SOP Log Sheet

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S: Facility Files- SOP Cover Sheet

9/13/16 JT

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TO: Dr. Brian Jenks
North Central R & E Center
5400 Hwy 83 South
Minot, ND 58701-7645

FROM: John Wise, IR-4 Regional Field Coordinator

SUBJECT: STANDARD OPERATING PROCEDURE APPROVAL
Version 2.0

DATE: April 10, 2018 (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 #	Revision Date	Submission/ Review Date
1	1.2	4/2016	4-10-17
2	1.3	4/2016	4-10-17
3	1.5	4/2016	4-10-17
4	1.5	4/2016	4-10-17
5	1.8	3/2018	3-29-18
6	1.5	4/2006	4-10-17
7	2.2	3/2018	3-29-18
8	1.7	3/2015	4-10-17
9	1.5	3/2015	4-10-17
10	1.3	4/2016	4-10-17
11	1.2	3/2004	4-10-17
12	1.2	4/2016	4-10-17
13	1.5	3/2018	3-29-18
14	1.6	4/2010	4-10-17
15	2.4	3/2018	3-29-18
16	1.6	4/2017	4-20-17
17	1.3	4/2006	4-10-17
18	1.2	3/2018	3-29-18
19	1.1	6/2015	4-10-17
20	2.0	4/2017	4-20-17

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IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

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North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #1: General requirements for the development and use of Standard Operating Procedures (SOPs) in field research (Rev. 1.2).

WRITTEN BY: Brian M. Jenks

PURPOSE: To provide guidance when conducting field research under Good Laboratory Practices (GLP).

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

PROCEDURES:

1. Studies that need to be conducted under GLP standards will have a Quality Assurance Unit (QAU) that will annually review the research to determine if the studies are being conducted per these procedures. The date of review will be noted.

2. Each SOP will be reviewed annually and revised as needed to assure they are current and accurate. All earlier revisions will be archived. SOPs will be submitted annually to the Regional Field Coordinator for approval with signed copies included in facility file.

3. All deviations from SOPs that would significantly affect results of the study must be signed off by the Study Director at IR-4 Headquarters.

4. 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. 3.0); minor revisions will receive the next decimal number (e.g. 1.2).

WRITTEN BY: (Name of person developing the SOP)

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

SCOPE: (Determines where and when the SOP is applicable)

PROCEDURES: (Describe the operating procedures in numerical order from beginning to end so that a person with some knowledge of the situation can carry out the procedures without any verbal input from other sources.

Revised 04/16
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Satoru Miyazaki Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #2: Responsibilities of the Field Research Director (Rev. 1.3).

WRITTEN BY: Brian M. Jenks

PURPOSE: To define the 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:
   a. Review draft protocol.
   b. Assure that the study is carried out according to an approved research protocol and SOPs.
   c. Assure that personnel, resources, facilities, equipment, materials and methods are available as scheduled for the conduct of the project.
   d. Make sure that all personnel conducting the study understand the research protocol and SOPs for the project.
   e. All deviations reported by the Quality Assurance Unit (QA) are responded to in writing.
   f. All raw data, summaries, and other items connected with the study that need to be retained are stored in archives.
   g. Maintain a copy of the master schedule from IR-4 Headquarters for all field research projects under the field research director's control.
   h. Submit a copy of the projected timetable to the Regional Research Coordinator before the start of the study.
   i. Maintain on file a current summary of qualifications, experience, and a job description for all key people engaged in the study.

Revised 04/16
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Satoru Miyazaki Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #3: Design and site selection for field studies (Rev. 1.5).

WRITTEN BY: Brian M. Jenks

PURPOSE: To assure plots are large enough to obtain the required data and samples with sufficient uniformity.

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

PROCEDURES:

1. Site selection will be made in accordance with agriculturally acceptable practices for the commodity.
2. The site will be large enough to accommodate the required number of replicates, buffer zones, and treatments in accordance with an approved research protocol and for the commodity to be grown under commercial conditions yielding samples of sufficient size for analysis where required.
3. Locate site with sufficient isolation to minimize contamination from external sources.
4. If the crop is not to be newly established, select a site that has a uniform stand for production.
5. Acceptable cultural practices should be performed prior to plot layout and marking.
6. The experimental design as specified by the research protocol should be used. If none is designated, then use a commonly accepted experimental design such as a complete randomized block design. The experimental design used should be documented. A minimum of 3 replications should be used.
7. Prepare a plot map showing the location of each plot on the site and the North azimuth. The plot map should contain permanent reference points, if possible, so that the plot can be relocated after the study is terminated. Retain the plot map in the IR-4 Field Data Book.
8. Lay out each plot on the site using a suitable measuring device to accurately locate the plots on the site.
9. Identify each corner with a marker (such as a flag) of sufficient visibility to be seen easily throughout the duration of the study.
10. The Field Research ID number and treatment (TRT vs. UNT) will be written on at least one stake or flag at the plot site.
11. Once the study is established, GPS coordinates for plot corners will be recorded using a hand-held GPS unit with accuracy of 1-2 meters. A permanent marker will be used to test the accuracy of the hand-held GPS units. The coordinates for this permanent marker will be initially identified by a professional survey company.

Revised 04/16
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Seton Rodriguez Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #4: Commodity establishment and maintenance (Rev. 1.5).

WRITTEN BY: Brian M. Jenks

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

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

PROCEDURES:

1. Use up-to-date information on the production of the commodity under study. Consult with an agricultural specialist familiar with the production practices for the commodity.
2. Use standard cultural practices and fertilize the soil at the site as necessary for normal commodity production.
3. Soil sampling will be conducted by pulling sporadic cores from the top 6 inches the plot. The cores will be sent to a laboratory to test for soil texture, N-P-K, pH, CEC, and OM.
4. Till the field as needed for the commodity.
5. Determine the correct species and variety to use as specified by the research protocol. If the variety is not specified, select a commercially acceptable variety for the study. If a commercial producer is providing the plants, try to select plants as uniform in growth and color as possible.
6. Identify each treatment in such a manner that it can be seen throughout the life of the study.
7. Irrigate or perform other agricultural practices as necessary to get the crop started.
8. If pesticides are applied to the crop to prevent losses due to pests not under study, they should be applied according to the relevant SOPs in this document. If this is a residue study, no pesticides should be applied that would interfere with the chemical analysis of the pesticide under study. If in doubt, call the study director or analytical laboratory identified in the field phase research protocol to receive the residue samples.

Revised 04/16
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Satom Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #5: Calibration of instruments and gauges (Rev. 1.8).

WRITTEN BY: Brian M. Jenks

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. For most situations, a pressure gauge is mounted on the field sprayer and treatments will be applied at 30-40 psi using XR flat fan nozzles. Studies that require other nozzles may require a slightly different pressure. However, the sprayer will be calibrated not by pressure per se, but by nozzle output which will be measured directly. Spray pressure is only important to record with reasonable accuracy because spray droplet size varies inversely with pressure. Droplet size (and therefore pressure) is not a critical factor in this procedure since both can vary significantly according to farmer practice with little to no evidence that such changes appreciably affect residue levels on the crop. Consequently, accuracy of the pressure gauge will be checked by verifying that the gauge reading and nozzle output are within manufacturer specifications.

2. Hourly air temperature, relative humidity, and wind speed are available from the North Dakota Agricultural Weather Network (NDAWN) which maintains a weather station usually within 1 mile of the NCREC. This will be one possible data source for application records. Hand-held air thermometers, soil thermometers, and anemometers may also be used. If an IR-4 study needs to be conducted away from the NCREC, hand-held weather equipment will be used. Data from the closest NDAWN station will be used, if necessary.

3. Daily temperature, rainfall, and relative humidity information may also be obtained from NDAWN.

4. A plastic syringe or graduated cylinder will be used to measure liquid test substances.

5. The balance used to measure dry test substances will be calibrated biennially prior to use by Northern Balance and Scale or another reputable company. Standard weights are verified by placing them on the balance after it has been serviced.

6. The digital thermometers used to monitor the temperature of test substance storage and sample freezer thermometers will be calibrated by comparing them to an NIST certified thermometer. The NIST certified thermometer will be sent for calibration biennially.

7. A written record will be kept of the dates and results of all accuracy and calibration tests. The thermometer is factory guaranteed to measure accurately within 1°C. The electronic balance should be accurate to within 0.1%. Wind speed should be accurate to within plus or minus 0.1 miles per hour.

8. All instruments giving inconsistent results or inaccurate within the above tolerances will be replaced or repaired as non-routine maintenance.

Revised 03/18
Submitted by Field Research Director: Brian Jenks Date: 3-29-18

Approved by Field Research Coordinator: Date: 4/10/18

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #6: Calibration of a liquid sprayer (Rev. 1.5)

WRITTEN BY: Brian M. Jenks

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 formulations under the IR-4 Project.

PROCEDURES:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary.

2. Choose the appropriate nozzle tip in accordance with gallonage and spray pressure guidelines on approved research protocol.

3. Determine whether all nozzles are discharging uniformly by spraying water through them at a uniform pressure and catching the discharge from each nozzle in a separate container for a given length of time. Begin timing the discharge after the system is fully charged and operating. Average the discharge of all nozzles. Replace any nozzle tip whose discharge varies by more than 5% from the average. Repeat the process until all nozzles are within 5% of the average discharge.

4. The sprayer (tractor, bicycle, or backpack) will be calibrated by measuring output over a given amount of time (ml/second/nozzle). Given nozzle output, nozzle spacing, and travel speed, gallons per acre applied can be calculated. Before application of the test substance, the sprayer speed will be calibrated on a surface similar to the test plot surface, preferably right next to the test plot. Sprayer speed typically will be calculated based on the time required to travel the length of the test plot. Sprayer speed will be adjusted to match the speed used to calculate gallons per acre.

5. If the tractor sprayer or bicycle sprayer malfunctions in any way, there are other sprayers available at the North Central Research Extension Center. Other sprayers are used routinely and are maintained in good working order. Under conditions where the mechanical sprayers cannot be used (i.e. field surface too wet), a backpack sprayer with a hand-held boom will be used to apply the treatment. Calibration of these alternative sprayers is essentially the same as in procedure above.

Revised 04/06
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Satu Miljoski Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #7: Receipt and storage of test substance and additives (Rev. 2.2).

WRITTEN BY: Brian M. Jenks

PURPOSE: To assure proper pesticide storage conditions.

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

PROCEDURES:

1. From the time of receipt of the test substance from the manufacturer until application of the test substance, the test substance will be stored at the North Central Research Extension Center. The maximum and minimum temperatures of the TS area in the NCREC Pesticide Storage Building or NCREC Admin Building will be recorded periodically using a digital max/min thermometer.

2. Following application of the test substance, the container will be returned (archived) to the same storage area as in procedure above.

3. All spray additives used will be stored with the TS/TS containers and will be stable at ambient temperature. The NCREC Pesticide Storage Building has a controlled temperature that fluctuates from 55 to 75 degrees Fahrenheit.

4. All spray additives will remain in the labeled original container. If the spray additive has to be removed from the original container the new container will be labeled adequately.

5. Spray additives will be carefully measured out to prevent cross contamination.

6. The spray additive will be disposed of properly if it becomes contaminated, changes in physical appearance from the original condition, or reaches the expiration date of 3 years after opening in the laboratory unless otherwise designated by the manufacturer.

Revised 03/18
Submitted by Field Research Director: Brian Jenks Date: 3-29-18
Approved by Field Research Coordinator: Date: 4/10/18

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #8: Measuring a pesticide formulation (Rev. 1.7).

WRITTEN BY: Brian M. Jenks

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

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

PROCEDURES:

1. Liquid formulations

    a. A plastic syringe or graduated cylinder will be used to measure liquid test substances. Air bubbles will be removed from the syringe before measuring out the required amount of test substance concentrate.

    b. Appropriate safety clothing and equipment will be worn while handling.

    c. The liquid test substance concentrate will be dispensed directly into the spray containers.

2. Dry formulations

    a. The balance will be calibrated prior to weighing the quantity for use in the study by following the directions of the manufacturer in the manual and in SOP #5.

3. A clean weighing container will be tared first and the amount of test substance weighed. The test substance will be dispensed directly into the spray container using a funnel as needed, or made into a slurry prior to dispensing into spray container to assure proper dissolution.

    a. Appropriate safety clothing and equipment will be worn while handling.

4. Precautions to Avoid Contamination of Samples

    a. The test substance will be stored at the North Central Research Extension Center from the time of receipt from the manufacturer until application. The test substance will be stored in its original container in an area designated for IR-4 storage.

    b. Any weighed or measured test products, if not used immediately, will be placed in clean sealed containers and labeled.

Revised 03/15
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Satom Mayorki Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #9: Procedures in the application of pesticides (Rev. 1.5).

WRITTEN BY: Brian M. Jenks

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 mixing, application, storage, and cleanup of pesticides will be properly trained.
2. Equipment used in pesticide application will be inspected and calibrated as indicated under SOP #6.
3. Personnel mixing and applying the pesticide should wear appropriate protective clothing as stated on the pesticide label. For general work this may include a Tyvek splash resistant coat or suit, unlined rubber gloves and boots, and eye protection for mixing and loading.
4. The pesticide concentrate will be measured out as indicated in SOP #8.
5. The test substance will be added to a small amount of carrier in the spray application container and brought up to volume with the carrier substance. The spray container will be closed tightly and agitated to adequately disperse the test substance throughout the carrier.
6. Immediately before applying the treatment, the sprayer will be calibrated as described in SOP #6, or rechecked. Then the spray container will be shaken for thorough mixing and the spray applied to the plot. Flags or stakes will be placed in the field as a guide for precise placement of each spraying pass.
7. Correct speed will be maintained while spraying the plot. Make sure all settings of pressure, speed, granular flow, etc. are set according to specification from the calibration as previously performed. Recheck the boom pressure after the sprayer has been turned on at the beginning of the plot, and before moving across the plot.
8. The test substance will be applied beginning with the lowest concentration and working up to the highest concentration.
9. Any pesticides applied for control of weeds or other pests will be applied to all plots according to directions on the pesticide label.

Revised 03/15
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Eston Mulligan Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #10: Cleanup of application equipment (Rev. 1.3).

WRITTEN BY: Brian M. Jenks

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

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

PROCEDURES:

1. All personnel involved in the mixing, application, storage, and cleanup of pesticides will be properly trained.
2. Application containers will be washed after use or disposed appropriately.
3. Leftover test substance solution will be disposed of in a manner consistent with sound environmental principles.

Revised 04/16
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Satoru Miyaoka Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #11: Procedures for cleaning the sprayer (Rev. 1.2).

WRITTEN BY: Brian M. Jenks

PURPOSE: To describe the procedures for cleaning the sprayer both 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 calibration runs.

2. The test substance will be applied beginning with the lowest concentration and working up to the highest concentration. Between applications of different rates of the same test substance, the sprayer will be rinsed with water only if required in the protocol.

3. Between treatments of different test chemicals or different formulations of the same chemical the sprayer will be rinsed with a dilute ammonia and water solution.

4. After application of the final treatment the sprayer will be rinsed with a dilute ammonia and water solution.

Revised 03/04
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Satoru Miyazaki Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #12: Procedures governing sprayer problems during application (Rev. 1.2).

WRITTEN BY: Brian M. Jenks

PURPOSE: To describe the procedures to follow in case something goes wrong during application of the test substance.

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

PROCEDURES:

1. The applicator should observe the spraying process to make sure the test substance is applied uniformly to the commodity.
2. If a sprayer malfunction such as a plugged nozzle, pressure loss, or hose break occurs, the applicator should try to determine when the malfunction happened and how to correct it before proceeding.
3. If the test substance is misapplied to any plot area, that area shall be well marked and avoided at harvest.
4. Appropriate individuals shall be notified and details recorded in the field notebook.

Revised 04/16
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Steven Wilczek Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
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SOP #13: Recording of raw data (Rev. 1.5).

WRITTEN BY: Brian M. Jenks

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

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

PROCEDURES:

1. The Field Research Director will take responsibility for seeing that all raw data, summaries, and other items connected with the study that need to be retained are stored in the study file.

2. Almost all raw data should be recorded in blue ink in a bound Inter Regional Research Project No. 4 Raw Data Notebook. Where specific data sheets are not available in the Notebook, data collected will be written on supplemental sheets and placed in the notebook (such as stand counts or crop injury ratings).

3. Corrections will be made by crossing through the item and initialing. The reason for change and date of change will also be noted.

4. No pages will be removed from the book.

5. All entries will be dated. Each filled page will be signed or initialed.

6. All data required by the Field Phase Research Protocol will be collected and recorded. The forms provided with the Field Phase Research Protocol will be carefully reviewed to make sure that all the required data are being collected.

7. The following information will be retained in the Field Data Book:

   a. Raw data including weather records, logs of instrument calibration, phytotoxicity, etc.
   b. Copies of summaries including original calculations and citations of sources of information used.
   c. Copies of reports including correspondence related to the reports.
   d. Copies of completed forms used during the study and for summaries of the study data.

Revised 03/18
Submitted by Field Research Director: Brian Jenks Date: 3-29-18
Approved by Field Research Coordinator: Date: 4/10/18

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #14: Method for collecting performance data (Rev. 1.6).

WRITTEN BY: Brian M. Jenks

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

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

PROCEDURES:

I. Phytotoxicity Data
   a. Consult the research protocol to determine the method and timing of the phytotoxicity data collection. Where phytotoxicity data is required, but no specific method is cited, then data may be collected as follows or as deemed appropriate by the Field Research Director:

   b. Where possible, take phytotoxicity data within 2-4 weeks after the test substance treatment. If symptoms occur during or after this period that warrant an additional reading, then phytotoxicity data should be taken as necessary.

   c. Phytotoxicity will be visually estimated by comparing the crop in the entire treated plot with crop in adjacent untreated areas and in the untreated plot. Crop in each plot will be assigned a percentage injury value using a scale of 0% = no injury to 100% = plant death. Stunting, chlorosis, necrosis, and stand reductions will be taken into account when estimating percentage injury.

II Efficacy
   a. Consult the research protocol to determine the method and timing of the pest data collection. Where efficacy data is required, but no specific method is cited, then data may be collected as follows or as deemed appropriate by the Field Research Director:
   b. Where possible, efficacy data (disease, insect, or weed control) may be taken within 2 to 4 weeks after treatment and within 2 weeks of expected harvest.
   c. Weed control will be visually estimated by comparing weed growth in the entire treated plot with that in adjacent untreated areas and in the untreated plot. Percentage weed control will be determined using a scale of 0% = no control to 100% = complete control, plant death.
   d. Where possible, disease and insect control will be evaluated in a manner appropriate for the specific organism.

Revised 04/10
Submitted by Field Research Director: Brian Jenks Date: 4-10-17
Approved by Field Research Coordinator: Eaton Mayski Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #15: Residue sample collection and storage (Rev. 2.4).

WRITTEN BY: Brian M. Jenks

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

SCOPE: All locations where studies are conducted to collect samples for residue analysis under the IR-4 Project.

PROCEDURES:

1. Consult the research protocol to establish specific dates for the collection of samples. These dates will be considered tentative and refined as necessary to accommodate a crop maturity that is earlier or later than expected due to environmental conditions for crop growth during the season.

2. Prior to sample collection, obtain a sufficient number of sample bags to collect all the samples. Plastic laminated cloth bags will be used.

3. Before entering the field, each sample bag will be labeled with waterproof ink to indicate the following information:

   a. Field ID
   b. Crop Fraction
   c. Test Substance
   d. Sample ID TRT #
   e. Harvest Date
   f. Sample Date
   g. Field Research Director: Name/Phone #

4. The following information will be printed and taped on the outside of each bag:
   a. Field ID
   b. Crop Fraction
   c. Test Substance
   d. Sample ID TRT#
   e. Harvest Date
   f. Sample Date
   g. Field Research Director: Name/Phone#

5. Samples will be direct harvested by small plot combine, if possible, with the untreated plot harvested first, followed by the lowest test substance rate, and finishing with the highest test substance rate. Standard procedures for cleaning plot combines prior to an IR-4 plot harvest will include: 1) use compressed air to clean inner surfaces including sieves, rotor, and conveyors, 2) use compressed air to clean outer surfaces including engine compartment, underbody and header platform, and 3) clean cutter sickle and bagger with ammonia solution. If seed cleaning and/or drying is necessary, seed will first be placed into new paper bags labeled with the appropriate treatment information. Seed may be dried at 90 to 150 F for up to 48 hours. Cleaning may be done using a small clipper mill. Finally, seed will be transferred to the permanent sample bags. If residue sampling is required before crop is combine harvestable, whole plants or plant parts would be collected by hand and dried for threshing. Drying would be in clean labeled paper bags or labeled cloth bags in a forced air dryer at 90 to 150 F for up to 48 hours. If drying is not necessary, seed cleaning may be done by hand immediately after sampling using appropriately sized cleaning sieves.

6. The scale used to weigh samples in the field will be calibrated bi-annually through one of the following methods: 1) the scale will be sent to the manufacturer for re-calibration, or 2) objects that weigh approximately 2-4 lbs will be weighed on a lab scale (recently calibrated by Northern Balance & Scale or other company) and compared to the field scale. If the field scale differs from the recently calibrated lab scale by more than 0.2 lb, the field scale will be sent to the manufacturer for re-calibration.

7. Special care will be exercised in the following aspects of sample collection:
   a. Contamination of the samples with the test substance under study will be avoided.
   b. Diseased portions of the plot will be avoided during harvest.
   c. Samples will be handled gently so as not to cause undue damage to the harvested seed.
   d. All tools used will be clean.

8. Samples will be kept in coolers during transportation from the test site to NCREC. If samples cannot be placed in freezer within 1 hour of harvest (unless drying is needed prior to going into freezer), coolers will contain some type of cooling agent to maintain integrity of the samples.

9. Attached to the freezer in which samples are stored will be a log indicating: Field ID No., date samples were collected, and number of sample bags for the project. Any removal of samples before shipment will be documented on the log with the following information: name of person removing the samples, the identity of the particular sample bags removed, date removed, and date returned.

10. At the NCREC, samples will be logged in and placed in frozen storage. The building where samples are stored will be locked after normal working hours and the storage temperature will be recorded periodically.

11. An electronic device with battery backup (Temperature Alert System) will monitor freezer temperatures. The device will send a message to research personnel if the freezer temperature rises above an acceptable level. If electricity is lost for an extended period of time, the freezers will be connected to a generator. If a freezer malfunctions, samples will be moved to a different freezer.

Revised 03/18
Submitted by Field Research Director: Brian Jenks Date: 3-29-18

Approved by Field Research Coordinator: Date: 4/10/18

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #16: Sample shipping procedures (Rev. 1.6).

WRITTEN BY: Brian M. Jenks

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

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

PROCEDURES:

1. The chemist at the residue laboratory will be notified of the shipment dates and method including carrier. Air freight shipments should be made on Monday or Tuesday to avoid potential weekend layovers. Shipment by freezer truck will be preferred, in which case dry ice will not be used. Otherwise, shipment will be as follows. Samples will be shipped within 14 days of harvest or next available time by ACDS.

2. Arrangements will be made with the carrier for sample shipment. Special packing instructions will be noted and observed.

3. If shipping method is not by freezer truck, note limits on quantity of dry ice, etc. set by the carrier. Obtain insulated containers of sufficient size and quantity to hold the residue samples and dry ice (where required) in a 1:1 to 1:3 weight ratio to commodity and pack the samples and dry ice in the containers just prior to shipment. The containers should have a sufficient bursting strength so as to withstand normal handling in shipping and storage.

4. Place a copy of the residue sample shipping form in every container. Distribute the copies of the residue sample shipping form as indicated on the form. The original of this form will be retained in the field notebook.

5. Shipping containers will be labeled with the following information:
   a. Return name and address of sender (B. M. Jenks).
   b. Name and address of residue lab receiving the samples.
   c. Number of the container (if more than one is used).
   d. "Dry ice" (if used) will be marked on two sides of each container.

6. Container lids will be taped firmly in place.

7. The carrier will be given samples and the phone number of the residue lab and asked to notify the lab for pickup when samples arrive at a remote terminal.

8. Provide the carrier with the samples for shipment.

Revised 04/17
Submitted by Field Research Director: Brian Jenks Date: 4-20-17
Approved by Field Research Coordinator: Eaton Mizerski Date: 4-26-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #17: Completion of forms (Rev. 1.3).

WRITTEN BY: Brian M. Jenks

PURPOSE: To assure that forms are completed accurately and properly document the results of the study.

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. Forms will be filled out as completely and accurately as possible. Sufficient detail will be provided on data collection methods to enable someone else to repeat the research.
3. The narrative portion of the forms and Field Data Book will be used to summarize findings or to explain any unusual findings or observations.
4. All existing forms will be used. New forms will be developed as needed to conform with SOPs.
5. The originals of completed report forms and accompanying documentation will be forwarded to the regional coordinator within a reasonable period of time after the study is completed.
6. The regional coordinator will review all forms and accompanying documents.
7. A copy of the Field Notebook and necessary documents will be archived by the Field Research Director at the NCREC.

Revised 04/06

Submitted by Field Research Director: Brian Jenks Date: 4-10-17

Approved by Field Research Coordinator: Peter Nyhuis Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #18: Facility file (Rev. 1.2).

WRITTEN BY: Brian M. Jenks

PURPOSE: To assure that facility file is current and complete.

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

PROCEDURES:

1. The facility file will contain either the original or certified copies of the following documents:
   a. SOP's and their revisions (Signed original SOPs should be sent to IR-4 HQ for archival).
   b. Organization chart and floor maps of the facilities. They will be reviewed periodically (at least every 5 years) and updated as needed.
   c. Personnel records such as CV's, job descriptions, and training records for individuals involved in IR-4 trials. They will be reviewed periodically (at least every 5 years) and updated as needed.
   d. Chemical storage log, test substance container inventory log, and freezer sample inventory log
   e. Equipment records for scales, weight sets, and thermometers, including calibration certificates and any repairs.
2. Long-term storage for the facility file will be a locked fire-proof filing cabinet located in the NCREC main office building.
3. Documents in the facility file will be archived at NCREC at least as long as specified in GLP Standards 160.195(b), or sent to IR-4 headquarters if space is limited.

Written 03/18

Submitted by Field Research Director: Brian Jenks Date: 3-29-18

Approved by Field Research Coordinator: Date: 4/10/18

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #19: Procedures for EPA inspection (Rev. 1.1).

WRITTEN BY: Brian M. Jenks

PURPOSE: To provide guidance for facilitating audits by the Environmental Protection Agency, Office of Compliance Monitoring.

SCOPE: Applies to all GLP research trials conducted at the NCREC.

PROCEDURES:

1. Prior to Inspection

    a. Notify the Study Director, Quality Assurance Officer, and other interested personnel of the pending audit or review as soon as possible.

    b. Personnel who may be associated with the trial(s) or facilities audit should be briefed and scheduled to be available for the audit.

    c. Prepare trial(s) and/or facilities personnel for the inspection.

    d. Be certain that all documents pertaining to the trial(s)/facilities inspection are available.

2. During Inspection

    a. Greet inspection team.

    b. Ask inspector(s) to provide credentials. The inspection team will also be asked for a proposed inspection agenda and expected duration.

    c. Personnel will provide information requested by inspectors.

    d. Proceed with inspection.

3. Post Inspection

    a. All personnel involved in the inspection should attend the exit interview.

    b. Any discrepancies brought up during the exit interview will be clarified if necessary and documented.

    c. A copy of all documents provided to the inspection team will be retained.

    d. After inspection all parties will be informed of inspection activities and findings. Remedial action for problems that may have been detected during the inspection will be discussed and a plan of action initiated.

    e. If required, inspection findings will be responded to in a timely manner.

Written 06/15

Submitted by Field Research Director: Brian Jenks Date: 4-10-17

Approved by Field Research Coordinator: Eldon Mayhew Date: 4-17-17

IR-4 Field Research - Standard Operating Procedure - Dr. Brian M. Jenks, FRD
North Central Research Extension Center, 5400 HWY 83 S, Minot, ND 58701

SOP #20: Data archiving, retention, and quality control review (Rev. 2.0).

WRITTEN BY: Brian M. Jenks

PURPOSE: To assure that data collected from a field trial is archived at IR-4 Headquarters and updated data are maintained at North Central Research Extension Center in Administration Building.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. The official archive for original raw data will be located at IR-4 Headquarters.

2. The Field Research Director or Research Personnel will make an exact copy of the original raw data including completed Field Data Books, logs, weather data, personnel forms, etc. These copies will be retained in a secure location at North Central Research Extension Center in Administration Building.

3. All information in the Field Research Director's file should be clearly identified.

4. All original raw data not included in a Field Data Book (e.g. logs, weather data, personnel forms, etc.) will be sent directly to IR-4 Headquarters with a chain of custody form including name of courier, tracking number and inventory of items sent.

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

6. The Field Research Director or Research Personnel will add any additional or changed pages to the Field Data Book copy on file and these updated pages will be used for all subsequent quality assurance audits.

Written 04/17
Submitted by Field Research Director: Brian Jenks Date: 4-20-17

Approved by Field Research Coordinator: Satom Mujaski Date: 9-26-17