

SOP Log Sheet

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FRD/LRD: Rebecca Tannenbaum
Submitter

Effective Date: 3/1/23

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(Circle one)

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Sign: [Signature]

Comment: _____



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IR-4 Southern Region

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March 1, 2023

Rebecca Tannenbaum
University of Florida - TREC
18905 SW 280 Street
Homestead, FL 33031-3314

Dear Rebecca,

I have received the cover page and table of contents for the 2023 Standard Operating Procedures (SOPs) for the IR-4 facility at University of Florida TREC in Homestead, FL. Thank you for reviewing the complete set of SOPs and submitting a revision for SOPs 4.1, 4.21, 9.1, and 9.4. I have reviewed the SOPs and I approve of the submitted revisions. The cover letter has been signed with an effective date of 3/1/2023.

Sincerely,

A handwritten signature in black ink, appearing to read 'Janine Spies'.

Janine Spies, Ph.D.
IR-4 Southern Region Field Coordinator



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Tropical Research and Education Center

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February 24, 2023

Janine Spies
1642 SW 23rd Drive
PO Box 110720
Gainesville, FL 32611-0720

Dear Janine,

Please review the cover page and table of contents for the 2023 Standard Operating Procedures (SOPs) for use at the IR-4 facility at TREC in Homestead, FL.

The SOPs have been reviewed and signed by me. I have included revisions for SOPs 4.1 v 8, 9.1 v 7, 9.4 v 8, and SOP 4.21 v 8.

Sincerely,

Rebecca Tannenbaum
Field Research Director/IR-4 Project
University of Florida/TREC
786.217.9273

STANDARD OPERATING PROCEDURES
FOR
MAGNITUDE OF THE RESIDUE-FIELD STUDIES

University of Florida
Tropical Research & Education Center
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<i>Adriana Jimenez</i>	2-24-23	AJ	2-24-23
Field Research Director	Signature	Initials	Date

<i>Janine Spies</i>	<i>Janine Spies</i>	JS	3/1/23
Regional Field Coordinator	Signature	Initials	Date

Effective Date of approval: DATE OF SIGNATURE BY REGIONAL FIELD COORDINATOR

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SOP 1.1 v 8

Title: General requirements for the development and use of Standard Operating Procedures.

Purpose: To provide guidance to scientists conducting field trial(s) in the development and use of Standard Operating Procedures for field research.

Scope: Locations conducting field trial(s).

Procedures:

1. All Standard Operating Procedures (SOPs) will be reviewed annually and revised as needed. Management and Field Research Director need only sign the title page which will indicate the effective date of all the SOPs, since SOPs are treated as a package. The index page includes the date that an individual SOP was revised.
2. Each facility where trial(s) are conducted in support of the registration of pesticides will develop SOP's for all phases of the research.
3. If a particular SOP is revised, the revision date will be changed in the SOP index. The index page indicates the revision date of each SOP. A revision provides a substantive change in the SOP. Correcting spelling and formatting errors do not constitute a revision.
4. Any deviation from the SOPs must be documented in the raw data and authorized by the Study Director. 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 not intended to describe every possible action that may be necessary for the complex and extremely variable operation of a field center. Actions specific to individual situations need to be explained and documented in the Field Data Notebook.
5. The effective date of the SOPs shall be the date that the regional field coordinator signs the SOP cover letter.
6. Numbering system for SOPs will be as follows:
 1. Administration
 2. Staff
 3. Facilities
 4. Equipment
 5. Test System Establishment & Maintenance
 6. Test Substance
 7. Data Handling

8. Residue Sample Handling
9. Reporting and Retention of Data
10. Disposal of Pesticides
11. Safety and Health Procedures
12. Procedures to Handle an EPA Audit or Inspection
13. Miscellaneous SOP's

The SOPs will be numbered sequentially, using the following format: SOP x.x v 7
Where x.x refers to the number of the SOP, e.g 1.2 and 'v' stands for version, in this case version 7.

7. The following is the format to be used in each Standard operating Procedure:

- Name of Test Facility
- Address
- SOP Number: SOP section number as a decimal; Version number, denoted by 'v': sequentially beginning with 1 for first use.
- Title: Title of SOP.
- Purpose: Brief description of the purpose of the SOP.
- Scope: Determines where and when the SOP is applicable.
- Procedures: Describe the operating procedures in numerical order from beginning to end so that an intelligent person with some knowledge of the process can carry out the procedures without any verbal input from other sources.
- Each SOP will be numbered individually.

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SOP 1.2 v 8

Title: Designation of Field Research Director (FRD) and responsibilities.

Purpose: To provide information on how a Field Research Director is designated and outline the responsibilities of the Field Research Director.

Scope: All test facilities where GLP trial(s) are conducted.

Procedures:

1. The FRD is designated by the Study Director based on the recommendation of the Regional Field Coordinator to conduct the trial(s). The Field Research Director shall be a scientist with appropriate training and experience to conduct the trial(s).
2. The Field Research Director will ensure that:
 - a. Draft protocols are reviewed and that the Study Director is informed of any potential issues.
 - b. The trial is carried out in accordance to an approved protocol and the GLP regulations.
 - c. Utilize personnel, resources, facilities, equipment, materials and methods as necessary for the conduct of the trial.
 - d. All personnel conducting the study understand the protocol, SOPs for the project, and GLP regulations.
 - e. All deviations and findings reported by the Quality Assurance Unit (QAU) are responded to through the IR-4 e-QA system or in writing.
 - f. All raw data, summaries and other items connected with the study that need to be retained are transferred to the archives at IR-4 Headquarters.
 - g. Maintain a current copy of a master schedule for all GLP projects under his/her direction.
 - h. Create copy of the following: 1) Completed Field Data books for all studies prior to shipping the books. Maintain on file a current record of training, education and experience, and a job description for all personnel engaged in the study. These records will be reviewed and updated as necessary before the initiation of the studies for the current year.
 - i. FRD is in charge of all equipment used in the trials.

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SOP#: 2.1 v7

Title: Staff

Purpose: Provide information to field locations about personnel requirements under Good Laboratory Practices.

Scope: All field facilities conducting trial(s) for the registration of pesticides.

Procedures:

1. The field facility will have on file current copies of a professional biography or CV, a position description, and training records for each person engaged in or supervising the trial(s).
2. The field facility will have a sufficient number of persons to carry out the trial(s) to its completion and the Field Research Director or designee will utilize trained personnel to conduct their portion of the trial(s).
3. The field facility will have a supply of safety equipment in 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 SOP's, regulations, other institution regulations, pesticide labels or the trial(s) protocol.
4. Where the application of restricted use pesticides is required in the trial(s), 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 of their institution.
6. Personnel documentation will be reviewed annually and revised as needed, or indicated by a dated signature that the document was reviewed.
7. In the event that a person's employment with the organization ends, their personnel records will sent for archiving at IR-4 HQ

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SOP 2.2 v 7

Title: Organizational chart.

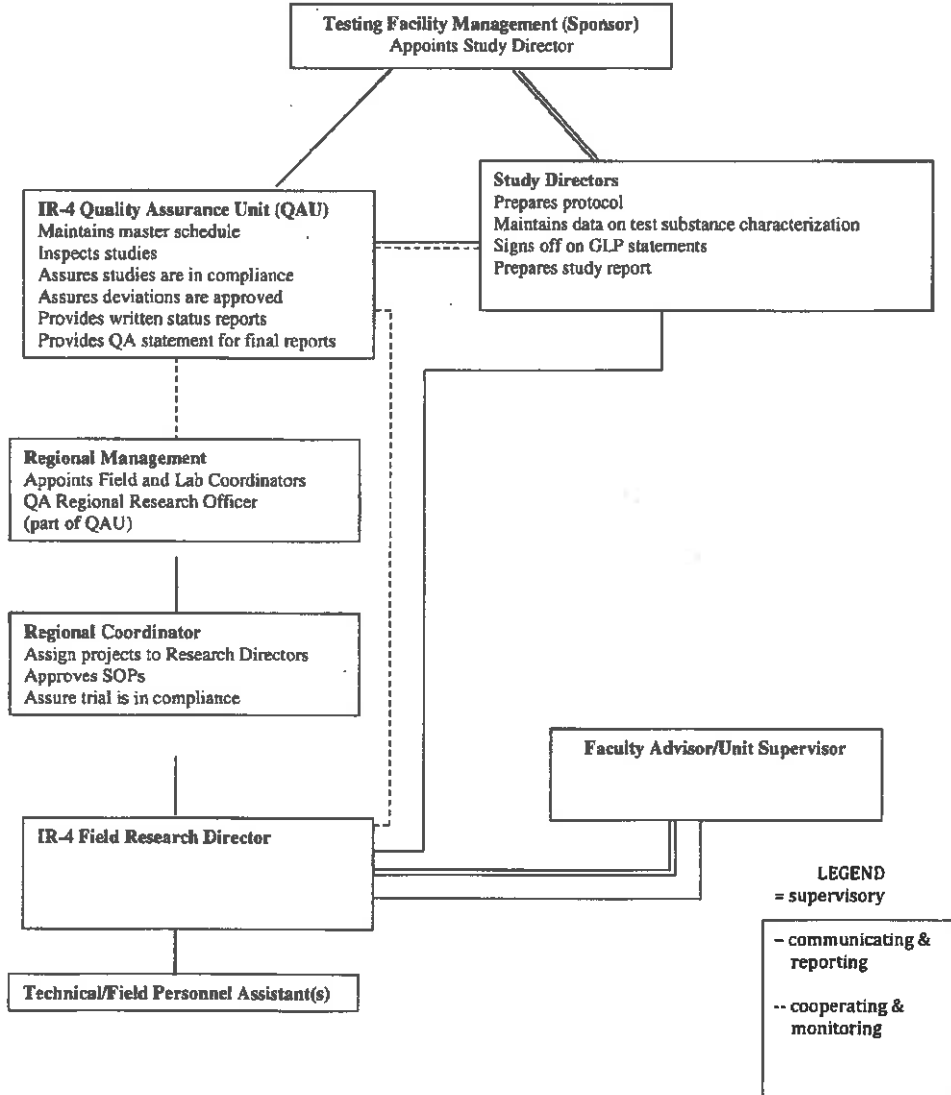
Purpose: To assist locations in the development of an organizational chart.

Scope: All field facilities conducting trial(s) for the registration of pesticides.

Procedures:

1. An organizational chart should describe the management structure of the institution performing the work. It should also document the reporting lines for personnel engaged in GLP studies both to the institution's management and to IR-4 Testing Facility Management.
2. Each block in the chart should show the title, and a brief description of the duties of each person.
3. The head of the unit (i.e. Department Chair, Director, etc.) should be included in the chart.
4. The chart should then show how the Field Research Director and the Quality Assurance Unit (QAU) independently report to the IR-4 Testing Facility Management.
5. Personnel engaged in the conduct of the trial(s) should then be shown on the chart with lines of supervision, communication, and cooperation indicated.

ORGANIZATIONAL CHART IR-4 TREC



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SOP 2.3 v 7

Title: Documentation of training.

Purpose: To assure that training for personnel involved in the study is properly documented.

Scope: All field facilities conducting trial(s) for the registration of pesticides.

Procedures:

1. All training of personnel engaged in GLP trials should be documented in a training record, to be kept at the field facility.
2. Training received from any source, should be noted as to the name of the event, date(s) of attendance, instructors name, and subjects covered. A copy of any type of certificates issued should be retained in the personnel files at the location.
3. Training on specific procedures and/or SOPs should also be documented. Record the name of the person giving the instruction, the name of the person receiving the instruction, the date given and a concise statement of the instruction, or SOP (e.g. Dr. X explained to Mr. Y how to label the sample bags as per SOP xx on 6/2/93).
4. Each person engaged in the conduct of the study should have read and understood those sections of the protocol and the standard operating procedures that pertain to their responsibilities. It should be recorded, in each respective training record, the names of the personnel and dates that the SOPs were explained to them. This information should be placed in the personnel file.

SOP 2.3 v 7

Reviewed by (initials)	Date	Comments
AJ	3-27-19	

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SOP 3.1 v 8

Title: Guidelines for adjuvant and test substance storage.

Purpose: To assure that all adjuvants test substances are stored in a manner consistent with GLP requirements.

Scope: Locations conducting IR-4 field trial(s) and pesticide storage.

Procedures:

1. Adjuvants and test substances will be stored in a dry, well-ventilated building which is separate from offices, laboratories and sample storage areas. The adjuvant and test substance area should be sufficient to allow storage of the adjuvant and test substances according to their label directions. Test substance and adjuvants will be stored in accordance with current policies and guidelines of the testing facility institution.
2. The temperature within the storage facility will be monitored by two temperature monitoring devices. Examples are Onset Hobos and min-max thermometers. The data from the hobo device should be downloaded once per month. (Minimum: once every 3 months) The Min-Max thermometer will serve as a backup to the main temperature monitoring device and shall be read and recorded at regular intervals, preferably twice a month.
3. The original containers for all GLP test substances and adjuvants must be retained until completion of the study and permission is given by the study director or regional coordinator to dispose of the containers.
4. The storage facility should have limited access by utilization of a lock and key so that only authorized persons may have access to GLP test substances.
5. Place highly visible, waterproof identification sign on the door of our storage section within the TREC pesticide shed to advise of the hazardous nature of the storage facility's contents.
6. Post the telephone numbers and names of personnel responsible for and knowledgeable of the contents of the storage facility.
7. Make accessible, materials such as (but not limited to, nor necessarily all) adsorptive clay, granulated activated charcoal, hydrated lime, and sodium hypochlorite for emergency treatment or detoxification of spills or leaks.

8. Store containers of test substances that could be damaged by moisture or water, off the floor.
9. Post a current inventory of all IR-4 test substances in the storage unit in an inside location accessible and visible to study personnel.
10. Test substances and adjuvants used under GLP may be stored separately or in a separately labeled area in the storage facility.
11. The storage area for the test substance's and adjuvants should be separate from but may be adjacent to non-IR-4 pest control substance to preclude contamination or mix-up.
12. Containers for adjuvants and test substances should be labeled as follows:
 - (1) Name of chemical
 - (2) Expiration Date (If no date exists for adjuvant, a date of 5 years will be assigned)
 - (3) PR number, in the case of test substance.
 - (4) Batch Lot ID
 - (5) Container number in case of multiple containers of the same substance.
 - (6) Date of Receipt
 - (7) Storage conditions and concentration.
13. Transport temperatures for adjuvant & test substance will be maintained by the use of Blue ice inside the transport cooler. A temperature reading device will be placed in the cooler to monitor temperature during transport. If it becomes necessary, adjuvant may be held in a secondary container to fit into cooler. Secondary container must be labeled as in #12.
14. When measuring the calculated amount of test substance or adjuvant for a spray mix, the Pipette or syringe used to extract the desired amount of chemical will not have been used prior and will not be used for any other product.
15. If it becomes necessary to use a temporary container for measurement of adjuvant, the remaining adjuvant must be discarded.
16. Test substances which are temperature sensitive will be stored in a locked box inside the IR-4 lab until the end of the trial. At the end of the trial the test substance will be moved to the UF/TREC pesticide storage facility. The lab will be temperature monitored by both Onset Hobos and a back up Min -Max thermometer.

SOP 3.1 v 8

Reviewed by (initials)	Date	Comments
RJ	3-27-19	

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SOP 3.2 v 7

Title: Site selection for field trial(s).

Purpose: To assure plots are large enough to obtain the required data or samples with sufficient uniformity and may be re-located after the trial(s) is terminated.

Scope: All locations conducting field trial(s).

Procedures:

1. Site selection will be made in accordance with the horticultural practices acceptable for the commodity and the requirements established by the protocol.
2. 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 to comply with protocol sample size requirements.
3. Locate site with sufficient isolation to prevent contamination of the test plots by spray drift sources such as commercial operations or other research trial(s).
4. Where samples for residue trial(s) are required, locate nontreated plots within the same area [preferably upwind and upslope of the treated plot(s)] but with enough isolation to produce nontreated, noncontaminated samples.
5. If the commodity is not required to be newly established, select a site that has commercial standards for production. Commercial farms off site may be used for residue trials.
6. Prepare a plot map, to be added to the raw data notebook that shows the location and dimensions of each plot on the site, the slope, and the North azimuth (which may vary slightly). The plot map should contain distances to permanent reference points (GPS or other known reference points) so that the plots can be re-located after the trial(s) is terminated.
7. Label each plot with the field ID number and treatment as a minimum. If statistical analysis is to be performed on the data, assign the replicates and treatments to the plot map using a commonly accepted statistical design with sufficient information to identify the replicate and treatment assigned to each plot.
8. Lay out each plot on the site using a suitable measuring device to accurately locate the plots on the site.
9. Identify both ends of each plot with a marker of sufficient visibility to be seen easily throughout the duration of the trial(s).

SOP 3.2 v7

Reviewed by (initials)	Date	Comments
RJ	3-27-19	

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SOP 3.3 v 6

Title: Greenhouse/shadehouse facilities.

Purpose: To assure that greenhouse/shadehouse facilities are properly maintained and in sufficient working order throughout the trial(s) to obtain data useful in the registration of pesticides in the GLP program.

Scope: All locations where greenhouse/shadehouse trial(s) are performed.

Procedures:

1. Each greenhouse/shadehouse must be sufficiently large enough to contain the entire trial(s) or a complete replicate of the trial(s) with sufficient space between plots to prevent contamination. Identify plots as described in SOP 3.2
2. Where more than one trial(s) is conducted in a greenhouse/shadehouse, there must be sufficient isolation between the trial(s) to prevent contamination or interference between trial(s).
3. Lighting, temperature, humidity, and shade should be sufficiently uniform at the trial(s) sites in the greenhouse/shadehouse to provide nearly uniform plant growth throughout the trial(s) sites.
4. The walls, floors, and ceilings of the greenhouse/shadehouse should be maintained in good condition. Floors, benches and isles should be free of debris, weeds and superfluous equipment and well-drained to prevent the buildup of excess moisture.
5. Greenhouses should be equipped so as to maintain temperature, lighting, and moisture conditions to simulate commercial greenhouse production techniques or as required by the study protocol.
6. Sufficient monitoring devices should be installed, in good working order, and calibrated to assure that the proper lighting, temperature and humidity conditions are maintained throughout the trial(s). All calibrations will be documented in the maintenance logs.
7. Document cultural practices used in the greenhouse and treatment locations in the raw data notebook.

SOP 3.3 v 6

Reviewed by (Initials)	Date	Comments
RJ	3-27-19	

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SOP 4.1 v 8

Title: Calibration and use of balances.

Purpose: To assure an accurate weighing of dry test substances or samples.

Scope: All field facilities where a dry material is weighed for use in a field, greenhouse or hothouse trial(s), or all facilities in which samples are being processed for freezing and/or shipping.

Procedures:

1. The balance should be calibrated routinely for weighing the quantity for use in the study by following the directions of the manufacturer in the manual. The calibration by use of "Known Weights" is acceptable provided that information is provided as to the manufacturer, date manufactured, and proper use of the weights.

1. To calibrate:
 - a. Clear the unit to the point at which readout is at zero.
 - b. Center the calibration weight on the weighing platform or pan. If adjustments are allowed, adjust the balance until the readout is +/- 2% of the declared value of the calibrated weight.
 - c. Annotate values on a log for retention in raw data. If recalibration is required and operator level adjustments are available, it should be done and the weights remeasured.
 - d. If operator level recalibration is either not available or not adequate, the unit should be routed for maintenance by a properly trained technician.

2. To measure a dry material formulation:
 - a. Select a clean glass jar or plastic container with a tight fitting lid or container suitable to hold the desired amount of pesticide and tare it on the scale following the manufacturer's directions. A weigh boat may be used to weigh the test substance before placing in another container for transport.
 - b. Select and wear or use appropriate safety equipment while handling pesticide concentrate.
 - c. Weigh the concentrate in the tared 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 for handling hazardous wastes.
 - d. Label the container to identify it as to the appropriate treatment or plot numbers.
 - e. If taring the container is not practical, then record the weight of the container, add the weight of the desired amount of pesticide to it and weigh out this amount.
 - f. Maintain a written record of the amount of the pesticide removed from the original container for each weighing for the study.

3. To measure a residue sample on a calibrated balance when required in the protocol (note use of a calibrated balance is no longer required for the residue fruit sample):
 - a. Ensure that sample is bagged so as to prevent direct contact with surface. Prior to weighing, ensure that the surface of the balance is clear and free of materials.
 - b. Starting with nontreated control first, weigh by centering on the balance and wait until readings settle. Annotate the reading and move on to the next one.
 - c. After finished, turn balance off, and maintain written record of the sample weights for the study.

4. Weights of appropriate mass shall be used to bracket target weight, during the measuring of formations.

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SOP 4.2 v 6

Title: Measuring liquid formulations.

Purpose: To assure an accurate measurement of liquid test substances.

Scope: All field facilities conducting field trial(s) for IR-4.

Procedures:

1. Obtain a clean cylinder or measuring device large enough to hold the volume of liquid needed, graduated in increments small enough to read to an accuracy within $\pm 1\%$ of the total volume required (i.e. if 100 ml is needed the smallest division on the cylinder should be 1 ml or less).
2. If the opening of the cylinder/device is too restricted to allow filling without danger of spillage, then do one of the following:
 - a. Use a clean container with a pour lip as an intermediate and fill the cylinder/device from it or
 - b. use a clean funnel that is large enough to allow filling the cylinder with a minimum of spillage or
 - c. use clean, calibrated pipettes or syringes for material transfer.
3. Select and wear or use appropriate safety equipment while measuring liquids.
4. Measure the liquid in the cylinder/device. Place the cylinder/device on a level surface and take the reading of the liquid in the cylinder/device at the bottom of the meniscus with the eye being level with the bottom of the meniscus. If using a pipette or syringe, read the liquid amount at the bottom of the meniscus with the eye being level with the bottom of the meniscus. Document the amount of test substance measured in the raw data book.
5. Pour the liquid into an appropriate container, fit with a leak proof lid and label as to contents and amount.
6. Cylinders/devices, glass re-usable pipettes, and syringes with metal detachable needles used to measure or transfer the test substance concentrate should be triple rinsed into the mixing container and then thoroughly washed with soap and water after use to ensure that they are clean and cross-contamination of pesticides does not occur in future use.

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SOP 4.3 v 6

Title: Calibration of thermometers.

Purpose: The purpose of this SOP is to establish procedures used when calibrating and reading thermometers.

Scope: The SOP is to be followed by IR-4 participating personnel when calibrating non-certified thermometers.

Procedures:

1. All temperature measuring devices at TREC used for studies or equipment associated with studies will be calibrated one time a year against a reference thermometer, either directly or by a recorded traceable chain.
2. Records of thermometer calibration will be maintained in a log.
3. All mercury and min/max thermometers will be identified by a unique number or code. The identification number or code will be placed on the measuring device such that it can easily be cross-referenced to calibration log records.
4. Measuring devices to be calibrated and the reference thermometer will be read side by side in water under conditions appropriate to the intended use.
5. Examples of temperature ranges to test may include:
 - a. room temperature (approx. 22°C)
 - b. ice slush (approx. 0°C)

Water baths will be contained in a pan or beaker deep enough for adequate immersion of the instrument. The ice bath should be made with chopped ice in water to form a tightly packed slush. The thermometers will be calibrated in the water bath temperatures in the order listed above.

6. The thermometers and the reference thermometer will remain in the water bath until a constant reading is reached. When the analyst feels confident that reading is constant, the values will be recorded in the log. The following information will be documented in the log:
 - a. date of calibration
 - b. initials of person doing calibration
 - c. reference thermometer reading at each temperature listed in 5
 - d. the thermometer reading at each temperature listed in 5
 - e. ID code number of the thermometer being calibrated

7. Temperature readings taken from the reference thermometer may be rounded to whole numbers (e.g.: If reference reads 38.2°C, the recorded value will be 38°C).
8. Each measuring device will be labeled following calibration and will include:
 - a. ID or code number
 - b. date of calibration
 - c. initials of person doing the calibration
 - d. temperature adjustment
9. If the reading of the laboratory thermometer is ± 1 degree of the reference reading, no temperature adjustment will be made and the label will read "OK". If the reading is more than 1 degree in relation to the reference thermometer, the proper adjustment will be made. For example: If the thermometer reads 20°C and the reference reads 22°C, the adjustment would be + 2°C at 22°C. When this thermometer is used, the individual would add 2°C to the 20°C observed reading and 22°C would be recorded as the temperature reading.
10. When recording a thermometer reading, the following information should also be included in the entry:
 - a. date
 - b. initials of individual conducting the activity
 - c. the thermometer ID or code number
11. Calibrated thermometers may be used to calibrate other temperature recording devices as long as they can be traced back to a reference calibration. These devices may include continuous thermographs used for walk-in digital displays on up-right freezer/refrigerator units, etc.

SOP 4.3 v 6

Reviewed by (initials)	Date	Comments
RJ	3-27-19	

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SOP 4.4 v 6

Title: Calibration and use of temperature datalogger devices.

Purpose: This document is for use by IR-4 personnel to define techniques used for calibrating, operating, and maintaining temperature monitoring devices.

Scope: The SOP describes the proper procedures used by IR-4 personnel to ensure that accurate calibration, operation, and temperature monitoring devices.

Procedures:

1. All temperature measuring devices at TREC used for studies or equipment associated with studies will be calibrated one time a year against a reference thermometer, either directly or by a recorded traceable chain. Prior to use, visually inspect the datalogger for cleanliness and that it is in good working condition. Check the power supply (if applicable, batteries should be replaced when the power indicator light does not blink while the unit is on). Record inspection and any maintenance performed in appropriate logs. Use the following methods or document the methods used as raw data.
2. All temperature measuring devices used for GLP studies will be calibrated one time a year against a reference thermometer, either directly or by a recorded traceable chain. Records of temperature monitoring devices calibration will be maintained in a log.
3. All dataloggers will be identified by a unique number or code. The identification number or code will be placed on the measuring device such that it can easily be cross-referenced to calibration log records.
4. Data logger(s) to be calibrated and the reference thermometer will be read side by side under conditions appropriate to the intended use.
5. Two temperatures will be noted. These are:
 - a. Room temperature reading as indicated by reference thermometer or other means of reliable temperature recording.
 - b. Cool/Cold, i.e. ambient to freezing as indicated by reference thermometer or other means of reliable temperature.
6. The data logger (s) and the reference thermometer will remain in the calibrating environment until a constant reading is reached. When the analyst feels confident that reading is constant, the values will be recorded in the log. The following information will be documented in the log:
 - a. date of calibration
 - b. initials of person doing calibration
 - c. reference thermometer reading
 - d. the datalogger temperature reading

- e. identification (ID) or code number of the datalogger being calibrated
7. Temperature reading taken from the reference thermometer may be rounded to whole numbers (e.g.: If reference reads 38.2°C, the recorded value will be 38°C).
 8. At the end of data collection period, the data should be printed out (hard copy). This hard copy will be placed in the appropriate Field Data Notebooks and submitted to the Study Director and/or retained in a file as raw data. The following information, at a minimum, should be included:
 - a. date
 - b. initials of person downloading the data
 - c. data ID or code number
 - d. units in measurements
 - e. Units will be appropriately maintained. If the unit fails, it will be serviced by a technician. The only exception is the changing of batteries and charts or the adjustment of sensor arms during calibration.

SOP 4.4 v 6

Reviewed by (initials)	Date	Comments
RJ	3-27-19	

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SOP 4.5 v 6

Title: Calibration of soil thermometers.

Purpose: The purpose of this SOP is to establish procedures used when calibrating and reading soil thermometers.

Scope: The SOP is to be followed by IR-4 participating personnel when calibrating soil thermometers.

Procedures:

1. All temperature measuring devices at TREC used for studies or equipment associated with studies will be calibrated one time a year against a reference thermometer, either directly or by a recorded traceable chain.
2. Records of thermometer calibration will be maintained in a log.
3. All soil thermometers will be identified by a unique number or code. The identification number or code will be placed on the measuring device such that it can easily be cross-referenced to calibration log records.
4. Examples of temperature ranges to test may include:
 - a. room temperature (approx. 22°C)
 - b. ice slush (approx. 0°C)Water baths will be contained in a pan or beaker deep enough for adequate immersion of the instrument. The ice bath should be made with chopped ice in water to form a tightly packed slush. The soil thermometers will be calibrated in the water bath temperatures in the order listed above.
5. The soil thermometers and the reference thermometer will remain in the water bath until a constant reading is reached. When the analyst feels confident that reading is constant, the values will be recorded in the log. The following information will be documented in the log:
 - a. date of calibration
 - b. initials of person doing calibration
 - c. reference thermometer reading at each temperature.
 - d. thermometer reading at each temperature.
 - e. ID code number of the soil thermometer being calibratedTemperature readings taken from the reference thermometer may be rounded to whole numbers (e.g.: If reference reads 38.2°C, the recorded value will be 38°C).
6. Values will be recorded and evaluated to determine if recalibration is required. If corrective measures are required, and available at operator maintenance level, then adjustments will be immediately made. If recalibration is above ability of operator level, the device should be dead lined and sent into manufacturer for repairs.

SOP 4.5 v 6

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SOP 4.6 v 7

Title: Calibration and Use of pH meters

Purpose: This document is for use by IR-4 personnel to define techniques used for calibrating, operating, and maintaining pH measuring devices.

Scope: The SOP describes the proper procedures used by IR-4 personnel to ensure that accurate calibration and operation of pH measuring devices.

Procedures for pH meters:

1. pH metering devices should be calibrated prior to use.
2. Records of pH calibration will be maintained in a log.
3. All pH meters will be identified by a unique number or code. The identification number or code will be placed on the measuring device such that it can easily be cross-referenced to calibration log records.
4. The pH meters will be inserted into known standard solutions, either made in the laboratory and certified by alternate calibrated devices or provided by a reliable provider. The pH meter will remain in the solution until a constant reading is reached, close to the known value of the solution. The instrument will then be calibrated. Examples of standards are:
 - a. pH 4.0; b. pH 7.0 and; c. pH 10.0.
5. When the analyst feels confident that reading is constant, the values will be recorded in the log. The following information will be documented in the log:
 - a. date of calibration
 - b. initials of person doing calibration
 - c. ID code number of the meter being calibrated.
6. Values will be recorded and evaluated to determine if recalibration is required. If corrective measures are required, and available at operator maintenance level, then adjustments will be immediately made. If recalibration is above ability of operator level, the device should be dead lined and sent into manufacturer for repairs.

SOP 4.6 v 7

Reviewed by (initials)	Date	Comments
RJ	3-27-19	

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SOP 4.7 v 7

Title: Calibration of RH/Temperature Device.

Purpose: The purpose of this SOP is to establish procedures used when calibrating and reading RH/Temperature monitoring equipment.

Scope: The SOP is to be followed by IR-4 participating personnel when calibrating

Procedures:

1. All relative humidity/temperature measuring devices at TREC used for studies or equipment associated with them will be a) either sent out annually to a company that will calibrate and certify the equipments accuracy or b) be calibrated annually to ensure their correct function and data yield.
2. Records of these calibrations will be maintained in a log.
3. All will be identified by a unique number or code. The identification number or code will be placed on the measuring device such that it can easily be cross-referenced to calibration log records.
4. The RH meters will be judged for accuracy by use of either a sling psychrometer (wet bulb), and/or standardized against other devices that provide traceability to sound and reasonable calibration. If standardizing is used, the analyst must provide the unique identification of the equipment the calibrated unit is being tested against.
5. The temperature device is not sent out for calibration will be calibrated according to SOP 4.5
6. When the analyst feels confident that reading is constant, the values will be recorded in the log. The following information will be documented in the log:
 - a. date of calibration
 - b. initials of person doing calibration
 - c. ID code number of the meter being calibrated.
7. Values will be recorded and evaluated to determine if recalibration is required. If corrective measures are required, and available at operator maintenance level, then adjustments will be immediately made. If recalibration is above ability of operator level, the device should be dead lined and sent into manufacturer for repairs.

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SOP 4.8 v 6

Title: Calibration of freezer and monitoring/emergency devices.

Purpose: The purpose of this SOP is to establish procedures used when calibrating and reading sensors in the sample freezers that allow the emergency monitoring device to protect against sample loss.

Scope: The SOP is to be followed by IR-4 participating personnel when calibrating the equipment used to ensure sample integrity in IR-4 sample freezers.

Procedures:

1. All temperature measuring devices at TREC used for studies or equipment associated with them will be calibrated on an annual basis to ensure their correct function and protection against sample loss.
2. Records of these calibrations will be maintained in a log.
3. Referring to the manual for guidance on procedure, a calibrated thermometer will be put into the freezer and then the automated machine will be checked to ensure that it is within (+/- 2°F) of the thermometer reading. If not, corrective action will be taken until reading is stable and in accordance with this method.
4. Phone contact numbers will be updated, and entered to the satisfaction of the programmer.
5. When the analyst feels confident that readings are correct, and the call out procedure is operable and updated, this will be recorded in the log.
The following information will be documented in the log:
 - a. date of calibration
 - b. initials of person doing calibration
 - c. ID code number of the freezer/monitor being calibrated and/or checked.

SOP 4.8 v 6

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SOP 4.9 v 7

Title: Maintenance and testing of backup generator for IR-4 freezers used to store residue samples.

Purpose: The purpose of this SOP is to establish procedures for maintenance and testing of the backup generator for the IR-4 freezer used to store residue field samples. This generator is to be used to provide electric power to the IR-4 freezers in the event of a prolonged power outage that would cause residue field samples to defrost – thus negating the integrity of the samples.

Scope: The SOP is to be followed by IR-4 personnel for maintenance and testing of the backup generator for the IR-4 freezer used to store residue field samples.

Procedures:

1. The permanent, stationary IR-4 backup generator will be tested or inspected annually to be sure it is in working order. The annual check will be performed by the maintenance department at TREC and UF at Gainesville.
2. A maintenance log will be maintained to verify the maintenance of the backup generator.
3. The generator is self-testing (programmable). Prior to turning the generator on (i.e., testing it), the oil and gas level will be checked and the generator visually inspected for loose connections etc. Testing of the backup generator will consist of turning the generator on and running it for a minimum of 5 minutes.
4. If the generator performs satisfactorily, it will be placed back into self-testing mode. If the generator does not perform satisfactorily it will be repaired and then retested as soon as possible.

4.9 v 7

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RJ	3-27-19	

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SOP 4.10 v 7

Title: Calibration of various metering/measuring/miscellaneous devices.

Purpose: The purpose of this SOP is to establish procedures used when calibrating or initializing various instruments used in IR-4 field trials. It will also cover repair and/or calibration of devices not readily calibrated by the operator.

Scope: The SOP is to be followed by IR-4 participating personnel when calibrating equipment such as wind meters, global positioning systems, stopwatches, variable volume pipettes or other similar devices.

Procedures:

1. Equipment used in deriving data for entry in GLP studies should be adequately calibrated to ensure that the data provided is reasonably sound.
2. Records of these calibrations/corrective measures will be maintained in a log.
3. All equipment such as stop watches or wind meters may be sent back to the manufacturer at the prescribed interval set either by the manual, or appropriate authority (company information, technical support recommendation, etc.) or if possible calibrated by the IR-4 staff on-site. Units will be identified by a unique number, code, or name. The identification for purposes of maintaining logs.
4. Information confirming the certified calibration will be kept with the maintenance logs. If calibrated on-site, then record the date of calibration, the initials of person doing calibration, and the ID code number of the equipment being calibrated and/or checked.
5. In the case of global positioning systems, the manual should be consulted on the method of initialization. Unit will be re-initialized bi-annually and annotated in its logs. Battery changes or physical work done on the will also be annotated.
 - a. Coordinates and times should be correct on the GPS unit to be certain that the correct position is being used. Coordinates should be determined by either the manual, or other credible sources (i.e. government topographical maps, information services, aviation maps, etc.).
 - b. If possible, the closest certified GPS site should be consulted, as their initialization numbers may yield better positioning results.
The following information will be documented in the log:
 - a. date of calibration
 - b. initials of person doing calibration
 - c. ID code number of the meter being calibrated.
6. A smart phone GPS may be used.
 - a. Download GPS map for phone type (ie: iphone, android)

- b. Download google earth (GE)
- c. Turn on phone's GPS function.
- d. Record waypoint at a permanent marker.
- e. Send GPS data (waypoint to GE)
- f. Use GE earth ruler to compare accuracy.

7. If any unit becomes aberrant or unusable (outside the margin of generally $\pm 5\%$ of intended readings), then the unit should be flagged from trial use and returned to manufacturer for repair and/or retired from service.

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SOP 4.11 v 6

Title: Calibration of a backpack sprayer (Method #1)

Purpose: To determine the delivery rate of a backpack boom sprayer and make adjustments as necessary to ensure an accurate application of the pesticide. To describe a banded application using a backpack sprayer.

Scope: All facilities where a back pack sprayer is used in the application of pesticides.

Procedures:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary. Use a calibration method suitable to the application and the equipment used such as that accompanying the sprayer manual or a TeeJet catalog. If none is available then proceed with the following steps.
2. Refer to the protocol for any specified application requirements. Select the appropriate type of nozzle, which is based on the pesticide formulation, the application method, the operating speed, the pressure setting and the dilution rate (gal/acre).
3. All sprayers should have a pressure gauge. Select the proper operating pressure.
4. 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.
5. Measure or establish travel speed of application:

e.g., 3.0 mph = 4.8 km/hr = 4.4 ft/sec. Travel 66ft in 15 sec.
4.0 mph = 6.4 km/hr = 5.9ft/sec. Travel 88.5ft in 15 sec.
Check speed of application and adjust to conform to established rate. When using backpack sprayers use a metronome to ensure correct walking speed.
6. Measure and/or establish width between nozzles or width of band application. In most instances, this will be 16, 18, or 20 inches.
7. Establish and/or measure gallons per acre. Standard procedures and/or protocols dictate pre-determined gallon per acre in most cases. Calibrate to this pre-determined figure.

Example: Protocol calls for material to be applied in 30 gallons of water per acre. (Use 1.0 gallon = 3785 ml, 1 acre = 43,560 sq. ft)

$$\frac{30 \text{ gal}}{1 \text{ acre}} = \frac{113,550 \text{ ml}}{1 \text{ acre}} = \frac{113,550 \text{ ml}}{43,560 \text{ sq. ft}}$$

8. Using speed and width established in 5 and 6 calculate square feet treated in 15 seconds @ X mph using:

band width (in ft) X ft traveled = treated square feet.
 band width = 20 in. = 1.666666666 ft
 ft traveled/15sec @ 3 mph = 66 ft

Example: 1.67ft X 66ft = 110.22 ft²

9. Using figure obtained from step 7 above set up a simple proportion to determine needed output for the established GPA.

Example:

$$\frac{113,550 \text{ ml}}{43,560 \text{ sq ft}} = \frac{x \text{ ml}}{110.220\text{ft}^2}$$

$$x = 287.32 \text{ ml}$$

Solve for x to get correct output from each nozzle for the established GPA or determine GPA from calibration chart already established for these calculations.

If the calculated GPA is within 5% of the desired GPA, as specified in the protocol, then the sprayer is calibrated and the same settings should be used in actual application. When the GPA must be changed, alter one or all of the following; nozzles, pressure or speed. Minor flow rate changes can be made with a slight pressure change or speed alteration. Major flow rate changes require selection of new nozzle sizes.

10. Applicators must carefully operate under the same conditions as during calibration. Besides checking the nozzles for their proper flow rate, the spray pattern should also be examined for its 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.
11. Follow the protocol for the timing and frequency of calibration of application/spray equipment. If no specific timing and frequency for recalibration is given then use the following procedure.
- a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).

- b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed within one to two days of the application.
- c. Rechecking will consist of a) setting the proper operating pressure; b) placing the sprayer and/or person spraying on level ground; c) checking the nozzle spacing for the correct application pattern and; d) determining whether the nozzle(s) are discharging uniformly by spraying water through them at a uniform pressure for a set time period (e.g., 15 sec), catching the discharged water, and measuring its volume to determine if it falls within -5% to +10% of the target application rate determined during the previous calibration.
- d. Compare the output with the target output to determine if it is within $\pm 5\%$ of the target application rate. If it is within -5% to +10% of the target application rate then make the application, if it is not, recalibrate the device

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SOP 4.12 v 6

Title: Calibration of a backpack sprayer (Method #2)

Purpose: To describe method of application by use of backpack sprayer when applying to trees. The material to spray on each treated tree is contained in separate bottles.

Scope: All facilities where backpack or CO₂ individual bottle sprayer is used in the application of pesticides.

Procedures:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary. Use a calibration method suitable to the application and the equipment used such as that accompanying the sprayer manual or a TeeJet catalog. If none is available then proceed with the following:
 - 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 dilution rate (gal/acre).
2. All sprayers should have a pressure gauge. Select the proper operating pressure.
3. Establish and/or measure gallons per acre. Standard procedures and/or protocols dictate pre-determined gallon per acre in some cases. Determine acreage by using tree spacing's to determine sq. footage. Reduce total volume per acre to 'per tree' amounts (e.g. 100 gal/A, 100 trees per acre = 1 gallon per tree). Repeat this process in determination of material and/or additive materials (e.g. 100 ml/A material, 1 tree = 1 ml material per tree).
4. Backpack sprayer calibration:
 - a. Fill backpack sprayer with the calculated volume of material to spray 3 trees plus enough to charge the sprayer hoses etc. Select a boom with enough nozzles to provide adequate dispersion and coverage. Set the psi of the back pack to a setting that will provide good penetration of the canopy and foliage.
 - b. Use a tree of the same species and size as the test site trees.
 - c. Consider the tree to have four sides. Using a steady motion, sweeping, up and down motion, spray all sides evenly and record the time it takes. Repeat this process for 3 trees. Calculate the mean time it took to spray each tree.
 - d. Measure the volume remaining in the tank which should roughly correspond to the material needed to charge the system.
 - e. Calculate the amount of material sprayed per tree to see if it is within -5% to +10% of the target application rate. If not re-test application timing and or spray technique to fall within -5% to +10% of the target application rate.

5. Backback sprayer application:

- a. Fill backpack sprayer with the calculated volume of material to spray 3 to 4 trees plus enough to charge the sprayer hoses etc. Select a boom with enough nozzles to provide adequate dispersion and coverage. Set the psi of the back pack to a setting that will provide good penetration of the canopy and foliage.
- b. Consider the tree to have four sides. Using a steady motion, sweeping, up and down motion, spray all sides evenly and record the time it takes. Repeat this process for the 3 to 4 trees. If need be, mix more solution to spray the required number of trees for the trial.
- c. Time the application per tree based on that found for the calibration. Measure the volume remaining in the tank which should roughly correspond to the material needed to charge the system.
- d. Post spray, calculate the amount of material sprayed per tree to see if it is within -5% to +10% of the target application rate.

6. CO₂ individual bottle sprayer verification:

- a. Fill 3 individual bottles with the calculated amount of solution needed per tree.
- b. Select a boom with enough nozzles to provide adequate dispersion and coverage. Set the psi of the back pack to a setting that will provide good penetration of the canopy and foliage.
- c. Consider the tree to have four sides. Using a steady motion, sweeping, up and down motion, spray all sides evenly.
- d. Verification that there is sufficient material in the bottle and that the spray technique is good is indicated by the fact that the starting point of the spray application and ending of the spray application are adjacent to each other. For example if you consider your start of the spray application to be 0° your ending point would be at or close to 359° (i.e., nearly full circle).

7. CO₂ individual bottle sprayer application:

- e. Fill individual bottles with the calculated amount of solution needed per tree. The individual amounts for each tree will be placed in separate solution bottles, with one for each tree treated. The bottle will bear the Trial #, the substance name, and the volumes of material and water.
- f. Select a boom with enough nozzles to provide adequate dispersion and coverage. Set the psi of the back pack to a setting that will provide good penetration of the canopy and foliage.
- g. Consider the tree to have four sides. Using a steady motion, sweeping, up and down motion, spray all sides evenly.
- h. Be cognizant of the starting and ending point of each application for each tree. For example if you consider your start of the spray application to be 0° your ending point would be at or close to 359° (i.e., nearly full circle)

- i. After completing the spray from one bottle (it is empty now), uncouple the bottle from the CO₂ source, recap, and move to the next tree.
 - j. After completion of the application, gather bottles and clean according to SOP.
8. Applicators must carefully operate under the same conditions as during calibration. When using the pressurized tanks, shake them before starting to spray. The test substance must be applied uniformly to the entire test area.
9. Follow the protocol for the timing and frequency of calibration of application/spray equipment. If no specific timing and frequency for recalibration is given then follow the following procedure.
- a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).
 - b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed within one to two days of the application.
 - c. Rechecking will consist of a) setting the proper operating pressure; b) placing the sprayer and/or person spraying on level ground; c) checking the nozzle spacing for the correct application pattern and; d) determining whether the nozzle(s) are discharging uniformly by spraying water through them at a uniform pressure and observing the spray pattern.
 - d. If the spray pattern is observed to be uniform (i.e., not clogged and/or distorted) then make the application, if it is not, replace the nozzle(s), recheck spray pattern and if satisfactory, make the application

SOP 4.12 v 6

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SOP 4.13 v 6

Title: Calibration of a backpack sprayer (Method #3)

Purpose: To describe method of application by use of backpack sprayer when applying to trees and multiple trees are sprayed from each test substance bottle.

Scope: All facilities where backpack sprayer is used in the application of pesticides.

Procedures:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary. Use a calibration method suitable to the application and the equipment used such as that accompanying the sprayer manual or a TeeJet catalog. If none is available then proceed with the following:
 - 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 dilution rate (gal/acre).
2. Select a boom with enough nozzles to provide adequate dispersion and coverage. Set the psi of the back pack to a setting that will provide good penetration of the canopy and foliage.
3. All sprayers should have a pressure gauge. Select the proper operating pressure.
4. Establish and/or measure gallons per acre. Standard procedures and/or protocols dictate pre-determined gallon per acre in some cases. Determine acreage by using tree spacing's to determine sq. footage.

Example: 10 ft in-row spacing x 15 ft between-row spacing = 150 ft² per tree
150 ft² per tree x 8 treated trees = 1,200 ft²
1,200 ft² / 43, 560 ft²/acre = 0.028 acre

Protocol calls for 150 gpa: 150 gpa x 0.028 acre to treat = 4.13 gallons needed

5. Calculate amount required per tree (e.g. flow rate in step 5 determined to be 150ml/15 seconds). Catch output during a 10 or 15 second interval at least 3 times for calibration.

Using output per given time, calculate how long it would take for you to spray required amount onto tree, e.g., 300 ml of solution is required per tree (300 ml soln. x 15 sec./150 ml = 30 seconds of spray per tree). If the container holds at least 1,000 ml and it is filled with 900 ml for spraying 3 trees then time your application with a stopwatch or other chronometric device. Each tree will be sprayed with 300 ml solution in 30 sec: therefore

3 trees will be sprayed per bottle, to spray each tree will take 30 sec, and to empty the bottle takes 90 sec.

6. The bottle will bear the Trial #, the substance name, and the volumes of material and water.
7. Consider the tree to have four sides. Using a steady motion, sweeping, up and down motion, spray all sides evenly. Continue until the calculated time (step 6) is up then move onto the next tree(s), etc. until all solution in the bottle is expended.
8. After completing the spray of one bottle, uncouple the bottle from the CO₂ source, recap, and move to the bottle.
9. After completion of the application, gather bottles and clean according to SOP.
10. Applicators must carefully operate the sprayer under the same conditions (e.g., same pressure) as during calibration. When using the pressurized tanks, shake them before starting to spray. The test substance must be applied uniformly to the entire test area.
11. Follow the protocol for the timing and frequency of calibration of application/spray equipment. If no specific timing and frequency for recalibration is given then follow the following procedure.
 - a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).
 - b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed within one to two days of the application.
 - c. Rechecking will consist of a) setting the proper operating pressure; b) placing the sprayer and/or person spraying on level ground; c) checking the nozzle spacing for the correct application pattern and; d) determining whether the nozzle(s) are discharging uniformly by spraying water through them at a uniform pressure for a set time period (e.g., 15 sec), catching the discharged water, and measuring its volume to determine if it falls within -5% to +10% of the target application rate determined during the previous calibration.
 - d. Compare the output with the target output to determine if it is within -5% to +10% of the target application rate. If it is within $\pm 5\%$ of the target application rate then make the application, if it is not, recalibrate the device.

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SOP 4.14 v 6

Title: Calibration and use of granular applicators.

Purpose: To determine the delivery rate of the granular applicator and make adjustments as necessary to ensure an accurate application of the pesticide.

Scope: All facilities where a granular applicator is used in the application of test substances.

Procedures:

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 free of debris.
2. Refer to the manual for the calibration method. If no method is available then proceed as follows.
3. 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.
4. Measure an area of 0.01 acre or 435.6 square feet in close proximity to the area to be treated. A simple method to calculate the distance is:

$$\frac{435.6}{(\text{width of application in feet})} = \text{feet to travel}$$

5. Determine the approximate setting of the openings and the approximate speed to operate the applicator for the desired amount of active ingredient/acre.
6. Operate the applicator over the measured distance and collect the output in the pan attached to the spreader.
7. Weigh the material from the pan and multiply by 100 to give the amount applied per acre.
8. Continue with steps 5 to 7 until the desired rate is achieved within 5% of the total/acre.
9. Example: Formulation= 15% G., rate= 10 lb a.i./acre
10 lb a.i. = 67 lbs formulation/acre or 10 lbs a.i./0.15
width of applicator = 10 ft $435.6 \div 10 = 44$ ft to travel or $435.6/10$

With the applicator set at the appropriate opening and operated at 4 mph over the 44 ft you get a weighing of 70 lbs. This is within the 5% limit so the applicator is calibrated.

SOP 4.14 v 6

Reviewed by (initials)	Date	Comments
AJ	3-27-19	

University of Florida
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SOP 4.15 v 6

Title: Delivery method by soil drench.

Purpose: To describe several methods by which soil drench applications may be made, i.e., tractor mounted application by a hand gun and individual containers (e.g., bucket, watering can) for each treated tree.

Scope: All facilities where a soil drench is used in the application of pesticides.

Procedures:

1. Unless otherwise instructed by the protocol, the Field Research Director will determine where the material will be incorporated.
2. Use a clean container that holds sufficient water/pesticide mix. Or, in the case of tractor mounted application handgun, ensure that tank has been cleaned out thoroughly and is in proper condition.
3. Water volume will be determined by determining first, the total GPA. Then, use spacing's to calculate the sq. ft per/tree. This sq. footage will be used to determined acreage, and then, total gallonage to be used per tree. For example: 10'ft by 12 ft per tree = 120 ft² per tree. GPA is 500 gallons per acre by protocol. Gallonage to use is: $(120 \text{ ft}^2) \times (1 \text{ Acre}/43,560 \text{ ft}^2) = 0.0027548 \text{ Acres}$. Then $(0.0027548 \text{ Acres}) \times (500 \text{ Gallons/Acre}) = 1.377 \text{ gallons per tree}$.
4. If done by tractor mounted handgun, the flow rate of the handgun should be caught, averaged, and used to calculate amount of time at specific psi required to produce the gallonage per tree that was determined in part 3. The tractor mounted handgun should be rechecked for discharge accuracy (i.e., within -5% to +10% of the target application rate) just prior to application of the test substance.
5. Follow the protocol for the timing and frequency of calibration of application equipment. If no specific timing and frequency for recalibration is given then follow the following procedure.
 - a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).
 - b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed within one to two days of the application.

- c. Rechecking the tractor mounted hand gun will consist of a) setting the proper operating pressure; b) placing the sprayer and/or person spraying on level ground; c) checking the hand gun for the correct application pattern and; d) determining whether the hand gun is discharging uniformly by spraying water through them at a uniform pressure for a set time period (e.g., 15 sec), catching the discharged water, and measuring its volume to determine if it falls within $\pm 5\%$ of the target application rate determined during the previous calibration.
- d. Compare the output with the target output to determine if it is within -5% to $+10\%$ of the target application rate. If it is within -5% to $+10\%$ of the target application rate then make the application, if it is not, recalibrate the device.
6. If application is done with individual containers for each treated trees, the container should be adequate enough in size to accommodate the amount of solution required per tree in Step 3. Rechecking consists of making sure the container is of adequate size to accommodate the amount of solution required.
7. Apply solution in a relatively uniform pattern around the indicated area of application (i.e. root zone, outer canopy, any other place designated by FRD, etc.). Consider the tree to have 4 sides. Apply in sweeping motion evenly to designated area on each of those 4 sides until time is reached (handgun) or vessel is empty (container delivery).
8. Containers will be cleaned in accordance with SOP.

SOP 4.15 v 6

Reviewed by (initials)	Date	Comments
RJ	3-27-19	

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SOP 4.16 v 7

Title: Operation and maintenance of farm equipment.

Purpose: To assure that the farm equipment under the control of the Field Research Director is in good working order. If the equipment is under private or staff control the Field Research Director may request equipment be repaired or replaced prior to use in maintenance of the field trial.

Scope: All locations where the farming operations are performed for the IR-4 staff for GLP studies.

Procedures:

1. If the Field Research Director or his designated representative has reason to believe the equipment to be used in crop maintenance operations is not in proper working order, he/she will visually inspect the equipment to see that it is in good working order, properly lubricated, and in good mechanical condition or request the owner or staff person responsible to do this and make needed maintenance or repairs.
2. Any necessary repairs or adjustments should be made prior to the use of the equipment in the trial(s).
3. The operator of the equipment should be familiar with its operation and safety precautions.
4. Whenever possible, manuals on the operation and records of the 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/owner/staff personnel and/or the Field Research Director.
5. Written records should be maintained for equipment used for the generation, measurement, or assessment of data under the control of the IR-4 Director in a GLP trial. The record should contain maintenance service dates and what was done and repair dates and type of repair. When equipment is shared, i.e., not under the control of the FRD, this should be indicated in the Compliance Statement of the field data book.
6. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.
7. For farm equipment cleaning procedures see SOP 6.4

SOP 4.16 v 7

Reviewed by (Initials)	Date	Comments
RJ	3-27-19	

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SOP 4.17 v 6

Title: Calibration of field instruments.

Purpose: To assure that all instruments and gauges used in a GLP trial(s) are accurate and in good working order.

Scope: All facilities where trial(s) are conducted.

Procedures:

1. Each gauge or instrument used in a GLP trial (e.g. temperature and humidity gauges, photometers etc.) that is not annually sent for certified calibration should be tested to determine that it is within the desired tolerance. If item is used continuously, it should be tested frequently enough to assure its continued accuracy (e.g., annually, every six months, monthly, after every 10 hrs. use etc.). If the item is used infrequently, it should be tested before it is first used each year in a GLP trial and as often thereafter as necessary to assure its accuracy.
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. If a manual is not available to describe how these should be tested, then record methods used in the relevant log or describe in an SOP.
5. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

SOP 4.17 v6

Reviewed by (initials)	Date	Comments
RS	3-27-19	

University of Florida
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SOP 4.18 v 6

Title: Pesticide application output calculations for fruit trees using a tractor sprayer – PTO driven airblast sprayer.

Purpose: To provide a standard method for calculating the output of pesticide and spray material (water) for fruit tree crops. Data may be in English or metric units.

Scope: All laboratories conducting studies for the registration of pesticides.

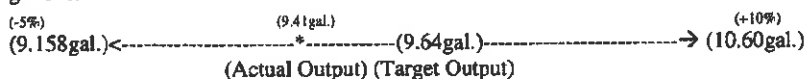
Procedures:

1. Determine acreage for treated area. This can be accomplished by determining the square footage per tree, which is (in row spacing) X (between row spacing) [e.g. (15ft)X(20ft) = 300ft²/tree]. Once the sq. ft per tree has been determined, multiply that number by the number of treated trees per plot [e.g. (300 ft²/tree)X(8 trees per treated plot) = 2,400ft² total for treated area]. Divide the resulting number by 43,560 ft² per acre to ascertain the acreage [e.g. (2400ft²) ÷ (43,560 ft²/Acre) = 0.0551 Acres]. The resulting figure is the treated acreage.
2. Consult the protocol to determine if a preset gallonage per acre has been given. If not, determine the gallonage that is most consistent with good agricultural practices for the particular crop being tested. After determining the GPA (gallons per acre), multiply this figure with the acreage as determined in step (1) [e.g. (0.0551 acres) X (175 gal/acre) = 9.64 gal H₂O to be used]. This amount is considered to be the "target amount" and represents the desired amount of soln to be distributed over the treatment area.
3. Determine the speed at which the application will be delivered. Convert the figure from mph/hr to ft/sec. (e.g., 1 mph = 88ft/60sec.; 2mph = 88ft/30sec.).
4. Determine the linear ft to be traveled during the application by measuring the in-row spacing per tree and multiplying by the number of trees [e.g. (15ft in row/tree)X(8 trees in treated area) = 120 ft linear]. The derived figure represents the distance to travel per pass. To apply uniform amounts to each side of trees, two passes are needed; one pass per side. To account for this, multiply the linear footage figure from the above example by two [e.g. (120 ft linear per pass)X(2 passes) = 240 ft total linear ft]. This will yield the total course length in linear feet.
5. Determine the time required to travel the total course length at the predetermined speed. This is done by multiplying the total linear ft by the speed [e.g. (240 ft total linear ft)X(30sec./88ft) = 81.8 seconds of operating time]. This will yield the Total Operating Time of the sprayer during the course length.

6. Fill the sprayer tank with a known amount of water (start vol.). Spray out a portion of this water using the number of nozzles and psi to be used in the trial, for a specific amount of time (e.g., 15 sec, 30 sec). Then measure and record what is left in the spray tank (final vol.). Do this 3 times for a calibration. Then subtract the initial amount (start vol.) of water in the tank (e.g., 10 gallons) from the amount left in the tank (final vol.) after test spraying (e.g., 6 gallons); this equals the amount sprayed (total sprayed vol.) during the specified time (e.g., 10 gallons initial - 6 gallons left in tank = 4 gallons sprayed in x seconds). This information then is used to calculate the amount of spray being applied per nozzle per second.

Another method for calibrating the spray applicator is to hook up tubing to each nozzle. Run sprayer at rpm used for operating speed and pressure to be used during the application (e.g., 200 psi). Collect and measure output from each nozzle. Calculate average output for each nozzle. Check for nozzle uniformity and calculate sprayer output. Using the output per minute per nozzle figure, determine the Total Output per Minute by multiplying the figure by the number of nozzles to be used [e.g. (0.46 gal/min/nozzle)X(15 nozzles) = 6.9 gal/min total].

7. Using the figures in steps (5) & (6), determine the gallonage that will actually be delivered to the treatment area by multiplying the Total Operating Time by the Total Output per Minute [e.g. (81.8 sec. of operating time)X(6.9 gal/60 sec. total output) = 9.41 gallons of H₂O actually delivered to treated area].
8. The acceptable range is described as being -5% to +10% of the "Target Amount" as described in step (2). If the actual amount derived in step (7) is found to be within the -5% to +10% limit, then the calculations are deemed valid, and acceptable for use in the field trial [e.g. Target Range in step (2.) is 9.64 gallons. Actual Output in step (7) is 9.41 gallons.



Actual output falls within the -5% to +10% range, and the calculations are within the acceptable parameters.

9. This method may be amended to accommodate different contingencies as they arise. If this is the case, the method shall be thoroughly written out in the raw data book.
10. Follow the protocol for the timing and frequency of calibration of application/spray equipment. If no specific timing and frequency for recalibration is given then follow the following procedure.

- a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).
- b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed within one to two days of the application. A recheck may consist of just one run.
- c. Rechecking may consist of a) filling the spray tank with water to a known point or to a known gallonage; b) setting the proper operating pressure; c) setting the rpm for the operating speed to be used during application; d) placing the sprayer on level ground; e) running the sprayer for a specific time period; f) refilling the spray tank with water to the known point or to a known gallonage and; g) calculating the output of the sprayer. Alternatively, rechecking may consist of one or more repetitions of the procedures in (6) and (7) above.
- d. Compare the output of the recheck with the target output to determine if it is within -5% to +10% of the target application rate. If it is within -5% to +10% of the target application rate then make the application, if it is not, recalibrate the device.

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SOP 4.19 v 6

Title: Pesticide application output calculations for fruit trees using a tractor sprayer handgun.

Purpose: To provide a standard method for calculating the output of pesticide and spray material (water) for fruit tree crops.

Scope: All laboratories conducting studies for the registration of pesticides.

Procedures:

1. Determine acreage for treated area. This can be accomplished by determining the square footage per tree, which is (in row spacing) X (between row spacing) [e.g. (15ft)X(20ft) = 300ft²/tree]. Once the sq. ft per tree has been determined, multiply that number by the number of treated trees per plot [e.g. (300 ft²/tree)X(8 trees per treated plot) = 2400ft² total for treated area]. Divide the resulting number by 43,560 ft² per acre to ascertain the acreage [e.g. (2400ft²) ÷ (43,560 ft²/Acre) = 0.0551 Acres]. The resulting figure is the treated acreage.
2. Consult the protocol to determine if a preset gallonage per acre has been given. If not, determine the gallonage that is most consistent with good agricultural practices for the particular crop being tested. After determining the GPA (gallons per acre), multiply this figure with the acreage as determined in step (1.) [e.g. (0.0551 acres) X (175 gal/acre) = 9.64 gal H₂O to be used]. This amount is considered to be the "target amount", and represents the desired amount of solution to be distributed over the treatment area.
3. Set the operational spray pressure (psi). Catch flow from handgun at 5-30 second intervals, measuring them out with a graduated cylinder and recording the amounts. Average them.
4. By determining the preset amount to be sprayed on per tree, and the output determined in step 2, determine the amount of time each tree needs to be sprayed at the calibrated psi.
5. Time by stopwatch or other chronometer.
6. Use sweeping motions and evenly disperse the amounts over the entire application area.
7. Follow the protocol for the timing and frequency of calibration of application/spray equipment. If no specific timing and frequency for recalibration is given then follow the following procedure.
 - a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).

- b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed within one to two days of the application.
- c. Rechecking will consist of repeating step (3.). If the output is within -5% to +10% of the calibration application rate then make the application. If output is not within -5% to +10% then recalibrate and then make the application.

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SOP 4.20 v 6

Title: Pesticide volume calculations for fruit trees.

Purpose: To provide a standard method for calculating the volume of pesticide and spray material (water) for fruit tree crops.

Scope: All laboratories conducting studies for the registration of pesticides.

Procedures:

1. Determine the treated acreage from the planting distance/spacing of the test site. This will be recorded in the site maps developed for each IR-4 pesticide trial.
2. Consult protocol to determine how the pesticide is to be measured out. If the protocol states that the desired rate is directly equivalent to a certain amount per sq. ft, then convert the sq. ft to acreage and compare against the acreage found in step (1) (e.g., The protocol directly denotes that 3 lbs. ai/treated acre equivalents to 4.5 fl. oz of the material per 1000 sq. ft, and 1000 sq. ft is $(1000 \text{ ft}^2 \div 43560 \text{ ft}^2/\text{acre})$ equal to 0.023 acres. The treated area from step (1) was determined to be (0.026 acres). So, $(0.026 \text{ acres}) \times (4.5 \text{ fl. oz}/0.023 \text{ acres}) = 5.09 \text{ fl. oz}$ of material for this acreage. Conversion to ml yields $(5.09 \text{ fl. oz}) \times (3785 \text{ ml per } 128 \text{ fl. oz}) = 150.5 \text{ ml material}$].
3. An alternate method may also be employed that derives the material amounts from the active ingredient per acre stated in the protocol. Consult the protocol to determine the ai/acre for the treated area.
4. Refer to the label of the test substance to determine the ai/gallon content of the material. After determining the composition of the material, use the equation below to ascertain the amount of material to be used:

$$\frac{(\text{treated acreage}) \times (\text{lbs ai/acre})}{(\text{lbs ai/gallon of material})} = \text{Amount Material to be Used}$$

(e.g., The treated acreage is (0.026 Acres). The protocol calls for 3 lbs ai/A. The label states that the material contains 2 lbs. ai/gal.). Using the equation yields...

$$\frac{(0.026 \text{ Acres}) \times (3 \text{ lbs ai/Acre})}{(2 \text{ lbs ai/gal.})} = 0.039 \text{ gal. material}$$

Which comes out as $(0.039 \text{ gal material}) \times (3785 \text{ ml}/1 \text{ gal.}) = 148.0 \text{ ml material to use}$.

5. Another alternative, when available, is to use the GPA (Gallons Per Acre) to determine pesticide amounts. This is available only when the protocol states the amount of material per gallon.
6. Where applicable, coverage shall be calculated to avoid running out of the spray soln. prior to completion of the application.
7. Determine the amount of extra H₂O needed by considering the amounts needed to charge application system, the possibility of accidental drainage, or any other considerations that would require extra amounts.
8. Once the amount of material needed to satisfy the protocol has been determined, couple this figure with the amount of actual output as determined in previous SOPs. For example, if it was determined that 52 ml of material were needed for the treatment area. The actual output of the sprayer during the application is 9.4 gallons of H₂O. It can be said that 52 ml material/9.4 gallons H₂O constitutes the protocol rate.
9. Increase the volume of H₂O to the necessary level (e.g., 12 gal H₂O) and compute the material amount by a ratio.

$$\frac{(52 \text{ ml of material})}{(9.4 \text{ gal. H}_2\text{O})} = \frac{(X \text{ ml of material})}{(12 \text{ gal. H}_2\text{O})}$$

$$X \text{ ml of material} = 66.4 \text{ ml of material}$$

10. The aforementioned methods may be amended to accommodate different contingencies As they arise. If this is the case, then the method shall be thoroughly written out in the raw data book.

SOP 4.21 v 8

Title: Determination of carrier volumes/gradient verification

Purpose: To provide guidelines for ensuring consistent volume in non-certified spray tanks or sprayers in the conduct of studies. If the spray tank is certified, no calibration is necessary; if not certified then follow one of the three calibration methods below.

Scope: All spray/mix tanks, or any other vessel used in the preparation of field application of test substances

Procedures:

1. Three methods are available for ensuring the consistent use of the same volume of water for field applications. These are: a. verification of existing gradients; b. use of water measuring stick, and; c. direct marking of water level. The first and second are used on large volume, non-disposable tanks. The third is used on disposable items only. For all methods, it is necessary to ensure that vessel is always level.
2. The Methods:
 - a. Verification of existing gradients:
 - (i.) Convert the gallons of the existing gradients to liters (i.e. 15gal. = 56.775 liters, 10gal. = 37.85 liters, 5gal. = 18.925, etc.). Identify the particular gradients already present and convert using the coefficient of 1 gallon = 3.785 liters = 3785 ml.
 - (ii.) Using a graduated cylinder, measure out these precise theoretic amounts, starting with the smallest and going to the largest in order.
 - (iii.) If the water level appears to be on the gradient in at least three (3) of the pre-existing gradients; then the gradients are considered reliable to use as a guide to filling for a field application.
 - b. Use of a water measuring stick:
 - (i.) Convert the gallons needed by calculations into the amount of liters. Use the same coefficient as stated in sect. (i.).
 - (ii.) Using a graduated cylinder, measure out the required water and mark with an indelible marker where the water line is on the measuring stick when the stick is put into the tank and firmly set against the bottom.
 - (iii.) Ensure that the stick is always on the bottom, and/or in the same place as it was when marking was done.
 - c. Direct marking of the water level:
 - (i.) Convert the gallons needed by calculations into the amount of liters. Use the same coefficient as stated in Part (a.) sect. (i.).
 - (ii.) Using a graduated cylinder, measure out the required water and mark with an indelible marker where the water line is on the inside or outside of the vessel.
 - (iii.) The vessel is now considered calibrated for this volume of water and can be used with certainty for filling other vessels.

- d. Calibration of 64 oz (1892.5 ml) pitcher for measuring carrier for spray tanks. Starting from the smallest amount to the largest, the precise theoretic amounts are added to a 64 oz/ 2000 ml pitcher. 2.5 ml in a 10 syringe with 0.5 ml increments, 90 ml in a 100 ml graduated cylinder with 1 ml increments, 1800 ml in a 2L graduated cylinder with 20 ml increments. A line is drawn on the pitcher with a permanent marker, so that 1892.5 ml can be measured for each half gallon of carrier.
3. At the completion of the field year, the measuring stick should be discarded. Similarly, the gradients must be recalibrated every year, or when circumstances may give cause for uncertainty as to the reliability them.
4. Verification of gradients will be documented in the appropriate logs.

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SOP 4.22 v 6

Title: Cleaning and maintenance guidelines for portable coolers/sample containers.

Purpose: To ensure that a proper cleaning routine is followed with regard to containers used for temporary sample storage.

Scope: All Containers used for the sampling, storage, shipping, or otherwise coming in contact with residue samples.

Procedures:

1. When cooling chests, transportation containers, or other reusable items are used for the handling and/or cooling of residue samples, they should be cleaned and inspected annually.
2. Once a year, the containers will be inspected for cracks, deficiencies, or anything that might jeopardize the integrity of the samples or ice packs that are placed inside.
3. A solution of soap and water should be used to clean the inside of the containers, and the Containers should be air dried and free of moisture prior to use.

4.22 v 6

Reviewed by (Initials)	Date	Comments
AJ	3-27-19	

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SOP 4.23 v7

Title: Storage freezers: cleaning and maintenance and remedial action if malfunction.

Purpose: To ensure that a proper cleaning routine is followed and a plan is in place if the freezer(s) malfunction.

Scope: All freezer storage used for the samples.

Procedures:

1. Sample storage freezer(s) will be cleaned annually. The following general procedure will be utilized:
 - a. Turn off freezer and allow to thaw.
 - b. Remove any accumulated water (either drain off or mop/sponge up).
 - c. Check rubber seals around the lid, if needed replace or repair.
 - d. Spray inside of freezer with dilute mixture of bleach and water.
 - e. Wipe down the sides and bottom of freezer and rinse with clean water.
 - f. Leave the lid open and let air dry until dry.
 - g. Hook freezer back up to electricity/turn on and allow to cool.
2. If one freezer malfunctions e.g., turns off and/or does not hold temperature, then transfer any samples from one freezer to the other functioning freezer. Be sure to separate treated and non-treated samples. Call for repair. Once repaired bring back on-line and place samples back into the freezer.
3. If the electricity at the testing facility goes out the backup generator will automatically turn on as programmed.

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SOP 4.24 v 1

Title: Calibration of battery powered electric backpack sprayer for a banded herbicide application.

Purpose: To determine the delivery rate of a battery powered electric backpack sprayer and make adjustments as necessary to ensure an accurate application of the pesticide. To describe a banded application using a backpack sprayer.

Scope: All facilities where an electric back pack sprayer is used in the application of pesticides.

Procedures:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary. Use a calibration method suitable to the application and the equipment used such as that accompanying the sprayer manual or a TeeJet catalog. If none is available then proceed with the following steps.
2. Refer to the protocol for any specified application requirements. Select the appropriate type of nozzle, which is based on the pesticide formulation, the application method, the operating speed, the pressure setting and the dilution rate (gal/acre).
3. All sprayers should have a pressure gauge.
4. For an herbicide application requiring a banded spray of 3 ft, measure the height from the nozzle to the ground which results in a 3 feet band of the spray applied. Either a low or high speed may be selected on the sprayer. But generally the low speed results in the best coverage on a banded application to avoid spraying the foliage.

5. Measure or establish travel speed of application:

e.g., 3.0 mph = 4.8 km/hr = 4.4 ft/sec. Travel 66ft in 15 sec.
4.0 mph = 6.4 km/hr = 5.9ft/sec. Travel 88.5ft in 15 sec.
Check speed of application and adjust to conform to established rate. When using backpack sprayers use a metronome to ensure correct walking speed.

6. Establish and/or measure gallons per acre. Standard procedures and/or protocols dictate pre-determined gallon per acre in most cases. Calibrate to this pre-determined figure.

Example: Protocol calls for material to be applied in 30 gallons of water per acre. (Use 1.0 gallon = 3785 ml, 1 acre = 43,560 sq. ft)

$$\frac{30 \text{ gal}}{1 \text{ acre}} = \frac{113,550 \text{ ml}}{1 \text{ acre}} = \frac{113,550 \text{ ml}}{43,560 \text{ sq. ft}}$$

7. Using speed and width established in 5 and 6 calculate square feet treated in 15 seconds @ X mph using:

band width (in ft) X ft traveled = treated square feet.
band width = 20 in. = 1.666666666 ft
ft traveled/15sec @ 3 mph = 66 ft

Example: 1.67ft X 66ft = 110.22 ft²

8. Using figure obtained from step 7 above set up a simple proportion to determine needed output for the established GPA.

Example:

$$\frac{113,550 \text{ ml}}{43,560 \text{ sq ft}} = \frac{x \text{ ml}}{110.220\text{ft}^2}$$

$$x = 287.32 \text{ ml}$$

Solve for x to get correct output from each nozzle for the established GPA or determine GPA from calibration chart already established for these calculations.

If the calculated GPA is within 5% of the desired GPA, as specified in the protocol, then the sprayer is calibrated and the same settings should be used in actual application. When the GPA must be changed, alter one or all of the following; nozzles, pressure or speed. Minor flow rate changes can be made with a slight pressure change or speed alteration. Major flow rate changes require selection of new nozzle sizes.

9. Applicators must carefully operate under the same conditions as during calibration. Besides checking the nozzles for their proper flow rate, the spray pattern should also be examined for its complete and symmetrical arrangement. Before the application, shake the tank before starting to spray. The test substance must be applied uniformly to the entire test area.
10. Follow the protocol for the timing and frequency of calibration of application/spray equipment. If no specific timing and frequency for recalibration is given then use the following procedure.
 - a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).

b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed within one to two days of the application.

c. Rechecking will consist of a) setting the proper operating pressure; b) placing the sprayer and/or person spraying on level ground; c) checking the nozzle spacing for the correct application pattern and; d) determining whether the nozzle(s) are discharging uniformly by spraying water through them at a uniform pressure for a set time period (e.g., 15 sec), catching the discharged water, and measuring its volume to determine if it falls within -5% to +10% of the target application rate determined during the previous calibration.

d. Compare the output with the target output to determine if it is within $\pm 5\%$ of the target application rate. If it is within -5% to +10% of the target application rate then make the application, if it is not, recalibrate the device

SOP 4.24 v 1

Reviewed by (initials)	Date	Comments
RJ	3-27-19	

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SOP 4.25 v 1

Title: Calibration of battery powered electric backpack sprayer for a foliar directed application.

Purpose: To determine the delivery rate of a battery powered electric backpack sprayer and make adjustments as necessary to ensure an accurate application of the pesticide. To describe a foliar application using an electric backpack sprayer.

Scope: All facilities where an electric back pack sprayer is used in the application of pesticides.

Procedures:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary. Use a calibration method suitable to the application and the equipment used such as that accompanying the sprayer manual or a TeeJet catalog. If none is available then proceed with the following steps.
2. Refer to the protocol for any specified application requirements. Select the appropriate type of nozzle, which is based on the pesticide formulation, the application method, the operating speed, the pressure setting and the dilution rate (gal/acre). The adjustable cone that accompanies the sprayer is usually nozzle is the best choice for a foliar application.
3. All sprayers should have a pressure gauge. Either a high or low speed may be selected, but generally a high speed results in the best coverage on a foliar application to ensure the best coverage.
4. Establish and measure gallons per acre. Standard procedures and/or protocols dictate pre-determined gallon per acre. Determine acreage by using tree spacings to determine square footage.

Example: 10 ft in row spacing x 15 ft between row spacing = 150 sq ft per tree.

150 sq ft per tree x 8 treated trees = 1,200 sq ft.

1,200 sq ft / 43,560 sq ft = 0.028 acre

Protocol calls for 150 gpa x 0.028 acre to treat = 4.13 gallons needed

5. Set the operational cone nozzle to the setting which which has the finest mist in order To coverage all of the foliage (ie: Papaya)
6. Establish the flow rate. Catch flow from the handgun at 15 second intervals 3 times. Time with a stopwatch. Measure the outputs in a graduated cylinder and the average them.

7. Determine the amount of time to spray each tree.

Gallons of carrier per plot/ number of trees per plot = Gallons / tree x 3785 = ml per tree

ml per tree/ discharge rate = Seconds per tree.

If the trees are too close to encircle while spraying, then they may be treated one side at a time. The total time to spray each tree would then be divided by 2 and then recorded as seconds per side.

8. Use sweeping motions to evenly disperse the amounts over the entire application area.

9. Rechecking the Discharge rate will consist of repeating step 6. If the output is within 5 % of the last full calibration, then make the next application. If the output is not within 5%, then a full calibration must be performed before making the next application.

10. Follow the protocol for the timing and frequency of application/spray equipment. If no specific timing and frequency for recalibration is given then follow the following procedures.

- a. Calibration of an application device/particular sprayer to be used in one or more field trials should be performed as per protocol or if not specified within the protocol may be performed within 14 days of application of test substance(s).
- b. However, to verify that the application device/particular sprayer is still properly calibrated (i.e., within -5% to +10% of the target application rate), a recheck of the equipment should be performed on the day of the application.
- c. Rechecking will consist of a) setting the proper operating pressure; b) placing the sprayer and/or person spraying on level ground; c) checking the nozzle for the correct application pattern and; d) determining whether the nozzle is discharging uniformly by spraying water through them at a uniform pressure for a set time period (e.g., 15 sec), catching the discharged water, and measuring its volume to determine if it falls within -5% to +10% of the target application rate determined during the previous calibration.
- d. Compare the output with the target output to determine if it is within -5% to +10% of the target application rate. If it is within $\pm 5\%$ of the target application rate then make the application, if it is not, recalibrate the device.

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SOP 5.1 v 6

Title: Commodity production and maintenance.

Purpose: Assure that commodities are grown under good management practices.

Scope: All locations developing data on vegetable, fruit, grain, oilseed or floral and nursery crops.

Procedures:

1. If the IR-4 staff is unfamiliar with the commodity being grown, they may refer to an up-to-date publication on the production of the commodity under trial(s). If they do, they should reference the source and add to the raw-data book. If no such publication exists, consult with cooperative agricultural specialist familiar with the production practices for the commodity and document the practices used to produce the commodity under simulated commercial conditions in the raw data notebook.
2. If not previously documented, determine pH, soil fertility, and soil characteristics requirements of the commodity. Obtain random samples of soil for testing from the trial(s) site. Have the soil tested to determine how well it will meet the requirements of the commodity (specify whether or not the testing was done under GLP in the raw data book.)
3. Lime, fertilize and/or condition the soil at the site as necessary to bring the soil within the requirements of the commodity.
4. Apply appropriate maintenance pesticides (preplant herbicide, soil insecticide, fungicide drench, soil-incorporated nematicide etc.) as required. Document maintenance chemicals in the field raw data notebook.
5. If pesticides are applied to the commodity to prevent losses due to pests not under trial(s), they should be applied according to the label directions. If this is a residue trial(s), no pesticide should be applied that would interfere with the chemical analysis of the pesticide under trial(s). If in doubt, consult the analytical chemist or analytical laboratory identified in the protocol to determine if a maintenance chemical may be used.
6. Perform other agricultural cultural practices as necessary to establish and maintain the commodity started.

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SOP 5.2 v 6

Title: Field preparation for seeding or transplanting.

Purpose: Assure that commodities are grown under good agricultural practices and provide a uniform crop for study.

Scope: All locations developing data on vegetable, fruit, grain, oilseed or floral and nursery crops.

Procedures:

1. If the IR-4 staff is unfamiliar with the commodity being grown, they may refer to an up-to-date publication on the production of the commodity under trial(s). If they do, they should reference the source and add to the raw-data book. If no such publication exists, consult with cooperative agricultural specialist familiar with the production practices for the commodity and document the practices used to produce the commodity under simulated commercial conditions in the raw data notebook.
2. If not previously document, determine pH, soil fertility, and soil characteristics requirements of the commodity. Have the soil tested to determine how well it will meet the requirements of the commodity (specify whether or not the testing was done under GLP in the raw data book.)
3. Lime, fertilize and/or condition the soil at the site as necessary to bring the soil reasonably within the requirements of the commodity.
4. Soil preparation of the field should mirror or follow local practices for the commodity.
5. Apply appropriate pesticides (preplant herbicide, soil insecticide, fungicide drench, soil-incorporated nematicide etc.). Apply and document application of pesticides as specified in other sections or subsections of these SOP's.

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SOP 5.3 v 6

Title: Method for seeding or transplanting.

Purpose: Assure that commodities are grown under good agricultural practices and provide a uniform crop for study.

Scope: All locations developing data on vegetable, fruit, grain, oilseed or floral and nursery crops.

Procedures:

1. Determine the correct species and variety to use as specified by the study protocol. If the variety is not specified, determine the variety most commonly used in the area by commercial producers and use it for the study. If a commercial producer is providing the plants, try to select plants as uniform in growth and color as possible.
2. Determine within and between row spacing and seed depth as specified in (name source, date) in protocol. Plant the seed or transplant in reasonably straight lines or rows with fairly accurate measurements to assure the commodity is planted according to specifications.
3. Identify each treatment row as to plot # and row # in such a manner so that it will be visual throughout the life of the study.
4. Irrigate or perform other agricultural practices as necessary to establish the commodity in the field.

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SOP 5.4 v 6

Title: Determining yield and quality.

Purpose: To assure that a measurement of yield or quality of the various treatments is taken if required to evaluate the effects of the treatments.

Scope: All locations conducting trial(s) where the protocol requires yield data. (Sample handling for residues is covered under another SOP).

Procedures:

1. Where possible, obtain an up-to-date copy of the United States standards for grades of the commodity under trial(s) from the Agricultural Marketing Service or other sources. If U.S. grade standards do not exist, then consult other sources and document the plant stage, fruit ripeness, or other characteristics needed to determine quality in the raw data notebook.
2. Check the protocol for information on time of harvest. If none, then follow commercial practices in the area for the time of harvest of the commodity. These practices should be documented in the raw data notebook.
3. Where grading standards are known or exist, the commodity should be graded accordingly at harvest to segregate the harvest to measure quality.
4. Each portion of the commodity, divided as to its quality standard, should be weighed or measured to determine yield. Written records should be kept of each measurement for each plot.
5. Various methods are utilized by various researchers to harvest a commodity. The method used if not specified in the protocol, should be recorded in the raw data notebook.

SOP 5.4 v 6

Reviewed by (Initials)	Date	Comments
AJ	3-27-19	

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SOP 6.1 v 6

Title: Adding a test substance concentrate to a carrier in the spray tank of a sprayer.

Purpose: To obtain the proper dilution and mixing of the concentrate in the spray tank.

Scope: All locations conducting field trial(s).

Procedures:

1. After the sprayer has been inspected and calibrated, empty the water from the tank.
2. Measure the amount of water needed to dilute the measured amount of concentrate into the spray tank or into a separate container. Make sure the spray mix will be enough to cover at least one treatment plot (preferably all plots in one treatment). Make sure the spray tank will hold the needed amount for application. Follow protocol for mixing instructions. If none is stated add up to the required water to the spray tank.
3. If needed (i.e. wettable powder formulation) make a slurry mix first by adding the concentrate to a small volume of water in a separate, clean container. Add the pesticide concentrate or slurry to the water in the spray tank. If needed or stated in the protocol triple rinse the container holding the pesticide concentrate (and slurry) using the other 1/2 of the water not in the spray tank and add the rinse water to the spray tank.
4. Add the remaining water to the spray tank. Close and tighten the lid.
5. If possible, agitate the spray mix before and during application to insure an even mix of the pesticide and water.

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SOP 6.2 v 6

Title: General procedures in the application of pesticides.

Purpose: To describe the practices common to all types of pesticide application.

Scope: All locations where studies are conducted in the field.

Procedures:

1. All personnel involved in the mixing, application, storage and cleanup of pesticides should be properly trained in accordance with current policies and guidelines of their institution.
2. Equipment used in the application of the pesticides should be inspected and calibrated as indicated under SOPs.
3. Personnel mixing and applying the pesticide should wear appropriate protective clothing as stated on the pesticide label or as indicated under SOPs.
4. The pesticide concentrate should be measured out as indicated under SOP's for a dry or liquid concentrate.
5. If the pesticide application is for maintenance of the plots, then apply the pesticide to all the plots in the study according to the directions on the pesticide label.
6. If the pesticide application involves the test substance, then procedures for handling the test substance as indicated in SOP should also be followed.

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SOP 6.3 v 6

Title: Procedures for the application of the trial(s) pesticide(s) in the field and greenhouse.

Purpose: To assure that the trial(s) pesticide(s) are applied uniformly to the plots.

Scope: All locations conducting field trial(s)

Procedures:

1. Ensure all settings of pressure, speed, granular flow etc. are set according to specification from the calibration as previously performed.
2. Just before entering each plot make sure you are traveling at the correct speed and turn on the sprayer or release the granules. Maintain the correct speed through the plot.
3. Apply the material according to the directions in the protocol or as specified on the label. If fumigants or mist blowers are used, follow instructions of the manufacturer of the equipment. If a fumigant is used, two people are required, one doing the actual application and one who can observe from a safe place to provide rescue assistance if necessary.
4. Calculations should be made to minimize the amount of spray material left in the spray equipment. This residue should be sprayed to a similar crop or disposed of according to current policies and guidelines of the research testing facility.
5. Where possible, apply the material beginning with the lowest concentration and work up to the highest concentration.

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SOP 6.4 v 7

Title: Cleanup of application equipment.

Purpose: To assure that pesticide application equipment is decontaminated without adversely affecting personnel or the environment.

Scope: All locations where pesticides are used.

Procedures:

1. Cleaning of equipment will be recorded in a maintenance log separately for each type of application equipment.
2. For granules, 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 in the pesticide log for the chemical, the amount of granular material used in the trial(s). 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.
3. For airblast, hand-gun, and backpack sprayers, unused spray material may be applied to an overplanting of the crop or to an open field with grass/weed vegetation at a distance adequate to prevent contamination of the test plot by drift or down slope movement of water. Triple wash sprayers and apply each wash to an overplanting of the crop or open field with grass/weed vegetation.
 - a. After the last application of a trial, or if the sprayer is used for multiple trials, and different chemicals in the same time period, the sprayer will be cleaned with a commercial sprayer cleaner.
4. If a crop overplanting is not available, then follow the disposal procedures for pesticide rinse water in accordance with current policies and guidelines of the institution.
5. Dispose of expendable protective clothing by placing the items in a container for incineration. Clean non-disposable items following the manufacturer's instructions or with water as appropriate.
6. If necessary, after the application equipment is dry, lubricate those parts requiring lubrication and return the equipment to storage.

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SOP 6.5 v 7

Title: Handling the test substance.

Purpose: To explain the procedures required in the receipt, removal, use, return and transfer of the test control and reference substances.

Scope: All locations where pesticides are used. For the purposes of this SOP, test substance also applies to control and reference substances.

Procedures:

1. When the test substance is received, the EPA registration number, lot/batch number, source, quantity, date of receipt, condition of the material, and storage location should be recorded in the raw data notebook. Each entry should be initialed and dated. The protocol number(s) may be recorded on the container.
2. The test substance should be stored in the pesticide storage facility until it is needed for use in the trial(s).
3. The storage temperatures of the test substance should be recorded in the raw data notebook.
4. When a test substance is used, the date, amount used, purpose of use, and initial/signature of the user should be recorded in the raw data notebook.
5. All test substance containers must be stored until notification by the study director that the containers may be discarded, unless returned to the registrant or sponsor.
6. Separate areas should be established for mixing and handling of the test substance and for storage of test substance mixtures.
7. Test substances and mixtures should be stored in a manner to prevent any possibility of contamination, deterioration, or damage during the conduct of the study. The test substance label should be consulted and followed for storage conditions.

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SOP 6.6 v 6

Title: Procedures to follow when a problem occurs in the application of the test substance.

Purpose: To explain the procedures required when something goes wrong during the application of the test substance in the trial(s).

Scope: All locations where pesticides are used. For the purposes of this SOP, test substance also applies to control and reference substances.

Procedures:

1. During application, the operator should observe the process to make sure that the test substance is being evenly distributed to the commodity.
2. If something goes wrong, for example, a nozzle is plugged or a hose breaks, 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. This portion should not be used for obtaining samples of the commodity for residue analysis. If the unaffected area is too small to obtain the samples required for analysis, then the trial(s) should be discontinued.
4. Appropriate individuals (e.g., the regional field coordinator, and the study director) should be notified of the incident, details recorded in the raw data notebook, and maintenance records retained in the assigned log.

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SOP 7.1 v 6

Title: Collection of raw data electronically.

Purpose: To assure that raw data collected electronically are verifiable if audited.

Scope: All locations conducting field or greenhouse trial(s).

Procedures:

1. Check the power supply on portable units to see that it will be adequate during the data collection and data transfer period.
2. Make sure the correct program for data collection is ready and available for use.
3. At the end of the data collection period, the data may be transferred to a storage system and/or immediately printed out with appropriate identification.
4. All remote sensing and other automatic data collecting and/or recording devices should be inspected and calibrated.
5. Prints or plots of data from these devices must be legible to persons with normal vision and dated and signed when printed or plotted.
6. Hard copies of computerized data and/or other written or plotted data sheets must be dated and signed, and retained in the file folder of the project.
7. If the data does not automatically provide output with the day, month, and year of occurrence, then each data sheet from a monitoring device should be marked in ink with the name of the dates (day, month, and year) of occurrence of the event measured, units of measurement and signed and dated by the person preparing the data sheet.

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SOP 7.2 v 6

Title: Recording of raw data.

Purpose: To assure that raw data collected and recorded are accurate and available for audit.

Scope: All locations conducting trial(s).

Procedures:

1. All raw data will be recorded in indelible ink.
2. Changes to the raw data can only be made by drawing a single line through the original entry so as not to obscure it. The date, signature (or initials) and reasons for change (brief description or error code) must accompany any change. Acceptable error codes include:

ME = Measurement Error	CE = Calculation Error
SP = Spelling Error	EE = Entry Error
WE = Wrong Entry	LE = Late entry
IE = Illegible Entry	IW = Inappropriate Word
TE = Transcription Error	AW = Accidental Write over
UE = Unnecessary Entry	PE = Pagination Error
NA = Not applicable	NR = Not Recorded
NI = New Information	

Other error codes can be used, however, the codes must be noted in the IR-4 FDB.

3. Pages containing raw data shall not be discarded.
4. Cross-reference instrument or statistical printouts when such data are retained in a separate location.
5. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data.
6. Make sure that all data required by the study protocol or by the forms provided in the field data book are collected and recorded.

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SOP 7.3 v 6

Title: Calculations for data presentation.

Purpose: To establish guidelines for computation and presentation of data.

Scope: Field sites conducting trials under the minor use pesticide program.

Procedures:

1. Results must be reported to correct number of significant figures reflecting an appropriate level of certainty.
2. In carrying measured quantities through calculations, the following rules are used:
 - a. Multiplication and division: the result should be round off as having no more significant figures than the measurement with the fewest significant figures.
 - b. Addition and subtraction: the result should be rounded off to the same number of decimal places as that of the term with the least number of decimal places.
3. Round-off rules:
 - a. If the first digit to be dropped is less than 5, round down.
 - b. If the first digit to be dropped is 5 or greater than 5, round up.
4. When a manual calculation involves two or more steps, you may retain at least one additional digit (insignificant figure) for intermediate answers. Round off at the last step in the calculations (end).
5. In using computer and/or calculator, calculation round off is usually done at the display and serial calculations are done with non-rounded numbers. You may appropriately round off (step 3 above) intermediate steps and the final results.

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SOP 7.4 v 6

Title: Method for collecting efficacy and phytotoxicity data.

Purpose: To describe the procedure used for taking biological field data.

Scope: All locations conducting field studies.

Procedures:

A. Phytotoxicity data:

1. Consult the protocol to determine the method and timing of the phytotoxicity data. If no method is cited then reference your method or proceed as follows:
 - a. Where possible, take phytotoxicity data within 24 hours before the initial pesticide treatment and within 48 hours after the treatment, 1 week later and at the termination of the study.
 - b. If symptoms occur during this period that warrant a reading, then additional phytotoxicity data should be taken as necessary.
2. Randomly select plants in the middle row of each plot and record a phytotoxicity rating of 0 to 5 for each plant with zero= plant healthy and five= plant dead.

B. Pest data:

1. Consult the protocol to determine the method and timing of the pest data. If no method is cited then reference your method(s) for each pest or proceed as follows:
 - a. Where possible, take pest data within 24 hours before the initial pesticide treatment and within 48 hours after the treatment and at various intervals thereafter depending on the pest life cycle and at the termination of the study.
2. Disease data: record the name of the disease(s) being observed. Record the symptom(s) for each disease. Randomly select plants in the middle row of each plot and record the severity of each disease in a rating system of 0 to 10 for each plant with zero= plant healthy and ten= plant dead.
3. Insect data: record the name of the insect(s) being observed. Record the damage symptom(s) for each insect. For damage symptoms-randomly select plants in the middle

row of each plot and record the severity of damage for each insect in a rating system of 0 to 10 for each plant with zero= plant healthy and ten= plant dead.

For insect pest population counts-take a random sample of the pest population (i.e. 5 leaves/plant of 5 plants/plot, 4 3-in dia. soil cores/plot, 100 apples/tree etc.) to insure an accurate reflection of the pest density/unit area.

4. Nematode data: record the name of the nematode(s) being observed. Record the damage symptom(s) for each nematode. For damage symptoms-randomly select 10 plants in the middle row of each plot and record the severity of damage for each nematode on each plant using one of the rating systems described by the following:

Barker, K.R., J.L. Townshend, G.W. Bird, I.J. Thomason and D.W. Dickson. 1986. Determining nematode population responses to control agents. In Kickey, K.D. (ed.). Methods for evaluating pesticides for control of Plant Pathogens. pages 283-296.

For nematode population counts-take a random sample of the pest population (i.e. root system of 2 plants/plot, 4 3-in dia. soil cores/plot, etc.) to insure an accurate reflection of the pest density/unit area as described by Barker et. al. cited above. Use a method suitable to extract the nematodes from the soil or plant sample and cite the method here. Count and record the number of nematodes by the various life stages/unit of soil or root.

5. Weed data: visually observe each plot and record the % of the area (to the nearest 5%) covered by weeds. Record the names of the 5 most prominent weed species and the area they cover (to the nearest 5%) in each plot. Randomly place a grid covering an area of 0.1 m² and divided by quadrants in the plot. Where possible, count the number of weeds in the grid. If weeds are too numerous to make counting the entire area possible within a reasonable period of time, then count the number of weeds in the lower left quadrant, multiply by 4 and record this value as the number of weeds in the grid.

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SOP 7.5 v 6

Title: Experimental design and data analysis.

Purpose: To assure that all efficacy, yield, and phytotoxicity data developed are statistically sound. In general, the protocols and trials are not designed nor set-up for statistical analysis. Each field trial is usually considered as one replication.

Scope: All locations conducting trial(s).

Procedures:

1. The experimental design as specified by the protocol should be used. If none is designated, then either the researcher should use a commonly accepted experimental design such as a completely randomized block design or set up so that non-treated control and treated plots are separated to avoid cross contamination of the non-treated control plots. The experimental design used should be documented in the raw data notebook.
2. A minimum of 3 replicates should be used (4 is preferred). No replicates or statistical analysis are required where the trial(s) is for magnitude of the residue only. For trials where efficacy and/or phytotoxicity data is collected then means may be calculated.
3. Draw a plot map showing the location of each plot in the site selected for testing.
4. If the experiment is to be statistically analyzed, assign the treatments to the plots using a random number table or random number generator. If the individual experiment or trial is not for statistical analysis then assign treatments set up so that non-treated control and treated plots are separated to avoid cross contamination of the non-treated control plots. Note the location of the treatments on the plot map.
5. Retain the plot map in the trial(s) folder.
6. Determine the level of significance for the trial(s).
7. Select an appropriate statistical package for data analysis and record sufficient information to identify the statistical package (i.e. Date, Revision no., Title, Authors, and Source etc.).
8. When the raw data are available for analysis, utilize the statistical package and follow instructions contained therein to conduct an analysis of variance and mean separation of the data.
9. Record the data as required on the appropriate forms and identify statistically significant differences in the data in the raw data note book.
10. Retain all data, analyses, notes etc. in the trial(s) folder with sufficient information to recalculate the data summaries and statistical analyses by another person without verbal input.

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SOP 7.6 v 6

Title: Data storage during the active life of the project.

Purpose: To assure that all data resulting from the trial(s) is retained and usable.

Scope: All locations conducting trial(s).

Procedures:

1. It is the responsibility of the Field Research Director and his/her technical staff to see that all raw data, summaries and other items connected with the trial(s) are properly retained prior to sending the data to IR-4 Headquarters and/or the Study Director for archiving.
2. The Field Research Director will see that a separate file or notebook containing all raw data, summaries, data logs, etc. connected with the trial(s) is maintained during the active life of each project for which he/she is responsible.
3. Dated and signed hard copies of electronic data, computerized summaries etc. should be placed in the file or field data notebook after the information is generated.
4. All notebooks, data sheets, summaries etc. should be clearly marked with the name of the project, project identification number, dates generated, name of investigator and other information that may be needed to understand the data and its source.
5. Computer generated raw data will be dated and initialed at the time of printing.

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SOP 8.1 v 7

Title: Sample collection, identification, and records.

Purpose: To assure proper collection and identification of residue samples.

Scope: At locations where trial(s) are conducted to obtain residue samples.

Procedures:

1. Consult the study protocol to establish specific dates and method 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 should be kept informed when the dates are changed.
2. Samples should not be taken during periods of inclement weather.
3. Representative samples of the crop in each plot must be taken by a recognized procedure. Follow the protocol or record in the data book the procedure used to ensure a representative sample.
4. Consult the study protocol to determine sample size and special instructions for the commodity.
5. Sample each replicate individually beginning with the nontreated plots and working up 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 samples with the test substance during the sampling, labeling, storage and shipping processes.
 - b. Avoid taking diseased or undersized crop parts.
 - c. Take care not to remove surface residues during handling, packing or preparation.
 - d. Be certain tools are clean.
 - e. Do not remove any soil or plant parts or trim the commodity unless it is so specified in the study protocol (leave stem in cherry, leave outer leaves of lettuce, etc. unless specified otherwise in the protocol.)
7. Plastic-lined cloth sampling bags with an identification tag sewn into the bottom stitching are usually provided to GLP cooperators for sample collection. If these bags have not been provided, a sampling bag suitable to protect the integrity of the sample should be used.

8. Prior to sample collection, obtain a sufficient number of sample bags to collect all the samples. Store the treatments separately by individual replicates.
9. Before entering the field, fill in the label attached to the bottom of the bag and indicate the study ID number on the tag. If no tag has been provided, then label each sample bag with with the following:
 - a. Field ID Number
 - b. Crop fraction
 - c. Test substance
 - d. Sample ID
 - e. Treatment no.
 - f. Harvest date
 - g. Sample date
 - h. Name of Field Research Director
 - i. Telephone number
10. All items listed in no. 9 should also be written in permanent marker on the outside of the sample bag, in the event that the bag tag becomes lost.
11. Sample bags will be provided by IR-4 headquarters or the regional office. They should be burst proof. Cloth laminated plastic bags are preferred.
12. Upon completion of the sampling, GLP shipping form(s) should be completed. The form should be signed and dated by the Field Research Director or person entering the data. Retain the original of the residue sample shipping form in the project file folder until the samples are shipped to the residue laboratory.

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SOP 8.2 v 6

Title: Reducing residue sample weight/size by cutting.

Purpose: To assure the integrity of the residue samples that are reduced in weight and/or size after collection (harvest).

Scope: All locations where residue samples are collected.

Procedures:

1. Consult the study protocol for any postharvest handling request and/or requirements of residue samples.
2. Follow the protocol and/or record in the data book the procedure used to reduce the weight and/or size of the residue samples and to ensure integrity of those samples.
3. If the protocol does not specify the order in which the samples are to be reduced in weight and/or size begin with the non-treated controls first, then the 1x, then the 2x, etc. Sample each replicate individually beginning with the nontreated plots and working up to the highest dosage. Treatments from each replicate should be individually packaged and labeled.
4. Take special care to do the following in the sample reduction process:
 - a. Avoid contamination of the non-treated field samples with the test substance during the sample weight and/or size reduction (e.g., cutting) process.
 - b. Take care not to remove surface residues during handling and weight and/or size reduction process. Gloves should be worn and change gloves and clean cutting implement(s) and cutting surface area between samples.
 - c. Do not remove any soil or plant parts or trim the commodity unless it is so specified in the study protocol (e.g., leave outer leaves of lettuce.)
 - d. Be careful to keep track of residue samples, i.e., remove harvested sample from plastic-bags one sample at a time, reduce sample in weight and/or size by cutting of the fruit from that sample, and then replace the reduced sample into the original sample bag. Reduced samples may be placed in Zip Loc plastic bags and then placed in the residue sample bags.

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SOP 8.3 v 6

Title: Packing and storage procedures.

Purpose: To assure the integrity of the samples after collection.

Scope: All locations where residue samples are collected.

Procedures:

1. If samples require refrigeration or freezing prior to movement to storage freezers and/or shipping to the residue laboratory, then containers with ice or dry ice in sufficient quantity to preserve the samples prior to storage should be taken to the site. Otherwise cartons or other suitable temporary boxes (e.g., cooler) of sufficient size and burst proof strength to hold the samples should be used.
2. Carefully place the sample as it is collected in the sample bag marked for that sample. Make sure that the labeling in the bag is enclosed with the sample.
3. Close the sample bag so as to prevent loss of the sample under reasonable storage, handling, and transportation conditions. Excess air should be expelled from the bag.
4. Place the sample bag in the appropriate container as determined in # 1 above. Physically separate treated and nontreated samples.
5. When sample collection is completed, the samples should be returned from the field to be placed in storage.
6. Consult the study protocol for the method, temperature, and maximum length of time for storage.
7. Samples identified for post-harvest processing should be processed or shipped to the processor as soon after collection as possible.
8. The storage temperature of the samples should be recorded frequently enough to ensure that the temperature is 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. It is preferred that a continuous recorder be used to monitor the temperature of the stored samples.
9. The refrigerator, freezer, or room where the samples are stored should be under lock and key with limited access and, where possible, only be used to store GLP samples.

10. Attached to the storage facility (i.e. freezer, refrigerator etc.) should be a log of the items inside indicating the Field ID No., Date samples collected, and Number of sample bags for each project. Removal of the samples prior to shipment should be recorded on the log sheet as to the name of the person removing them, what sample bags or parts thereof where removed, date removed and date returned.
11. Freezer temperature does not have to be recorded during time when no residue samples are being stored.

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SOP 8.4 v 6

Title: Sample shipping procedures.

Purpose: To assure that residue samples are removed from storage and shipped to the residue laboratory with a minimum loss of integrity.

Scope: All locations where residue samples are stored.

Procedures:

1. If possible, one week before the samples are to be shipped, contact (e.g., telephone, fax, email) the chemist at the residue laboratory and notify him/her of the Pr. No., shipment dates and method of shipment including the carrier. Ask for any special instructions in shipping the samples. Air freight shipments should be made on Monday or Tuesday to avoid potential weekend layovers, and shipment during holidays should be avoided.
2. Complete residue sample shipping form(s), make copies and send or fax them to the study director, regional coordinator and residue chemist.
3. Make arrangements with the carrier for shipment of the samples and determine any special packing instructions etc. that is required to preserve the sample integrity. Some carriers require dry ice, note any limits on quantity of dry ice etc. that may be set by the carrier.
4. Obtain insulated containers, if necessary, of sufficient size and quantity to hold the residue samples and if necessary, dry ice in a 1:2 to 1:5 weight ratio (sample:dry ice) to commodity. 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. Place the copy of the residue sample shipping form in a waterproof container and place it in one of the sample shipping containers.
6. Label each container with the following information:
 - a. Study Identification Number, Pesticide, and Commodity
 - b. Return Name and Address of the sender
 - c. Name and Address of the residue laboratory receiving the samples
 - d. Number of the container if more than one is used
 - e. If being shipped via FedEx or UPS, affix "Experimental Samples-Perishable" on each carton
 - f. Where used, affix "Dry Ice" on two sides of the container(USE FEDEX DRY ICE LABEL)

- g. When appropriate, label as box ___ of ___
7. Tie or tape lids of each container firmly in place.
 8. 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.
 9. Provide the carrier with the samples for shipment.

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SOP 9.1 v 7

Title: Raw data report forms.

Purpose: To review the forms used to report raw data.

Scope: All locations conducting trial(s)

Procedures:

1. IR-4 Headquarters provides a field data book to each of the cooperators for each trial undertaken. Detailed instructions are provided in the book.
2. All forms should be filled out legibly and mistakes should be crossed with a single line, initialed, dated, and the reason for change given.
3. Blank forms may be photocopied as needed.
4. The reporting forms as provided in the field data book should be filled out as completely as possible at the time the data is collected.
5. Each location should use the forms provided or develop new forms where needed. The new forms should be placed in the field data book.
6. If a particular form or section of the form does not require a response, make a slashed line (diagonal line from the top of the page or field to the bottom). Initial and date on the slashed line or sign and date at the bottom of the page.
7. If data are recorded elsewhere, other than the raw-data book, date and sign data entries, and include in the data book.

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SOP 9.2 v 6

Title: Handling completed report forms that transcend two or more trial(s).

Purpose: To explain how report forms can be completed for one trial(s) and serve as raw data for other trial(s).

Scope: All locations conducting trial(s).

Background: Where a Field Research Director is conducting multiple trial(s) during the year, there may be an opportunity to utilize one form for data that pertains to more than one trial(s). Examples may be logs of various types such as weather data and sprayer calibration/pesticide application where the same test substance is used at the same time at the same rates on two or more crops (i.e. preplant herbicide, foliar insecticide). There are provisions within the GLPs for substitution of verified copies for original records. However, the retention of all original raw data is also a requirement. The following procedures are designed to meet the GLP and FIFRA requirements where copies of data are used.

Procedures:

1. Each form that is to be used for data common to more than one trial(s) should contain a notation at the bottom of the form as to the trial that has been designated as the one containing the raw data. This should read: "The original is in IR-4 field data book No. _____".
2. When the form is completed, it should be photocopied. Each copy should contain a notation that this is a true copy. The copy should be signed, dated and placed in the field data books for the other trial(s) that utilize the same data.

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SOP 9.3 v 7

Title: Disposition of raw data from the trial(s).

Purpose: To assure that raw data are sent to the archives. To provide for the testing site to retain a copy of the original raw data, at their option.

Scope: All locations conducting trial(s) where the original raw data is not archived at IR-4 Headquarters.

Procedures:

1. The Field Research Director may make a copy of the original raw data including completed field data books, correspondence, protocol deviation forms, logs, and any accompanying documentation such as weather charts etc. The original raw data and true copies of site specific documents from the trial(s) should be forwarded by the Field Research Director to the Regional Field Coordinator. After review (i.e., Regional Coordinator, QA) the data may then be submitted to the IR-4 Headquarters and/or the Study Director.
2. The Regional Field Coordinator or designee will review the documents received in #1 for completeness and accuracy of reporting. The Field Coordinator or designee will follow up to obtain any missing data or correct deficiencies.
3. The quality control team may place a copy of the raw data from #1 in folders or notebooks. Copies of field data notebooks and other pertinent trial records will be retained at the field facility.

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SOP 9.4 v 8

Title: Retention of data.

Purpose: To assure that each location conducting trial(s) retains all data and documents connected with the trial(s) until the trial (s) is completed and then forwards any raw data on to the Regional Coordinator and/or IR-4 Headquarters for archiving.

Scope: All locations conducting trial(s).

Procedures:

1. The Field Research Director will see that the trial(s) file containing the raw data, originals or true copies of reports, logs, etc. are submitted to Headquarters for archiving and a copy of site specific documents are retained at the field facility to assure that raw data is not lost.
2. The Field Research Director should maintain appropriate files with the copies of data or place copies of trial-specific data in the Field Centers copies of the trials. The following is a list of information that may be retained:
 - a. Copies of raw data including pest counts, yield, phytotoxicity, weather records, logs of instrument calibration and test substance receipt, distribution, etc.
 - b. Copies of summaries including calculations and copies of information used from referenced sources.
 - c. Copies of reports and correspondence related to the conduct of the trial(s).
 - d. Copies of completed forms used during the trial(s) and for summaries of the trial(s) data.
 - e. Historical Standard Operating Procedures.
 - f. Master schedule of all GLP trial(s) conducted at the facility.
 - g. Organizational charts, training records, job descriptions and CVs (current, out of date, or former employees).
 - h. Copies of equipment maintenance logs.
 - i. Any samples as required by the study protocol or the Study Director.

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SOP 10.1 v 6

Title: Disposal of pesticides.

Purpose: To assure that pesticide concentrate, spray solutions, rinse water, and containers are disposed of with minimal environmental contamination and in accordance with federal, state and local regulations.

Scope: All locations conducting field trial(s).

Procedures:

1. Where institutional policies and guidelines do not exist, the following procedures should be followed:
 - a. Follow procedures in the protocol. Generally, containers cannot be disposed of under GLP until the study is completed. If it is necessary to dispose of the container prior to the end of the trial(s), the Study Director should be consulted.
 - b. Where possible, the pesticide concentrate and containers should be returned to the registrant or manufacturer. Transportation must be according to all Federal, State, and local laws and regulations.
 - c. Follow label directions for use or disposal of the pesticide if option 2.b is not available.
 - d. If no label directions exist for disposal, arrangements should be made with a licensed waste disposal firm for pickup and disposal of the pesticide and/or the empty containers.
2. Disposal of pesticide rinse water, unused spray solutions and other dilute pesticide waste after a test application.
 - a. Check State and local laws and regulations to determine any procedures that may exist for proper disposal of pesticide solutions.
 - b. Dispose of the dilute pesticide waste in the field by adding carrier (e.g., water) to the spray tank and spraying on an overplanting of the crop or an open field of grass/weeds. All pesticide solutions should be mixed with the intent of limiting the problem of excess solutions.
 - c. Triple rinse the spray tank/container (i.e., add carrier to the tank and spray out).

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SOP 11.1 v 6

Title: Safety and health procedures in handling pesticides.

Purpose: To assure that personnel handling pesticides are doing so in a safe manner and if an accident occurs, danger is minimized.

Scope: All locations conducting field trial(s) (including greenhouse).

Procedures: Where institutional policies and guidelines do not exist, the following procedures should be followed:

- a. A supply of soap/detergent and water should be readily accessible for cleanup in the case of an emergency.
- b. All personal protective equipment and clothing as required by the label or written SOPs should be worn in the handling of pesticides for storage, mixing and application. Emergency personal protective equipment (e.g. coveralls, self-contained breathing apparatus) must be available when handling hazardous pesticides such as restricted use pesticides.
- c. Appropriate weather conditions for the application of the pesticide should prevail otherwise the pesticide applications should be delayed.
- d. All precautions should be taken to avoid applying pesticides to or near sensitive areas or where drift to these areas may occur.
- e. Prior to application, the equipment should be checked to make sure there are no leaks in the pump or tanks, hose connections, or worn spots in the hoses. All spray tanks should have lids. Filling the spray tank should be done carefully so it does not run over. All machinery should be shut down if necessary to adjust or repair any moving parts. Never blow out nozzles, hoses, or clogged lines by mouth. Inspect all pesticide containers for leaks, tears, or holes before handling. Do not mishandle or abuse containers and thereby create hazards and/or emergencies by carelessness.
- f. All pesticides should be mixed in quantities which are adequate for the job to avoid excess dilute solutions after the job is completed. Cleanup procedures should be established whereby excess sprays can be safely discarded preferably by spraying the material on an overplanting of the commodity and/or an open field of grass/weeds. The equipment should be washed off both inside and outside and all pesticides and pesticide containers should be returned to a storage area immediately after use.

- g. At the end of the working day employees who have applied or mixed pesticides should take a shower and change clothes. Clothing should be washed after the end of the day. In no case should the same clothing, including shoes, be worn on a second day after it has been worn during a pesticide application.
- h. A pesticide-treated area, greenhouse, or field should not be entered until adequate time, as specified on the label of the pesticide, has re-entry to the treated area. For persons who regularly handle organophosphates and/or large quantities of carbamates, a cholinesterase level should be determined at least monthly throughout the pesticide application season.
- i. Do not permit unauthorized persons in the pesticide storage area.
- j. Do not store pesticides next to food, feed, seed, fertilizer, or other articles intended for consumption or use by humans or animals. Do not store food, beverages, tobacco, clothing, eating utensils, or smoking equipment in any area where pesticides are present.
- k. Do not drink, eat food, smoke, apply cosmetics, or use tobacco in areas where pesticides are present.
- l. Wear unlined protective gloves while handling containers and mixing or measuring pesticides.
- m. Do not put fingers in mouth or rub eyes while working with pesticides.
- n. Wash hand thoroughly with soap and water immediately after handling pesticides and, especially before eating, smoking, or using the toilet.
- o. Treated field should be posted with warning signs.
- p. Pesticide storage areas should be properly ventilated.

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SOP 12.1 v 6

Title: Procedures to follow prior to an EPA inspection.

Purpose: To provide guidance to study personnel in responding to a request for an EPA audit or review by Office of Compliance Monitoring (OCM).

Scope: All locations conducting field trial(s).

Procedures:

1. Notify the Study Director, Quality Assurance Officer, and other interested personnel of the pending audit or review as soon as possible.
2. Arrange to have available the personnel who may be associated with the trial(s) or facilities audit.
3. Make sure someone who is authorized to accept the Notice of Inspection is going to be present at the start and finish of the inspection.
4. Prepare trial(s) and/or facilities personnel for the inspection.
 - a. Review position descriptions with technical personnel so they understand and can explain their role in the trial(s).
 - b. Discuss possible questions that may likely come up about the trial(s) or facility and make sure every one understands what to expect.
 - c. Request personnel to answer the questions only to the extent needed and not to provide extraneous information. Do not volunteer information; keep answers direct and to the point.
 - d. Make certain that technical personnel know the safety precautions needed for the work area.
 - e. Be certain that all documents pertaining to the trial(s)/facilities inspection are available. This would include:
 - (1) Master schedules for both the field research director, Quality Assurance Research Officer and possibly their counterparts at the region and IR-4 headquarters.
 - (2) Study Protocol and Standard Operating Procedures
 - (3) Raw data, correspondence and logs.
 - (4) Training records, CVs, job descriptions, etc. of personnel assigned to the trial(s).
 - (5) Appropriate chain of custody documents for samples and freezer logs and storage temperature documentation.
 - (6) Documentation of the characterization of the test substance, receipt, and handling, and storage records.
 - (7) Calibration logs on equipment such as balances and application equipment.
 - (8) Archives or storage of records and logs indicating removal and replacement of documents. Have accessible organizational charts, a map of the facility and any information

SOP 12.1 v 6

Reviewed by (Initials)	Date	Comments
RJ	3-27-19	

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SOP 12.2 v 6

Title: Procedures to follow during an EPA inspection.

Purpose: To provide guidance to study personnel in responding to a request for an EPA audit or review by OCM.

Scope: All locations conducting field trial(s).

Procedures:

1. Greet the inspection team and follow any institutional procedures for signing in. Escort the entire group to a conference or meeting room.
2. At the opening of the conference ask the lead inspector for his credentials and for any opening statements.
3. Introduce the facility personnel present and state their function in the facility or trial(s). Identify the person responsible who will accept the Notice of Inspection.
4. Distribute organizational charts, map of the facility and any other information previously prepared to make the inspection go smoother.
5. Ask the lead inspector for his/her agenda for the inspection as well as a list, if possible, of personnel for interview during the inspection.
6. Explain any housekeeping rules such as the use of safety equipment in work areas etc. to avoid any possible misunderstandings.
7. Proceed with the inspection.
 - a. Provide documents requested and provide explanations needed.
 - b. Keep notes of observations and of all interviews.
 - c. Keep management informed of the progress of the inspection and the findings.

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SOP 12.3 v 6

Title: Procedures to follow after the EPA inspection.

Purpose: To provide guidance to study personnel in responding to a request for an EPA audit or review by OCM.

Scope: All locations conducting field trial(s).

Procedures:

1. If possible, have on-hand all personnel involved in the inspection present for the closeout conference.
2. Clarify any discrepancies brought up during the exit interview and offer supporting documentation for clarification.
3. If you have corrected any problems during the inspection make sure the corrections are so noted in the inspector's logbook
4. Have someone present during the close-out take accurate notes.
5. Be sure you obtain a copy of the list of documents or other materials that may be taken as exhibits by the inspectors.
6. Debrief management, staff, and the Study Director with an explanation of any problems found.
7. Assign responsibility for preparation of possible solutions to problems and obtain time estimates for implementation.
8. Prepare any replies to the regulatory agency as necessary within a timely basis and keep interested parties such as management and the Study Director informed.

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SOP 13.1 v 1

Title: Borrowed or seldom used equipment

Purpose: To assure that borrowed or seldom used equipment is in satisfactory condition to comply with IR4 GLP studies.

Scope: All locations conducting field trial(s).

Procedures:

1. Inspect equipment for obvious problems, i.e. loose conditions, cracked hoses, etc.
2. Ascertain, if possible, last known use and document.
3. If equipment is spray equipment, clean it.
4. If to be used in spray application, calibrate and adjust or replace those parts not functioning properly. Record all actions.
5. Enter date equipment was returned to original source. .

