# SOP Log Sheet

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**FRD/LRD:** Stephen C. Smith
**Submitter**

**Effective Date:** 3/3/25

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# STANDARD OPERATING PROCEDURES

# FOR

# MAGNITUDE OF THE RESIDUE-FIELD STUDIES

N. C. State University IR-4 Field Research Center
3800 Castle Hayne Road
Castle Hayne, NC 28429
2025 Season

Stephen C. Smith
Field Research Director

(Signature) 2-25-25 (Date)

Kristen Searer-Jones
Regional Field Coordinator

Kristen Searer-Jones (Signature) 3/3/2025 (Date)

Effective Date: 3-3-25

N. C. State University IR-4 Field Research Center
3800 Castle Hayne Road
Castle Hayne, NC 28429

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2025 Season

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Effective Date 3-3-25

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Standard Operating Procedures Table of Contents
2025 Season

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*All SOP's have been reviewed prior to signing:

Date: 3-10-25

Effective Date 3-3-25

N. C. State University IR-4 Field Research Center
3800 Castle Hayne Road
Castle Hayne, NC 28429

**SOP#:** 1.1 **Revision Number:** 16

**Title:** General requirements for the development, use, retirement, and retention of standard operating procedures (SOPs).

**Purpose:** To provide guidance to scientists conducting field trials in the development, use, retirement, and retention of SOPs for field research.

**Scope:** Locations conducting field trials.

**Procedures:**

1. Each facility where trial(s) are conducted in support of the registration of pesticides will develop SOP's for all phases of the research.

2. Generic SOPs may be provided to each facility and these SOPs will be revised to accurately reflect that facilities policies, procedures and methods. Where generic SOPs are not available, the Field Research Director (FRD) will see that the required SOPs are developed and approved prior to the use in any GLP studies.

3. The SOPs will be approved by the IR-4 Regional Field Coordinator (RFC) or other appropriate approving official and this will become the effective date. The title page should show the signature or initials of the approving official, and the date signed by the approving official.

4. Each SOP will be reviewed annually and revised when necessary. The effective date and revision number must be changed to reflect the revision. The revision number should begin with 1 and increase sequentially with each revision. One copy of each retired SOP will be retained by the FRD and placed into the NCSU IR-4 Center archives. All other copies will be destroyed. Retained copies of SOPs will be placed in a separate file from the active SOPs so as not to be used by mistake.

5. Any deviations from the SOPs must be documented in the raw data and authorized by the Study Director (SD).

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3800 Castle Hayne Road
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**SOP#:** 1.2 **Revision Number:** 14

**Title:** Numbering system for standard operating procedures (SOPs).

**Purpose:** To provide a general outline for SOPs via a numbering system.

**Scope:** All SOPs should follow the numbering system to provide uniformity in the system.

**Procedures:** The numbering system for SOPs is as follows:

1. General
2. Personnel
3. Facilities
4. Equipment
5. Test System Establishment and Maintenance
6. Test Substance
7. Data Handling
8. Residue Sample Handling
9. Reporting and Retention of Data
10. Disposal of Test Substances
11. Safety and Health Procedures
12. Procedures to Handle an EPA Audit or Inspection

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Castle Hayne, NC 28429

**SOP#:** 1.3 **Revision Number:** 16

**Title:** Format for use in developing SOPs.

**Purpose:** To assure a uniform format in the development of SOPs.

**Scope:** Applies to all SOPs developed by scientists for use in the conduct of trials under GLP .

**Procedures:** The following is the format to be used for each standard operating procedure (SOP):

Name of Test Facility (centered)
Address (centered)
1 space
SOP Number: (SOP section number as a decimal); Revision Number: (sequentially beginning with 1 for first use)
1 space
Title: (Title)
1 space
Purpose: (Brief description of the purpose of the SOP)
1 space
Scope: (Determines where and when the SOP is applicable)
1 space
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 page will have document name and page number. Pages will be numbered 'page ___of $X$', with $X$ being total number of pages in the SOP.

N. C. State University IR-4 Field Research Center
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Castle Hayne, NC 28429

**SOP#:** 1.4 **Revision Number:** 16

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

**Purpose:** To provide information on how the FRD is designated and outline the responsibilities of the FRD.

**Scope:** All test facilities where GLP trials are conducted.

**Procedures:**

1. The FRD is designated by the Study Director (SD) based on the recommendation of the Regional Field Coordinator (RFC) to conduct the trials. The FRD at the NCSU IR-4 Field Research Center shall be a scientist with appropriate training and experience to conduct the trials.

2. The FRD will ensure that:

   a. The trial is carried out in accordance to an approved protocol and the GLP regulations.

   b. Personnel, resources, facilities, equipment, materials and methods as necessary for the conduct of the trial are utilized.

   c. All personnel actively participating in the trials understand the trial protocols, facility SOPs, and GLP regulations.

   d. All findings reported by the Quality Assurance Unit (QAU) receive appropriate responses.

   e. All raw data, summaries and other items connected with the trials that need to be retained are archived per NCSU IR-4 Center SOP 9.5.

   f. A current copy of a master schedule for all GLP trials under his/her direction is maintained.

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**SOP#:** 2.1 **Revision Number:** 18

**Title:** Personnel.

**Purpose:** Provide information regarding personnel requirements under Good Laboratory Practices (GLPs).

**Scope:** All field facilities conducting trials for the registration of pesticides

**Procedures:**

1. The NCSU IR-4 Center will have on file current copies of a professional biography or curriculum vitae (CV), a position description, and training records for any person that records data in GLP trials and any person that supervises trial participants.

2. The NCSU IR-4 Center will have a sufficient number of persons to carry out the trials to completion and the Field Research Director (FRD) or designee will utilize trained personnel to conduct their portion of the trials.

3. There will be a supply of safety equipment in working order and sufficiently clean to protect the health and safety of the personnel connected with the trials as required by regulations, pesticide labels or the trial protocols.

4. Where the application of restricted use pesticides is required, the applicator must be certified or under the direct supervision of a certified applicator, depending on regulations associated with the particular product.

5. Personnel handling pesticides should be trained in accordance with the current policies and guidelines of N. C. State University.

6. Personnel documentation will be reviewed periodically and revised as needed.

7. When a person's employment with the organization ends, their training records and curriculum vitae will be archived, per NCSU IR-4 Center SOP 9.5.

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3800 Castle Hayne Road
Castle Hayne, NC 28429

**SOP#:** 2.2 **Revision Number:** 25

**Title:** Organizational chart, facility locations, and facility layout.

**Purpose:** To assist in the development of an organizational chart and clarifying facility locations and layout.

**Scope:** All field facilities conducting trials 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.

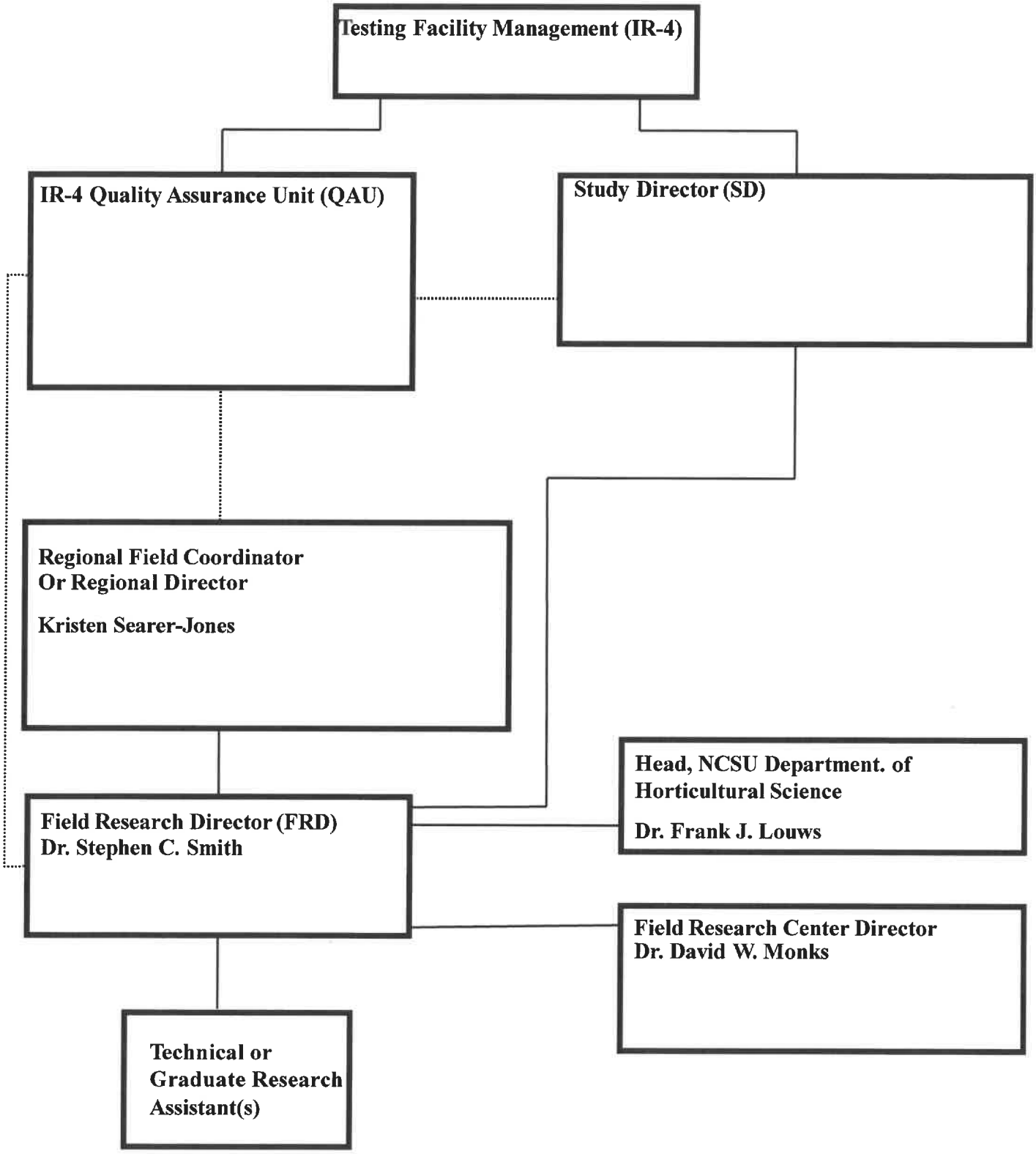
2. Each block in the chart should show the title, and a brief description of the duties of each person.

3. The head of the unit (i.e. department chair, director, etc.) should be included in the chart. This person should be the one who appoints the Field Research Director (FRD) at the institution.

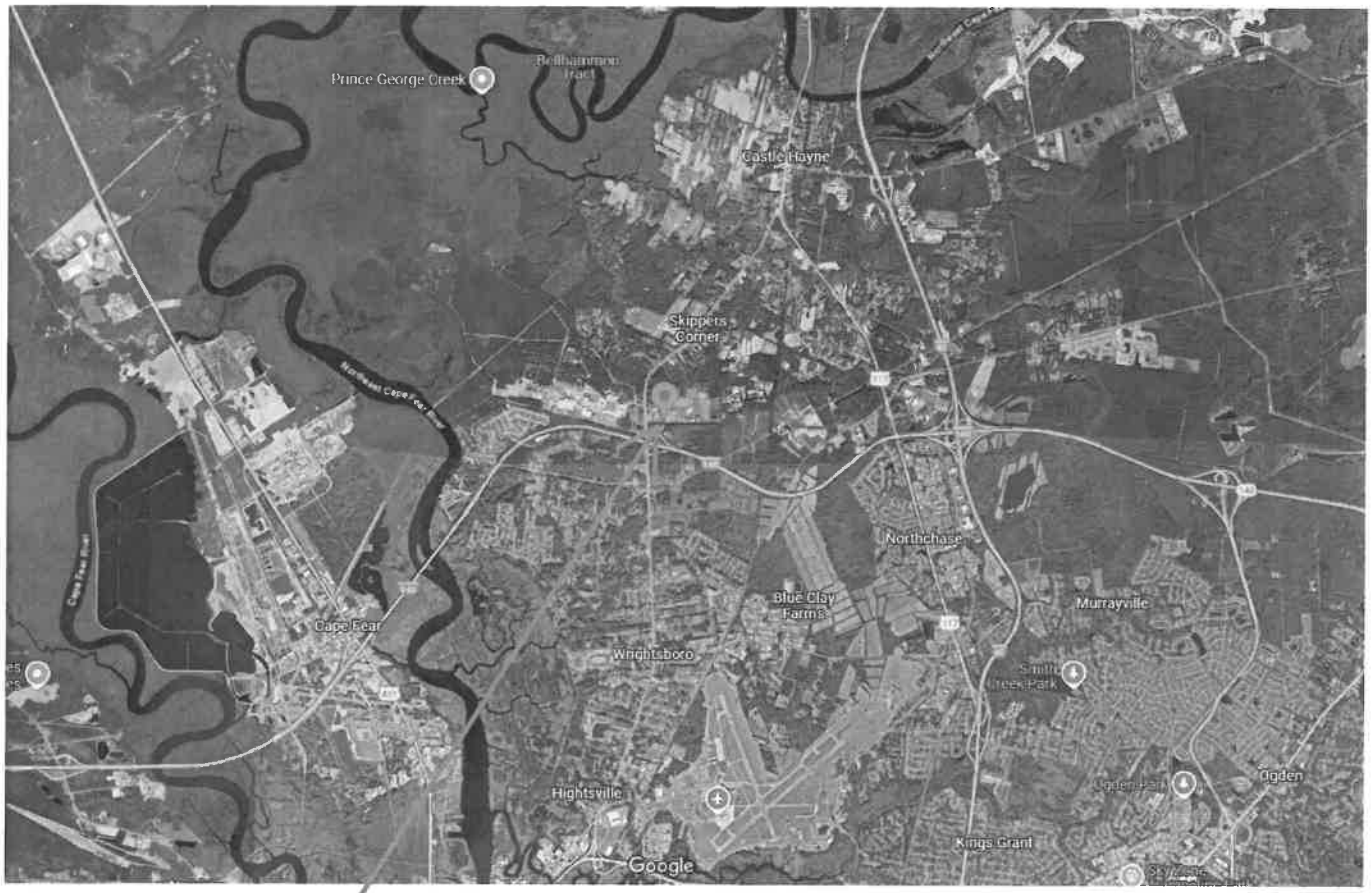
4. The chart should show how the FRD and the Quality Assurance Unit (QAU) independently report to the IR-4 testing facility management.

5. Personnel engaged in the conduct of the trials should then be shown on the chart with lines of supervision, communication, and cooperation indicated.

6. Maps to primary facilities and facility layout should be present so that an intelligent person can find equipment, supplies, and records without verbal instruction. Maps to off-site equipment storage (ex. storage at other research stations or field labs) will be stored in facility files.


Testing Facility Management (IR-4)
IR-4 Quality Assurance Unit (QAU)
Study Director (SD)
Regional Field Coordinator
Or Regional Director
Kristen Searer-Jones
Head, NCSU Department. of
Horticultural Science
Dr. Frank J. Louws
Field Research Director (FRD)
Dr. Stephen C. Smith
Field Research Center Director
Dr. David W. Monks
Technical or
Graduate Research
Assistant(s)
Lines of supervision and communication
Lines of communication only

# NCSU IR-4 Field Research Center
3800 Castle Hayne Road
Castle Hayne, NC 28429




3800 Castle Hayne Rd
NCSU IR-4 Field Research Center

**NCSU IR-4 Field Research Center** 3800
Castle Hayne Road
Castle Hayne, NC 28429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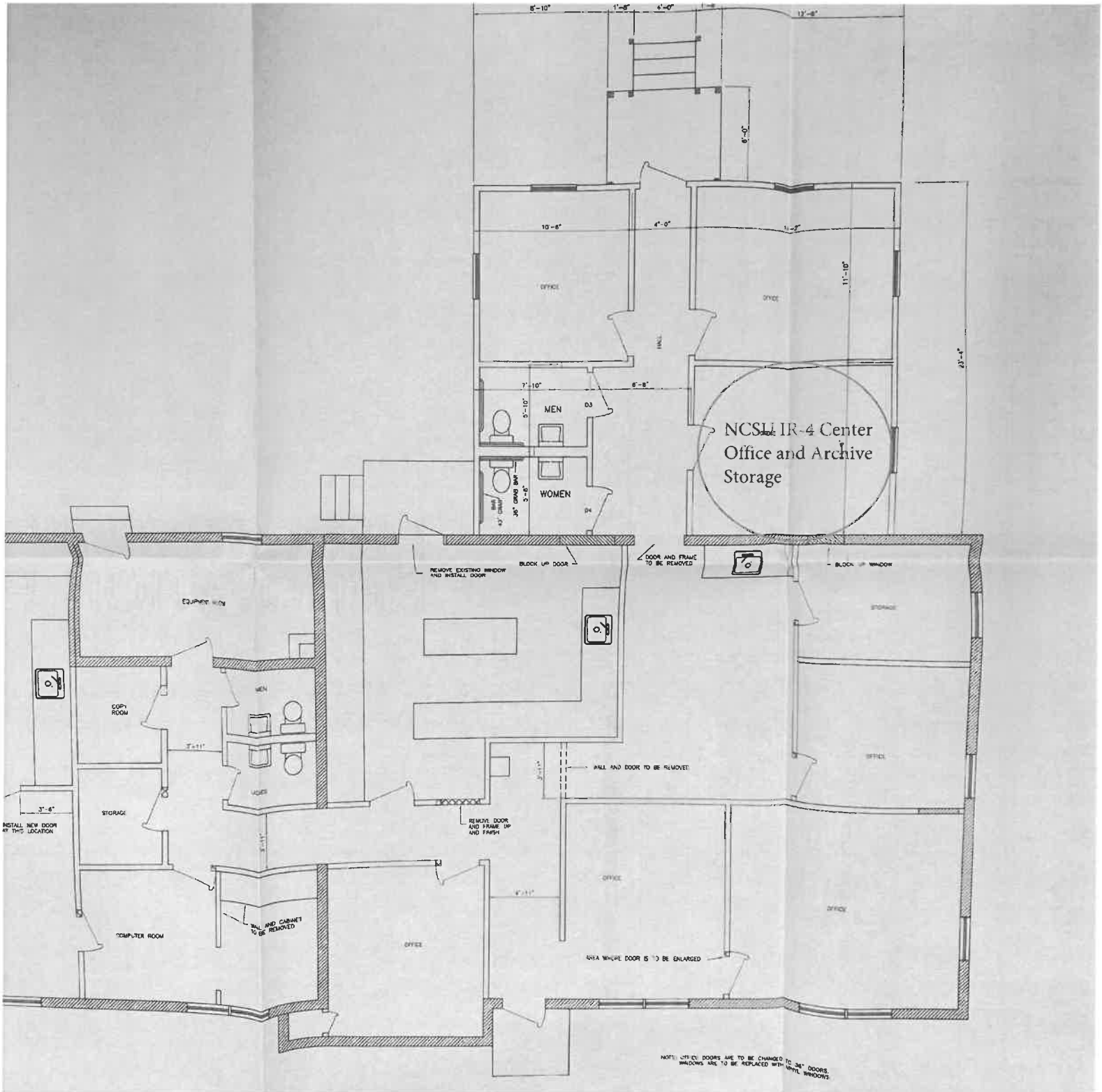
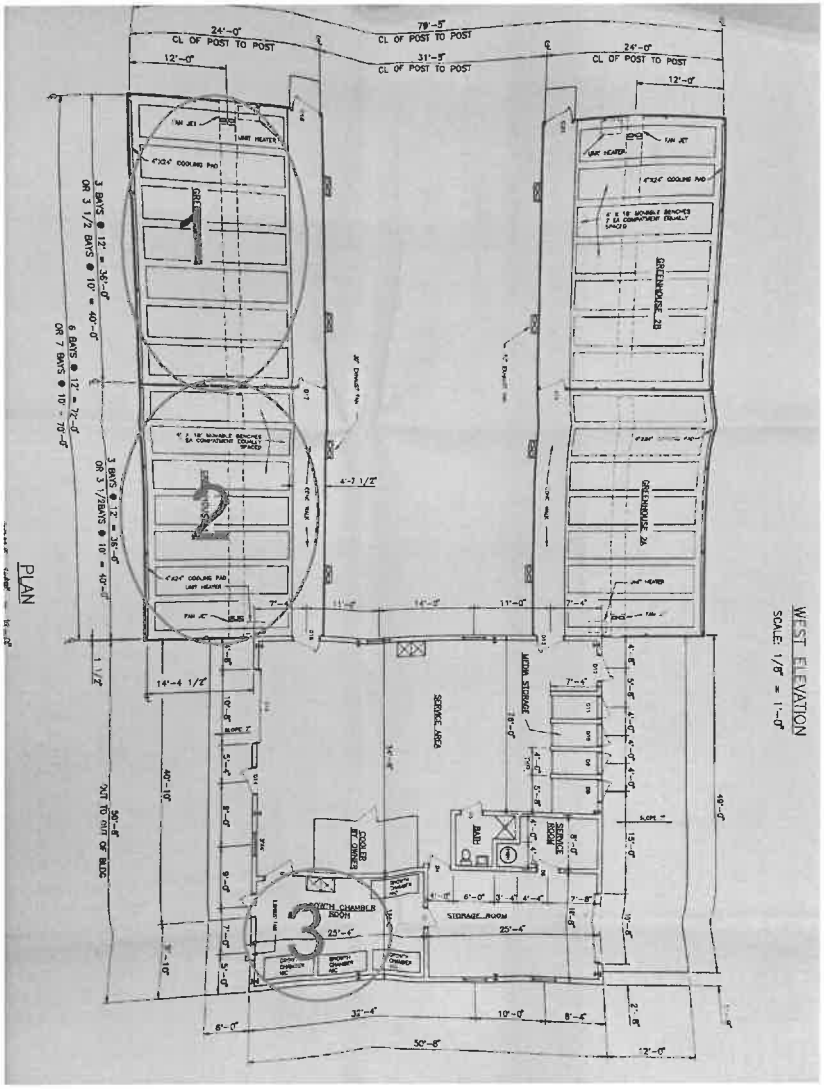
1. NCSU IR-4 Center Office Space and Archives (CALS 880)
2. NCSU IR-4 Center Supply Building 1
3. NCSU IR-4 Center Supply Building 2
4. NCSU IR-4 Center Pesticide Storage
5. NCSU IR-4 Center Sample Freezers (CALS 880A)
6. HCRS-CH Greenhouse 3 (CALS 880A)
7. HCRS-CH Greenhouse 4 (CALS 880A)

# NCSU IR-4 Field Research Center
## Building CALS 880, 3800 Castle Hayne Road
## Castle Hayne, NC 28429



# NCSU IR-4 Field Research Center

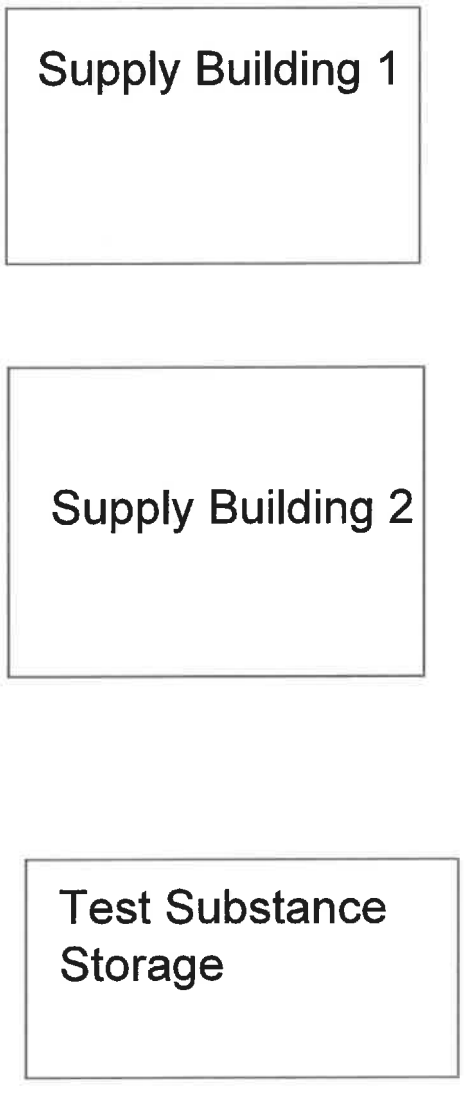
## Building CALS 880A, 3800 Castle Hayne Road
## Castle Hayne, NC 28429




1. HCRS- CH Greenhouse 4
2. HCRS- CH Greenhouse 3
3. NCSU IR-4 Center Sample Freezers

# NCSU IR-4 Field Research Center

NCSU IR-4 Center Supply and Test Substance Storage Buildings, 3800 Castle Hayne Road
Castle Hayne, NC 28429



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**SOP#:** 2.3 **Revision Number:** 14

**Title:** Documentation of training.

**Purpose:** To assure that training for personnel involved in GLP trials is properly documented.

**Scope:** All field facilities conducting trial(s) for the registration of test substances.

**Procedures:**

1. All training of personnel engaged in GLP trials should be documented in a training record.

2. Training received from any source, should be noted as to the title or description of the event, date(s) of attendance, instructor or organizer name, trainee name, and subject(s) covered. A copy of any type of certificates issued should be retained in the personnel files.

3. Training on specific procedures and/or standard operating procedures (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 6/2/18).

4. Each person that collects data or supervises participants in GLP trials should have read and understood those sections of the protocol and the SOPs that pertain to their responsibilities. The Field Research Director (FRD) should record in their respective training records, the names of the personnel and dates that the SOPs were explained to them.

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**SOP#:** 3.1 **Revision Number:** 22

**Title:** Guidelines for test substance and adjuvant storage and labeling.

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

**Scope:** Locations conducting IR-4 field trials and where institutional guidelines do not exist for storage of test substances and adjuvants that are used in GLP trials is required.

**Procedures:**

1. Test substances and adjuvants that are used in GLP trials will be stored in a dry, well-ventilated area which is separated from offices, laboratories and sample storage areas. This area should be sufficient to allow storage of the products according to their label directions. Products will be stored in accordance with current policies and guidelines of N. C. State University.

2. The temperature range within the storage facility will be monitored by a data logger and a backup device. Data from the data logger and backup device will be collected and/or recorded approximately monthly.

3. The original containers for all GLP test substances must be returned to the manufacturer or retained until completion of the study and/or permission is given by the Study Director (SD) or Test Facility Management to dispose of the containers.

4. The storage facility should have limited access.

5. Items listed below will be the responsibility of University officials overseeing the pesticide storage complex where the test substances are stored:

    a. Place highly visible, waterproof identification signs in and around the pesticide storage complex to advise of the hazardous nature of the storage facility's contents.

    b. Make accessible, materials such as adsorptive clay, granulated activated charcoal, hydrated lime, etc. for emergency treatment detoxification of spills or leaks.

6. Do not keep products on the floor of the storage area.

7. Storage areas and mixing areas should be separate to prevent potential

contamination or mix-up.

8. Test substance containers shall have the following information present on the label or container surface:

    a. Name
    b. Batch or lot number
    c. Expiration date
    d. Storage conditions specific to the test substance
    e. Container identification (when appropriate i.e., 1 of *X*, 2 of *X*, etc.)

9. Adjuvants used in GLP trials will have the following listed on the container label:

    a. Name
    b. Concentration
    c. Storage conditions
    d. Expiration date

    If adjuvants that are used in GLP trials do not have a known expiration date at the time of receipt, an expiration date will be assigned by NCSU IR-4 personnel. This date will be no more than 5 years from the date of receipt.

10. The integrity of test substances and adjuvants and the prevention of product contamination will be of paramount concern at the NCSU IR-4 Field Research Center. Practices and procedures related to these issues will include, but not be limited to, the following:

    a. The physical properties of the test substance and adjuvant (i.e., color consistency, odor, etc.) will be examined at each use. If any attribute is questionable, the product will not be used. If a test substance is deemed unusable, the SD will be immediately notified for guidance. If an adjuvant is deemed unusable, an appropriate adjuvant may be substituted for use. This substitution will be thoroughly documented in the Field Data Book (FDB) and/or appropriate log(s).
    b. Test substance integrity will be further protected by controlling the temperature range to which it is exposed. This will be done at the NCSU IR-4 Field Research Center by transporting the test substance to the test site inside a cooler, using blue ice if needed to avoid excessive temperatures. Since adjuvants often arrive in large containers the NCSU IR-4 Field Research Center can use smaller secondary containers of the adjuvant to facilitate transportation. Secondary containers must be labeled with the same information

required on the original adjuvant container (See #9 above).

c. When measuring the calculated amount of test substance or adjuvant for a spray mix, the pipette or syringe used to extract the desired amount will not have been previously used and will not be used for any other product.

d. The pipette or syringe used to extract a desired amount of a test substance or adjuvant will only be used once, with the exception of multiple aliquots of the same syringe described in SOP 4.2.

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**SOP#:** 3.2 **Revision Number:** 16

**Title:** Site selection for field trials.

**Purpose:** To help ensure plots are large enough to obtain the required data or samples with sufficient uniformity and can be relocated after the trials are terminated.

**Scope:** Locations conducting field trials.

**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. It should also be large enough 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 trials.

4. Where samples for residue trials are required, locate nontreated plots within the same area (preferably upwind and up slope 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.

6. Prepare a plot map containing all required items from protocol and Field Data Book (FDB) instructions. Include this map in the FDB.

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. Identify both ends of each plot with a marker of sufficient visibility to be seen easily throughout trial duration.

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Castle Hayne, NC 28429

**SOP#:** 3.3 **Revision Number:** 17

**Title:** Greenhouse/shadehouse facilities.

**Purpose:** To assure that greenhouse/shadehouse facilities are properly maintained and in sufficient working order throughout the trials to obtain data useful in the registration of pesticides under GLP guidelines.

**Scope:** All locations where greenhouse/shadehouse trials are performed.

**Procedures:**

1. Lighting, temperature, humidity, and shade levels should be sufficiently uniform in the greenhouse/shadehouse to provide nearly uniform plant growth throughout the trial area. If, due to size of trial or structure(s), a trial requires more than one structure, lighting, temperature, humidity, and shade levels should be sufficiently uniform across each to provide nearly uniform plant growth throughout the trial. Plots will be identified as described in SOP 3.2.

2. 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 should be well-drained to prevent the buildup of excess moisture.

3. Sufficient monitoring devices should be in place, in good working order, and calibrated to assure that the proper environmental conditions are maintained throughout the trials. Calibrations and maintenance activities for GLP-maintained equipment will be documented in the maintenance logs for these items.

4. Where more than one trial is conducted in a greenhouse/shadehouse, there must be sufficient isolation between the trials to prevent contamination or interference between trials.

5. Greenhouses should be equipped so as to maintain environmental conditions to simulate commercial greenhouse production techniques or as required by the study protocol.

6. Document cultural practices used in the greenhouse/shadehouse in the raw data notebook and/or greenhouse/shadehouse logbook(s).

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**SOP#:** 4.1 **Revision Number:** 19

**Title:** Calibration and use of balances.

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

**Scope:** All field facilities where a dry material is weighed for use in a field, greenhouse or hothouse trials.

**Procedures:** The methods, materials, and schedules for routine inspection, cleaning and calibration will be:

1. Standard weights will be calibrated/standardized every two years or less. Documentation of calibration/standardization will be kept on file.

2. Prior to each use, the user will visually inspect the balance for cleanliness. Any dirt or chemicals within the chamber or on the pan must be cleaned immediately.

3. Prior to each use, balance accuracy checks should be performed, using two standard weights that bracket (in the weight range of the chemical samples being weighed) the amount to be weighed. Record declared weights and actual weights of standards as raw data.

    a. If the measured weights of both standard weights are within 2% of the standard weights, proceed with weighing.

    b. If the measured weight of either standard weight differs by more than $\pm$ 2% from the standard weight, recalibrate the balance.

    c. If, after recalibration, the measured weights of both standard weights are within $\pm$ 2% of the standard weights, proceed with weighing and record the measured weights.

    d. If, after recalibration, the measured weight of either standard weight still differs by more than $\pm$ 2%, replace the defective weight. If it is determined that the problem is the balance, it should be serviced before further use.

    e. If desired test substance amount is less than lowest certified weight available, the two lowest certified weights will be used to ensure balance is working accurately. Similarly, if the desired test substance amount is greater than the largest certified weight available, the two highest certified weights will be used

to ensure balance is working accurately.

4. Select an appropriately-sized vessel to hold the desired amount of test substance and tare it on the scale following the manufacturer's directions in the appropriate technical manual.

5. If taring the vessel is not practical, record the weight of the vessel, add the weight of the desired amount of test substance to it and weigh out this amount.

6. Select and use appropriate safety equipment while handling the test substance.

7. Any interim container used for the test substance should be adequately labeled to prevent possible confusion at mixing.

8. Remedial actions to be taken in case of failure or malfunction include:

    a. Any problem should be immediately reported to the Field Research Director (FRD), documented, and placed in the balance records as non-routine procedures.

    b. If the problem cannot be corrected by instructions from the manufacturer's manual, a service representative should be notified. All corrective actions taken shall be documented in the balance records as non-routine procedures.

9. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

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**SOP#:** 4.2 **Revision Number:** 20

**Title:** Measuring liquid formulations.

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

**Scope:** All field facilities conducting field trials for IR-4.

**Procedures:**

1. Obtain a clean cylinder, syringe, pipette or other measuring device large enough to hold the volume of liquid needed. Effort will be made to use a device with the greatest accuracy and smallest increments available. If the device can not hold the entire volume of liquid needed, multiple aliquots can be obtained using a single device.

2. If the opening of the graduated cylinder is too restrictive to allow filling without danger of spillage, 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.

3. If mouth of container is too restrictive to allow access of measuring device, use a clean container as an intermediate from which to draw the aliquot. After use, dispose of unused liquid properly, do not return it to the original container.

4. Select and use appropriate safety equipment while measuring liquids.

5. When amounts >60 ml are required, measurement with a graduated cylinder is permitted. Place the graduated cylinder on a level surface and take the reading of the liquid in the graduated cylinder 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.

6. Any graduated cylinders used to measure or transfer the test substance concentrate, as described in #5 above, should be triple rinsed and then thoroughly washed with water plus soap (or ammonia) after use to ensure that they are clean and cross-contamination of pesticides will not occur. Finally, rinse thoroughly to remove soap (or ammonia) from cylinder.

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3800 Castle Hayne Road
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**SOP#:** 4.3 **Revision Number:** 19

**Title:** Calibration and use of a liquid sprayer.

**Purpose:** To determine the delivery rate of a liquid sprayer and adjust as necessary to ensure an accurate application of the test substance.

**Scope:** All facilities where a liquid sprayer is used in the application of test substances.

**Procedures:** The methods, materials, and schedules for routine inspection and calibration should include:

1. Always calibrate the spray equipment before initial test substance application. For trials with multiple applications, recheck the calibration at subsequent applications to confirm accurate delivery ( e.g., $\pm$ 5% of initial calibration or as specified by the protocol), or recalibrate the equipment.

2. Prior to use, visually inspect pumps, hoses, pipes, fittings, regulators, pressure gages, and tanks for obvious wear or potential leaks and repair or replace as necessary as part of routine maintenance. Record any maintenance performed in the appropriate log(s).

3. Refer to the protocol for any specified application requirements.

4. Gallons per minute (GPM) can be calculated by collecting output, in milliliters (ml) from each nozzle for 15 seconds; multiplying this value by four and then dividing by 3,785. Output from each nozzle will not vary by more than 5% from highest to lowest. Any part(s) contributing to values outside this range will be cleaned or replaced. This procedure will be repeated until output of all nozzles fall within required parameters.

5. Once application speed, in miles per hour (MPH), nozzle spacing (NS), in inches, and sprayer output, in GPM, are known, gallons per acre (GPA) can be calculated with the following formula:

$$GPA = \frac{GPM * 5940}{MPH * NS}$$

6. Speed will be determined by timing (sec) the movement of the sprayer over a known distance (D) or by selecting a speed, calculating the needed

pass time, and adjusting until this pass time is matched. This can be done by using the following formula:

$$MPH = \frac{(D/sec)}{1.47}$$

7. Operator must carefully operate equipment, during application, under the same conditions as during calibration. Ensure solution is thoroughly mixed before application and continue to agitate during application, if possible. The test substance must be applied uniformly to the entire test area.

8. Thorough cleaning of the applicator will be done after each period of use and when changing test substances.

9. Remedial action to be taken in case of failure or malfunction should include:

    a. Immediately report malfunction to the Field Research Director (FRD).

    b. If problems occur during application, refer to SOP 6.5.

    c. Any repairs and replacement of parts, other than changing nozzles and/or strainers while creating a different setup of the boom, will be documented as non-routine maintenance in the appropriate maintenance log(s).

    d. Changes of nozzles and/or strainers while creating a different setup of the boom will be documented as routine maintenance in the appropriate log(s)

10. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

11. When not in use, equipment used for GLP trials will be kept in a secure location to avoid the possibility of contamination and tampering. This may include a locked cover on the bed of a truck.

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**SOP#:** 4.4 **Revision Number:** 14

**Title:** Calibration and use of granular applicators.

**Purpose:** To determine the delivery rate of the granular applicator and adjust as necessary to ensure an accurate application of the test substance

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

**Procedures:** The methods, materials, and schedules for routine inspection and calibration should include:

1. Prior to use, determine that the applicator is in good working order and mechanical condition. Make sure that the openings to release the granular material are not clogged and are free of debris.

2. Ground driven applicators:

   Applicator can be calibrated using the following method:

   a. 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:

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

   b. Approximate setting of the openings to operate the applicator for the desired amount of active ingredient/acre.

   c. Wear protective clothing as required by product label and fill the spreader with enough material to ensure proper operation. Operate the applicator over the measured distance and collect the output.

   d. Weigh the material collected and multiply by 100 to give the amount applied per acre.

   e. Repeat steps "a" to "d" until the desired rate is achieved within 5% of the total/acre

   All calibration data and calculations should be recorded in the raw data

3. Non-ground driven applicators.

   Applicator can be calibrated using the following method:

   a. 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:

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

b. Determine how long it will take the applicator to travel that distance.

c. Determine the amount of material needed to treat 0.01 acre and the approximate setting of the openings to operate the applicator for the desired amount of active ingredient/acre.

d. Turn the applicator on and operate it for the time required to travel the distance determined above while collecting the output from the applicator.

e. Weigh the collected material from the applicator and multiply by 100 to give the amount applied per acre.

f. Continue to change settings of the openings on the applicator until the desired rate is achieved within 5% of the total/acre.

4. Applicators must carefully operate under the same conditions as during calibration. All discharge openings should be unobstructed and flow from each should be identical. The test substance must be applied uniformly to the entire test area.

5. Thorough cleaning of the applicator will be done after each period of use and when changing test substances.

6. Remedial action to be taken in case of failure or malfunction should include:

   a. Immediately report malfunction to the Field Research Director (FRD).

   b. If problems occur during application, refer to SOP 6.5.

   c. Any repairs or replacements will be documented as non-routine maintenance in the appropriate log(s).

7. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

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**SOP#:** 4.5 **Revision Number:** 20

**Title:** Calibration, use, and cleaning of an air blast sprayer

**Purpose:** To determine the delivery rate of air blast sprayer and adjust as necessary to ensure an accurate application of the test substance

**Scope:** For use when calibrating an air blast sprayer for applying test substances to tree/bush crops.

**Procedures:**

1. Sprayer hoses, pipes, connections, pressure gages, pressure regulator and tank will be examined for leaks. The sprayer will be examined for worn belts and pulleys.

2. The sprayer will be calibrated to deliver a volume per area within the guidelines specified by the protocol. Always calibrate before initial test substance application and after any application parameter changes, if applicable. For multiple applications, recheck the calibration to confirm accurate delivery (e.g., $\pm$ 5% of initial calibration or as specified by the protocol)

3. To calculate speed of sprayer, in miles per hour (MPH), the following formula can be used:

$$MPH = \frac{(D/Sec)}{1.47}$$

Where D is distance of tract used measured in feet and Sec is seconds.

4. With air blast sprayers, nozzle spacing (NS) can be determined by dividing row spacing by the number of nozzles used and dividing that number by 2 if you will be applying test substance by making two passes per row (one on each side).

5. Gallons per minute (GPM) can be calculated by averaging the output, in milliliters (ml) from each nozzle for 15 seconds, multiplying this value by four and then dividing by 3785. As these amounts are immediately mixed and blown onto the crop after exiting the sprayer, the NCSU IR-4 Field Research Center does not set range limits regarding the consistency of output among the tips of an airblast sprayer.

6. Once application speed, in miles per hour (MPH), nozzle spacing (NS), in inches, and sprayer output, in GPM, are known, gallons per acre (GPA) can be calculated with the following formula:

$$GPA = \frac{GPM * 5940}{MPH * NS}$$

7. When the application is made to trees, the nozzle arrangement will be to direct approximately 2/3 of the spray pattern to the top one-half of the trees and approximately 1/3 of the pattern to the lower one-half of the trees. When the application is made to bush or cane crops that have fruit on all portions of the plants, the spray pattern should be equally distributed to entire plant.

8. Operator must carefully operate equipment, during application, under the same conditions as during calibration. Ensure solution is thoroughly mixed before application and continue to agitate during application.

9. The sprayer will be cleaned immediately after use by rinsing the tank with clean water. The sprayer will then be partially filled with tank cleaner (or ammonia) plus water and operated to clean spray lines, tank, and nozzles. Finally, the tank will be partially filled with water and operated to remove cleaner residue from lines, tank, and nozzles. Cleaning of the sprayer will be documented in the maintenance records for the sprayer.

10. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

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**SOP#:** 4.6 **Revision Number:** 21

**Title:** Calibration of backpack sprayer for foliar test substance applications to erect crops.

**Purpose:** To determine the delivery rate of a backpack sprayer for accurate pesticide application when multiple nozzles are used to direct insecticides and fungicides on the crop.

**Scope:** For use when calibrating a backpack sprayer for applying directed sprays of test substances to erect crops (i.e., blueberry, tomato, pepper)

**Procedures:**

1. Always calibrate before initial test substance application. For multiple applications, recheck the calibration to confirm accurate delivery ( e.g., ± 5% of initial calibration or as specified by the protocol), or recalibrate.

2. Prior to use, visually inspect hoses, pipes, fittings, regulators, pressure gages, and tanks for obvious wear or potential leaks and repair or replace as necessary. Record inspection and any maintenance performed in the appropriate log(s).

3. Refer to the protocol for any specified application requirements.

4. With directed applications to erect crops, nozzle spacing (NS) can be determined by dividing row spacing by the number of nozzles used and dividing that number by 2 if you will be applying test substance by making two passes per row (one on each side).

5. Gallons per minute (GPM) can be calculated by averaging the output, in milliliters (ml) from each nozzle for 15 seconds, multiplying this value by four and then dividing by 3785. Output from each nozzle will not vary by more than 5% from highest to lowest. Any part(s) contributing to values outside this range will be cleaned or replaced. This procedure will be repeated until output of all nozzles fall within required parameters.

6. Once application speed, in miles per hour (MPH), nozzle spacing (NS), in inches, and sprayer output, in GPM are known, gallons per acre (GPA) can be calculated with the following formula:

$$GPA = \frac{GPM * 5940}{MPH * NS}$$

7. Speed will be determined by timing (sec) the movement of the sprayer over a known distance (D) or by selecting a speed, calculating the needed pass time, and adjusting until pass time is matched. This can be done by using the following formula:

$$MPH = \frac{(D/sec)}{1.47}$$

8. Operator must carefully operate equipment, during application, under the same conditions as during calibration. Ensure solution is thoroughly mixed before application and continue to agitate during application if possible. The test substance must be applied uniformly to the entire crop.

9. Remedial action to be taken in case of failure or malfunction should include:

    a. Immediately report malfunction to the Field Research Director (FRD).

    b. If problems occur during application, refer to SOP 6.5.

    c. Any repairs and replacement of parts, other than changing nozzles and/or strainers while creating a different setup of the boom, will be documented as non-routine maintenance in the appropriate maintenance log(s).

    d. Changes of nozzles and/or strainers while creating a different setup of the boom will be documented as routine maintenance in the appropriate log(s)

10. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

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**SOP#:** 4.7 **Revision Number:** 13

**Title:** Operation and maintenance of farm equipment.

**Purpose:** To assure that the crop or commodity under study is grown under simulated commercial conditions, in a quantity sufficient for the trial and in a good state of health.

**Scope:** All locations where the farming operations are performed for trials are conducted under good laboratory practices (GLPs)

**Procedures:**

1. The maintenance records for any equipment used to prepare soil, cultivate, apply fertilizer, and irrigate the crop where the trial is conducted will be kept by the research station personnel and will be the only records available on that equipment.

2. Just prior to the initiation of the use of the equipment (tractor, plow, disk, harrow, planters, harvester etc.) it will be visually inspected to see that it is in good working order, properly lubricated, and in good mechanical condition.

3. Any necessary repairs or adjustments should be made prior to the use of the equipment in the GLP trials.

4. The operator of the equipment should be familiar with its operation and safety precautions.

5. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

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**SOP#:** 4.8 **Revision Number:** 16

**Title:** Calibration of field instruments.

**Purpose:** To assure that all instruments and devices used in good laboratory practices (GLP) trials are accurate and in good working order.

**Scope:** All facilities where GLP trials are conducted.

**Procedures:**

1. Each GLP-maintained instrument (e.g. min/max thermometers, data loggers, etc.) should be tested annually to determine that it is within the desired tolerance.
2. Thermometers will be verified against a NIST certified thermometer at two temperature reference points covering the intended working range. The instruments should be allowed to equilibrate for at least 5 minutes before readings are taken and recorded for both test and reference thermometers at any reference point. Acceptable tolerance is ± 3F.
2. A written record of the dates and results of the tests and of the acceptable tolerance for each instrument should be kept in the appropriate log.
3. Those instruments and/or devices that give inconsistent results or are not accurate to within desired tolerances should be replaced.
4. If a manual is not available to describe how an instrument or device should be tested, record the testing procedure used in the relevant log or describe in a standard operating procedure (SOP).
5. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

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**SOP#:** 4.10 **Revision Number:** 10

**Title:** Calibration and use of Nikon Monarch® Laser 800 optical range-finder.

**Purpose:** To establish guidelines for the calibration and use of a Nikon Monarch® Laser 800 in order to accurately measure distance related to the establishment of IR-4 magnitude-of-residue trials at the NCSU IR-4 Field Research Center.

**Scope:** All facilities where trials are conducted under good laboratory practices (GLPs).

**Procedures:**

Calibration: Calibration of the Nikon Monarch® Laser 800 optical range-finder shall be done annually to determine if it is within the desired tolerance.

1. Ensure that the unit displays information when activated by pressing the POWER button. If so, continue to Step 3. If no display occurs or the battery image is blinking or not present, continue to Step 2.
2. Check and replace battery, if necessary
3. Using a tape measure or other accurate measuring device, establish two 'close-range' known distances. One should have a distance that measures in whole yards (i.e., 10.0 yds) while the other should have a distance that measures in 0.5 yards (i.e., 10.5 yds). A third 'long-range' distance will also be established (i.e., 100 yds). Place an object at each distance that is large enough to easily find in the optical range-finder crosshairs. Ensure that the object has a flat surface that will reflect the laser directly back to the optical range-finder. Avoid bright-colored objects and/or extremely bright conditions during this operation, as the reflectance in these circumstances may produce an incorrect readout from the device.
4. Make sure the display is reading in yards. If meters are displayed, hold the MODE button down for more than 1.5 seconds. This will switch the units.
5. Record distance values from the range-finder display.
    a. Stand with range-finder at one end of the pre-measured distance for whole yards, aim at the object placed at other end of this distance and press the POWER button to gain a read out of the distance. Repeat at least once for consistency and accuracy. Record the displayed value in the appropriate log. If values for whole yards do not match the measured distance, do not use range-finder for determining distances. Use some other device.

b. Stand with range-finder at one end of the pre-measured distance for half yards, aim at the object placed at other end of this distance and press the POWER button to gain a read out of the distance. Repeat at least once for consistency and accuracy. Record the displayed value in the appropriate log. If values for half yards do not match the measured distance, do not use range-finder for determining distances. Use some other device.

Use:

1. To determine the distance to an object, do the following:
   a. Press the POWER button to turn on the unit.
   b. Make sure the object is large enough for the range-finder to detect and display a reading. If this is not the case, place an object that is large enough to be detected at/on the initial object. Be sure not to create angles of deflection when placing the second object (see Calibration, Step 3, above).
   c. While viewing the object through the range-finder, press the POWER button to create a displayed distance. Repeat at least once for consistency and accuracy. Record the distance in the appropriate place.

Safety Precautions:

1. Never look directly at the laser beam or directly at the sun when using the range-finder.
2. Do not operate with other optical elements, such as binoculars, lenses, etc.
3. Do not disassemble unit.
4. If unit's body cover is damaged, or if it emits a strange sound due to dropping or from some other cause, immediately remove the battery and stop using.
5. Do not press POWER button when not using the unit.
6. Do not leave within reach of small children.
7. Remove water, sand, or mud immediately with soft, clean, dry cloth.
8. Do not attempt to use range-finder under water.
9. Do not swing by lanyard or leave in unstable situation. Both could result in damage to the unit.
10. Do not leave unit in direct sunlight or in situations of extreme heat.

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**SOP#:** 4.11 **Revision Number:** 7

**Title:** Operation, maintenance, and cleaning of borrowed equipment.

**Purpose:** To assure that borrowed equipment is operated, maintained, and cleaned appropriately for the conduction of good laboratory practice (GLP) trials.

**Scope:** All locations where borrowed equipment is required to conduct GLP trials.

**Procedures:**

1. Upon receipt of equipment, ensure that any available owners/operation manuals are reviewed and understood. Receipt of equipment is to be documented in the appropriate log.

2. Visually inspect the equipment to ensure that it is in good working order. Any discovered problems should be repaired prior to use. These repairs are to be documented in the appropriate log

3. Prior to use, any surface of the equipment that may contact the test substance or target should be cleaned. This cleaning is to be documented in the appropriate log.

4. Perform required task(s) with the equipment.

5. After each use, the equipment is to be cleaned according to the equipment owner's manual or an appropriate standard operating procedure (SOP). This cleaning and any SOP that was used shall be documented in the appropriate log.

6. Document return of the equipment in the appropriate log.

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**SOP#:** 4.12 **Revision Number:** 10

**Title:** Operation, cleaning, and maintenance of forced-air drying ovens.

**Purpose:** This document is for use by IR-4 personnel to define procedures used for operating, cleaning, and maintaining the forced-air drying ovens at the NCSU IR-4 Field Research Center.

**Scope:** This standard operating procedure (SOP) describes the proper procedures used by IR-4 personnel to ensure proper operation, cleaning, and maintenance of the forced-air drying ovens at the NCSU IR-4 Field Research Center.

**Procedures:**

I Operation

1. Inspect each unit and review the maintenance log for each to ensure unit trays and other surfaces were cleaned, according to this SOP, after previous use. If there is any question on whether or not cleaning was performed, the unit will be cleaned prior to operation.

2. To activate unit turn on the power switch(es) for the oven(s) to be used. Use a separate drying oven for each treatment from which samples were collected. Set each oven to the desired drying temperature, per protocol, by using the appropriate button or dial and allow unit to reach this temperature. While temperature is adjusting, verify proper operation of each unit (good air flow from exhaust, normal operational sounds, etc.). If there is any concern about the operation of a unit, shut it off and contact service provider. Any repairs and maintenance to drying ovens will be documented in the appropriate NCSU IR-4 Field Research Center logs.

3. Personnel detecting a problem or malfunction of a drying oven are responsible for documenting both the problem and the repair in the appropriate NCSU IR-4 Field Research Center logs. If another unit needs to be used, follow steps 1 and 2 for the new unit.

4. Line each drying tray that will be used with a single layer of paper to prevent portions of the samples from falling from the tray. Mark each paper with the corresponding sample identification number or letter, to reduce chances of confusion and/or contamination.

5. To dry samples, follow the procedures described in NCSU IR-4 Field Research Center SOP 8.4, "Forced-air drying of RAC (Raw Agricultural Commodity) Samples". All sample drying will be documented in the

appropriate NCSU IR-4 Field Research Center logs.

II Cleaning

1. After drying is complete and samples removed, all paper liners are to be discarded. Retention of paper liners may lead to contamination of subsequent samples.

2. Drying trays are to be cleaned by:
   a. removing the trays from the drying oven(s)
   b. rinsing each tray thoroughly with ammonia
   c. rinsing ammonia residue off with a thorough water rinse
   d. air-drying trays completely prior to replacing them into drying oven(s)

3. While trays are air-drying, the interior surfaces of the oven(s) will be cleaned by:
   a. sweeping or vacuuming out any debris
   b. wiping surfaces with clean rag(s) carrying ammonia + water
   c. wiping surfaces with clean rag(s) carrying water only
   d. wiping surfaces with clean, dry rag(s)

4. Cleaning of drying oven(s) will be documented in the appropriate NCSU IR-4 Field Research Center logs.

III Maintenance

1. Each drying oven will be serviced as needed, using local dealer/distributor of the drying oven(s). All maintenance activities will be documented in the appropriate NCSU IR-4 Field Research Center logs.

2. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

IV Verification of oven temperature

1. Each oven should be tested annually to determine that it is within the desired tolerance.

2. Oven temperature settings will be verified against a NIST certified thermometer at two temperature reference points covering the intended working range. The oven temperature should be allowed to equilibrate for at least 30 minutes before readings are taken and recorded for both oven setting and reference thermometer. Acceptable tolerance is ± 10F.

3. A written record of the dates and results of the tests and of the acceptable tolerance for each oven should be kept in the appropriate NCSU IR-4 Field Research Center logs.

4. If oven temperature is not consistent with NIST then a GLP min/max thermometer should be used to monitor temperature when drying samples.

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**SOP#:** 4.13 **Revision Number:** 6

**Title:** Calibration, use, and cleaning of a drip injection application system

**Purpose:** To determine the delivery rate of a drip irrigation system and injection application equipment and make adjustments as necessary to ensure an accurate application of test substance(s)

**Scope:** For use when calibrating and using a drip injection application system for applying test substances through an irrigation system to various fruit and vegetable crops.

**Procedures:**

1. Set up the drip application system so that the treated and non-treated plots are irrigated at the same time, but that the injection port for the test substance only flows to the treated plot(s). Connect all hoses, pipes, pressure gages, etc. so that the system is ready to apply water. Connect the test substance mix tank containing water to the CO2 source for the application and connect it to the injection port. Charge the drip system and the test substance mix tank to the desired pressures and examine the entire system and drip lines to confirm there are no large leaks and the drip lines are pressurized to the ends of the runs. Make repairs as needed.

2. Confirm the output of the drip system by charging the lines to the desired pressure. Locate 3 emitters from different locations in the test area (near, middle and far from the closest point of the system) for each drip line. Remove the surrounding soil so that a collection vessel can be placed to capture the emitter output without disturbing the drip line. Collect the emitter output for a set period of time (e.g. 3 minutes), then measure and record the discharge from each of the emitters. Repeat for a total of 3 collections. Calculate average discharge per emitter, and overall average for the system.

   There is variability in individual emitters due to manufacturing procedures or other uncontrollable factors. As long as the overall water distribution within the plot appears relatively consistent, large variability between emitters is not a concern. If in doubt, contact the Study Director.

   Once the drip system output is confirmed, the system can be shut off.

3. Calibrate the application injection equipment flow by connecting an output line to the test substance mix tank filled with water. The output line will have a shut-off valve and an in-line orifice to regulate the flow of the test substance. The desired mix tank flow rate will be achieved by changing the

pressure and/or the in-line orifice to match the injection volume and/or time listed in the protocol. Pressurize the application container to the desired pressure and capture the output flow for a set time (e.g. 30 seconds), then measure and record the output. Repeat for a total of 3 captures. Verify that the output for the three runs does not vary by more than ±5% of the mean and that this output will deliver the appropriate calculated volume/time needed for the application. Calibration procedures and output amounts will be documented in the Field Data Book and other logs, as appropriate.

**NOTE:** The test substance mix tank pressure MUST be higher than the drip line application pressure, or the test substance will not flow into the drip system.

4. To make a test substance application, mix the calculated volume of test substance(s) and water in the test substance mix tank. Connect the test substance output hose (with valve closed) to the drip line injection port and pressurize the drip system to the desired pressure. Visually confirm the system is operating normally (no large leaks, pressurized to the ends of the runs, etc.). Connect the propellant (e.g. $CO_2$) to the test substance mix tank and pressurize to the desired pressure. Once it is confirmed that the drip system and test substance mix tank are at the correct pressures, open the valve on the application hose and begin timing the application. If the application is calculated to take more than 30 minutes, the test substance application container should be agitated periodically to ensure test substance mixing.

   Once the liquid runs out of the test substance application container, record the elapsed time and shut off the valve in the application injection line. Continue to run the drip system to allow the test substance to be uniformly applied. If the protocol does not specify a volume/time for post application water, then the system should be run for at least 30 minutes after the test substance application.

5. The test substance mix tank and application line will be cleaned immediately after use. The mix tank will be rinsed with clean water. The tank will then be partially filled with enough water to flow through the application hose and orifice for 2 minutes. After this initial rinse, the process will be repeated with a tank cleaner (or ammonia)/water mixture and then with clean water one more time. Cleaning will be documented in the maintenance records for the equipment.
6. Personnel currently operating the equipment are responsible for the maintenance and remedial action taken in case of malfunction.

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**SOP#:** 4.14 **Revision Number:** 2

**Title:** Use, verification, and maintenance of GPS device.

**Purpose:** To establish guidelines for the verification of a GPS device (Tablet and Google) in order to record plot location of IR-4 magnitude-of-residue trials at the NCSU IR-4 Field Research Center.

**Scope:** All facilities where trials are conducted under good laboratory practices (GLPs).

**Procedures:**

Use

1. Open Google Maps or Google Earth application on device
2. Zoom in on current location.
3. Select current location and record coordinates.

Verification

The accuracy and precision of the GPS device shall be verified annually to determine if it is within the desired tolerance.

1. Locate a permanent marker clearly visible in Google Maps or Earth
2. Use GPS device to record coordinates of permanent marker at least three times.
3. Export GPS coordinates to Google Maps or Earth
4. Use Google Maps or Earth ruler tool to compare recorded locations versus known location. All recorded locations must be within 30 feet of known location for device to be considered accurate and precise. If locations are not within 30 feet reinstall application and repeat above steps. Note any operations in appropriate log.
5. Record date, time, coordinates and proximity in appropriate log

Maintenance

1. All device maintenance activities will be documented in appropriate log.
2. Personnel currently operating the device are responsible for the

maintenance and remedial action taken in case of malfunction.

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**SOP#:** 5.1 **Revision Number:** 16

**Title:** Commodity production and maintenance.

**Purpose:** Assure that commodities are grown under best management practices.

**Scope:** All locations developing data on test crops.

**Procedures:**

1. Refer to an up-to-date publication on the production of the commodity under trial. If no such publication exists, consult with agricultural specialist (i.e., University extension specialist, county/regional extension agent, grower, etc.) familiar with the production practices for the commodity.
2. It is imperative that any maintenance activities in trials conducted under good laboratory practices (GLPs) be performed in ways that will not impact sample integrity
3. A soil sample will be obtained and analyzed for soil texture, organic matter, cation exchange capacity and pH from the trial site.
4. Lime, fertilize and/or condition the soil at the site as necessary to bring the soil within the requirements of the commodity. This will normally be performed by research station personnel or grower, depending on the trial location.
5. 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.
6. If maintenance pesticides are applied to the commodity, they should be applied according to the label directions. For residue trials, no pesticide should be applied that would interfere with the chemical analysis of the pesticide under evaluation. If in doubt, consult the analytical chemist, analytical laboratory or study director identified in the protocol to determine if a maintenance chemical may be used.
7. Perform other cultural practices as necessary to establish and maintain the commodity.

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**SOP#:** 5.2 **Revision Number:** 13

**Title:** Method for seeding or transplanting.

**Purpose:** Help ensure that an appropriate and high quality crop is produced in trials conducted under good laboratory practices (GLPs).

**Scope:** All locations developing data on test crops.

**Procedures:**

1. Determine the correct species and variety to use as specified by the study protocol. If the variety is not specified, select a variety commonly used in the area by commercial producers. With transplanted crops, plants as uniform in growth and color as possible will be used.

2. Determine within and between row spacing and seed/transplant depth as specified in cooperative extension services recommendations.

3. If exact seeding rate cannot be attained due to seed size and/or equipment capabilities, thinning of the emerged crop to the proper population will be performed.

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**SOP#:** 5.3 **Revision Number:** 12

**Title:** Determining yield or 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 trials where the protocol requires yield data. (Handling of residue samples is covered under NCSU IR-4 Field Research Center SOP 8.1).

**Procedures:**

1. Check the protocol for information on time of harvest. If none, follow commercial practices in the area for the time of harvest of the commodity. These practices should be documented in the raw data notebook.

2. Where grading standards are known or exist, the harvested commodity should be graded, accordingly.

3. 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.

4. 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.

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**SOP#:** 6.1 **Revision Number:** 17

**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 good laboratory practice (GLP) trials

**Procedures:**

1. After the sprayer has been inspected and calibrated, empty the water from the tank.

2. Measure the amount of water needed for the application into a separate container. Make sure this amount is sufficient to cover the entire plot. Make sure the chosen spray tank will hold this amount and the calculated amount of test substance concentrate needed.

3. For some formulations (i.e. wettable powder), it may be necessary to make a slurry mix first by adding the concentrate to a small volume of water in a separate, clean container prior to placing in the spray vessel. To do this, remove a generous amount of water from the calculated amount for total mix. Add the slurry to the water in the spray tank. Using the remainder of the water that was removed earlier, thoroughly rinse the container that held the slurry into the spray tank.

4. Add the remaining portion of the removed water to the spray tank, so that final volume and mix match the mix calculations. Close and tighten the lid. Rinse the outside surface of the spray tank with clean water, if needed.

5. Agitate the spray mix before and, if possible, during application to insure an even mix of the test substance and water.

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**SOP#:** 6.2 **Revision Number:** 14

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

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

**Scope:** All locations conducting field trials

**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 travelling at the correct speed and turn on the sprayer or release the granules. Maintain the correct speed for each pass 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 will do the actual application and one will 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. If the material being applied is US EPA registered, unused spray material should be applied to an overplanting of the crop at a distance adequate to prevent contamination of the test plot by drift or downslope movement of water. In the event that no overplanting of the crop exists, unused material may be placed along the field edge so long as contamination prevention measures listed above are followed.

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**SOP#:** 6.3 **Revision Number:** 17

**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. 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. Record in the appropriate log the amount of granular material used in the trial(s).
2. Liquids-If labeled, unused spray material should be applied to an overplanting of the crop at a distance adequate to prevent contamination of the test plot by drift or downslope movement of water. In the event that no overplanting of the crop exists, unused material may be placed along the field edge so long as contamination prevention measures listed above are followed. If excess spray material is applied to an overplanting of crop, this area will be marked as treated and consumption prohibited.
3. In a suitable area away from aquatic areas or danger of aquatic contamination, rinse the granular applicator to remove pesticide dust from the inside and outside. After application of a liquid mixture, the container/tank will be rinsed with water, then rinsed with water plus tank cleaner or ammonia, then rinsed with water again. The entire spray system will be flushed through with a tank cleaner (or ammonia) /water mixture, then flushed through with water alone. Cleaning should be recorded in the appropriate log(s).
4. Follow the disposal procedures for pesticide rinse water in accordance with current policies and guidelines of the state.
5. Properly dispose of expendable protective clothing. Clean non-disposable items such as respirators, face shields, and other equipment by following the manufacturer's instructions or with soap and water as appropriate.
6. After the application equipment is dry, lubricate those parts requiring lubrication and return the equipment to storage.

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**SOP#:** 6.5 **Revision Number:** 14

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

**Purpose:** To explain the procedures required in the event of a malfunction during the application of the test substance.

**Scope:** All locations where test substances are used. For the purposes of this SOP, the term '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. In the event of a malfunction (i.e., a nozzle is plugged or a hose breaks), the operator should take immediate action to correct the situation.
3. The affected portion of the plot should be flagged.
4. Appropriate individuals (e.g., the study director) should be notified of the incident, details recorded in the raw data notebook, and maintenance records retained in the appropriate log(s).

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**SOP#:** 7.1 **Revision Number:** 12

**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 trials.

**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 beginning of data collection, provide verification that the system is working by collecting data from the first plot electronically and also have someone record the data by hand. At the end of the data collection period, the printout of the electronic data and the hand-collected data should be signed by the person collecting the data. If both sets of data are in agreement a signed and dated statement to that effect should be written in the maintenance log.
4. Prompts should be used as much as possible to avoid any confusion in collecting the data. Where feasible, the prompts should state the plot # from which the data is being collected, the current date, and the type of data being collected.
5. Data should be taken in an orderly fashion to avoid any confusion.
6. At the end of the data collection period, the data should be transferred to a storage system and immediately printed out with appropriate identification. This hard copy must be dated and signed then stored in the trial(s) file folder.
7. All remote sensing and other automatic data collecting and/or recording devices should be inspected and calibrated.
8. Prints of data from these devices must be legible to persons with normal vision and dated and signed when printed or plotted.
9. Hard copies of computerized data and/or other written or plotted date sheets must be dated and signed, and retained in the file folder of the

project.

10. Each data sheet from a monitoring device should be marked in ink with the name of the trial ID number, dates (day, month, 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 **Revision Number:** 13

**Title:** Recording of raw data on paper.

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

**Scope:** All locations conducting trials.

**Procedures:**

1. All raw data will be recorded in indelible ink.
2. Changes to the raw data should 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 | NR = Not Recorded |
| IE = Illegible Entry | IW = Inappropriate Word |
| TE = Transcription Error | AW = Accidental Write-over |
| UE = Unnecessary Entry | PE = Pagination Error |
| LE = Late Entry | NA = Not Applicable |
| NI = New Information | |

   Other error codes can be used, however, the codes must be noted in the IR-4 Field Data Book.
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 **Revision Number:** 15

**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 must be rounded off as having no more significant figures than the measurement with the fewest significant figures.

   b. Addition and subtraction: the result is rounded off to the same number of decimal places as that of the term with the least number of decimal places.

3. When a manual calculation involves two or more steps, retain at least one additional digit (insignificant figure) for intermediate answers. Round off at the end.

4. When using computer(s) or calculator(s), serial calculations should be done with unrounded numbers and the final result is to be rounded.

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**SOP#:** 7.4 **Revision Number:** 13

**Title:** Method for collecting efficacy and phytotoxicity data.

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

**Scope:** All locations conducting field trial(s).

**Procedures:**

A. Phytotoxicity data:
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:

1. Where possible, take phytotoxicity data within 48 hours after the treatment, 1 week later and at the termination of the trial(s). If symptoms occur during this period that warrant a reading, then additional phytotoxicity data should be taken as necessary.

2. Assign a phytotoxicity rating of 0 to 100 for each plot; 0 = plant healthy. 100 = plant dead. 1 thru 99 = the percentage necrosis, yellowing and/or stunting of the plants in each plot.

B. Pest data:
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:

1. Where possible, take pest data within 48 hours after the treatment and at various intervals thereafter depending on the pest life cycle and at the termination of the trial(s).

2. Disease data - Record the name of the disease(s) being observed. Record the symptom(s) for each disease. Randomly select 5 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. Zero = plant healthy. Ten = plant dead. One through nine = the percentage disease appearing on the plant. it there are less than five plants/row, record data from 5plants/plot or all the plants in a plot.

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 5 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. Zero = plant healthy. Ten = plant dead. One through nine = the percentage damage appearing

on the plant. If there are less than five plants/row, record data from 5 plants/plot or all the plants in a plot.

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 diam. 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. (See article in Appendix)

   If there are less than 10 plants/row, record data from all the plants in a row.

   For nematode population counts-take a random sample of the pest population (i.e. root system of 2 plants/plot, 4 3-in diam. 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 used. 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 percentage (%) of the area (to the nearest 5%) covered by weeds. Record the names of the 5 most prominent weed species and percentage control of each species relative to the non-treated check (to the nearest 5%) in each plot. Randomly place a grid covering an area of 1 $ft^2$ and divided by quadrants in the plot. Where possible, count the number of weeds in the grid. If weeds are too numerous, 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 **Revision Number:** 13

**Title:** Experimental design and data analysis.

**Purpose:** To assure that all efficacy, yield, and phytotoxicity data developed are statistically sound.

**Scope:** All locations conducting trial(s)

**Procedures:**

1. The experimental design as specified by the protocol should be used. If none is designated, then the researcher should use a commonly accepted experimental design such as a complete randomized block design. 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 trials is for magnitude of the residue only.

3. Randomly assign the treatments.

4. Select an appropriate statistical package for data analysis and record sufficient information to identify the statistical package (i.e. Date, Revision no., Title, Authors, Source etc.) and determine the level of significance for the trial(s).

5. 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.

6. Report the data as required by protocol, in tabular and/or narrative format.

7. 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 **Revision Number:** 18

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

**Purpose:** To assure that all data resulting from the trials is retained and usable.

**Scope:** All locations conducting trials.

**Procedures:**

1. It is the responsibility of the Field Research Director (FRD) to see that all raw data, summaries and other items connected with the trial are properly retained prior to sending the data to IR-4 National Headquarters or the Regional Field Coordinator (RFC).

2. The FRD will see that all raw data, summaries, data logs, etc. connected with a trial are maintained during the active life of each project for which they are responsible. Afterwards, all raw data that is not directly used in a report is to be archived per procedures described in SOP 9.5. This also pertains to dated and signed hard copies of electronic data, computerized summaries etc. These should be placed in the file as soon as possible after the information is generated.

3. All notebooks, data sheets, summaries etc. should be clearly marked with the project identification number and any other information that may be needed to understand the data and its source.

4. Computer software or on line programs (i.e., SAS) used in the trial should be noted in the data book and information on the title, source, revision or other identifying information should be recorded and the data maintained and updated as needed and filed in the data book.

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**SOP#:** 8.1 **Revision Number:** 16

**Title:** Sample collection, identification, and records.

**Purpose:** To assure proper collection and identification of residue samples,

**Scope:** At locations where trials 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 (SD) and Quality Assurance Unit (QAU) should be kept informed when the dates are changed.

2. Collection of samples during periods of inclement weather should be avoided if possible.

3. Representative samples of the crop in each plot must be taken by a recognized procedure. Follow the protocol or record in the Field Data Book (FDB) 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 treatment individually beginning with the untreated plot(s) and progressing through to the highest dosage.

    a. If sampling in this manner is not possible, cross-contamination of samples must be avoided and methods used to prevent cross-contamination should be thoroughly described in the FDB.

    b. Samples may also be simultaneously collected from different plots by different persons. If this is done, it should be clearly explained in the FDB.

    c. Samples from each treatment 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. Take care not to allow the outer surface of the sample bag to contact treated crop during harvest

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, outer leaves on 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.

It is highly recommended that for 'juicy' commodities (berries, fruits and vegetables that have been sectioned to reduce sample weight, etc.), a large resealable bag be added inside the sample bag to prevent these juices from freezing into the cloth of the sample bag.

8. Prior to sample collection, obtain a sufficient number of sample bags to collect all the samples required by protocol.

9. Before entering the field, use waterproof ink to fill in the label attached to the bottom of the bag and indicate the study ID number and bag number on the tag if more than one is used for the plot sample. Use of pre-printed adhesive labels is permitted provided that all required information is present. If no tag has been provided, then label each sample bag with waterproof ink with the following:

a. PR Number
b. Commodity (Crop)
c. Chemical
d. Replicate Number
e. Date sampled
f. Investigator Name/Address/Phone Number
g. Container Number (if more than 1 container for a sample)

If a resealable plastic bag is used, as indicated in Step 7, this card SHOULD

NOT be placed inside the resealable plastic bag, as it may become frozen into the sample and create difficulties at the analytical laboratory.

10. Sample bags should be burst proof. Cloth laminated plastic bags are preferred.

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**SOP#:** 8.2 **Revision Number:** 19

**Title:** Residue sample packing and storage procedures.

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

**Scope:** All locations where residue samples are collected.

**Procedures:**

1. If samples require refrigeration or freezing prior to shipping to the residue laboratory, containers with ice or blue ice in sufficient quantity to preserve the samples prior to storage should be taken to the site. Otherwise cartons of sufficient size and burst proof strength to hold the samples should be used.
2. Carefully place the sample as it is collected (or cleaned according to protocol, if required) in the sample bag marked for that 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 placed in storage as soon as reasonably possible. If the time between sample collection and placement in the freezer is expected to be greater than one hour, temperature will be monitored using a device such as a minimum/maximum thermometer.
6. Consult the study protocol for the method, temperature, and maximum length of time for storage, if listed. If specifications are not given in the protocol, samples will be stored in a freezer at <32 F and shipped as soon as possible.
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 will be continuously monitored using a maximum/minimum thermometer, or other calibrated device, and will be recorded in a freezer temperature log.

9. The sample storage freezer(s) should have limited access.

10. A log should be maintained for the items inside the storage equipment (i.e. freezer, refrigerator etc. ) indicating the trial number, sample ID number, collection date with initials and removal date with initials. Removal of the samples prior to shipment should be recorded in the log with the name or initials of the person removing them, what sample bags or parts thereof were removed, date removed and date returned.

11. Freezer temperature does not have to be recorded if no residue samples are being stored.

12. An alarm system will be used to monitor the temperature range of the samples.

    a. The alarm system will be tested at least every 2-3 months by removing the sensor from the freezer and allowing it to exceed 32F and trigger the notification process that has been programmed into it. These tests, including documentation of 'pass' or 'fail', will be recorded in the appropriate facility logs.

    b. In the event of a test 'failure', the operator is to refer to the alarm user's manual located in the NCSU IR-4 Field Research Center archives for troubleshooting guidance. If no resolution of the problem can be achieved, call manufacturer for assistance.

13. If freezer malfunctions and samples cannot be maintained at the desired temperature, samples will be moved to a functioning freezer. Transfer of the samples to the functioning freezer should be well documented by recording the date of transfer, sample trial number, and number of samples and the new location. Storage temperature in the new storage equipment should be monitored, including logbook entries, as stated above.

14. A log shall be kept for any and all freezer repairs or maintenance activities. These activities shall be denoted as Routine or Non-routine in the log.

15. Maintenance and repair activities shall include but not be limited to any electrical or structural activities as well as simple defrosting of the freezer(s). Defrosting will also be recorded in the log. Defrosting should be performed in the absence of samples. However, if samples are present when defrosting is necessary, all samples and blue ice will be removed. Samples will be transferred to another freezer and documentation of the transfer will be shown as described in # 13 above. Blue ice will be allowed to completely thaw and dry prior to replacement in the freezer so that no condensation is present that may create extra ice build

up in the freezer(s).

16. Personnel conducting maintenance or repairs are responsible for documentation of actions taken.

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**SOP#:** 8.3 **Revision Number:** 17

**Title:** Sample shipping procedures.

**Purpose:** To assure that residue samples are removed from storage and shipped to the residue laboratory with a minimal or no loss of integrity.

**Scope:** All locations where residue samples are stored.

**Procedures:**

1. For overnight shipments, make arrangements with the carrier for shipment of the samples and determine any special packing instructions that are required to preserve the sample integrity. Note any limits on quantity of dry ice that may be set by the carrier. Obtain insulated containers, if necessary, of sufficient size and quantity to hold the residue samples and dry ice (where required) in a sample : dry ice weight ratio of at least 1:3 and 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. Air freight shipments should be made on Monday or Tuesday to avoid potential weekend layovers, and shipment during holidays should be avoided.

2. For freezer truck shipment, contact shipping company and arrange shipping date

3. Notify the laboratory with trial number, shipment date and method of shipment including the carrier and carrier schedule.

4. Complete appropriate residue sample shipping forms and send them, via fax or e-mail, to the Study Director (SD), Regional Field Coordinator (RFC) and residue chemist.

5. Place copies of the completed residue sample shipping forms and any chain-of-custody forms that may be required in waterproof containers and place in each of the sample shipping containers.

6. Label each container with the following information:
   a. Study Identification Number
   b. Return Name and Address of the sender
   c. Name and Address of the residue laboratory receiving the samples
   d. Affix "Experimental Samples-Perishable" sticker on each carton
   e. Where used, affix "Dry Ice" on two sides of the container

f. When appropriate, label as box _____ of ____.

7. Tie or tape lids of each container firmly in place.

8. Provide the carrier with the samples for shipment.

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**SOP#:** 8.4 **Revision Number:** 7

**Title:** Forced-air drying of RAC (Raw Agricultural Commodity) samples.

**Purpose:** To assure that all forced-air dried samples meet protocol specifications.

**Scope:** This SOP describes the procedure that will be used at the NCSU IR-4 Field Research Center for drying commodities to meet protocol sample moisture requirements.

**Procedures:**

I DETERMINE DRY MATTER PERCENTAGE OF CROP

1. Turn on drying oven(s) and set to desired temperature, per protocol. Document this temperature and temperature calibration information for each oven used for inclusion in the Field Data Book (FDB). Drying information for PRE samples may be documented using NCSU IR-4 Field Research Center Form 8.4-A.

2. A few days ahead of Raw Agricultural Commodity (RAC) sampling, collect PRE samples. Collect at least two samples for each drying oven to be used. Identify PRE samples with a different nomenclature than the RAC samples of the trial. If protocol lists RAC samples alphabetically (A,B,C...), PRE samples should be identified numerically (1,2,3...). Sample identification must be maintained while samples are drying. Document the procedure for maintaining identification in the FDB. At the NCSU IR-4 Field Research Center, this is typically done by writing the sample identification onto the paper (See SOP 4.12) that lines the drying tray.

3. Record 'wet weight' of each sample.

4. Place each PRE sample in drying oven in a way to ensure maximum air flow through the PRE samples. At the NCSU IR-4 Field Research Center, this will typically be a screen-bottom tray with a paper lining. Document this configuration for inclusion in the FDB.
5. Monitor these samples and record the weights as they dry. When no appreciable change in sample weight occurs (1% or less), record final, 'dry weight' of PRE samples.

6. Dry matter percentage can now be calculated:

a. The formula:

**((wet weight – dry weight)/wet weight)*100**

results in moisture percentage of the PRE sample

| **EXAMPLE** |
| --- |
| ((400g wet sample – 100g dry sample)/ 400g wet weight) = 75% moisture |

b. The formula:

**100% - % moisture**

results in dry matter percentage of the PRE sample

| **EXAMPLE** |
| --- |
| 100% – 75% moisture = 25% dry matter |

## II DETERMINE TARGET DRY WEIGHT FOR RAC SAMPLES

1. Using the dry matter percentage calculated from above, the following formula:

**(dry matter portion of PRE samples * wet weight of RAC sample)**
**dry matter portions required by protocol**

results in the acceptable dry weight range of the RAC sample.

| **EXAMPLE:** |
| --- |
| **PRE sample dry matter percentage = 25** |
| **RAC sample is 4 lb and protocol requires dry matter of 80% to 90%** |
| (0.25 * 4 lb)/.8 = 1.25 lb, the RAC sample weight at 80% dry matter. |
| and |
| (0.25 * 4 lb)/.9 = 1.11 lb, the RAC sample weight at 90% dry weight. |

## III DRYING THE RAC SAMPLES

1. Use temperature settings that match the setting used in Part I. Drying information for RAC samples may be documented using NCSU IR-4 Field Research Center Form 8.4-B.

2. Use separate ovens to dry samples from different treatments. Document oven information (brand, model, and serial number) for inclusion in the FDB.
3. Record 'wet weight' of each RAC sample and calculate desired 'dry weight' range for each, per protocol, using formula in Part II.
4. Sample identification must be maintained while samples are drying. Document the procedure for maintaining identification in the FDB. At the NCSU IR-4 Field Research Center, this is typically done by writing the sample ID onto the paper that lines the drying tray.
5. Place each RAC sample in drying oven in a way to ensure maximum air flow through the RAC samples. At the NCSU IR-4 Field Research Center, this will typically be a screen-bottom tray with a paper lining. Document this configuration for inclusion in the FDB.
6. Monitor each sample as it dries, until the weight reaches the desired range calculated for each RAC sample.
7. Verify moisture percentage of the sample:
   a. The formula

$$100 * \left[ \frac{\text{(final RAC dry weight)-(RAC wet weight * Dry matter portion of PRE)}}{\text{(final RAC dry weight)}} \right]$$

results in moisture percentage of the dry RAC sample

**EXAMPLE:**
**RAC wet weight = 4 lb**
**Final RAC dry weight = 1.18 lb**
**Dry matter portion of PRE sample = .25**

$$100 * \left[ \frac{(1.18)-(4.0 * 0.25)}{(1.18)} \right] = 15.25\ \%\ \text{moisture}$$

   b. The formula:

**100% - % moisture**

results in dry matter percentage of the RAC sample

| **EXAMPLE** |
| --- |
| 100% – 15.25 % moisture = 84.75 % dry matter |

8. All calculations and any forms used during the drying shall be inserted into the FDB.

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**SOP#:** 9.1 **Revision Number:** 18

**Title:** Raw data report forms.

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

**Scope:** All locations conducting IR-4 good laboratory practice (GLP) trials

**Procedures:**

1. IR-4 Headquarters or other trial sponsor will provide a Field Data Book (FDB) to each of the cooperators for each trial undertaken. Detailed instructions are provided in the book and in the IR-4 Field Data Book Guidance Document located on the IR-4 webpage: FDBGuidanceDoc.pdf (ncsu.edu).
2. Paper forms should be filled out legibly and mistakes should be crossed with a single line, initialed, dated, and the reason for change given or an assigned code for reason. Suggested codes for these entries can be found in NCSU IR-4 Field Research Center SOP # 7.2.
3. Blank forms may be photocopied as needed.
4. The forms provided in the FDB 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 FDB.
6. If a particular paper form or section of paper form does not require a response, make a diagonal line across this form or section. Initial and date the diagonal line. Empty fields of two or more lines require a diagonal line with initials and date.
7. For IR-4 sponsored trials, number each page (i.e. Part *X*, Page *Y*) within each section of the raw data book, where *X* is the section of the field data notebook and *Y* is the current page number in that section. For trials with non-IR-4 sponsors, pagination will be done per sponsor requirements, if different from the IR-4 format.

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**SOP#:** 9.2 **Revision Number:** 14

**Title:** Handling completed report forms that transcend two or more trials.

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

**Scope:** All locations conducting trials.

**Background:** Where a field research director (FRD) is conducting multiple trials during the year, there may be an opportunity to utilize one form for data that pertains to more than one trial. Examples may be logs of various types, 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 procedure is designed to meet the GLP and FIFRA requirements where copies of data are used.

**Procedure:** Each copy that is to be used for data common to more than one trial should contain a notation as to the trial that has been designated as the one containing the original raw data. This should read: "NCSU IR-4 Center, TRUE COPY, Location of Original _____". The copy should be initialed, dated and placed in the Field Data Books (FDBs).

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**SOP#:** 9.3 **Revision Number:** 20

**Title:** Disposition of paper raw data from trials

**Purpose:** To assure that raw data are sent to the archives.

**Scope:** All locations conducting trials where the original raw data is not archived at IR-4 Headquarters.

**Procedures:**

1. The field research director (FRD) will make an exact copy of the original completed paper field data book (FDB); including correspondences, protocol deviation forms, logs, and any accompanying documentation such as weather charts etc. The original FDB will be forwarded by the FRD to the regional field coordinator (RFC).

2. The FRD will retain the true copy of the FDB from #1 at the field facility. This copy shall be authenticated by stamping all pages as TRUE COPY or preferably, by including an index that lists trial number, name of trial and number of pages in each section along with researcher's signature. This copy may be discarded only after a report on the data has been submitted to EPA or the related study is cancelled. Researchers may also obtain guidance on discarding copies of raw data.

3. Any original document that is copied, in part or in total, to help complete raw data requirements of a field trial will be archived at the NCSU IR-4 Field Research Center per procedures described in SOP 9.5.

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**SOP#:** 9.4 **Revision Number:** 17

**Title:** Retention of data.

**Purpose:** To assure that all data and documents connected with good laboratory practice (GLP) trials are archived.

**Scope:** All locations conducting GLP trials

**Procedures:**

1. The field research director (FRD) will see that a true copy or original of any site-specific documents are retained at the field facility to assure that raw data is not lost. These retained documents are to be archived per procedures described in SOP 9.5. Per federal requirements (CFR40, Title 40, Chapter I, Subchapter E, Part 160.195), all archived data and documents are to be retained for five years or the life of the registration, whichever is longer.

2. The FRD should maintain a file and items placed in the file should be identified as to the trial they pertain to or the dates when the items were in use. The following is a list of information that should be retained:

    a. True 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.

    d. Copies of completed forms used during the trial and for summaries of the trial data.

    e. Historical standard operating procedures (SOPs).

    f. Master schedule of all GLP trials conducted at the facility.

    g. Organizational charts, training records, job descriptions and CVs (current, out of date, or former employees).

h. Copies of computer software and/or information sufficient to identify outdated computer software or programs that were used in trial so data developed from these programs can be repeated if necessary in the reconstruction of the trial.

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**SOP#:** 9.5 **Revision Number:** 7

**Title:** NCSU IR-4 Field Research Center Archives

**Purpose:** To assure that any original data and/or records used in IR-4 trials that are kept at NCSU are protected and trackable.

**Scope:** NCSU IR-4 Field Research Center original data and records

**Procedures:**

1. The NCSU IR-4 Field Research Center archives are located in fire-proof cabinets housed in N. C. State University IR 4 Field Research Center 3800 Castle Hayne Rd, Castle Hayne NC 28429

2. The NCSU IR-4 Field Research Center field research director (FRD) is responsible for the archives and will designate who shall have access to the archives. These individuals, along with initial permission date and permission termination date, will be noted in the archive log.

3. A log will be used to record removal and return of records to and from the archives.

4. Per federal requirements (CFR40, Title 40, Chapter I, Subchapter E, Part 160.195), all archived data and documents are to be retained for five years or the life of the registration, whichever is longer.

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**SOP#:** 10.1 **Revision Number:** 20

**Title:** Disposal of test substances.

**Purpose:** To assure that test substance 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 trials

**Procedures:**

1. Where institutional policies and guidelines do not exist, the following procedures should be followed.

2. Disposal of test substance concentrate and/or containers.

   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 study, the study director must be consulted. More guidance on IR-4 test substance container disposal is available by opening advisory 2005-01 at the following link:

      https://www.ir4project.org/fc/fc-researcher-resources/field-researchers/

      Researchers may also visit

      https://ir4app.cals.ncsu.edu/Ir4FoodPub/SubstanceDispoSch

      to obtain a listing of containers that he/she may discard.

   b. Where possible, the test substance 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 test substance 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 test substance and/or the empty containers.

3. Disposal of test substance rinse water, unused spray solutions and other dilute test substance waste.

   a. Check State and local laws and regulations to determine any procedures that may exist for proper disposal of test substance solutions.

   b. Dispose of the dilute test substance waste in the field by adding to the spray tank and spraying on an overplanting of the crop where no contamination of plots will occur and where this procedure does not violate any laws or regulations. In the event that no overplanting exists, unused material may be placed along the field edge so long as contamination prevention measures are taken. All test substance solutions should be mixed with the intent of limiting excess amounts of solutions. If excess spray material is applied to an overplanting of crop, this area will be marked as treated and consumption prohibited.

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**SOP#:** 11.1 **Revision Number:** 17

**Title:** Safety and health procedures in handling test substances.

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

**Scope:** All locations conducting field trials (including greenhouse).

**Procedures:**

1. Where institutional policies and guidelines do not exist, the following procedures should be followed.
2. All personal protective equipment and clothing as required by the label or written SOPs should be worn in the handling of test substances for storage, mixing and application. Emergency personal protective equipment (e.g. coveralls, self-contained breathing apparatus) must be available when handling hazardous test substances such as restricted use pesticides.
3. Appropriate weather conditions for the application of the test substance should prevail. Schedule application for desirable conditions, if possible.
4. All precautions should be taken to avoid applying test substances to or near sensitive areas or where drift to these areas may occur.
5. 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 it is necessary to adjust or repair any moving parts. Never blow out nozzles, hoses, or clogged lines by mouth. Inspect all test substance containers for leaks, tears, or holes before handling. Do not mishandle or abuse containers and thereby create hazards and/or emergencies by carelessness.
6. All test substances 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, if labeled. In the event that no overplanting exists, unused material may be placed along the field edge so long as contamination prevention measures are taken. If excess spray material is applied to an overplanting of crop, this area will be marked as treated and consumption prohibited. The equipment should be washed off both inside and outside

and all test substances and test substance containers should be returned to storage as soon after use.

7. Treated areas should not be entered until adequate time, as specified by information on the test substance, has elapsed.

8. Do not permit unauthorized persons in the test substance storage area.

9. Do not store test substances 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 test substances are present.

10. Do not drink, eat food, apply cosmetics, or use tobacco in areas where test substances are present.

11. Wear protective gloves while handling containers and mixing or measuring test substances.

12. Do not put fingers in mouth or rub eyes while working with test substances. Personnel should avoid touching moustache, if applicable, to avoid inhalation of compound(s).

13. Test substance storage areas should be properly ventilated.

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**SOP#:** 12.1 **Revision Number:** 14

**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.

**Scope:** All locations conducting field trials under good laboratory practices (GLPs).

**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 trials and/or facility 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 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 trials or facility and make sure everyone 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 inspection are available. This would include:

        1.) Master schedules for Field Research Director (FRD).

        2.) Study protocol and standard operating procedures (SOPs).

        3.) Raw data, correspondence and logs.

4.) Training records, C.V.s, job descriptions, etc. of personnel.

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.

5. Have accessible organizational charts, a map of the facility and any information specific to the facility or area that will make the inspection go smoothly.

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**SOP#:** 12.2 **Revision Number:** 13

**Title:** Procedures to follow during an EPA inspection.

**Purpose:** To provide guidance to study personnel in responding to a request for an EPA audit.

**Scope:** All locations conducting field trials

**Procedures:**

1. Greet the inspection team and follow any institutional procedures for signing in. Provide name tags and 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 trials. 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 smoothly.
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 **Revision Number:** 13

**Title:** Procedures to follow after an EPA inspection.

**Purpose:** To provide guidance to study personnel in responding to a request for an EPA audit.

**Scope:** All locations conducting field trials.

**Procedures:**

1. Make sure that all personnel involved in the inspection are 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#:** 6.4 **Revision Number:** 19

**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. Upon notification of test substance arrival at receiving point, NC State IR-4 personnel will collect test substance as soon as possible, preferably within 24 hours.
2. The test substance should be stored in the pesticide storage facility until it is needed for use in the trial(s). When a test substance is removed or transferred to a different location, removal date, return date, and the amount removed are to be recorded. The temperature range that the test substance is exposed to while out of storage will also be recorded. Removal of test substance prior to the day of use is allowable so long as proper documentation, according to item #4 below, is performed.
3. The storage temperatures of the test substance should be recorded in the raw data. A data logger along with another backup device, will be used for continuous monitoring of pesticide storage temperature. Calibration of these instruments is outlined in SOP 4.8.
4. When a test substance is removed from storage, the following should be recorded in the test substance use log:
    a. complete trial number
    b. test substance name and lot/batch number
    c. removal date and initials
    d. temperature range while out of storage
    e. return date and initials
    f. amount removed and initials
5. The original containers for all GLP test substances must be returned to the manufacturer or retained until completion of the study and/or permission is given by the study director to dispose of the containers.