2020 Standard Operating Procedures

For

Magnitude of Residue Field Studies

IR-4 Program

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2020 Standard Operating Procedure
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10-1.0
Standard Operating Procedures

PURPOSE: To provide guidelines for creating, revising, and maintaining Standard Operating Procedures.

PROCEDURES:

1. All Standard Operating Procedures (SOPs) will be reviewed annually by the Field Research Director and revised as needed. The Western Regional Field Coordinator will annually approve the SOPs and will sign and date the SOP index page upon approval. The index should also have the name and signature of the Field Research Director. The Western Regional Field Coordinator need only sign the title page which will indicate the effective date of all the SOPs, since SOPs are treated as a package. The signed SOPs will be kept in the WSU-Mount Vernon IR-4 files.
2. All SOPs will be uniquely identified by number. Each number will begin with the SOP category, sequential SOP number and version number. An example of this is 20-2.1, where the first number (”20”) is the general category (e.g. data); the second number (“2”) indicates the SOP number within that category (e.g. 20-2 is recording raw data); the third number (“.1”) is the revision number.
3. These SOPs are designed to be general guidelines for procedures and instructions to follow if procedures have become a standard set of actions. This cannot be all-inclusive. These SOPs are intended to describe procedures to perform tasks related to the conduct of a Good Laboratory Practice (GLP) study. Actions specific to unique activities noted in the study protocol will be explained and documented in the Field Data Notebook (FDN).

10-2.0
Responsibilities of the Field Research Director (FRD)

PURPOSE: To provide information on the responsibilities of the Field Research Director

PROCEDURES:

1. The Field Research Director has the responsibility for the following:
   a. Review draft protocols and inform the Study Director of any potential issues.
   b. Read and understand the signed protocol prior to initiation of a GLP trial.
   c. Assure that the study is carried out according to an approved protocol.
   d. Assure that personnel, resources, facilities, equipment, materials, and methods are available as scheduled for the conduct of the project.
   e. Maintain all equipment, instruments, and gauges according to GLPs as outlined in these SOPs.
   f. Make sure that all personnel conducting the study understand the protocol and SOPs for the project.

g. All deviations from the protocol or SOPs are to be promptly reported to the Study Director.
h. All findings reported by the Quality Assurance Unit (QAU) are responded to in writing.
i. Create electronic copy of the following:
    i. Completed Field Data Notebooks for all studies prior to shipping the books to Western Region.
    ii. All original raw data, supporting data, summaries and other items connected with the study.
j. Maintain on file a current record of training (including webinars), education and experience, and a job description for all personnel engaged in the study. These records will be reviewed and updated as necessary (generally annually) before the initiation of the studies for the current year.

10-3.0
Field Research Personnel

PURPOSE: To provide information concerning requirements for all trial personnel and related records under GLPs.

PROCEDURES:

1. WSU-Mount Vernon will have on file a current summary of the training and experience and a brief description of duties or responsibilities for each person engaged in or supervising the study. This training file will be maintained in the WSU-Mount Vernon IR-4 records, Building #4032.
2. The Field Research Director will determine that the person or persons conducting the study are of sufficient number to carry out the study to its completion and are sufficiently trained to conduct their portion of the study.
3. WSU-Mount Vernon will have a supply of safety equipment in reasonable working order and sufficiently clean to protect the health and safety of the personnel connected with the project as required by the Health and Safety SOPs, regulations, other institution regulations, pesticide labels or the study protocol.
4. When the application of restricted use pesticides is required in the study, the applicator must be certified or under the direct supervision of a certified applicator.
5. Personnel handling pesticides should be trained in accordance with the current policies and guidelines.
6. GLP training or related technical training for full-time IR-4 field personnel will take place at a minimum of once every three years. Training can be from a private GLP trainer or from any GLP training session organized or offered by IR-4. GLP webinar trainings offered by the Western Region will be attended, if possible. Informal or formal training related to field and/or greenhouse research (non-GLP) may be included with a CV, but generally is not included on the GLP Training Summary.

10-4.0
FRD Responsibilities Relating to Quality Assurance and Quality Control Audits

PURPOSE: To ensure that data generated during the study meets the requirement of EPA's Good Laboratory Practices (GLPs).

PROCEDURES:

1. All QA procedures/activities will be conducted according to, and in compliance with, SOPs of the Quality Assurance Unit (QAU).
    a. QAU picks certain trials each year for a critical phase inspection. The FRD will respond through the eQA system.
    b. After the FDN is sent to the Western Region, the WR Field Research Coordinator or a designee performs a quality control review of the notebook. The FRD will respond to the QC audit through e-mail to make any necessary changes.
    c. After QC the FDN goes to QA. The FRD will respond to any findings through the eQA system, making sure to use the most current version of the Field Data Notebook (available on the Western Region website).
2. Responses to Facility Inspections findings will also be made through the eQA system.

20-1.0
Archives and Retention Files

PURPOSE: To ensure that all original raw data generated under GLP's are safely maintained at the IR-4 Headquarters Archive, and at WSU-Mount Vernon retention file.

PROCEDURE:

1. The official archive for original raw data will be located at IR-4 Headquarters. Original raw data for the current season will be kept in a locked file cabinet in the WSU – Mount Vernon IR-4 office in building #4032. True copies of original, raw data will be made and retained at the WSU – Mount Vernon facility.
2. The procedure for transferring raw data to the archive at IR-4 Headquarters will be as follows:

    At the end of the current field season, after the last study has been completed, original raw data that have been generated for all current studies will be compiled in preparation for shipping to IR-4 Headquarters for archiving. These original items, such as annual standardization of temperature monitors for test substance storage and freezers, and standardization of instruments that record environmental data, will be included, as well as any other supporting original raw data or documentation that apply to the studies. These originals will then be sent to IR-4 Headquarters for archiving and a copy of the originals inventory list will be provided to the IR-4 Western Region office. A chain-of-custody

form will be generated and include an inventory of all documents being sent to headquarters. The chain-of-custody form will include the name of the courier and the tracking number. The transfer should be by certifiable mail or some other traceable mechanism (e.g. FedEx). Also, a scanned copy of the completed chain-of-custody form is kept in the WSU – Mount Vernon retention file and is also sent to the IR-4 Western Region Field Office. HQ retains the original, signed chain- of-custody form.

3. Retention Files of facility records and documents will be maintained at WSU – Mount Vernon (located in a "FireKing", or similar file cabinet, with a one-hour fire rating). Access to all retention files is restricted to personnel involved in IR-4 studies. The WSU -Mount Vernon facility retention files may contain the following:
   a. True copies of original raw data; true copies of signed and dated SOPs; copies of supporting data associated with the facility such as work orders for freezers and balance maintenance; personnel and GLP training records; and QA facility inspection reports.
   b. All retention files will be maintained as digital copies on a backed up remote access server.
4. Copies of older field data notebooks may be discarded if no longer needed. This will be determined by reviewing the project status, as listed in "Test Substance Container Disposal Approval" database on the IR-4 Headquarters website, for individual field researcher directors. Field Data Notebook retention copies can be discarded if the project status indicates: Use Registered; Tolerance Established; Request Withdrawn; Mfg. Will Not Support; or Use Cancelled.

20-2.0
Recording of Raw Data

PURPOSE: To ensure that raw data collected and recorded is accurate and available.

PROCEDURES:

1. It is the responsibility of the Field Research Director to see that all data and other items connected with the study are stored in a clean, secure place and are accessible by authorized study personnel.
2. During the active season, raw data may be located in the following areas:
   a. Freezer activity logs are in each individual FDN.
   b. Test Substance activity logs, indicating amount removed with date and user initials will be in each individual FDN. For multiple trials using the same test substance container, a true copy of the use log will be placed in the appropriate notebooks with the original in one of the FDNs.
   c. Equipment maintenance logs will be maintained and kept separate from Databook page that are used to record any repairs on equipment relevant to trial (Part 6M).
   d. All supporting data forms (e.g. field maintenance activity, etc.) will be located in the desk of the IR-4 the Field Research Director.

   e. The field data notebooks will remain in the custody of the field personnel responsible for the study

3. All raw data will be recorded in dark ink. Blue ink is preferred as it distinguishes the original from true copies.
4. If forms are generated to replace or supplement the forms in the IR-4 Field Data Notebook (e.g. freezer logs, chemical storage log), the forms must contain all information on the original form and, once completed, will be included in the field data notebook. Before use, modified FDN pages must first be approved by the Western Region Regional Field Coordinator or designee.
5. Corrections will be made by crossing through the item, initialing, dating and giving reason or code for the correction. Blank spaces in the field data notebook will be lined out; if there is no reason given for the line-out, it means the space was not needed.
6. Initial and date each activity as it occurs.
7. Electronic data, computerized summaries, etc., can be used in individual field data notebooks.
8. All notebooks, data sheets, summaries, etc. should be clearly marked with the name of the project, field ID number, date and initials of the person responsible for the data, and other information that may be needed to understand the data and its source to aid in reconstructing of activities associated with the GLP study
9. Make sure that all data required in the study protocol is collected, recorded and retained. Carefully review the protocol to ensure that all required data is collected.
10. Transcribed data in the field data notebook must indicate location of the original and be verified by someone other than the transcriber with date and initials.
11. Pagination and the application of Field ID# labels in the FDN are not considered raw data and do not require a date and initial; these components are considered data organization. Copies of original documents or data will be indicated as such with a stamp or written statement that reads: “This is a true copy of the original”; with the location of the original provided/noted.

20-3.0
Method for Collecting Additional Data

PURPOSE: To describe the procedure used for noting phytotoxicity and describing soil information data.

PROCEDURES:

1. Phytotoxicity Data
   Consult the protocol to determine the method and necessity of the phytotoxicity data (consult Study Director if evaluation methods are not included in protocol). If this data is optional, collection may or may not be done; however, in general, the crop should be checked periodically for evidence of phytotoxicity and crop vigor. Include the rating scale or other reference guide used to assess phytotoxicity and crop vigor.
2. Soils Data

Soils data can be found at a USDA Web Soil Survey internet site (websoilsurvey.sc.egov.usda.gov), and data downloaded and printed for inclusion in the FDN.

20-4.0
Significant Figures and Rounding

PURPOSE: To provide guidelines for determining significant figures in a measurement and to provide guidelines and ensure consistency in rounding numbers in a calculation.

PROCEDURE:

1. When reporting a measurement, record only to the degree of accuracy of the equipment or instrument or device used to make the measurement. For example, if a scale can weigh to a tenth of a gram, record 3.2 grams, not 3.20 grams. Remember, however, that conventional scientific notation allows that four tenths of a gram (.4) is commonly written as "0.4".
2. Knowing the correct number of significant figures in a measurement will help determine the proper method for rounding when making a calculation.

   Rules for Determining the Number of Significant Figures:

   Start counting from the leftmost, non-zero digit and count through the last digit, including zeros, to the right. However, zeros that serve as place holders (i.e. zeros to the left of the decimal point in numbers greater than one, or zeros to the right of the decimal point in numbers less than one) are not counted. Zeros between two non-zero digits are always counted.

Examples:	7.6	= 2 sig. fig.	2001	= 4 sig. fig.
	0.055	= 2 sig. fig.	200	= 1 sig. fig.
	2.00	= 3 sig. figs.	200.0	= 4 sig. fig.
	2,000	= 1 sig. fig.	2.0×10^2	= 2 sig. fig.
	0.406	= 3 sig. fig.	2.00×10^2	= 3 sig. fig.
	0.4060	= 4 sig. fig.	2.01×10^2	= 3 sig. fig.
	47,000	= 2 sig. fig.	0.001×10^2	= 1 sig. fig.

3. Conversion numbers, such as the number of square feet in an acre or the number of grams in a pound, are constants (they are not measurements made with an instrument to be used in a calculation). They do not affect the accuracy of a calculation nor do they determine the number of significant figures in the result of a calculation.
4. Rounding: Do not round any numbers within a calculation; round only the final result. When rounding, look at the digit following (i.e. to the right of) the digit that is to be the last value recorded:

   If it is less than 5, drop it and all the digits to the right of it. If it is greater than 5, increase by 1 the digit to be rounded.

   If it is 5, round in the direction that will result in an even number (i.e. if already even,

drop the 5; if odd, round up by one to make an even number).

Examples (rounding to 4 places):	64.534 = 64.53	64.575 = 64.58
	64.538 = 64.54	64.505 = 64.50
	64.585 = 64.58	64.495 = 64.50

5. In calculations involving significant figures, the answer is reported with significant figures that reflects the reliability of the least precise measured value or operation. Although both involve significant figures, the rounding rule for addition and subtraction is different than the rule for multiplication and division. (Do not round any numbers within a calculation; round only the final result.)

30-1.0

Site Selection and Plot Design

PURPOSE: To ensure plots will meet all protocol-specified requirements for a specific crop.

PROCEDURES:

1. Site selection will be made in accordance with the local horticultural practices acceptable for the commodity.
2. Locate site with enough isolation to minimize contamination from external sources such as commercial operations or other research studies.
3. The site will be large enough to accommodate the required number of replicates, buffer zones and treatments in accordance with an approved study protocol and for the commodity to be grown under simulated commercial conditions yielding samples of sufficient size and quality for analysis where required. The experimental design as specified by the protocol should be used. Individual plots should be large enough so that no more than 50% of the harvestable area will be needed to provide samples.
4. If the crop is established, select a site that has a uniform stand density.
5. Lay out each plot on the site using a suitable measuring device to accurately locate the plots on the site.
6. Prepare a plot map showing the location of each plot at the site. The plot map should include the dimensions and locations of treated and untreated plots; the plant and row spacing, the number of rows and/or beds and their direction; the amount and direction of slope, the North azimuth, and the dimensions and locations of buffer zones. Reference points are usually power poles, fence lines, irrigation hydrants, roads, etc. Distance from the permanent reference point(s) to at least two corners of each plot should be marked on the plot map to facilitate location of the plots.
7. Use stakes or flags to accurately indicate the plots on the site.
8. Identify each plot with an individual designation specific to that plot (e.g. untreated plot is TRT 01; treated plot is TRT 02). In addition, each stake, flag or card shall contain the Trial Identification Number and the name of the test substance.

30-2.0

Commodity Establishment and Maintenance

PURPOSE: To ensure that crops are grown under common and current agricultural practices and to provide a uniform crop for the study/trial.

PROCEDURES:

1. Follow the practices required to produce the commodity under standard commercial conditions.
2. Determine that soil and fertility of the trial site is adequate for the commodity.
3. When field trials are conducted in commercial production fields, accepted grower practices will be considered as standard procedures unless the protocol states differently. Communicate clearly to the cooperating commercial grower the scope of the trial and the data required from the plots; encourage him/her to call before conducting an activity in the plots that may affect the integrity of the GLP trial. When trials are conducted at WSU-MV, accepted commercial practices should be performed as necessary to bring the soil reasonably within the requirements of the commodity. If necessary, prepare the soil as is commonly defined as "accepted grower practices" in the region where the commodity is being grown.
4. Determine the correct species and variety to use as specified by the study protocol. If the variety is not specified, select a variety commonly used in the area by commercial producers. If a commercial producer is providing the plants, try to select plants as uniform in growth and color as possible.
5. Determine within and between row spacing and seeding depth as commonly used in the area by commercial producers for the commodity. Plant the seed or transplant in reasonably straight lines or rows with accurate measurements to assure the commodity is planted according to specifications.
6. Irrigate, fertilize, or perform other agricultural practices, as necessary, to get the commodity established and maintain growth and health of the crop throughout the life of the trial.
7. Before application of maintenance pesticides to the test crop, take into consideration any conflicts with the test substance to ensure that they will not interfere with residue analysis in the laboratory. If in doubt, contact the Study Directory identified in the protocol for approval.

40-1.0

Test Substance and Adjuvant Maintenance

PURPOSE: To ensure test substances and adjuvants are received and maintained according to GLP standards.

PROCEDURE:

1. When the GLP test substance is received fill out the appropriate FDB form, Part 4.A.

with all information given on the label and the MSDS. Check information given in MSDS and container label against protocol Test Substance Information #13, if any information does not match or if any is missing contact Study director and/or the chemical company.

2. The test substance container label must contain the chemical name, batch number, expiration date and storage conditions, if anything is missing, contact study director, obtain information and add to container label. The protocol number(s), date of receipt, and study personnel initials will be recorded on the container before it is placed in chemical storage.
3. Adjuvant Containers must be labeled with following information:
    - Date received
    - Identity and concentration of the adjuvant
    - Recommended storage conditions
    - Expiration date (if no expiration date is supplied by manufacture, assign an expiration date up to 4 years from date of receipt)
4. The test substance and adjuvants will be stored in the WSU-Mount Vernon IR-4 pesticide storage cabinet in the Chemical Storage Building # 4021 until it is needed for use in the study. The IR-4 pesticide storage cabinet will have limited access and must be separate from areas where the pesticide is mixed and where mixtures are stored. Dry materials will be weighed prior to use in Room #141, Building ARTB #4015. Liquid materials will normally be measured in the field immediately prior to use.
5. Test substances, adjuvants, and mixtures should be stored in a manner to prevent contamination, deterioration, or damage during the conduct of the study. The test substance label and/or Material Safety Data Sheet should be consulted.
6. Temperature monitoring of the storage facility will be conducted to verify that test substances have not been exposed to temperatures outside of approved ranges. An electronic temperature monitoring device (Onset HOBO Pendent) will be placed in the IR-4 storage cabinet at WSU-Mount Vernon. The following checks should be made at start-up and at approximately monthly intervals during the life of the field phase of each study (generally from March through July):
    a. Check the battery. Replace if the level is less than 25%.
    b. Set the devices to take a reading every hour.
    c. At the end of the data collection period, the data should be transferred to a storage system and immediately printed out with appropriate identification. This hard copy should be signed, dated, and stored in records kept at WSU-Mount Vernon.
    d. A max/min thermometer may also be used as a backup to the electronic device. If used, the thermometer should be reset after the data from the electronic device has been downloaded and printed.
7. Post a current inventory of all GLP pesticides and adjuvants in the storage unit in an inside location accessible and visible to study personnel. This inventory will include the pesticide common name, toxicity signal word, the number and kind of containers, and the date each was received.
8. The test substance activity log found in the FDN (Part 4B) will be maintained that contains test substance name, trial ID, date and removed, date and time replaced, and initials of person involved.
9. All unused GLP pesticides and empty containers should be returned to the storage facility at the completion of their use and retained until the study is completed and

disposal is approved by the study director.

10. When test substance is removed from storage facility for longer periods of time for use in the field precautions must be taken to insure test material is maintained at proper condition. Test material may be kept in a cooler with blue ice to insure conditions are within approved the range and a temperature recording device will be used to monitor temperatures.

40-2.0

Calibration and Use of balances

PURPOSE: To ensure test substance is weighed accurately.

PROCEDURE:

1. The Mettler AJl00 balance (Room 140, Building #4015) will be used to weigh dry pesticides to be used in GLP studies. The scale will be calibrated annually by an instrument calibration service. Shortly after the scale has been calibrated, it will be used to check the accuracy of the bracketing weights by weighing them on the scale and recording the actual and stated weight.
2. The balance calibration will be checked prior to each weighing by using standard weights which bracket the amount of test substance to be weighed. Record the actual and measured masses of the standard weights used to bracket. If the measured mass is not within ± 1 % of actual mass, recheck and if measured mass is still not within ± 1 % the balance must be re-calibrated.
3. Select a clean weighing boat or other container suitable to hold the desired amount of pesticide and tare it on the balance following the manufacturer's directions in the operating instructions posted by the balance.
4. Select and wear or use appropriate safety equipment while handling pesticide concentrate.
5. Weigh the concentrate in a tarred container. Return excess to original pesticide container if this procedure does not affect the integrity of the contents or dispose of the excess by using appropriate methods.
6. Transfer the weighed product to a sealable plastic bag (e.g., Whirl-pack bag) for transport to the study site. Label the bag to identify it with the Field ID#, test substance, weight of test substance, and plot number. Maintain a written record of the amount of the pesticide removed from the original container for each weighing for the study and maintain a master sheet (FDN Part 4B) of the pesticide removed for each container used.

40-3.0

Measuring a Liquid Formulation

PURPOSE: To ensure an accurate dosage in application of test substance.

PROCEDURES:

1. Liquid pesticides will normally be measured in the field to minimize the generation of pesticide rinsate. Plastic syringes will be the primary measuring device used for this purpose. Alternatively, graduated cylinders may be used, either in the field or in the lab. When using a cylinder, measure the liquid at the bottom of the meniscus.
2. In most cases, it is best to use plastic syringes/cylinders large enough to hold the volume of pesticide needed for the treatment and graduated in 1 ml increments for a volume over 10 mls. and .25 ml increments for smaller volumes. For larger amounts that require a larger measuring device that is in greater increments, measure to closest increment, do not estimate. If the volume is too large the total volume can be divided into smaller aliquots for separate measurement.
3. If the opening of the pesticide container is too restrictive to allow measurement of the pesticide from the original container without danger of spillage, then use a clean container with a pour lip as an intermediate and fill the syringe/cylinder from it.
4. Plastic syringes will be used for only one liquid formulation, then triple- rinsed and disposed of after use. Add the rinsate to the spray tank. If used, cylinders will not be disposed of after use, but they will be clean and used for only one liquid formulation. After use, cylinders will be triple-rinsed and the rinsate added to the spray tank. Cylinders will be thoroughly washed in hot soapy water after return from the field to ensure that they are clean.
5. If measured in the lab, the pesticide should be transferred from the cylinder into a clean jar with a spill-proof lid, and labeled as to amount, treatment, and plot numbers receiving treatment. Make sure that as much liquid as possible is transferred to the jar. When emptied into the spray tank the container should be triple-rinsed and rinsate added to the spray tank.
6. Maintain a written record of each volume of the GLP pesticide removed from the original container during the study and maintain a master sheet of the pesticide removed for each container used (FDN Part 4B).

40-4.0
Sprayer Calibration

PURPOSE: To determine the delivery rate of sprayer to ensure accurate application of the test substance according to the protocol.

PROCUDURES:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary.
2. Calibrate the boom using the following procedures:

a. Refer to the protocol for any specified application requirements.
b. Select the appropriate type of nozzle, which is based on the pesticide formulation, the application method, the operating speed, the pressure setting, and the delivery rate (gal/a).
c. All sprayers must have a pressure gauge. Select the proper operating pressure.
d. Place the sprayer on level ground. Adjust the boom height and nozzle spacing for the correct application pattern. Determine whether all nozzles are discharging uniformly by spraying water through them at a uniform pressure. Catch the discharge from each nozzle in a separate container over a timed interval (e.g., 30 seconds) beginning after the nozzles are discharging. If the discharge between nozzles varies widely, replace those nozzle tips that give a much larger or much smaller discharge. As a guide, the discharge of any given nozzle should be within ±5% of an average nozzle discharge. Repeat the procedure above until all nozzles are discharging relatively uniformly.
e. Use the following formula in a manner described above to determine nozzle flow rate in ml/min or gal/min (GPM):

$$\frac{\text{ml}}{\text{min}} = \frac{\text{(avg ml caught for each nozzle)}}{\text{(30 seconds of catch)}} \text{ x } \frac{\text{60 seconds}}{\text{1 min}}$$

$$\text{GPM} = \frac{\text{ml}}{\text{min}} \text{ x } \frac{\text{gal}}{\text{3785 ml}}$$

f. When using TeeJet nozzles, refer to the nozzle calibration charts to determine the appropriate speed. Or, the following formula can be used to determine the required speed:

$$\text{MPH} = \frac{\text{GPM x 5940}}{\text{GPA x nozzle spacing in inches}}$$

*for a band application, the nozzle spacing equals the band width.

1. Check the MPH of the tractor by measuring a distance of at least 25 ft. Operate the tractor or ATV over the measured distance at the calculated MPH from above and record the time it takes to travel the distance. Calculate the actual MPH from the following formula:

$$\text{MPH} = \frac{\text{feet traveled x 0.682}}{\text{time in seconds of travel}}$$

3. If the calculated delivery rate (GPA) is within 5% of the desired delivery rate (as specified in the protocol), then the sprayer is calibrated, and the same settings should

be used in actual application. If the delivery rate must be changed, alter one or all of the following: nozzles, pressure, or speed. Minor changes in delivery rate can be made with a slight pressure change or speed alteration; major delivery rate changes require selection of new nozzle sizes.

4. Applicators must carefully operate under the same conditions as during calibration. Besides checking the nozzles for their proper delivery rate, the spray pattern should also be examined for a complete and symmetrical arrangement. When using the pressurized tanks, shake them before starting to spray. The test substance must be applied uniformly to the entire test area.
5. When using a backpack sprayer most applications will be calibrated at a walking speed of 2 mph. Walking speed should be verified immediately prior to pesticide application at the trial site. To determine the correct walking speed, divide the distance to be sprayed (in feet) by the speed (2.93 feet per second = 2 mph). A metronome set at about 71 beats per minute may be a useful aid to maintain a constant walking speed.
   Example: To determine the time necessary to spray a 50-ft plot, divide 50 ft by 2.93 ft/sec = 17.06 seconds.

40-5.0
Granular Applicator Calibration

PURPOSE: To determine the delivery rate of granular applicator to ensure accurate application of the test substance according to the protocol.

PROCEDURE:

1. Determine that the spreader is in good working order and good mechanical condition. Make sure that the openings to release the granular material are not clogged and are free of debris.
2. Wear protective clothing as necessary and fill the spreader at least half full of the material to be applied. Attach a pan under the spreader to catch the material as it is released.
3. Measure an area of 0.01 acre (or 435.6 ft) in close proximity to the area to be treated. A simple method to calculate the distance is:

$$\frac{435.6\ \text{sq. ft}}{\text{width of application in ft}}$$

4. Determine the approximate setting of the openings and the approximate speed to operate the applicator to deliver the desired amount of product/a.

5. Operate the applicator over the measured distance and collect the output in the pan attached to the spreader.
6. Weigh the material from the pan and multiply by 100 to give the amount product applied per acre.
7. Repeat steps until the delivery rate is within 5% of the desired rate (as specified in the protocol).

   Example: The pesticide formulation is a 15% granular product to be applied at a rate of 10 lbs. a.i./A.

$$\frac{10\text{ lbs a.i.}}{\text{Acre}} \text{ x } \frac{1\text{ lb product}}{.15\text{lbs a.i.}} = 67\text{lbs product/acre}$$

   Width of applicator = 10ft

$$\frac{435.6\text{ sq. ft.}}{10\text{ ft. width}} = 44\text{ ft to travel}$$

   With the applicator set at the appropriate opening and operated at 4 mph over the 44 ft, assume you collect of 70 lbs. of product. This is within the ±5% limit (63.7 to 70.4 lbs./a), so the applicator is calibrated correctly.

40-6.0

Application of Test Substance

PURPOSE: To provide guidelines for test substance application methods that ensures study integrity and compliance with regulatory requirements.

PROCEDURE:

1. Application equipment should be inspected and calibrated as described under SOP's 40-4.0 and 40-5.0.
2. Make sure all settings of pressure, speed, granular flow, etc., are set according to specifications from the calibration as previously performed.
3. When multiple rates of the pesticide are to be used, apply the test material beginning with the lowest concentration and proceeding to the highest concentration.
4. Prior to entering each plot, make sure you are traveling at the correct speed before turning on the sprayer or releasing the granules. Time your entry into the plot to ensure that the boom/granular applicator are charged, and that pesticide is being applied correctly. Maintain the correct speed through the plot.
5. Turn off the sprayer or stop granular flow just after leaving the plot.
6. Calculations should be made to minimize the amount of spray material left in the spray tank. This residual material should be sprayed to a similar crop or disposed of according to current policies and guidelines of the research testing facility.

7. The application width should be adequate to deliver the test substance to the edge of each plot, or as stipulated in the protocol.

40-7.0

Cleanup of Application Equipment

PURPOSE: To ensure that rest substance application equipment is clean and ready for use.

PROCEDURE:

1. For granules:
    a. Remove any excess granules and return them to the original container (if this procedure does not affect the integrity of the contents) or dispose of the excess by using appropriate methods for handling hazardous wastes. Note the amount of granular material used for the study in the pesticide log for that chemical.
    b. In a suitable area away from aquatic areas or danger of aquatic contamination, hose down the granular applicator to remove pesticide dust from the inside and outside.
2. For spray applications:
    a. Excess spray material should be applied to an overplanting of the crop.
    b. Triple rinse spray tank, then fill with clean water and run through system until tank is empty. When possible, warm soapy water will be used for first rinse.
    c. Spray off exterior of spray equipment with clean water
3. For all applications:
    a. Place expendable protective clothing in a container for disposal.
    b. Clean non-disposable items following the manufacturer's instructions or with soap and water as appropriate.
    c. After the application equipment is dry, lubricate those parts requiring lubrication and return the equipment to storage.

40-8.0

Problems During Applications of Test Substance

PURPOSE: To outline the procedures required when something goes wrong during the application of the test substance.

PROCEDURE:

1. During application, the applicator should observe the process to make sure that the test substance is being evenly distributed to the commodity.

2. If something goes wrong during an application (i.e., a plugged nozzle or a broken hose), then the operator should take immediate action to correct the situation.
3. The affected portion of the plot should be carefully marked off and staked to indicate the area affected.
4. Appropriate individuals, including the Study Director and Regional Field Coordinator, should be notified of the incident to determine impact of the error and how to proceed with the study.
5. Details should be recorded in the field data notebook. This is usually accomplished by filing a deviation form.

50-1.0

Residue Sample Collection

PURPOSE: To ensure residue samples are collected according to protocol and within GLP guidelines.

PROCEDURE:

1. Consult the study protocol to establish specific dates for the collection of samples. If these dates are based on uncontrolled events (fruit size, spray applications, etc.), then tentative dates should be established and refined as necessary. The Study Director and Quality Assurance Unit should be kept informed when the dates are changed.
2. Prior to sample collection, obtain a sufficient number of sample bags to collect all the samples as outlined in the protocol.
3. Label each sample bag with at least the following information: Field ID number, crop, test substance, field research director, treatment number, and date harvested.
4. Consult the study protocol to determine sample size and special instructions for the commodity.
5. Sample each replicate individually beginning with the untreated plots and proceeding to the highest dosage. Treatments from each replicate should be individually packaged and labeled.
6. Take special care to do the following in the sample collection process:
    a. Avoid contamination of the field sample with the pesticide under study during the sampling, labeling, storage, and shipping processes.
    b. Avoid contact of samples bags to treated soil surface.
    c. Avoid taking diseased or undersized crop parts.
    d. Take care not to remove surface residues during handling, packing, or preparation.
    e. Be certain tools are clean.
    f. Do not remove any soil or plant parts or trim the commodity (e.g., leave the stem in the cherry, leave the outer leaves on the lettuce, etc.), unless specified otherwise in the protocol.
    g. Samples should not be taken during periods of inclement weather.

7. Representative samples of the commodity in each plot will be collected in accordance with the protocol. If a method is not specified in the protocol, plants to be sampled will be selected as randomly as possible from among the full length and width of the plot(s), excluding the plants at the end of the plots.

50-2.0

Post-Sampling Crop Destruct

PURPOSE: To ensure that a crop sprayed with an unregistered pesticide (test substance) does not enter the channels of trade and is inaccessible for human and animal consumption.

PROCEDURES:

1. Field Research Director will decide the method for crop destruction. The criteria to consider includes accessibility of the crop to the public, wildlife, and/or livestock; the grower/cooperator involved in the trial; the feasibility of pursuing other options.

2. When a study is undertaken at a grower/cooperator site, the grower/cooperator and the FRD must have an agreement and understanding about what will be done with the treated crop for which no tolerance has been established. In most cases, the FRD will harvest and dispose of the contents of the treated crop in a manner that ensures it will not be consumed (e.g. compost, burn, trash bin).

3. For trials conducted at WSU – Mount Vernon a different set of circumstances exists. These plots are considered off-limits and non-accessible to un-informed personnel or the public. They are under close supervision of IR-4 personnel; flagging and signage clearly mark the trial plot locations. At WSU and other University sites, it will be up to the discretion of the FRD to determine how best to dispose of the treated crop understanding that the treated crop cannot be consumed by humans and/or animals

4. Whether a plot is at a grower field or on university property, include in the field data notebook an explanation of when (e.g. the date) and how the crop was destroyed or why no crop destruction was necessary.

50-3.0

Sample Storage and Freezer Use

PURPOSE: To ensure integrity of samples and regulatory compliance while in storage.

PROCEDURE:

1. All samples will be frozen as soon as possible following their collection, unless other storage requirements are specified in the study protocol.
2. There are separate freezers for treated and untreated samples. The freezer for untreated samples, (Whirlpool Model ET18NK, Serial # EC5266756) marked "UTC" is located in building #4015 room 145. The second freezer (Amana Model ESU30HW, Serial # 9311931131) marked "TRT" is located in building #4015 room 141 and is for treated samples only.
3. When samples are placed in freezers the time and date the samples were placed in the freezer, the field ID number, the number of samples and their sampling code, and the initials of the person placing the samples in the freezer will be entered on the appropriate log form in the field data notebook. Removal of the samples will be recorded on the log form and include: time and date removed, Field ID #, and person's initials. The Freezers will remain locked during sample storage.
4. Temperature monitoring of the freezers will be conducted to verify that residue samples are maintained within the limits as prescribed by the study protocol or within limits to preserve the commodity and the pesticide residues as close to the condition at harvest as is feasible. Two Hobo dataloggers are kept in each freezer designated as A and B. Data loggers will be set to take temperature reading every half hour. At the end of the data collection period, the data should be transferred to a storage system and immediately printed out with appropriate identification. This hard copy should be signed, dated, and stored in the IR-4 records kept at WSU-Mount Vernon.
5. Freezers will be cleaned and tested for serviceability annually, prior to harvest of residue samples.
6. Steps must be taken to assure that samples remain frozen in the event of a freezer failure. Samples will be stored in freezers for the shortest time, samples will usually be kept in freezer for one or two days. In addition, jugs of water will be frozen in the freezer prior to addition of the samples so that if freezers fail the frozen jugs will maintain freezing temperatures for longer than without the jugs. And freezers temperature will be checked at the beginning and end of each workday that samples are in freezers
7. In the event of a mechanical failure of one of these freezers while samples are in storage, the samples will be moved to the operational freezer. Sample condition will be noted at the time of movement.

50-4.0

Sample Shipping Procedures

PURPOSE: To ensure that residue samples are shipped to the residue laboratory with a minimum loss of integrity.

PROCEDURE:

1. As per protocol, contact the residue laboratory by phone, fax or e-mail, and notify them of the shipment dates and method of shipment.
2. Complete Form 8A (“Residue Sample Shipping Information”) and Form 8B (“Residue Sample Chain of Custody”). Include a true copy of 8B and a blank copy of 8C (“Sample Arrival Check Sheet”) in each shipping container (preferably within a plastic bag). Retain the original of Form 8A and Form 8B in the field data notebook.
3. Make arrangements with the carrier for shipment of the samples and determine any special packing instructions that are required to preserve the sample integrity. Note any limits on quantity of dry ice, etc., that may be set by the carrier.
4. Obtain insulated containers of sufficient size and quantity to hold the residue samples and dry ice (when required) in an approximate 3:1 ratio (dry ice: sample, by weight). Pack the samples and dry ice in the containers just prior to shipment. The containers should have sufficient bursting strength so as to withstand normal handling in shipping and storage.
5. Label each container with the following information:
   a. Name and address of the sender
   b. Name and address of the residue laboratory receiving the samples
   c. Number of the container if more than one is used
   d. Affix "Experimental Samples--Perishable" on each carton
   e. When used, affix "Dry Ice" on two sides of the container
6. Provide carrier with the phone number of the residue laboratory receiving the samples and request the carrier to notify the laboratory when the samples arrive at a remote terminal for pickup.
7. Once the samples are picked up by the carrier, fax or scan a true copy of Form 8A (“Residue Sample Shipping Information”) and Form 8B (“Residue Sample Chain of Custody”) to the Study Director, WSU IR-4 Field Center Director and the Western Region Regional Field Coordinator. Retain all originals in the field data notebook.

60-1.0

Equipment Maintenance

PURPOSE: To ensure application equipment used in GLP studies is functioning and in good condition.

PROCEDURE:

1. Just prior to the initiation of the use of the equipment (i.e., tractor, ATV, plow, disk, harrow, planters, harvester, etc.) the Field Research Director or his/her designated representative will visually inspect the equipment to see that it is in good working order, properly lubricated, and in reasonably good mechanical condition.
2. Any necessary repairs or adjustments should be made prior to the use of the equipment in the study.

3. The operator of the equipment should be reasonably familiar with its operation and safety precautions.
4. Manuals on the operation and maintenance of the equipment and the name, address, and telephone number of a parts supply company should be kept in a place accessible to the operator and the Field Research Director.
5. Manuals for vehicles (as available) are maintained at WSU-Mount Vernon in the Farm Shop, Building #4009. Manuals for equipment (as available) are maintained at WSU-Mount Vernon in IR-4 Office, Building #4032.
6. A written record will be maintained for each piece of equipment used in a GLP study. The maintenance record should contain a description of the equipment and the type(s) of service or repair, the date(s) it was performed an whether SOP was followed. In addition, non-routine maintenance entries should describe nature of defect, how and when it occurred, and remedial action taken. This record will be maintained in the WSU-Mount Vernon Maintenance Log file, IR-4 Office, Building #4032.

60-2.0

Calibration of Instruments and Gauges

PURPOSE: To ensure that instruments crucial to GLP data requirements are standardized and maintained to provide accurate readings.

PROCEDURE:

1. Each gauge or instrument used in a GLP study (i.e., temperature and humidity gauges, photometers, max/min thermometers etc.) should be tested to determine that it is reasonably accurate. Most instruments need to be calibrated or checked at minimum annually.
2. A written record should be kept of the dates and results of the tests and of the acceptable tolerance for each instrument.
3. Those gauges or instruments that give inconsistent results or are not accurate to within desired tolerances should be repaired or replaced.
4. Pressure gauges are considered accurate unless giving questionable or inconsistent results as judged by WSU IR-4 personnel. There is no reliable method in place for standardizing gauges and replacement will occur on an "as needed" basis when personnel deem necessary.
5. Thermometers, hobos, and other such instruments will be calibrated according to manufacturer specifications, if possible. If calibration is not possible, each instrument will be tested against two similar instruments or an NIST certified device to determine if it is providing accurate readings (tolerance of ±5% or ± 5° F). If beyond tolerance, it will be tested again against a third, similar instrument. If again found to be beyond the tolerance, the instrument should be replaced.

6. A NIST thermometer is used when standardizing temperature recording devices used in GLP trials. The NIST is used for comparison only and is not used when calculating the average of the temperature recording devices. Either a new NIST thermometer will be purchased with certification that it is accurate or the one currently being used will be re-certified every 3 years
7. Onset Hobo Pendant temperature loggers that are used to record temperature of freezers, and test substance storage and transportation must be standardized annually. For loggers used in freezers use HOBOware software to set Pendent data loggers to record every minute and place loggers and NIST thermometer in freezer. After approximately one-hour remove from freezer, immediately read and record the temperature of the NIST thermometer, then download all the Temp Loggers and print out a graph for each. The graph should be "zoomed-in" to show the stabilized temperature (i.e. where the readings become constant), and then printed. The reading of each unit must be within ± 5° F of the NIST thermometer. Repeat this process for test substance storage loggers but place in these loggers in IR-4 test substance storage cabinet for one hour. The NIST thermometer reading is not used when calculating the average of the HOBOS; it is used for comparison only, but also must be within ±5° Fahrenheit of the average of the HOBOS. The primary instrument used to measure air temperature, relative humidity, and wind speed is a digital Kestrel 5500 by Nielsen-Kellerman. Prior to standardization change the battery. Standardization is accomplished by comparing readings from three sperate instruments while under the same conditions.
    a. To standardize for air temperature and relative humidity, line up the instruments on the same surface, at the same level, under the same conditions. After about 15 minutes, take readings from all instruments, for each environmental parameter (i.e. air temp, RH). Results should be within 5% of the average of all three instruments for rH and be within ±5° Fahrenheit. Instruments should be standardized in the approximate range of the temperatures likely to occur in the unit. Record the results in the maintenance log.
    b. To standardize for wind speed, align the instruments next to each other on the same surface, aiming them all in the same direction. Conducting the standardization indoors with a fan directed towards the meters facilitates the standardization process. Read all three meters concurrently and document. Results should be within ± 2 mph of the average of all three instruments. It is recommended to standardize the wind function of the Kestrels under a range of wind speeds. For example, for a given standardization, altering the distance from the fan that the instruments are placed will give different wind speeds. Record the results in the maintenance log.