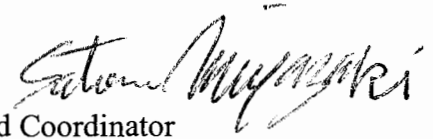


**MICHIGAN STATE**  
UNIVERSITY

**TO:** **Mark Ciernia**  
North Dakota State University  
Department of Plant Sciences  
Loftsgard Hall  
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**FROM:** Satoru Miyazaki, IR-4 Regional Field Coordinator

**SUBJECT:** STANDARD OPERATING PROCEDURE APPROVAL

**DATE:** April 1, 2014 (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	Revision #	Revise or Review Date
1	1.11	3-20-14
2	1.13	3-20-14
3	2.8	3-20-14
4	3.3	3-20-14
5	5.0	3-20-14
6	5.0	3-20-14
7	1.15	3-20-14
8	4.1	3-20-14
9	2.3	3-20-14
10	1.9	3-20-14
11	2.1	3-20-14
12	1.8	3-20-14
13	2.2	3-20-14
14	2.1	3-20-14
15	3.1	3-20-14
16	2.8	3-20-14
17	1.13	3-20-14
18	1.10	3-20-14
19	1.6	3-20-14
20	1.9	3-20-14
21	2.2	3-20-14
22	1.0	3-20-14



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**Standard Operating Procedures**

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Approved by Field Research Director: Mark Ciernia Date: 03/21/14  
 Approved by Field Research Coordinator: Eaton Mizuki Date: 4/1/14

**IR-4 Field Research  
Standard Operating Procedure**

SOP #1: General requirements for the development and use of Standard Operating Procedures (SOPs) for use in field research (Rev. 1.11).

WRITTEN BY: Mark Ciernia

PURPOSE: To describe the use and maintenance of SOPs at the Fargo location.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. Studies to be conducted under GLP standards at the Fargo location will have a set of Standard Operating Procedures (SOP) that describe the operations, policies, and procedures particular to the field research at that location. These SOPs will also cover in general those IR-4 trials conducted at other NDSU research sites as well as sites provided by farmer cooperators.
2. Each SOP will be reviewed annually and revised as needed. Each SOP will contain a date of revision/review and a date at which that SOP goes into effect.
3. The Field Research Director will archive each year's set of original, approved SOPs in a locked, fire resistant container.
4. The Study Director at IR-4 Headquarters will be notified of any deviation from the SOPs that would affect the integrity of a trial.
5. The following is the format to be used for each SOP:

SOP #: (SOP number in numerical order [1 to n]. Title: (SOP title). Revision #: (serially beginning with 1.0 after the initial draft). Major revisions will receive the next larger whole number (e.g. 2.4 to 3.0); minor revisions will receive the next decimal number (e.g. 1.1 to 1.2).

WRITTEN BY: (Name of person developing the SOP)

PURPOSE: (Brief description of the purpose of the SOP)

SCOPE: (Determines where the SOP is applicable)

PROCEDURES: (Describe the operating procedures in numerical order from beginning to end so that they can be reasonably understood by someone somewhat familiar with IR-4 procedures and agricultural research).

**IR-4 Field Research  
Standard Operating Procedures**

SOP #2: Responsibilities of the Field Research Director (Rev. 1.13).

WRITTEN BY: Mark Ciernia

PURPOSE: To list the general responsibilities of the Field Research Director.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. The Field Research Director has the following duties:
  1. Assure that the study is carried out according to an approved research protocol.
  2. Assure that personnel, resources, facilities, equipment, materials, and methods are available as scheduled for the conduct of the project.
  3. Ensure that all personnel significantly involved in the study are trained, and understand the research protocol, Field Data Book, and SOPs for the project.
  4. Promptly notify the Study Director of any protocol deviations, SOP deviations or other events that threaten the integrity of the study.
  5. Respond to all audits submitted by the Quality Assurance Unit (QA) as instructed.
  6. Retain copies of the Field Data Books, research protocols, QA audits, paper and electronic communications, and Facility Files at the Fargo location. Original raw data forms are incorporated into the Field Data Books as much as possible. Copies of forms may be kept as paper or electronic copies or both.
  7. Submit the estimated timetable and GLP Certification letter to the Field Research Coordinator in a timely manner.
  8. Maintain on file a current summary of experience and a job description for all key people engaged in the study.
  9. Annually revise and/or review SOPs. Create new SOPs as needed for the Fargo location.
  10. Maintain a set of Facility Files for the Fargo location (see Sop#20).
  11. Keep current on GLP training and IR-4 policy.
  12. Offer draft protocol input.

Reviewed 03/20/14 Effective date: 05/01/13

## **IR-4 Field Research Standard Operating Procedures**

SOP #3: Design and site selection for field studies (Rev. 2.8).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure plots are suitably located, easily identifiable, and large enough to obtain the required data and residue samples.

SCOPE: All field studies under the IR-4 Project.

### PROCEDURES:

1. NDSU currently operates a research farm located just northwest of Fargo and dedicated to weed science. An area of this land has been assigned to IR-4 residue work and allows for a 2 year rotation with a cover crop occupying the land in the off year. Residue trials may be conducted on another research site if this one is not available or suitable for any particular trial. Research land is also available on the NDSU campus and at the Prosper Research Farm located about 20 miles northwest of Fargo.
2. On rare occasions it may be necessary to conduct a trial on a farmer cooperator's field somewhere in the Fargo region. In such cases the cooperator may be relied upon to provide much of the agronomic information like soil characteristics, variety selection, planting dates etc. The Field Research Director will be responsible for helping the cooperator in obtaining the required information.
3. The plot site will be large enough to accommodate the required number of replicates, buffer zones and treatments in accordance with the research protocols. Normal RAC plots are planned to be at least twice as large as needed to meet sample size requirements under normal growing conditions. Adverse environmental conditions can, of course, reduce yields and require a larger proportion of the plot be harvested to provide adequate sample sizes. Larger plots are needed to produce samples for processing or decline trials.
4. Acceptable cultural practices for tillage, fertilization, and planting will be performed prior to plot layout and marking. Depending on which planter is used the actual planted area might be slightly larger than the intended plot size so tillage around the plot edges can trim the plot back to the correct dimensions.
5. The research protocol will describe the experimental design for the trial. Unless the research protocol specifically requires yield or efficacy data plot replicates are usually not required.
6. Sometime after the plot is established a plot map is drawn as part of the Field Data Book. The plot map will contain permanent reference points so that the plot can be relocated after the study is terminated. A GPS locator may be used to document permanent plot reference points. Its use, verification, and maintenance are addressed in SOP #5.
7. Each plot on the map is assigned a treatment number corresponding to the treatment number given in the research protocol. Another separate plot number will be necessary if the treatments are replicated.

**IR-4 Field Research  
Standard Operating Procedures**

SOP #3: Design and site selection for field studies (Rev. 2.8).

PROCEDURES: Continued

8. Each plot is laid out using a suitable measuring device to accurately locate plot corners on the site. A couple efficiencies in the application process can be attained by manipulating plot size. Problems with sprayer overlaps or skips can be avoided by matching the plot width to a multiple of the sprayer boom width. Trials with the same requirement for sprayer speed will have the same speed calibration if plot lengths are the same.
9. In trials with post-emergent applications plots may be planted wider than necessary and trimmed back to the correct width later in the season in order to alleviate the border effect on plot edges.
10. The four corners of each plot are identified with a flag or wooden stake of sufficient visibility to be seen easily throughout the duration of the study. Each stake or flag is labeled with a permanent marker to show the project and treatment numbers as indicated in the research protocol.

## **IR-4 Field Research Standard Operating Procedures**

SOP #4: Commodity establishment and maintenance (Rev. 3.3).

WRITTEN BY: Mark Ciernia

PURPOSE: Assure that commodities are grown under good agricultural research practices to provide a healthy, uniform crop for study.

SCOPE: All field studies developing data and/or residue samples under the IR-4 Project.

### PROCEDURES:

1. The Field Research Director will become familiar with the agronomic requirements of the crop under study. As much as possible the selection of a crop variety and its establishment and maintenance will be based on sound agricultural practices that mimic the practices of area producers.
2. The study site will be soil tested to determine pH, organic matter, fertility levels, and other soil characteristics required by the research protocol. The soil sample may be obtained in the fall of the previous season. In cases where the commodity is planted by a cooperator or other researcher, this information will be obtained from the cooperator or researcher insofar as it is available.
3. Standard cultural practices for fertilization and tillage will be implemented. Some fertilizer may be applied in the fall of the previous season.
4. Research protocols normally require the use of commercially accepted varieties but the specific variety selection is left to the discretion of the Field Research Director.
5. The Field Research Director will be familiar with the accepted planting techniques for the crops under study. Generally the plot width for crops planted in wide rows (corn, dry bean) is a number of feet equal to the row spacing times the number of rows. Crops planted in narrow rows (small grains, canola) are considered solid seeded and the plot width is measured in feet but does not include a number of rows.
6. Irrigation is currently not available at the NDSU Weeds, Campus, or Prosper research farms.
7. If pesticides are applied to the crop to control pests not under study, they will be applied according to label directions. Maintenance chemicals are currently not required to be applied under GLP procedures. Maintenance pesticides must be applied equally to all plots within a study with the exception of spot spraying for perennial weeds. For residue studies, pesticides cannot be applied that would interfere with the chemical analysis of the pesticide under study. If there are questions about maintenance pesticides the Study Director will be contacted.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #5: Operation of instruments and gauges (Rev. 5.0).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure that all instruments and gauges used in field research studies are accurate and in good working order.

SCOPE: All facilities where field studies are conducted under the IR-4 Project.

PROCEDURES:

1. A liquid filled pressure gauge is mounted on the boom of the field sprayer and treatments will be applied within the pressure range recommended by the nozzle manufacturer. However, the sprayer will be calibrated not by pressure per se but by nozzle output which will be measured directly. Accuracy of the pressure gauge will be checked by normal sprayer calibration procedures considering the manufacturer's specifications for pressure and volume. If the pressure gauge varies significantly within these specifications it will be replaced.
2. Some sprayers have a speedometer but since each application pass is timed to determine speed the actual speedometer reading is only a guide and may vary slightly from the theoretically desired speed used in application rate calculations.
3. A Kestrel digital thermometer/hygrometer/ anemometer will be used to record air temperature, relative humidity, and wind speed at the time of application. A standard soil thermometer will be used to measure soil temperature at application. All thermometers used in the study will be checked annually against 2 points of an NIST traceable standard thermometer to verify their accuracy. Thermometers may need a label or number to individually identify them. The thermometers currently in use and their accuracies are as follows:

<b>Thermometer ID</b>	<b>Manufacturer/Supplier</b>	<b>Accuracy</b>
Kestrel 3000 #1	Nielson – Kellerman	± 2F
Kestrel 3000 #2	Nielson – Kellerman	± 2F
Min/max #1	Fisher Scientific	± 2F
Min/max #2	VWR	± 2F
Min/max #3	VWR	± 2F
Min/max #4	VWR	± 2F
Soil #1	Gemplers	± 2F
Soil #2	Gemplers	± 2F
Freezer #1	VWR	± 2F
Freezer #2	VWR	± 2F

These thermometers are battery powered and their only maintenance would be battery replacement as needed.



**IR-4 Field Research  
Standard Operating Procedure**

SOP #5: Operation of instruments and gauges (Rev. 5.0).

PROCEDURES: Continued

4. Factory calibrated glass syringes will be used to measure liquid pesticides and adjuvants. A set of glass syringes of different volumes and pipeting needles are kept for GLP use. After each use syringes are cleaned by washing in soap and water, rinsing with water and acetone, rinsing again with distilled water, and air dried. Mixing glassware and utensils are cleaned in a similar manner.
5. A pH measurement of the carrier water is taken at each application. Currently an I.Q. Scientific Instruments, Inc. handheld pH meter model # IQ120 is used for pH readings and is calibrated according to the manufacturer's instructions at each use. A maintenance and use log for this meter is included in the Field Data Books.
6. Daily temperature and precipitation information can be obtained from the nearest North Dakota Agricultural Weather Network (NDAWN) station. NDAWN weather stations are located on the NDSU campus and the Prosper research sites. The NDAWN weather data is suitable for the Weather & Irrigation section of the Field Data Book.
7. The scale used for weighing dry materials is checked before each use using a set of standard weights. Standard weights and observed weights read from the scale are recorded in the Field Data Book and bracket the weight of the test substance
8. A professional balance and scale service regularly cleans, repairs and calibrates scales on the NDSU campus and is employed to periodically service the scale used for these studies.
9. As soon as possible after the scale has been serviced the set of standard weights will be checked as well by weighing each standard weight on the scale. The observed weight of each standard weight will be recorded.
10. If checking the scale immediately prior to weighing the test substance indicates balance malfunction another balance will be obtained from a nearby laboratory. Many such alternative scales are used throughout Waldron and Loftsgard Hall at North Dakota State University. A substitute scale will be checked and used as described above, however it probably will not have a maintenance and repair log associated with it.
11. The Field Research Director will keep a written record of all scale maintenance and standard weight accuracy tests as required by GLP guidelines.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #5: Operation of instruments and gauges (Rev. 5.0).

PROCEDURES: Continued

12. A handheld GPS instrument may be used to record the latitude and longitude coordinates of plot corners to fulfill the permanent landmark measurement to plot corners requirement. The unit currently used is a Magellan Explorist 200 which has an accuracy of 3 meters. Points of specific longitude and latitude have been established by Fargo city engineers on the NDSU campus. The handheld unit can be checked against 2 of these points prior to each year's use and the results recorded in a GPS Verification and Maintenance Log. Coordinates in the verification procedure are given in the DEG/MIN/SEC format and the coordinates for plot corners on the plot map are given in the DEG.DDDDD format. Experience has shown that the accuracy of the unit at each use can increase over a short period of time so every effort will be made to record the more accurate readings.
  
13. All instruments giving results inconsistent or inaccurate within tolerances will be replaced or repaired.

## **IR-4 Field Research Standard Operating Procedure**

SOP #6: Calibration of a liquid sprayer (Rev. 5.0).

WRITTEN BY: Mark Ciernia

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

SCOPE: All field studies using liquid sprayers under the IR-4 Project.

### PROCEDURES:

1. Pumps, hoses, pipes, fittings, regulators, gauges, switches, tanks, etc. will be inspected for obvious wear or malfunction and repaired or replaced as necessary.
2. Spraying Systems Co. offers a large selection of TeeJet spray nozzles and technical advice for many different application situations. An appropriate nozzle will be chosen considering the research protocol requirements for spray volume, the manufacturer's specifications for pressure and volume, pesticide class, and drift potential.
3. Prior to any calibration the sprayer must be fully operational and the plumbing system primed. To determine whether all nozzles are discharging uniformly water is sprayed through them for a given length of time at a uniform pressure. The discharge is captured separately from each nozzle in a container such as a graduate cylinder. The discharge of all nozzles is averaged and any nozzle tip whose discharge varies by more than 5% from the average is replaced. The process is repeated until all nozzles are within 5% of the average discharge. This final set of matching nozzle tips will be kept intact and in order throughout all required applications unless subsequent calibrations show unacceptable nozzle variability.
4. The sprayer will be calibrated according to output and speed. Once a matching set of nozzle tips is obtained the operating pressure at the sprayer boom is determined by running the sprayer for a given length of time and collecting the water discharged from individual nozzles. A container of adequate size, preferably similar to the container used for the actual application, will be filled with water, attached to the sprayer plumbing system, pressurized with CO<sub>2</sub>, and run long enough to prime the plumbing system. The sprayer will then be operated for a given length of time and the water collected from individual nozzles. The length of time for water collection will vary with the nozzle size and output volume but should be greater than the predetermined pass time for the application. A graduated cylinder will be used to measure this volume. If necessary the pressure will be adjusted in order to collect the desired volume of water according to the volume output specifications and delivery rate calculations for that particular application. When the correct pressure is determined that delivers as close as possible to the targeted water volume the sprayer is ready for the preapplication discharge calibration checks required in the Field Data Book.
5. Speed calibrations will be conducted at the test site in a field margin or buffer area. The exact pass time can be figured ahead of time using the plot length and the theoretical sprayer speed. Flags or fiberglass poles are placed in the field a distance apart equal to the plot length. The sprayer driver can then operate the sprayer in a simulated spray pass as a helper uses a stopwatch to time the pass as the spray boom hits the markers at either end of the pass. The sprayer speed will be adjusted to achieve as close as possible to the desired pass time.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #6: Calibration of a liquid sprayer (Rev. 5.0).

PROCEDURES: Continued

6. A 4-wheel all-terrain vehicle (ATV) with mounted sprayer is the implement of choice for IR-4 applications. The ATV sprayer has been adapted with several different and interchangeable booms and is preferred for its flexibility, ease of transport, adaptation to larger plot sizes, and close imitation to commercial applications. Each spray boom is given a unique identifier. The ATV also works well as a maintenance chemical applicator. Since the ATV itself does not generate data points the sprayer Maintenance, Use, and Repair log will reflect routine and nonroutine maintenance on only the components of the sprayer that influence the application such as the boom, plumbing, nozzles, pressure gauges, and CO<sub>2</sub> components.
7. A hand held 4 nozzle spray boom has been used on rare occasions for IR-4 applications. It is pressurized with CO<sub>2</sub> and its output and speed is determined essentially the same as the ATV except sprayer speed is achieved by walking.
8. Sometimes IR-4 projects require the production of a large processing sample. If an unusually large plot is needed to produce such a sample the ATV may be impractical and a tractor mounted field sprayer may be used. This sprayer would be pressurized with a PTO driven roller pump or could be rigged to use CO<sub>2</sub> pressurized canisters. The calibration procedures are the same as for the ATV and in some situations this sprayer could substitute for the ATV.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #7: Receipt and storage of test substances (Rev. 1.15).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure proper test substance container documentation and storage conditions.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. Test substances are normally shipped to the Plant Sciences Dept. at NDSU and received by the Field Research Director. All relevant information from the manufacturer that accompanies the test substance will be kept for the Field Data Book. If more than one container is provided for a project each container will be numbered or otherwise individually identified to keep the containers separate. If a project has more than one trial an individual container may be assigned to an individual trial.
2. A separate test substance receipt form is kept as part of the Fargo Facility File.
3. Test substance container labels are required to have the test substance name, CAS or code number, batch or lot number, expiration date, and storage conditions. If any of this information is missing from the container label, and it can be obtained from literature accompanying the test substance, the Field Research Director will transcribe the information onto the label and initial and date the entry.
4. From the time of receipt of the test substance from the manufacturer until after the final application of the test substance, the test substance container will be stored in the chemical storage room 113A Waldron Hall. The minimum and maximum temperatures of this room will be recorded periodically using a digital min/max thermometer. IR-4 test substances are kept together in a locked tool box.
5. Sometime following the last application of the test substance(s) the test substance container(s) will be moved to North Dakota State University's new Pesticide Storage Facility. This facility was opened in 2009 and is kept locked at all times. The TS containers from the same year and an inventory sheet will be kept in a cardboard box in cage #8 of the herbicide room. Temperature of this facility is maintained at close to 70F and is not monitored by the Field Research Director.
6. Some manufacturers have requested that the test substance container be returned to them at the end of the trial. If this is the case it will be documented in the Field Data Book.

Revised 03/20/14 Effective date: 04/01/14

**IR-4 Field Research  
Standard Operating Procedure**

SOP #8: Measuring and mixing pesticide formulations (Rev. 4.1).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure an accurate dosage in the mixing of pesticides for field research.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. Test substances are weighed or measured in room 113 Waldron Hall which is adjacent to the test substance storage room. If the field site is remote enough from Fargo mixing may be done on site. In such cases test substances will be transported in a secure container and the temperature monitored.
2. IR-4 prefers that one spray tank or container be used for mixing even for applications with multiple passes. Three gallon stainless steel pop canisters are suitable for this purpose for normal size plots. The canister will be labeled with the project, treatment, and application numbers. Very large plots using a field sprayer may require the spray tank be used as the tank mix container.
3. Fargo city water is normally used as the carrier. Information about Fargo's water treatment and quality is kept in the Fargo Facility File.

Liquid formulations:

1. Treated plots are typically sprayed in 1 to 3 passes. The volumes of water, test substance, and any adjuvants are determined as part of the volume, mixing, and dilution calculations and recorded in the Field Data Book. The amount of water needed is measured with a graduated cylinder and poured into the tank mix container. The water temperature can be taken at this time and an additional water sample measured for pH as described in SOP #5.
2. A factory calibrated glass syringe will be used to measure liquid test substances and adjuvants. A set of glass syringes and pipeting needles are kept for GLP use. The syringe will be pumped several times to remove all air bubbles before measuring out the required amount of test substance. It may be necessary to dispense the test substance more than once to get the total amount of pesticide into the container. The container will be sealed and agitated to thoroughly mix the solution.
3. If an adjuvant is part of the mix it will be added last and the tank mix canister agitated again.

Dry formulations:

1. The balance will be checked according to SOP#5 prior to weighing the test substance.
2. A clean weighing container will be tared first and then the amount of test substance weighed out. The test substance will be dispensed directly into the labeled tank mix container using a funnel if needed.
3. The predetermined volume of water will be measured with a graduate cylinder and added to the canister which is then agitated to disperse the test substance into solution. Any adjuvants are added last and the container agitated again. Water temperature and pH measurements are done similar to the liquid mixing procedure.

Reviewed 03/20/14 Effective date: 05/01/13

**IR-4 Field Research  
Standard Operating Procedure**

SOP #9: Procedures in the application of pesticides (Rev. 2.3).

WRITTEN BY: Mark Ciernia

PURPOSE: To describe the procedures used in pesticide application.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. All personnel involved in the storage, mixing, application, and cleanup of pesticides will be properly trained and familiar with the pesticide label. The North Dakota State Dept. of Agriculture provides training in all aspects of pesticide use and NDSU employees who apply pesticides must be certified by that agency.
2. In the case of row crops, the sprayer is usually centered over one of the rows for guidance and the sprayer boom positioned so it covers the intended number of rows. For solid seeded crops or soil applied treatments flags are positioned at both ends of the plot for each pass for the sprayer to center on for uniform pesticide placement. If the treatment is applied at a more mature crop stage it might not be feasible to drive the sprayer through the plot. In those cases an offset boom can be used so that the sprayer can be driven down the edge of the plot and the spray boom overhang the crop. The boom height is set by measuring from the nozzle tip to the target.
3. Flags used for the pass time documentation are placed in the treated plot at either end so that they can be observed by the timekeeper as they are hit by the sprayer boom.
4. Immediately before applying the treatment the sprayer boom pressure will be checked and then the system blown clear of any remaining water. The spray mixture container will be agitated for remixing and attached to the sprayer system. Just prior to the application the sprayer boom is primed with the test substance mixture and the pressure gauge checked again.
5. Correct speed should be maintained as well as possible while spraying the plot. The sprayer boom should be turned on right before actually entering the plot and turned off right after leaving the plot.
6. The test substance will be applied beginning with the lowest concentration and working up to the highest concentration for multiple rate trials.
7. If the test substance is to be applied as a dry material one option would be to apply a measured weight of pesticide to a measured area of the test plot. The entire amount of dry material would be applied through some kind of dispensing apparatus that can distribute the test substance uniformly. In such cases spray volume and pass time calibrations are not relevant to the trial.

Reviewed 03/20/14 Effective date: 05/01/13

**IR-4 Field Research  
Standard Operating Procedure**

SOP #10: Cleanup of application equipment (Rev. 1.9).

WRITTEN BY: Mark Ciernia

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

SCOPE: All locations where pesticides are used under the IR-4 Project.

PROCEDURES:

1. All personnel involved in the storage, mixing, application, and cleanup of pesticides will be properly trained.
2. Usually a small volume of spray mixture is left over in the spray canister after the application. This amount will be discarded by pouring on the soil in a field margin away from the test plot. Any pesticide left in the spray boom is blown out on the edge of the field downwind from the test plots.
3. Mixing glassware and utensils are cleaned as described in SOP #5.



**IR-4 Field Research  
Standard Operating Procedure**

SOP #11: Procedures for cleaning the sprayer (Rev. 2.1).

WRITTEN BY: Mark Ciernia

PURPOSE: To describe the procedures for cleaning the sprayer between treatments and after completion of treatments.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. The sprayer will be rinsed with water prior to application of the first treatment. This is usually accomplished by nozzle and pressure checks or output calibration procedures.
2. Between treatments of the same chemical but different rates the sprayer will be rinsed with water.
3. Between treatments of different chemicals or different formulations of the same chemical the sprayer will be rinsed with a dilute ammonia and water solution or other spray tank cleaner.
4. After application of the final treatment the sprayer boom will be blown clear in an area downwind from the plot area. Any pesticide mixture remaining in the spray canister will be discarded on the soil at a field margin. A dilute ammonia solution or other spray tank cleaner will then be added to the canister and pumped through the spray boom to clean the entire system. Finally the sprayer will be rinsed again with clean water.
5. If a field sprayer is used the cleaning process is essentially the same with the spray tank serving as the pesticide canister.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #12: Procedures governing misapplications (Rev. 1.8).

WRITTEN BY: Mark Ciernia

PURPOSE: To describe the procedures to follow in case of a misapplication of the test substance.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. The applicator and timekeeper should observe the application process to make sure the test substance is applied uniformly to the test plot.
2. If a sprayer malfunction such as a plugged nozzle, pressure loss, or hose break occurs the applicator should try to determine when and how the malfunction happened and how to correct it before proceeding.
3. If the test substance is misapplied to any plot area, that area will be well marked for the duration of the trial and avoided at harvest or destroyed prior to harvest.
4. The Study Director will be notified and details recorded in the Field Data Book if the integrity of the study is compromised by a misapplication.
5. The Field Research Director is responsible for the above.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #13: Recording of raw data (Rev. 2.2).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure that raw data forms are filled out clearly and completely.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. The Field Research Director will be responsible for seeing that all raw data forms, facility files, copies, correspondence, e-mails and other documents relevant to the study are secured in that trial's Field Data Book.
2. All raw data will be recorded in ink.
3. Corrections will be made by crossing through that entry and initialing. The reason for change and date of change will also be noted. The Field Data Book's instructions give many error codes used for explaining mistakes. Others that may be appropriate are **ID** = incorrect date, **WL** = wrong entry line, and **WN** = wrong number.
4. Raw data pages must be retained in the Field Data Book. Pages may be temporarily removed to make copies or to facilitate keeping different logs. Some pages originate from the Fargo Facility Files which are kept separate until they are included in a Field Data Book.
5. All entries will be dated where prompted in the Field Data Book or where the date is different from the page date. Each page will be signed or initialed by the Field Research Director as prompted.
6. All raw data sheets, correspondence, etc. in the Field Data Book will be clearly marked with the project number, dates generated, page number, name or initials of investigator and other information that may be needed to understand the data and its source.
7. The forms provided with the Field Data Book will be carefully reviewed to make sure that all the required data are being collected.
8. Unused portions of the raw data forms including unused parts of tables or unused lines of narratives will be lined out, dated, and initialed. If the entire page is initialed and dated then it is not necessary to initial and date every lineout on that page. A whole page will be lined out, dated, and initialed if that page is not relevant to the trial.

Reviewed 03/20/14 Effective date: 05/01/13

**IR-4 Field Research  
Standard Operating Procedure**

SOP #14: Method for collecting performance data (Rev. 2.1).

WRITTEN BY: Mark Ciernia

PURPOSE: To describe the procedure used for taking field performance data.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

Phytotoxicity Data:

1. The research protocol may give specific directions for phytotoxicity data collection. If no specifics are given proceed as follows:
2. When possible take phytotoxicity data within 2 weeks after each application of the pesticide although appearance of symptoms varies with the pesticide (fungicides and insecticides by themselves are rarely phytotoxic) and environmental conditions. If symptoms occur after this period that warrant an additional reading then phytotoxicity data should be taken as necessary.
3. Phytotoxicity will be visually estimated by comparing the crop in a treated plot(s) with the crop in the untreated plot. The crop in treated plots will be assigned a percentage injury value using a scale of 0% = no injury to 100% = plant death. Stunting, misshapen or discolored leaves, and stand reductions will be taken into account when estimating percentage injury. Adjuvant use could also influence injury symptoms.

Efficacy/Weed Control Data:

1. Weed control efficacy data is rarely required in residue trials but if it is and the research protocol does not give specific instructions proceed as follows:
2. Where possible, take weed control data within 2 to 4 weeks after the treatment. Timings of weed control evaluations vary somewhat with the type of application (preemergence vs. postemergence), activity of the herbicide, the environment, and any residual effects of the herbicide. Additional evaluations at later dates may be necessary to see the full effect of the weed control.
3. Weed control will be visually estimated by comparing weed growth in the treated plot(s) with that in the untreated plot. Percentage weed control will be determined using a scale of 0% = no control to 100% = complete control.

Revised 03/20/14 Effective date: 04/01/14

**IR-4 Field Research  
Standard Operating Procedure**

SOP #15: Residue sample collection (Rev. 3.1).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure that residue samples are collected in a proper fashion.

SCOPE: All field studies producing samples for residue analysis under the IR-4 Project.

PROCEDURES:

1. The research protocol may require specific dates or treatment to harvest intervals (PHI) for the collection of samples. If the protocol does not require a specific PHI and the crop is instead to be harvested at commercial maturity, the harvest date will be determined by the Field Research Director.
2. Prior to sample collection, a sufficient number of sample bags will be obtained to collect all the samples. The Field Research Coordinator provides IR-4 plastic lined cloth bags for this purpose. Processing samples may require larger sample bags.
3. Before harvest each sample bag will be labeled with waterproof ink to indicate the following information (although IR-4 sample bag labels vary somewhat):
  1. Project number
  2. Crop fraction
  3. Test substance
  4. Sample number
  5. Treatment number
  6. Harvest date
  7. Sample date
  8. Name and phone number of the Field Research Director
4. The following information will be printed on index cards for each bag:
  1. Project number
  2. Crop fraction
  3. Test substance (untreated samples will be labeled "untreated")
  4. Rate (the rate for untreated samples will be NA)
  5. Treatment date(s) (NA for untreated samples)
  6. Treatment number
  7. Sample number
  8. Harvest date
  9. Sample date
  10. Field Research Director name, address, and phone number
5. Each card will be placed inside a ziplock plastic bag and placed inside the sample bag at harvest.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #15: Residue sample collection (Rev. 3.1).

PROCEDURES: Continued

6. Research protocols often provide alternative harvest scenarios to accommodate different harvest environments. Seed crop samples will be harvested by small plot combine, if possible, with the untreated plot harvested first, followed by the lowest pesticide rate of the treated plot, and finishing with the highest pesticide rate. Seed may be combined directly by cutting a diagonal swath across the plot or plants may be hand harvested, usually from a minimum number of areas in the plot, and brought to the combine or portable thresher to thresh the seed. Regardless of which method is used the seed is collected in a clean bucket or grain pan. If more than enough seed is collected this bulk amount is stirred and enough removed to provide the required sample. Excess seed is discarded. Sometimes mowing and windrowing (swathing) the commodity is necessary before combining if the crop needs field drying to meet the PHI interval or it's a routine agronomic practice for that crop. If necessary each seed sample is cleaned immediately after threshing using appropriately sized hand cleaning sieves and/or fanning cleaner before it is placed into harvest bags. These sieves screen out both large and small foreign material such as straw, chaff, and weed seeds. It may be expedient to clean large (processing) seed samples by using a clipper mill. If the crop is unable to be mechanically harvested in the field whole plants or plant parts would be representatively selected, placed into new grocery bags or plastic trash bags labeled with the appropriate treatment information, and artificially dried at 90 to 100 F for up to 48 hours. The plant parts would then be mechanically threshed, cleaned with a small clipper mill or hand cleaning sieves, and seed transferred to the permanent sample bags. Drying temperature and duration will be documented.
7. Root crops will be dug from the soil most likely by hand using clean tools. If a large (processing) sample is required a mechanical harvester may be employed. Harvesting will progress from the untreated plot to the lowest rate of the treated plot and so on. Any cleaning of plant parts or soil from the samples will be done according to the research protocol. The root material and/or any required above ground plant parts will be placed into labeled sample bags until enough is collected to satisfy protocol requirements.
8. Sometimes vegetative plant parts such as leaves, stems, or forage are needed for RAC samples. If so these samples are collected according to the research protocol using clean tools and methods. If straw is required from a grain crop it can be collected from that straw that is ejected from the plot combine or thresher.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #15: Residue sample collection (Rev. 3.1).

PROCEDURES: Continued

9. Special care will be exercised in the following aspects of sample collection:
  1. Samples must be representative of the test plot and duplicate samples will be collected in separate harvest operations.
  2. Untreated samples are always collected first and handled separately from treated samples.
  3. Diseased or otherwise damaged portions of the plot will be avoided during harvest.
  4. Samples will be handled gently so as not to cause undue damage to the harvested portion.
  5. All tools, handling equipment, and protective clothing will be clean at the start of each treatment. The plot combine or thresher will be visually inspected and allowed to run empty for a period of time to help facilitate cleanout. Likewise any seed cleaning tools will be inspected before starting each treatment.
  6. Sometimes sample materials are reduced in size by cutting and collecting partial plant parts. In such cases the bed of a pickup provides a convenient work surface but it will be covered with clean plastic sheeting or a tarp to prevent contamination. Cutting boards will be cleaned prior to each sampling.
10. The research protocol usually specifies the handling of residue samples from field to freezer. Samples will preferably be weighed in the field to assure the weights meet protocol requirements. Currently samples need not be weighed with a GLP compliant scale. If cooling of the samples is needed they will be placed in insulated plastic coolers with containers of blue ice. Samples are nearly always transported via pickup truck and treated samples are always kept separate from untreated ones. If transport to the freezer can be done within the protocol defined time limit sample cooling would not be necessary although inclusion of blue ice in the insulated coolers is routine.
11. Untreated samples may be harvested and delivered to the freezer before treated samples are harvested to help assure prompt sample delivery to the freezer and help avoid contamination.
12. All relevant harvest procedures are recorded in the Field Data Book.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #16: Residue sample storage (Rev. 2.8).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure the integrity of residue samples in frozen storage from harvest to shipment.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. Currently there are 2 freezers, one upright and one chest, located in room 110 Waldron Hall and used for temporary residue sample storage. The freezers are identified as IR-4 Freezer #1 and IR-4 Freezer #2.
2. IR-4 Freezer #1 is dedicated to untreated sample storage and Freezer #2 is dedicated to treated sample storage. Both freezers are labeled as such.
3. During the time residue samples are stored in the freezer a maximum/minimum temperature log will be kept by the Field Research Director. A digital max/min freezer thermometer will be used to periodically read the temperatures. Usually there is a temperature spike when fresh samples are added to the freezer, samples are removed and replaced during packaging, or the freezer door is otherwise opened for a short period of time. This is considered part of normal procedures and does not threaten the integrity of the residue samples.
4. The Field Research Director will be responsible for keeping a freezer maintenance and repair log as well as a freezer contents log with log-in and log-out dates of residue samples.
5. The freezers will be kept locked whenever they contain residue samples with access controlled by the Field Research Director.



**IR-4 Field Research  
Standard Operating Procedure**

SOP #17: Sample shipping procedures (Rev. 1.13).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure that residue samples are removed from storage and shipped to the residue laboratory while maintaining sample integrity.

SCOPE: All locations where residue samples are stored under the IR-4 Project.

PROCEDURES:

1. Prior to sample shipment the researcher at the residue laboratory will be notified via telephone or e-mail of the impending shipment. Samples are preferably shipped via freezer truck so the shipment date depends somewhat on the trucker's schedule. If the research protocol requires sample shipment immediately following harvest samples will be packaged in insulated coolers or lined cardboard boxes with an adequate quantity of dry ice and shipped by overnight express. Air freight shipments should be avoided on Fridays to prevent potential weekend layovers.
2. The Residue Sample Chain of Custody Form from the Field Data Book will be completed and a copy will be placed in a waterproof plastic bag and packed with each container of samples. Additional copies will be forwarded to IR-4 personnel as directed on the form.
3. Arrangements will be made with the carrier for sample shipment. Special packing instructions will be noted and observed.
4. For overnight shipments containers of sufficient size and quantity will be obtained to hold the residue samples and dry ice. The samples and dry ice will be packed in the containers just prior to shipment. Insulated plastic coolers or cardboard boxes, if handled correctly, work well for shipments containing dry ice. Dry ice is considered a hazardous shipping material and is subject to US Department of Transportation regulations. IR-4 also provides guidance on shipping with dry ice.
5. Shipping containers will display labels with the following information:
  1. Return name and address of sender.
  2. Name, address, and phone # of the residue lab receiving the samples.
  3. Project number, crop, test substance, treatment number(s), sample number(s), and box number.
  4. "IR-4 Experimental Samples-Perishable"
  5. Boxes containing dry ice require a dry ice specific hazardous warning label.
6. Container lids will be taped firmly in place and the sample boxes loaded in a vehicle for transport to the carrier.
7. The carrier will be given the samples and the destination of the residue lab. A bill of lading or similar form will be obtained from the carrier to include in the Field Data Book.

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**IR-4 Field Research  
Standard Operating Procedure**

SOP #18: Completion of forms (Rev. 1.10).

WRITTEN BY: Mark Ciernia

PURPOSE: To assure that forms are completed accurately and properly capture the required data of the trial.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. Forms will be filled out in ink with legible writing. Mistakes will be struck over with a single line, identified by code as to the type of error, dated, and initialed.
2. Unused pages will be lined out, dated, and initialed by the Field Research Director. Unused sections or parts of forms such as unused cells of a table or a blank section of a narrative will also be lined out, initialed, and dated if necessary.
3. Forms will be filled out as completely and accurately as possible. Sufficient detail will be provided with the intention that data reviewers will be able to reconstruct the research materials and methods.
4. New forms will be developed as needed to comply with SOP, GLP, research protocol, or facility file requirements.
5. The originals of the completed Field Data Book and any accompanying documentation will be forwarded to the Field Research Coordinator within a reasonable period of time after the field trial is completed and according to IR-4 Project policy.
6. The Field Research Director will make a copy of each completed Field Data Book to retain at North Dakota State University.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #19: SOP review (Rev. 1.6).

WRITTEN BY: Mark Ciernia

PURPOSE: Annual review, revision, or addition of SOPs

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. Sometime between the completion of the Field Data Books and the beginning of the next season's work the Field Research Director will review all SOPs and incorporate any changes necessary to enhance the SOP's compliance with research protocols or GLP procedures or to better explain procedures at the Fargo location. Each year the entire SOP set requires the approval of both the Field Research Director and the Regional Field Coordinator.
2. Minor SOP revisions will be assigned the next higher tenths number, e.g. Rev. 1.2 will become Rev. 1.3. Major revisions will be given the next higher tens number, e.g. Rev. 1.7 will become Rev. 2.0. The date of the SOP revision or review, as well as an effective date, will be noted at the bottom of that SOP's page.
3. If any new SOPs are required they can be added at this time and the date noted at the bottom of the page.
4. The SOP Index will be changed to show any of the above changes.

**IR-4 Field Research  
Standard Operating Procedure**

SOP #20: The Facility File (Rev. 1.9).

WRITTEN BY: Mark Ciernia

PURPOSE: The development of those forms generating data common to the Fargo location or otherwise required for GLP compliance.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

1. The Facility File is a set of forms developed by the Field Research Director and can generally be divided into 2 groups. One group consists of those forms containing data common to more than one study at the Fargo location. The original and copies of these forms will appear in the Field Data Books. The other group contains those forms referring to the organization and personnel at the Fargo site and may not contain data relevant to the Field Data Book.
2. Data forms or logs in the Facility File will include:
  1. Thermometer Check
  2. Scale Repair, Calibration, and Standard Weight Check
  3. Sprayer Calibration, Use, and Repair
  4. Test Substance Storage Temperature
  5. Pesticide and Fertilizer History
  6. Freezer Temperature
  7. Freezer Contents
  8. Freezer Maintenance and Repair
  9. GPS Check and Maintenance
  10. pH Meter Maintenance and Use
3. Forms particular to the Fargo site include:
  1. Test Substance Container Inventory
  2. Test Substance Receipt
  3. IR-4 Organizational Chart
  4. Floor plans for Waldron and Loftsgard Halls at North Dakota State University
  5. Curriculum Vitae for Mark Ciernia and Kirk Howatt
  6. Job Descriptions for Mark Ciernia and Kirk Howatt
  7. Training Records for Mark Ciernia and Kirk Howatt
  8. SOPs for the current year including an index
  9. Fargo water treatment and quality
4. The Field Research Director is responsible for creating any new facility files or editing the templates of existing facility files.

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**IR-4 Field Research  
Standard Operating Procedure**

SOP #21: Seed Treatment (Rev. 2.2).

WRITTEN BY: Mark Ciernia

PURPOSE: To describe procedures used in seed treatment studies.

SCOPE: Seed treatment trials under the IR-4 Project.

PROCEDURES:

1. Seed treatments differ from other applications in that the pesticide in question is not applied to a crop using some kind of liquid sprayer. Seed treatments can be applied directly to the seed either as a liquid or dry formulation and the rate is usually stated as a certain amount of active ingredient applied to a certain weight or volume of seed.
2. For research purposes small amounts of seed can be treated with a liquid seed treatment by applying the pesticide, usually through some kind of single orifice applicator, onto the seed as it tumbles in a canister for a short period of time. The seed and liquid must mix together long enough to distribute the treatment throughout the batch of seed but not so long that the treatment dries on the seed as pesticide can then be lost by abrasion. After treatment the seed can be stored in a clean container and the canister and application mechanism must be cleaned.
3. Dry seed treatment formulations are normally applied to the seed right before planting and consist of a prescribed amount of pesticide added to a certain weight or volume of seed and the two mixed together in some kind of container before placing into the planter unit. The container should be of such material that the seed treatment does not adhere to it and the maximum amount of pesticide is retained on the seed. After planting each treatment each planter unit will have to be cleaned, most likely using compressed air, with as much disassembly as necessary to remove any pesticide residue from that unit.
4. Some trials have had the treatment procedure performed by the pesticide manufacturer or other laboratory and the treated seed is then shipped to the research site. In between receipt of treated seed and planting the seed is kept in cool storage and the temperature monitored.

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**IR-4 Field Research  
Standard Operating Procedure**

SOP #22: Field Personnel (Rev. 1.0)

WRITTEN BY: Mark Ciernia

PURPOSE: To define the roles of other personnel involved in field trials.

SCOPE: All field studies under the IR-4 Project.

PROCEDURES:

4. The Field Research Director is responsible for all aspects in the conduct of field trials.
5. If another person other than the Field Research Director is responsible for entering raw data any time during the trial then the Field Research Director will keep on file a current vita for that person. Any training or supervision necessary in performing this duty will be provided by the Field Research Director.
6. Other personnel helping in an incidental or temporary nature will not be considered actively involved in the field trial although they will require supervision while assisting in IR-4 field work.