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TO: Graig Reicks
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FROM: Nicole Soldan, IR-4 Regional Field Coordinator

SUBJECT: STANDARD OPERATING PROCEDURE (5.1) APPROVAL

DATE: August 21, 2023 (Effective Date)

Per 40CRF160 Good Laboratory Practice Standards (GLP), this is to notify you that your Standard Operating Procedure (SOP) in use is approved. Please retain this document with your SOP to fulfill GLP requirements.

SOP	Rev. #	Last Revision Date	SOP	Rev. #	Last Revision Date	SOP	Rev. #	Last Revision Date
1.1	5.1	5-31-23	6.1	retired	2018	9.3	retired	2014
1.2	5.0	3-28-22	6.2	5.0	3-28-22	9.4	retired	2014
1.3	retired	2022	6.3	5.0	3-28-22	10.1	retired	2022
1.4	5.0	3-28-22	6.4	5.0	3-28-22	10.2	retired	2018
2.1	retired	2022	6.5	5.0	3-28-22	10.3	retired	2018
2.2	5.0	3-28-22	6.6	5.0	3-28-22	11.1	retired	2006
2.3	5.1	5-31-23	6.7	5.0	3-28-22	11.2	retired	2006
3.1	retired	2018	7.1	retired	2014	11.3	retired	2006
3.2	5.1	5-31-23	7.2	5.0	3-28-22	11.4	retired	2006
3.3	retired	2018	7.3	5.0	3-28-22	11.5	retired	2006
3.4	5.0	3-28-22	7.4	5.0	3-28-22	11.6	retired	2006
3.5	retired	2018	7.5	retired	2014	12.1	retired	2022
4.1	5.1	7-26-23	7.6	5.0	3-28-22	12.2	retired	2022
4.2	5.0	3-28-22	7.7	retired	2018	13.1	retired	2022
4.3	5.1	7-27-23	8.1	retired	2022	13.2	retired	2022
4.4	5.0	3-28-22	8.2	5.0	3-28-22	14.1	5.0	3-28-22
4.5	retired	2018	8.3	5.0	3-28-22	14.2	5.0	3-28-22
4.6	5.0	3-28-22	8.4	5.0	3-28-22	14.3	5.0	3-28-22
5.1	5.0	3-28-22	8.5	5.0	3-28-22			
5.2	retired	2022	8.6	5.0	3-28-22			
5.3	retired	2022	9.1	retired	2014			
5.4	retired	2022	9.2	retired	2014			

IR-4 North Central Region Field Coordinator's Office

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STANDARD OPERATING PROCEDURES FOR GLP TRIALS

South Dakota State University
Department of Agronomy, Horticulture, and Plant Science
Brookings, SD 57007

These SOP's replace Version 5.0 (dated April 13, 2022). They are submitted as a complete set (Version 5.1) and become effective when approved by the IR-4 Regional Field Coordinator.

SOP's Reviewed by:

Graig Reicks — 8/9/23

Graig Reicks
Field Research Director

Date

SOP's Approved by:

Nicole Soldan — 8-21-23

Nicole Soldan
Regional Field Coordinator

Date

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APPENDIX

1 IR-4 Research Protocol
2 IR-4 Operational Handbook

RETIRED SOPS

NUMBER	TITLE	YEAR RETIRED
1.3	Format for use in developing SOPs	2022 (see SOP 1.2)
2.1	Personnel	2022
3.1	Guidelines for pesticide storage	2018 (see SOPs 6.6 and 13.2)
3.3	Site selection for field studies	2018 (see SOP 5.1)
3.5	Archives facility	2018 (see SOP 7.2)
4.5	Operation and maintenance of farm equipment	2018
5.2	Method for seeding or transplanting	2022
5.3	Commodity maintenance	2022
5.4	Determining yield or quality	2022
6.1	General procedures in the application of pesticides	2018
7.1	Collection of raw data electronically	2014 (See SOP 7.6)
7.5	Completion of summary forms	2014 (See SOP 7.2)
7.7	Data storage during the active life of the project	2018 (see SOP 7.2)
8.1	When to obtain residue samples	2022
9.1	Raw data report forms	2014 (see SOP 7.2)
9.2	Use of report forms for raw data	2014 (see SOP 7.2)
9.3	Handling completed report forms that transcend two or more studies	2014 (see SOP 7.2)
9.4	Disposition of raw data from the study	2014
10.1	General procedures-Archives	2022 (see SOP 7.2)
10.2	Retention times for documents in archives	2018 (see SOP 7.2)
10.3	Information to be retained in the archives	2018 (see SOP 7.2)
11.1	Location and scope of Quality Assurance Research Unit (QARU)	2006
11.2	Responsibilities of the Quality Assurance Research Unit	2006
11.3	Procedures for inspection of a study by the QARU	2006
11.4	Quality assurance report and follow-up at the research testing facility	2006
11.5	Quality Assurance Overview	2006
11.6	Non-Complicance with Good Laboratory Practices	2006
12.1	Disposal of pesticides	2022 (see SOP 6.6)
12.2	Guidelines for the disposal of pesticides	2022 (see SOP 6.6)
13.1	Safety and health procedures in handling pesticides	2022 (see Section 6)
13.2	Guidelines for storing and handling pesticides safely	2022 (see Section 6)

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Version: 5.1
Revised: 5/31/23

Title: 1.1 General requirements for the development and use of Standard Operating Procedures.

Purpose: To provide guidance to scientists conducting studies in the development and use of Standard Operating Procedures in field research.

Scope: All field studies conducted under Good Laboratory Practices (GLPs)

Procedures:

1 The IR-4 Field Research Director (FRD) will develop a standard operating procedure (SOP) for each phase of research. Each one of these documents will be known as an individual SOP.

2 All of the individual SOPs will collectively be known as a complete set of SOPs. The complete set of will be assigned a Version (i.e. Version 5.0), which will appear on the Cover Page.

3 The complete set of SOPs will be reviewed annually by the FRD and revised as needed. After the annual review, a new cover page will replace previous one that will feature the signature of the FRD and the date that the review was completed. If minor revisions are made, a new version (i.e. 5.1, 5.2, etc.) will replace the previous one on the cover page. The Regional Field Coordinator (RFC) must also sign and date the new cover page once they've approved the changes made to the SOPs.

4 Individual SOPs will each be numbered in a similar manner as the complete set of SOPs, with the version near the top of each page. The only difference is that the complete set of SOPs may carry a higher number (i.e. 5.1) than an individual SOP (i.e. 5.0) if the individual SOP wasn't changed in the most recent revision.

5 If major revisions are made, then the complete set of SOPs and all individual SOPs within the set will be renumbered (i.e. 6.0).

6 Any deviations from the SOPs should be noted in the raw data notebook and signed by the Study Director.

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Version: 5.0
Revised: 3/28/22

Title: 1.2 SOP Format

Purpose: To provide a standard for the individual SOP's.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1. **Each individual SOP will begin with a whole number that corresponds to the phase of the study to which the individual SOP applies. For example, all test substance application SOP's will have a whole number of 6. A decimal (i.e. 6.1, 6.2, etc.) will further identify the individual SOP.**

Personnel and SOPs

1._ Field Research Director and SOPs
2._ Other personnel and their training

Test Substance and Application

3._ Miscellaneous (includes spray additives)
4._ Calibration of Measuring and Application Equipment
5._ Field Preparation and Agronomic Practices
6._ Application of Test Substances
7._ Data Collection for Current Trials

Sample Collection, Storage, and Shipping

8._

EPA Insepections

14._

2. Each individual SOP will have the following format

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Version:
Revised: Date

Title: 1.1

Purpose:

Scope:

Procedures:

Title: 1.4 Designation of Field Research Director and responsibilities.

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 The Regional Field Coordinator will designate a Field Research Director for each trial.

2 The Field Research Director has the responsibility for the following:

a Assure that the study is carried out according to the protocol while following SOPs.

b Assure that personnel, resources, facilities, equipment, materials and methods are available as scheduled for the conduct of the project.

c Make sure that all personnel conducting the study understand the protocol and SOP's for the project.

d All deviations reported by the Quality Assurance Research Officer are responded to in writing.

e All raw data, summaries and other items connected with the study that need to be retained are stored in archives.

f Maintain a master schedule for all GLP projects under his/her direction.

g Submit annually a copy of the master schedule to the Quality Assurance Research Officer within 30 days after receiving project assignments.

h Designate the location for the Archives if necessary.

Title: 2.2 Organizational Chart

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's)

Procedures:

1. An organizational chart should be developed which reflects the management of the facility and the reporting lines of the personnel engaged in the GLP studies.
2. Each block in the chart should show the name and title of the person filling that position.
3. The chart must show how the Field Research Director reports to the Study Director and the National Director, and the relationship between the QAU and the other parts of the organization.

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Version: 5.1
Revised: 5/31/23

Title: 2.3 Documentation of GLP Training

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

Scope: All field studies conducted under Good Laboratory Practices (GLPs)

Procedures:

1 The FRD must be GLP trained in all phases of research conducted at the testing facility. The FRD must also have a C.V. that appears in the Field Databook that outlines their GLP qualifications. Training received from workshops and/or conferences (name of event and dates attended).should also be included on the C.V.. A copy training certificates from the events should be retained in the personnel files at the location.

2 When the FRD is unable to conduct a critical phase of the research, they can appoint an alternate whom they feel confident to perform the task correctly without supervision. The alternate must be GLP trained prior to conducting the task. They must also be familiar with the SOPs that may come into play during that task. The alternate only needs training in the phase of the research they are conducting. For example, it's not necessary for them to understand sample collection SOPs if they are applying a test substance.

3 Documentation of training is required and will be summarized on the alternate's C.V., which will appear in the field databook. This should include the SOPs reviewed with the alternate and the date. It should also include technical training, such as actually witnessing the alternate correctly perform the task with minimal assistance.

4 GLP training is not required for tasks where the FRD or an alternate (as described above) is directly supervising employees. A common scenario would be labor-intensive sample collection that would be difficult for one person to execute. A C.V. is not required for directly supervised employees. However, they are not allowed to record in the field databook. Their training still needs to be recorded in the FDB, along with names of the individual(s) performing the task. For example, ***Joe Smith picked samples A and B. He was shown the method and was periodically inspected to ensure he was performing the task correctly.***

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Version: 5.1
Revised: 5/31/23

Title: 3.2 Guidelines for spray additives in GLP trials

Purpose: To assure that all spray additives are used and stored in a manner consistent with GLP requirements.

Scope: All field studies conducted under Good Laboratory Practices (GLPs)

Procedures:

1. Store in a location that has limited access and according to storage requirements on the adjuvant label.

2. If the additive has a suspicious appearance or smell, discontinue its use in GLP trials. The Study Director should be notified of the situation. The date when the issue was noticed and the Study Director was contacted should be recorded in the field databook.

3. Proper labeling will include: name, concentration, required storage conditions (from the label or Safety Data Sheet), and expiration date. If storage requirements and expiration date are not specified, attach a sticker to the container stating "Store Ambient" and add an expiration date up to 60 months from date of receipt.

4. Spray additives can be subdivided into secondary containers for ease of use. These secondary containers must be properly labeled per the original container and now take on all the requirements of an original container.

5. Since adjuvants don't require constant temperature monitoring, the original or secondary container can be brought to the field as long as it's not exposed to excessive heat or cold for extended periods of time. This shouldn't be an issue if the container is stored in an insulated cooler while traveling inside a climate-controlled vehicle. When the vehicle is stopped for an extended time, either roll down the windows or put the cooler containing the adjuvant in a shaded area.

6. Only newly opened syringes may be dipped directly into adjuvant containers. If a measuring error is made with the syringe, the adjuvant can be added back to the container as long as there's no reason to believe that it has been contaminated. Multiple draws with the same syringe are permitted as long as the syringe hasn't come into contact with any potential contamination source. If there's a reason to believe that contamination could have occurred, the spray additive may no longer be used for GLP studies.

7. If dispensing the adjuvant directly into the sprayer tank, keep the syringe a sufficient distance above the spray solution to avoid contaminating the syringe by splashing. If there's reason to believe that a syringe was contaminated, use a new one.

8. With the exception of temperature monitoring (see Procedure 5), SOPs 4.1 and/or 4.2 will be followed when measuring spray additives.

Title: 3.4 Greenhouse/shadehouse facilities

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

Scope: All greenhouse/shadehouse studies conducted under Good Laboratory Practices (GLP's)

Procedures:

1 Each greenhouse/shadehouse must be sufficiently large enough to contain the entire study or a complete replicate of the study with sufficient space between plots to reasonably prevent contamination.

2 Where more than one study is conducted in a greenhouse/shadehouse, there must be sufficient isolation between the studies to reasonably prevent contamination or interference between studies.

3 Lighting, temperature, humidity, and shade should be sufficiently uniform at the study sites in the greenhouse/shadehouse to provide nearly uniform plant growth throughout the study sites.

4 The walls, floors, and ceilings of the greenhouse/shadehouse should be maintained in good condition. Floors, benches and isles should be free of debris, weeds and superfluous and well-drained to prevent the buildup of excess moisture.

5 Greenhouses should be equipped so as to maintain temperature, lighting, and moisture conditions to simulate commercial greenhouse production techniques or as required by the study protocol.

6 Sufficient monitoring devices should be installed, in good working order, and calibrated periodically to assure that the proper lighting, temperature and humidity conditions are maintained throughout the study.

7 Document cultural practices used in the greenhouse and treatment locations in the raw data notebook.

Title: 4.1 Calibration and use of an analytical balance.

Purpose: To assure an accurate dosage in the application of pesticides.

Scope: All field studies conducted under Good Laboratory Practices (GLPs).

Procedures:

Balance Calibration

1 Calibrate the analytical balance using certified standard weights immediately prior to weighing the test substance. Always use two standard weights, where one is lighter and one is heavier than the amount of test substance being measured.

2 Place a clean, empty container (that will hold the measured test substance) on the balance pan and press the appropriate button to tare the balance. Next, place a standard weight inside the container (preferred) or near the container. Do not touch the standard weights with bare hands. Instead, use a tweezers, clean disposable gloves, or some type of clean laboratory wipe.

3 For the scale calibration to be acceptable, the balance must read within +/-1% of the value stated on the standard weight. Ideally, calibration data will be recorded directly into the field data book instead of keeping a separate log.

4 Analytical balance is inspected annually by a professional calibration business. The report from this inspection is placed in the field databook.

Using the Balance

5 Dry test substances are typically measured on the SDSU campus directly into a separate container for transport to the trial location. Refer to SOP 4.2 for more details on measuring the substance.

6 Weigh the concentrate into the tared container on the balance. Dry substances should be measured to at least three significant figures. For example, if trying to measure 5.00 g, the balance should read three decimal places. In this case, a measurement between 4.995 and 5.004 g would be acceptable.

7 If the balance malfunctions or doesn't pass calibration prior to weighing a test substance, either repair the balance or find an alternative. If a repair is made, then details of this repair will be recorded in the field databook. If an alternate balance is used, record the identity (preferably make/model/serial #) in the field databook. If available, add a professional inspection report of the alternate balance (step 4).

Calibrating the Standard Weight Set

8 Recertify standard weights annually by recording the weight of each standard as soon as possible following professional calibration of the balance. Again, this value should read +/-1% of the value stated on the standard weight.

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Version: 5.0
Revised: 3/28/22

Title: 4.2 Measuring test substances

Purpose: To assure an accurate dosage in the application of a pesticide.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 The original test substance container can be taken to the trial site as long as the container is out of temperature-monitored storage for <24 hrs. If out for storage >24 hrs., then temperature monitoring should occur.

2 If the test substance is measured at the storage location and then dispensed into a secondary container, the secondary container should be labeled with the appropriate trial ID number. Also include the TRT ID if there's more than one treated plot per trial. An exception to this rule is when the secondary containers each have the same amount of substance and it came from the same original container.

4 Measuring devices for liquid formulations shall be graduated in increments within 2% of the total volume being measured for that component of the tank mix (i.e. 50 mL is the smallest volume that can be measured with a device having 1 mL increments). If a meniscus forms in the measuring device, take the reading at the bottom of the meniscus.

5 Syringes or pipettes are the preferred measuring devices because they typically don't require rinsing to remove the substance inside of them. A liquid formulation can also be measured with a graduated cylinder. However, these instruments must be triple rinsed to fully-remove the substance inside. Additional rinses maybe necessary if it appears that three rinses didn't remove all of the substance. This rinsate must be dispensed into the sprayer tank for application to the treated plot. Secondary containers must also be rinsed in the same manner to fully-remove the substance inside.

6 Wear appropriate safety equipment while handling pesticide concentrate. This can vary depending on the toxicity of the pesticide, so refer to the pesticide label for guidance.

7 Return excess to the original container if the integrity of the substance has not been compromised or dispose of the excess by using appropriate methods.

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Version: 5.1
Revised: 7/27/23

Title: 4.3 Calibration of a Sprayer and Calculation of Tank Mix Components

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

Scope: All field studies conducted under Good Laboratory Practices (GLPs).

Procedures:

1 The protocol sets the standards and schedules for sprayer calibration. In addition, the field databook has prompts to enter the required calibration data. If a calibration fails, either fix the sprayer (preferred) or find an alternate sprayer to make the application.

2 All GLP calibrations will be performed with a clean sprayer and results will be documented in the field databook. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary. All repairs and maintenance will be documented in the field data book, whether they require a new calibration or not. The following repairs typically require a new calibration:

- Replacement of spray delivery components (pumps, hoses, valves, pressure gauges, nozzles, nozzle bodies, screens, etc.)

These situations don't require a new calibration if an output or speed recheck is within the acceptable range:

- Switching back and forth between different CO_2 regulators, as long as the pressure adjustment screw hasn't been adjusted since the most recent calibration.
- Switching to a different tank if it's the same type of tank
- Wheel or tire-related maintenance (i.e. adding air, replacing an inner tube, etc.) as long as a single speed recheck is within an acceptable range

3 All sprayers should have a pressure gauge. Select the appropriate type of nozzle to meet the protocol requirements. If none are listed in the protocol, then refer to the test substance label. In the field databook, include the reason for selecting the spray parameters.

DELIVERY RATE CALCULATION

4 For the **output calibration**, adjust the boom height and nozzle spacing for the correct application pattern. Calibrations are often performed without knowing the exact crop height because the trial site is too far away to measure the crop. Therefore, it's acceptable to estimate the crop height when setting the nozzle height for calibration. However, a single recheck at the trial site prior to application should be performed at the correct nozzle height. Determine whether all nozzles are discharging uniformly by spraying water through them at a uniform pressure. Visually evaluate the spray pattern for its complete and symmetrical arrangement. Then catch the discharge from each nozzle in a separate container over a timed interval (usually 20 - 60 seconds, but no less than 15 seconds) beginning after the nozzles are discharging.

5 The **speed calibration** can be performed over the same paths that the applicator will traverse when spraying the plot. Alternatively, a course the same length as the plot about to be treated, over similar terrain, and within a similar crop canopy can be used for speed calibration. Each application pass must be timed with a stopwatch to confirm that the target rate was applied. If practical, the individual applying the test substance may also operate the stopwatch. These timings will be recorded in the field data book.

6 The delivery rate will be automatically calculated in the electronic field databook (eFDB). If for some reason the eFDB is not functioning properly, then calculate the output with the following equation:

$$\underset{\text{nozzle sec}}{\overset{\text{Output calibration}\downarrow}{\frac{11.3\ \text{mL}}{}}} \times \frac{\text{gal}}{3785\ \text{mL}} \times \underset{\text{20 in.}}{\overset{\text{Nozzle spacing}\downarrow}{\frac{\text{nozzle}}{}}} \times \underset{\text{4.17 ft}}{\overset{\text{Speed calibration}\downarrow}{\frac{\text{sec}}{}}} \times \frac{12\ \text{in}}{\text{ft}} \times \frac{43560\ \text{ft}^2}{\text{acre}} = \frac{\textbf{18.72 gal.}}{\textbf{acre}}$$

If manual calculations are performed, the calibration data will also be entered into the eFDB as soon as possible. Once entered into the eFDB, the results (automatically calculated by the eFDB) will supersede the manual calculations, even if a different result is achieved. All manual calculations will still be retained as an item in the eFDB.

VOLUME, MIXING, AND DILUTION CALCULATIONS

7 The volume, mixing, and dilution calculations will also be automatically calculated in the eFDB. If for some reason the eFDB book is not functioning properly, then the following method may be used to manually calculate the mixing and dilutions.

A. Plot area in acres.

50 feet x 100 feet = 5000 $feet^2$

$$5000\ feet^2 \times \frac{1\ Acre}{43560\ feet^2} = 0.115\ Acre$$

Add a minimum of 10% extra area to avoid running out of spray solution

- 20% was used in the example.

0.115 Acre x 1.20 = 0.138 acres

B. Total amount solution applied over plot area.

$$0.138\ acres \times \frac{18.72\ gallon}{1\ Acre} \times \frac{3785\ ml}{1\ gallon} = 9759\ ml\ total$$

C. Amount of test substance. Protocol calls for 1420 ml product / acre.

$$\frac{1420\ ml}{acre} \times 0.138\ A = 196\ ml\ product$$

D. Amount of adjuvant. Protocol calls for 0.5 % surfactant v/v.

0.005 x 9759 ml = 49 ml surfactant

E. Amount of water to add to tank mix.

Amount of water = Total volume – product – surfactant

9514 ml water = 9759 ml – 196 ml product – 49 ml surfactant

8 If manual calculations are performed to determine the tank mix components, and these quantities are actually added to the tank, then they will be used to calculate the rate per acre following the application. If the eFDB calculates different tank mix components after the fact, these will be considered irrelevant.

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Version: 5.0
Revised: 3/28/22

Title: 4.4 Calibration and use of Granulator Applicators

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Determine that the spreader is in good working order and good mechanical condition. Make sure that the openings to release the granular material are not clogged and free of debris.

2 Refer to manufacturer's manual for the calibration method. If no method is available then proceed as follows:

3 Wear protective clothing as necessary and fill the spreader at least half full of the material to be applied. Attach a pan under the spreader to catch the material as it is released.

4 Measure an area of 0.01 acre or 435.6 square feet in close proximity to the area to be treated. If the applicator width is 10 ft, a simple method to calculate the distance is:

$$435.6 \text{ ft}^2 / 10 \text{ ft} = 44 \text{ feet to travel}$$

5 Determine the approximate setting of the openings and the approximate speed to operate the applicator for the desired amount of active ingredient/acre.

6 Operate the applicator over the measured distance and collect the output in the pan attached to the spreader.

7 Weigh the material from the pan and multiply by 100 to give the amount applied per acre.

8 Continue with steps 5 to 7 until the desired rate is achieved within 5% of the total/acre.

9 Example: Formulation= 15% G., rate= 10 lb A.I./acre

$$\frac{10 \text{ lbs A.I.}}{\text{acre}} \times \frac{100 \text{ lbs formulation}}{15 \text{ lbs A.I.}} = \frac{67 \text{ lbs formulation}}{\text{acre}}$$

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

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Title: 4.6 Calibration of instruments and gauges.

Purpose: To assure that temperature monitoring devices used for measurement in a GLP study are reasonably accurate and in good working order.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Electronic measuring instruments used in GLP studies should be periodically tested to confirm reasonable accuracy. Calibration against a standard once per year will be acceptable.

2 Thermometers containing mercury or a mercury alternative (i.e. non-electronic) do not require annual calibration.

3 Calibration points will bracket the working ranges of the instrument during the trial. The working range, for example, are the min. and max. temperatures recorded in the chemical storage facility from test substance receipt until the final test substance application.

4 A good method is to calibrate data loggers against an ASTM or NIST calibrated thermometer inside a freezer for the low range and inside an oven for the high temperature range.

6 Each calibration point should be within +/-1°C of the standard thermometer.

8 A written record should be kept of the dates and results of the tests and of the acceptable tolerance for each instrument.

9 Those instruments that give inconsistent results or are not accurate to within desired tolerances should be repaired or replaced.

10 Refer to the manufactures' manual for the calibration method. If no method is available onsite, then contact the manufacture directly on how to proceed.

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Title: 5.1 Site selection and field preparation for field studies

Purpose: To assure plots are large enough to obtain the required data or samples with sufficient uniformity and can be relocated after the study is terminated.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Locate site with sufficient isolation to minimize contamination from external sources such as commercial operations or other research studies.

2 Permanent reference points are typically required on a plot map. A Global Positioning System (GPS) may be used to define these reference points. It the trial protocol does not specify a minimum number of GPS points to record, then a minimum of 2 opposite corners per plot in the trial shall suffice.

3 GPS accuracy will be checked against a known latitude and longitude reference point before and after taking readings of the GLP trials each year. Three readings will be taken for each GPS accuracy check. Between each reading, the operator will walk away from the reference point (>30 ft) with the GPS in hand, wait at least 1 minute, and then return to record another point. Three points will be recorded for a calibration, with each point within 2m of the standard.

4 If soil sampling is performed to describe the soil characteristics, then a soil probe should be used to collect soil cores randomly throughout the trial location. The soil sample should be of the top 6 in. of the soil profile and consist of 10-15 cores. A single soil sample can represent multiple trials as long as they belong to the same soil series in the USDA-NRCS Soil Survey.

5 Mark trial boundaries with taller orange flags that stand above the crop canopy and can be seen from a far distance. The centers of each application pass are then marked shorter (<1 ft tall) flags of different colors (i.e. blue for pass 1, yellow pass 2, blue pass 3, yellow pass 4). A taller flags are often placed a few feet to the left or right of the shorter flags as a reference when to start or stop the stopwatch for each pass. These flags should be tall enough for the boom to hit them when making the applications.

6 Each plot should have at least one corner marked with the trial number and treatment number. This can be done with permanent markers on brightly colored wooden stakes.

Title: 6.2 Adding the pesticide concentrate to the water 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 field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

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

2 Add about half of the water to the spray tank.

3 If needed (i.e. wettable powder formulation) make a slurry mix first by adding the concentrate to a small volume of water in a separate, reasonably clean container. Add the pesticide concentrate or slurry and adjuvants (if needed) to the water in the spray tank. Triple rinse the container holding the pesticide concentrate (and slurry) using the remaining water not in the spray tank and add the wash water to the spray tank.

4 Add the remaining water to the spray tank. Close and tighten the lid.

5 Thoroughly shaking the sprayer tank by hand just prior to application coupled with the natural splashing inside the sprayer tank during the application usually provides sufficient agitation. Sometimes a protocol and/or pesticide label will recommend constant agitation during the application. If this is a requirement, a spraying system capable of constant agitation will be used.

6 All personnel involved in storage, mixing, application, and cleanup of pesticides will be properly trained.

7 As stated in the test substance measurement SOP (SOP 4.2), wear appropriate safety equipment while preparing the tank mix. This can vary depending on the toxicity of the pesticide, so refer to the pesticide label for guidance.

Title: 6.3 Procedures for the application of the test substance in the field.

Purpose: To assure that the test substance is applied uniformly to the plots.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Application equipment should be inspected and calibrated according to guidelines outlined in Section 4.

2 Make sure all settings of pressure, speed, granular flow etc. are set according to specification from the calibration as previously performed.

3 Where possible, apply the material beginning with the lowest concentration and work up to the highest concentration.

4 Just before entering each plot make sure you are traveling at the correct speed and turn on the sprayer or release the granules. Maintain the correct speed through the plot.

5 Turn off the sprayer or stop granular flow just after leaving the plot.

6 Record pass times in the Field Data Book.

7 Appropriate weather conditions for the application of the pesticide should prevail otherwise the pesticide applications should be delayed.

8 All precautions should be taken to avoid applying pesticides to or near sensitive areas or where drift to these areas may occur.

9 All pesticides should be mixed in quantities which are adequate for the job to avoid excess dilute solutions after the job is completed. Cleanup procedures should be established whereby excess sprays can be safely discarded preferably by spraying the material on an overplanting of the commodity.

10 As stated in the test substance measurement SOP (SOP 4.2), wear appropriate safety equipment while preparing the tank mix. This can vary depending on the toxicity of the pesticide, so refer to the pesticide label for guidance.

11 At the end of the working day, employees who have applied or mixed pesticides should take a shower and change clothes. This clothing should be washed separately. If an excessive amount of pesticide soaked through protective suit, the contaminated article of clothing underneath should be removed as soon as possible and discarded.

Title: 6.4 Application of pesticides in the greenhouse.

Purpose: To assure that the study pesticide(s) are applied uniformly and at the correct dosage.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Application equipment should be inspected and calibrated according to guidelines outlined in Section 4.

2 Make sure all settings of pressure, speed, granular flow etc. are set according to specification from the calibration as previously performed.

3 Where possible, apply the material beginning with the lowest concentration and work up to the highest concentration.

4 Apply the material according to the directions in the protocol or as specified on the label. If fumigants or mist blowers are used, follow instructions of the manufacturer of the equipment. If a fumigant is used, two people are required, one doing the actual application and one who can observe from a safe place to provide rescue assistance if necessary.

5 Calculations should be made to minimize the amount of spray material left in the spray tank. This residue should be sprayed to a similar crop or disposed of according to current policies and guidelines of their institution.

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Title: 6.5 Cleanup of application equipment

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

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. Note in the pesticide log for the chemical, the amount of granular material used in the study.

2. In a suitable area away from aquatic areas or danger of aquatic contamination, hose down the granular applicator to remove pesticide dust from the inside and outside. Triple wash spray machines and apply each wash to the overplanting of the crop.

3. Spray-Excess spray solution should be applied to an overplanting of the crop.

4. If a crop overplanting is not available, then follow the disposal procedures for excess spray solution in accordance with current policies and guidelines of their institution.

5. Add water to the sprayer tank until at least half-full. Add the recommended amount of a specialized sprayer cleaning solution to this water. Hand-shake the sprayer tank vigorously for at least 1 minute. Invert the tank and shake again for another 1 minute. Run this solution through the sprayer hoses and nozzles for at least 1 minute. Then, pour the remaining solution all over the sprayer, especially the boom and nozzles. Rinse the sprayer tank 3 more times with fresh water, each time pouring the water over the sprayer to rinse off the cleaning solution. Run fresh water through the sprayer hoses and nozzles for at least 1 minute.

6. Dispose of expendable protective clothing by placing the items in a container for incineration. Clean non-disposable items following the manufacturer's instructions or with soap and water as appropriate.

7. After the application equipment is dry, lubricate those parts requiring lubrication and return the equipment to storage.

Title: 6.6 Storage, handling, and disposal of test substances

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

Storage Conditions

1 Test substance should be placed in proper storage as soon as possible to protect its integrity. This information is often found on the pesticide label. If not available, store in a room that stays dry, has relatively ambient temperatures, and keep out of direct sunlight.

2 GLP test substances should be stored separately from other pesticides and locked in separate containment within the room.

3 Temperature recording with calibrated equipment (Refer to Section 4 for guidance) should begin within 2 days of receipt and continue until the final application. If using a manual min/max thermometer (as opposed to a datalogger), the minimum and maximum temperature needs to be recorded from the time a container enters the chemical storage facility through the final GLP application. At minimum, this can be a single measurement. However, more frequent measurements (i.e. weekly) are preferred.

4 Store pesticides according to rules of the institution. These rules include having adequate ventilation, fire protection, and a highly visible warning sign placed on the door. Unless supervised, only authorized personnel should be in the pesticide storage room.

5 Make accessible, materials such as adsorptive clay, granulated activated charcoal, hydrated lime, and sodium hypochlorite for emergency treatment or detoxification of spills or leaks.

Identification Test Substances in Storage

5 When the test substance is received, the name on container, batch/lot number, date of receipt, expiration date, quantity received, amounts used, and purpose of use should be recorded in a log. Other information required by the study protocol will also be recorded in the field data book. Each entry should be initialed and dated.

6 Each container should be labeled with a unique ID, which is usually the study number. If more than one container is received for a study, label as 1 of x, 2 of x, etc.

Test Substance Disposal

6 All test substance containers must be stored until notification, which usually found on the IR-4 Website.

7 Dispose of pesticides either by applying them to a field in a legal manner or through a State pesticide disposal program.

Title: 6.7 Procedures to follow when a problem occurs in the application of the Test Substance.

Purpose: To explain the procedures required when something goes wrong during the application of the test substance in the study.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

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

2. If something goes wrong such as a nozzle is plugged or a hose breaks, then the operator should take immediate action to correct the situation.

3. The affected portion of the plot should be carefully marked off and staked to indicate the area affected. This portion should not be used for obtaining samples of the commodity for residue analysis. If the unaffected area is too small to obtain the samples required for analysis, then the trial should be discontinued.

4. Appropriate individuals should be notified of the incident and details should be recorded in the raw data notebook.

Title: 7.2 Recording of raw data and handling this data during QC Field Data Book review.

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures: 1 Under most circumstances, raw data will be recorded into the field data book (FDB) by people who've been GLP trained for that phase of the trial. Raw data shall be recorded in ink, preferably blue, so copies and originals can easily be distinguished.

2 Date and initial each time an entry is made.

3 If a document from a printer, copier, or fax machine constitutes raw data, then this printout should be initialed and dated in ink.

4 Corrections will be made by single line crossing through the item and initialing and dated. A reason for the correction will be noted.

5 No pages containing data will be removed from the book. Transcribing data is discouraged.

6 If a particular form or section of the form does not require a response, make a slashed line (diagonal line from the top of the page or field to the bottom). Initial and date on the slashed line or sign and date at the bottom of the page. If the requested data are not applicable, give an explanation.

7 Where raw transcends two or more studies, copies can be used to reduce the amount of paperwork. One study should be designated as one to contain the raw data. Each copy should contain a notation that reads:

True and Exact Copy
Initial_Date____
Original in_____

8 Make sure that all data required by the study protocol or by the forms provided or used are collected and recorded. Carefully review the forms to make sure that all the required data is being collected. This data includes but are not limited to:

9. Number each form as directed within each part of the raw data book.

10. All completed FDB's will be submitted to the Regional Field Coordinator (RFC) to review for completeness and accuracy. The RFC or designee will follow up to obtain any missing data or correct deficiencies with the Field Research Director's consent.

11. For the most part, original documents will be included in the FDB's to minimize the number of original documents kept at our testing facility. Original documents kept at our testing facility will be those of lesser importance that could easily be recreated if lost or accidentally destroyed, such as organizational charts and facility maps.

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Title: 7.3 Method for collecting efficacy and phytotoxicity data.

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance.

Procedures: A. Phytotoxicity data:

1 Consult the protocol to determine the method and timing of the phytotoxicity data. If no method is cited then reference your method or proceed as follows:

2 Where possible, take phytotoxicity data within 1 week after each treatment or, if specified, by the phytotoxicity rating schedule in the specific IR-4 protocol being followed. If symptoms occur during this period that warrant a reading, then additional phytotoxicity data should be taken as necessary.

3 Often, quality photos of the phytotoxicity with a brief description are sufficient. Consult the Study Director to determine whether more detail and/or measurements are necessary.

B. Pest data:

1 Consult the protocol to determine the method and timing of the pest data. If no method is cited then reference your method(s) for each pest or proceed as follows:

2 Where possible, take pest data within 24 hours before the initial pesticide treatment and within 48 hours after the treatment and at various intervals thereafter depending on the pest life cycle and at the termination of the study.

3 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 thru nine= the percentage disease 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.

4 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 thru 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.

5 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.

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 here. Count and record the number of nematodes by the various life stages/unit of soil or root.

5 Weed data-Visually observe each plot and record the % of the area (to the nearest 5%) covered by weeds. Record the names of the 5 most prominent weed species and the area they cover (to the nearest 5%) in each plot. Randomly place a grid covering an area of 0.1 M^2 and divided by quadrants in the plot. Where possible, count the number of weeds in the grid. If weeds are too numerous to make counting the entire area possible within a reasonable period of time, then count the number of weeds in the lower left quadrant, multiply by 4 and record this value as the number of weeds in the grid.

Title: 7.4 Experimental design and data analysis

Purpose: To assure that all efficacy, yield, and phytotoxicity data developed is statistically sound

Scope: All field studies conducted under Good Laboratory Practices (GLP'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 study is for magnitude of the residue only.
3. Draw a plot map showing the location of each plot in the site selected for testing as described under SOP 3.2.
4. Randomly assign the treatments to the plots using a random number table or random number generator. Note the location of the treatments on the plot map.
5. Retain the plot map in the study folder.
6. Determine the level of significance for the study.
7. Select an appropriate statistical package for data analysis and record sufficient information to identify the statistical package (i.e. Date, Revision no., Title, Authors, Source etc.).
8. When the raw data are available for analysis, utilize the statistical package and follow instructions contained therein to conduct an analysis of variance and mean separation of the data.
9. Record the data as required on the appropriate forms and identify statistically significant differences in the data in the raw data note book.
10. Retain all data, analyses, notes etc. in the study folder with sufficient information to recalculate the data summaries and statistical analyses by another person.

Title: 7.6 Collection and recording of data from electronic devices

Purpose: To describe methods for handling data from remote sensing and other data collecting devices.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1. All remote sensing and other automatic data collecting and/or recording devices should be inspected and calibrated according to their respective SOP. If not required by GLP, some form of inspection and/or calibration is highly-recommended.
2. Check the power supply on portable units to see that it will be adequate during the data collection and data transfer period.
3. Make sure the correct program for data collection is ready and available for use.
4. Electronic data must be legible to persons with normal vision.
5. The original printout should have an initial and date of the person who performed the printing. If this original printout is inserted into the field data book.

Title: 8.2 Method of sample collection

Purpose: To assure that a sample representative of the commodity is taken.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Follow the procedures stated in the protocol to collect representative samples of the plot.

2 Avoid sampling the outer 5 ft of the plot.

3 Sample each replicate individually beginning with the untreated plots and working up to the highest dosage. Treatments from each replicate should be individually packaged and labeled.

4 Take special care to do the following in the sample collection process:

- a Avoid contamination of the field sample with the pesticide under study during the sampling, labeling, storage and shipping processes.
- b Have one person hold the sample collection bag above the ground or place sample collection bag inside a container to avoid contaminating the outside of the bag when sampling treated plots.
- c Avoid taking diseased or undersized crop parts.
- d Take care not to remove surface residues during handling, packing or preparation.
- e Do not transport samples in a vehicle used to transport pesticides.
- f Be certain tools are clean.
- g 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 of lettuce on etc. unless specified otherwise in the protocol.

5 Harvesting plant material in the field and transporting it back to SDSU for subsequent threshing is acceptable as long as the following criteria have been met:

- a Harvested plant material to be threshed into samples should be in separate containers, each labeled with the Trial ID and the Sample ID.
- b Temperature monitoring is not necessary if harvested plant material is threshed and samples are collected on the harvest date.
- c Transportation should occur in a climate-controlled vehicle with a roof.
- d Treated and untreated containers should be physically separated in the vehicle.

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Title: 8.3 Method of sample collection using a combine or small bundle thresher

Purpose: To assure that a sample representative of the commodity is taken.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Many protocols state that grain or seed samples can be collected with a combine or thresher. In this case, we must ensure that the machine is free of grain and/or other plant material from previous harvests. Cleaning a plot combine or small bundle thresher with a liquid solution that one might use to clean smaller sampling equipment is usually not practical on these large machines.

2 If one can not see the entire inside of a harvesting machine to confirm its cleanliness, which is often the case with a plot combine, a sample should be collected from the plot that that meets the sampling requirement stated in the protocol. This sample is considered a cleanout sample and should be run through the machine and properly discarded.

3 In some harvesting machines, one can see inside the entire machine to confirm its cleanliness. This is usually the case with the small bundle thresher. Collecting a cleanout sample (as described in Procedure 2) is not necessary.

4 Sample Collection Method 1 consists of feeding hand-collected plant material to a stationary combine or small bundle thresher operating outside the plot area. Follow the sample method collection method in the protocol.

5 Sample Collection Method 2 consists of driving the combine through the plot and letting the machine collect the sample. Normally, a combine pass diagonally across the plot will suffice for a collecting a sample. Collect a second sample from the plot making a pass in the opposite direction.

6 If the sample is too large, randomly remove handfuls of sample until it is of sufficient size. Record the approximate initial sample weight. The excess sample can be discarded on the ground of the respective plot for eventual destruction by tillage.

7 Use the same procedure to harvest the treated plot or the treated plot of the next highest dosage.

Title: 8.4 Sample identification and records

Purpose: To specify how samples are to be identified and the records needed.

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures: 1 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.

2 Prior to sample collection, obtain a sufficient number of sample bags to collect all the samples with the treatments stored individually by individual replicates and a separate untreated check sample as large as a single treatment combined over the replicates.

3 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 sampled. Each sample bag should also contain a card within a waterproof bag or container the following information (same info. as found on outside of bag):

a. Field ID#
b. Crop Fraction
c. Test substance
d. Sample ID
e. Date harvested
f. Date sampled
g. Field Research Director Name/Phone#

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

5 Upon completion of the sampling, GLP shipping form(s) should be completed. Retain the original of the residue sample shipping form in the project file folder until the samples are shipped to the residue laboratory.

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Title: 8.5 Sample storage and freezer malfunction procedures.

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 If samples can be delivered to the freezers within approximately one hour of sampling, then immediate cooler storage on ice is not necessary.

2 If storing samples temporarily in a cooler, use separate coolers for treated and untreated samples. As a rule of thumb, a 20 lb. bag of ice will be placed in each 48 qt. cooler. If samples will be in a cooler with ice for more than one hour, then temperature monitoring with a calibrated min/max thermometer will need to occur. Even if placed in coolers on ice, make every attempt to get the samples in the freezers as soon as possible.

3 Samples should be shipped to the analytical lab as soon after collection as possible. Consult the study protocol for the method, temperature, and maximum length of time for storage. If specifications are not given in the protocol use as a rule of thumb for maximum temperature and storage times: -20°C. and 30 days for frozen commodities, 4°C. and 14 days for refrigerated commodities and 25°C. and 2 days for commodities held at room temperature. For frozen commodities, also place a test tube containing ice in the freezer, and monitor if any thaw happened during the sample storage period.

4 Each freezer will contain a calibrated temperature recording device (Refer to Section 4 for guidance) that will alert all Sample Rescuers with a recorded voice message and email to their mobile device when temperatures inside the freezer become warmer than -10°C. Even if a person receiving an alert is not available to tend to the freezer emergency, that person should still contact others to ensure the message wasn't missed.

5 A freezer warning doesn't require that the samples be immediately moved to another freezer. However, the temperature should be monitored closely to determine whether the freezer is truly malfunctioning.

6 If a malfunction is suspected, the first option will be to move the samples to the other main freezer, which usually stores the opposite treatment. A divider, boxes, bags, etc. could be used for separation of treated and untreated samples.

7 The freezers are in Room 053 of McFadden Biostress Laboratory, which has a backup generator. In the highly unlikely event that both freezers should fail, do not open the freezer doors. Contact the Study Director immediately for guidance.

8 If the FRD leaves the Brookings area while samples are being stored in the freezer, they should have an alternate person trained on how to manage a freezer malfunction.

9 Data that will be recorded during a freezer malfunction include the following:

- The name or ID of the freezer that malfunctioned
- Date and time that the malfunction alert was received
- Date and time that the samples were removed from the malfunctioned freezer and added to a functioning freezer or cooler with dry ice
- The min/max temperature of the malfunctioned freezer after the samples were removed, and whether water from the test tube filled with ice was observed.
- The day and time when samples were returned to the main freezer(s)

10 All freezers should be under lock and key and only used to store GLP samples.

11 In addition to the automated temperature monitoring system, each freezer should also contain a calibrated min/max thermometer or data logger as a backup. If using a min/max, this should be reset once per day, if possible. Temperatures will be recorded from the min/max each time it is reset.

12 An annual check of the alarm system will be performed prior to the sampling season. The freezers will be unplugged to see if everyone receives an alert on their mobile device. The results of these checks will be documented.

13 Attached to the storage facility (i.e. freezer, refrigerator etc.) should be a log of the items inside indicating the Trial ID#, contents (e.g. treated sunflower seed), day/time in, and day/time out. An initial should accompany each entry.

14 All people who receive freezer failure alerts should have keys to all buildings and rooms necessary to successfully execute a sample rescue.

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Title: 8.6 Sample packaging and shipping procedures.

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Contact the Chemist at the residue laboratory by telephone or e-mail before the shipment, if possible. Inform this person of the project number, the sample ID's being shipped, shipment dates, and the name of the carrier. Ask for any special instructions in shipping the samples. Air freight shipments should be made on Monday or Tuesday to avoid potential weekend layovers.

2 Complete residue sample shipping form(s), make copies and send them to the study director, regional coordinator and residue chemist.

3 Make arrangements with the carrier for shipment of the samples and determine any special packing instructions etc. that is required to preserve the sample integrity. Note any limits on quantity of dry ice etc. that may be set by the carrier.

4 When shipping with dry ice, use highly-durable insulated coolers and pack in a 1:4 commodity:dry ice weight ratio. Dry ice should be underneath, on top, and around as many sides of the sample(s) as possible. Fill voids with packaging material such as crumpled paper. Aim to have the sample received by the residue laboratory within 24 hrs. of packaging. Shipments should be made on Monday or Tuesday, but absolutely no later than Wednesday.

5 Place the copy of the residue sample shipping form in a waterproof container and place it in one of the sample shipping containers.

6 Label each container with the following information:

a. Return Name and Address of the sender
b. Name and Address of the residue laboratory receiving the samples.
c. Number of the container if more than one is used.
d. Affix "Experimental Samples-Perishable" on each carton

Title: 14.1 Procedures to follow prior to an announced EPA inspection.

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

Procedures:

1 Arrange to have available the personnel who may be associated with the study or facilities audit.

2 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.

3 Prepare study and/or facilities personnel for the inspection.

a. Discuss position descriptions with technical personnel so they understand and can explain their role in the study.

b. Discuss possible questions that may likely come up about the study or facility and make sure every one understands what to expect.

c. Request personnel to answer the questions only to the extent needed and not to provide extraneous information. Do not volunteer information; keep answers direct and to the point.

d. Make certain that technical personnel know the safety precautions needed for the work area.

e. IR-4 Headquarters typically mails all raw data from the trials targeted for inspection. These documents should kept in as safe place through inspection and then immediately mailed back to Headquarters after the inspection.

4 Have accessible organizational charts, a map of the facility and any information specific to the facility or area that will make the inspection go smoother (restaurants, motels etc.)

Title: 14.2 Procedures to follow during an EPA inspection.

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

Scope: All field studies conducted under Good Laboratory Practices (GLP's).

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 study. Identify the person responsible who will accept the Notice of Inspection.

4 Distribute organizational charts, map of the facility and any other information previously prepared to make the inspection go smoother.

5 Ask the lead inspector for his/her agenda for the inspection as well as a list, if possible, of personnel for interview during the inspection.

6 Explain any housekeeping rules such as the use of safety equipment in work areas etc. to avoid any possible misunderstandings.

7 Proceed with the inspection.

a. Provide documents requested and provide explanations needed.

b. Keep notes of observations and of all interviews.

c. Keep management informed of the progress of the inspection and the findings.

Title: 14.3 Procedures to follow after the EPA inspection.

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance.

Procedures:

1 Make sure that all personnel involved in the inspection are present for the closeout conference.

2 If the inspector's comments are in error, call this to the inspector's attention. Remember the close out conference is not the forum for any debate.

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 or record the conference on tape if taping is acceptable to the inspectors.

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.