temperatures, generally  $<25^{\circ}\text{C}$  (refrigeration at approximately  $4^{\circ}\text{C}$  is preferred but not required), until they are shipped.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 stalks	Cane
B	01	Untreated	NA	12 stalks	Cane

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C	02	PPP	X (+1)	12 stalks	Cane
D	02	PPP	X (+1)	12 stalks	Cane

#### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 stalks	Cane
B	01	Untreated	NA	12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane
	02	PPP		12 stalks	Cane

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples C and D.

#### 18.3 PROCESSING RESIDUE SAMPLE INVENTORY: Trial XX only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROXIMATE WEIGHT RANGE OF SAMPLE	CROP FRACTION
PA	01	Untreated	NA	600-700 lb.	Cane
PT	02	PPP	X ( $\pm 1$ )	600-700 lb.	Cane

### 19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

See below for instructions for handling residue samples (not for processing) that are to be sent directly to an analytical laboratory and instructions for handling processing samples that are to be sent to a processing facility.

#### 19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT: (Samples not for processing)

The methods used in harvest, sample handling, and storage will be outlined generally in SOP's, and described fully in raw data.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Shipment of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). If field trial personnel transport the samples to the analytical laboratory directly from the plots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the

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samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, telephone log, Field Data Book communication note, etc.

**Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory. Send samples to: @@@**

## **19.2 PROCESSING SAMPLE HANDLING AND SHIPMENT:**

If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in an environment with controlled temperatures, generally <25 °C (refrigeration at approximately 4°C is preferred but not required), until they are shipped. **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: @@@

## **19.3 PROCESSING:**

Immediately prior to processing sugarcane, remove representative “grab” samples of untreated and treated canes from the larger samples (approximately 2-4 lb. for each sample). Place the “grab” samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Using simulated commercial practices (provide detailed description of equipment and procedures) produce refined sugar and blackstrap molasses from samples PA and PT. Collect one sample each of refined sugar and blackstrap molasses from each cane sample. Refined sugar samples should weigh approximately 2-4 lb each. Molasses samples should have a volume of approximately 1000-2000 ml each.

Place samples in appropriate containers and label. **Divide each sample of molasses into separate containers of 50-150 grams. It is also acceptable to divide refined sugar samples into multiple containers. Each portion of the divided sample should be representative of the whole sample.** Identify each sample with correct Processing ID number, Test Substance (common chemical name), complete sample ID (see table in this section), and processing date(s).

Maintain all frozen processed samples at temperatures generally less than -18 °C until shipped, allowing for normal variations of less than 24 hours’ duration due to freezer cycling, sample movement, etc. Freezer logs will be used to document all sample additions to and removals from storage. All storage temperatures are to be monitored and documented.

**For express shipments (overnight carriers such as Federal Express or Airborne),** contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **For shipments via freezer truck (ACDS), it is acceptable to contact the laboratory prior to shipment, on the day of shipment, or on the day after the samples have been loaded on the truck.** Document the notification made to the sample destination by use of e-mail, telephone log, field data book communication note, etc. Shipment of frozen samples will be by freezer truck or “express” shipment. Shipments sent via “express shipment” (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory (see IR-4 Advisory 2007-01 for more information). For analysis, send samples to: @@@

## **19.4 PROCESSED RESIDUE SAMPLE INVENTORY:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	APPROX. WGT./VOL. RANGE OF SAMPLE	CROP FRACTION
GA	01	Untreated	NA	2-4 lb.	Stalks

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GT	02	PPP	X (+1)	2-4 lb.	Stalks
RSA	01	Untreated	NA	2-4 lb.	Refined sugar
RST	02	PPP	X (+1)	2-4 lb.	Refined sugar
BMA	01	Untreated	NA	1000-2000 ml	Blackstrap molasses
BMT	02	PPP	X (+1)	1000-2000 ml	Blackstrap molasses

## **20. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be **recorded directly and promptly** into the IR-4 Field Data Book or equivalent raw data notebook.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

- 20.01- Names of all personnel conducting specific research functions
- 20.02- Amendments and deviations from protocol and standard operating procedures
- 20.03- Test site information
- 20.04- Plot maps
- 20.05 - Test substance receipt, use and container/substance disposition records
- 20.06- Test substance storage conditions (including temperatures)
- 20.07- Data regarding calibration and use of application equipment
- 20.08- Treatment application data
- 20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)
- 20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)
- 20.11- Residue sample shipping information
- 20.12- Description of crop destruction, or explanation for lack of destruction
- 20.13- Daily Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from the date of planting or transplanting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. These records do not need to be determined under GLP standards. If the protocol requires that transplants are treated with the test substance prior to transplanting, then weather records are required from the date of seeding. If transplants are used for an IR-4 trial but no test substance applications are made prior to the transplanting, then temperature/humidity records are NOT required for the period prior to transplanting.
- 20.14- Pass times (if applicable) and other data to confirm amount of material applied to plots
- 20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
- 20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

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## **20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:**

At a minimum, collect and maintain the following raw data:

- 20.1.01- Names of all personnel conducting specific research functions
- 20.1.02- Deviations from protocol and standard operating procedures
- 20.1.03- Date cane samples received
- 20.1.04- Storage temperatures until samples are processed into refined sugar and blackstrap molasses
- 20.1.05- Processing Methodology (SOPs are acceptable)
- 20.1.06- Data collected and observations made during processing of samples into refined sugar and blackstrap molasses
- 20.1.07- Storage temperatures of cane, refined sugar, and blackstrap molasses samples until shipped
- 20.1.08- Date cane, refined sugar, and blackstrap molasses samples are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Raleigh, NC (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Raleigh, NC. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

## Taro

### 17. RESIDUE SAMPLE COLLECTION:

#### Foliage Samples:

**All trials except decline trial:** Collect two samples from each foliage subplot. Each sample should be representative of the entire subplot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect foliage from at least 12 plants. Each sample should be collected during a separate run through the entire plot. Each foliage sample should weigh a minimum of 4 lb (but preferably not more than 6 lb). Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE FOLIAGE.

#### Corm Samples:

**All trials except decline trial:** Collect two samples from each corm subplot. Each sample should be representative of the entire subplot (except plot ends). At X ( $\pm 1$ ) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect corms from at least 12 plants. Corm samples should weigh a minimum of 4 lb (but preferably not more than 6 lb). Avoid sampling from plot ends.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Wash the corms following local commercial practices. Document the technique used to wash the samples. If necessary, reduce the sample weight by cutting the corms longitudinally into quarters with a clean, uncontaminated knife on an uncontaminated surface. Retain at two opposite quarters of each corm. Process untreated sample first. Record the length of time from completion of the sample reduction to placement in a cooler for each sample in Field Data Book Part 7.A.2.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Trials except Decline Trial XX@@:

SAMPLE	TRT#	TREATMENT	DAYS AFTER	MINIMUM	CROP
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ID			LAST APPLIC.	SAMPLE SIZE	FRACTION
FA	01	Untreated	NA	12 plants / 4 lb.	Foliage
FB	01	Untreated	NA	12 plants / 4 lb.	Foliage
FC	02	PPP	X (+1)	12 plants / 4 lb.	Foliage
FD	02	PPP	X (+1)	12 plants / 4 lb.	Foliage
CA	01	Untreated	NA	12 plants / 4 lb.	Corm
CB	01	Untreated	NA	12 plants / 4 lb.	Corm
CC	02	PPP	X (+1)	12 plants / 4 lb.	Corm
CD	02	PPP	X (+1)	12 plants / 4 lb.	Corm

#### 18.2 Decline Trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
FA	01	Untreated	NA	12 plants / 4 lb.	Foliage
FB	01	Untreated	NA	12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
	02	PPP		12 plants / 4 lb.	Foliage
CA	01	Untreated	NA	12 plants / 4 lb.	Corm
CB	01	Untreated	NA	12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm
	02	PPP		12 plants / 4 lb.	Corm

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples FC, FD, CC, and CD.



**10. TEST SYSTEM/CROP:**

TI - Use a commercial variety. Report: variety, age of plants, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; see Section 23 **for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

**12. TEST SITE PREPARATION:**

Select a test site that has been maintained following good local agricultural practices for the production of ti including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and fertilizer history of a minimum of 1 year and preferably 3 years.

**17. RESIDUE SAMPLE COLLECTION:**

**All trials except decline trial:** Collect two samples from each plot. Gather each sample in a manner to assure a representative, impartial sample of the entire plot (except plot ends). Each sample should be collected during a separate run through the entire plot.

**Leaf harvest:**  $X(\pm 1)$  days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest one marketable leaf from at least 12 plants. Impartially select a range of leaves that would be typical of leaves harvested for use as food wrappers or for flavoring during cooking.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**Root harvest:**  $X(\pm 1)$  days after the last application to the subplot used for root sampling, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, harvest one marketable root from at least 12 plants. Impartially select a range of roots. Roots may be cleaned as per commercial practice. Document any cleaning of the root in the field data book.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

**All trials, for all samples:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using **IR-4 plastic lined cloth residue sample bags**, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

**18. FIELD RESIDUE SAMPLE INVENTORY:**

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**18.1 All Trials except Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 plants	Leaves
LB	01	Untreated	NA	12 plants	Leaves
LC	02	PPP	X(+1)	12 plants	Leaves
LD	02	PPP	X(+1)	12 plants	Leaves
RA	01	Untreated	NA	12 plants	Roots
RB	01	Untreated	NA	12 plants	Roots
RC	02	PPP	X(+1)	12 plants	Roots
RD	02	PPP	X(+1)	12 plants	Roots

**18.2 Decline Trial XX@@:**

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
LA	01	Untreated	NA	12 plants	Leaves
LB	01	Untreated	NA	12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
	02	PPP		12 plants	Leaves
RA	01	Untreated	NA	12 plants	Roots
RB	01	Untreated	NA	12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots
	02	PPP		12 plants	Roots

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples LC, LD, RC, and RD.

## Wasabi

### 17. RESIDUE SAMPLE COLLECTION:

**All trials except decline trial:** Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). At X (+1) days after the last application, starting with the untreated plot or utilizing separate personnel for the treated and untreated plots, collect at least 12 stems. Each sample should be collected during a separate run through the entire plot. Stem samples should weigh a minimum of 2 lb and top (leaf and petiole) samples should weigh a minimum of 1 lb. Avoid sampling from plot ends.

In any trial in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance on the day of the last application.

Remove tops and roots. Remove tops (leaves with petioles) and package the tops separately from stems. If excessive soil adheres to the stems, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method).

If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the stem briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE STEMS.

**Decline trial XX@@ only:** Insert instructions here or delete if there is no decline trial.

**All trials:** Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated plot(s) before proceeding to the treated plot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Regional Field Coordinator (Section 23). Identify each sample bag\*\* with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

\*\*When using IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:

**Field ID Number; Crop Fraction; Test Substance** (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

### 18. FIELD RESIDUE SAMPLE INVENTORY:

#### 18.1 All Field trials except decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
SA	01	Untreated	NA	12 Stems / 2 lb.	Stems
SB	01	Untreated	NA	12 Stems / 2 lb.	Stems
SC	02	PPP	X (+1)	12 Stems / 2 lb.	Stems
SD	02	PPP	X (+1)	12 Stems / 2 lb.	Stems
TA	01	Untreated	NA	12 Plants / 1 lb.	Tops
TB	01	Untreated	NA	12 Plants / 1 lb.	Tops
TC	02	PPP	X (+1)	12 Plants / 1 lb.	Tops
TD	02	PPP	X (+1)	12 Plants / 1 lb.	Tops

#### 18.2 Decline trial XX@@:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
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SA	01	Untreated	NA	12 Stems / 2 lb.	Stems
SB	01	Untreated	NA	12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
	02	PPP		12 Stems / 2 lb.	Stems
TA	01	Untreated	NA	12 Plants / 1 lb.	Tops
TB	01	Untreated	NA	12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops
	02	PPP		12 Plants / 1 lb.	Tops

\*Sample IDs are out of sequence in order to maintain consistency among trials for Samples SC, SD, TC, and TD.