SOP Log Sheet

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State: MI **City:** East Lansing

Location: NCR Field MSU Hausbeck

FRD/LRD: Mary Hausbeck
Submitter

Effective Date: 4/10/18

Description of Material (s): 2018 SOPs

Reviewed By: Juliet Chompeon 4/26/18
Sign/Date

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Comment:

S: Facility Files- SOP Cover Sheet

9/13/16 JT

MICHIGAN STATE
UNIVERSITY

TO: Dr. Mary K. Hausbeck
Michigan State University
Dept. of Plant Pathology
140 Plant Biology Lab
East Lansing, MI, 48824

FROM: John Wise, IR-4 Regional Field Coordinator

SUBJECT: STANDARD OPERATING PROCEDURE APPROVAL

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 #	Submission Date	SOP	Revision #	Review Date	SOP	Revision #	Submission Date
1.1	1.2	4-15-16	6.2	1.0	4-15-16	9.7	1.2	4-15-16
1.2	1.3	2-27-18	6.3	1.1	4-15-16	9.8	1.2	4-15-16
1.3	1.1	2-27-18	6.4	1.2	4-15-16	10.1	Retired 2015	
1.4	1.1	4-15-16	6.5	1.0	4-15-16	10.2	1.1	4-15-16
1.5	1.3	2-27-18	7.1	1.4	4-15-16	10.3	1.2	4-15-16
2.1	1.0	4-15-16	7.2	1.2	4-15-16	10.4	2.1	4-15-16
2.2	1.0	4-15-16	7.3	1.2	4-15-16	10.5	2.2	4-15-16
2.3	1.0	4-15-16	7.4	2.2	2-27-18	10.6	1.0	8-22-16
2.4	1.0	4-15-16	7.5	1.2	4-15-16	11.1	1.1	4-15-16
2.5	1.1	4-15-16	7.6	1.2	4-15-16	11.2	1.1	4-15-16
2.6	1.0	4-15-16	7.7	1.0	4-15-16	11.3	1.0	4-15-16
3.1	1.0	4-15-16	7.8	1.1	4-15-16	12.1	1.0	4-15-16
3.2	1.0	4-15-16	7.9	1.0	4-15-16	13.1	1.1	4-15-16
3.3	1.4	4-15-16	7.10	1.0	4-15-16	13.2	1.0	4-15-16
4.1	1.2	4-15-16	8.1	1.1	4-15-16	13.3	1.0	4-15-16
4.2	1.2	2-27-18	9.1	1.0	4-15-16			
5.1	1.0	4-15-16	9.2	1.1	4-15-16			
5.2	1.1	2-27-18	9.3	1.0	4-15-16			
5.3	1.0	4-15-16	9.4	1.0	4-15-16			
5.4	1.0	4-15-16	9.5	1.3	4-15-16			
6.1	1.1	4-15-16	9.6	1.0	4-15-16			

IR-4 NORTH CENTRAL REGION RESEARCH CENTER
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Department of Plant, Soil, and Microbial Sciences
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East Lansing, MI 48824-1311

APPROVAL OF REVISIONS IN THE CURRENT YEAR OF STANDARD OPERATING PROCEDURES

DATE OF REVIEW	SOP NUMBER(S) REVISED	SIGNATURE OF FIELD RESEARCH DIRECTOR	SIGNATURE OF IR-4 FIELD RESEARCH COORDINATOR
2-27-18	1.2, 1.3, 1.5, 4.2, 5.2, 7.4	Mary Hausbeck	

Department of Plant, Soil, and Microbial Sciences
Michigan State University
East Lansing, MI 48824-1311

Submitted by Field Research Director: Mary Hausbeck MH Date: 3-27-18

Approved by Regional Field Coordinator: John Wise JCW Date: 4/10/18

INDEX OF STANDARD OPERATING PROCEDURES

Department of Plant, Soil, and Microbial Sciences
Michigan State University
East Lansing, MI 48824-1311

Submitted by Field Research Director: Mary Hausbeck MH Date: 2-27-18

Approved by Regional Field Coordinator: John Wise JW Date: 4/10/18

INDEX OF STANDARD OPERATING PROCEDURES

Department of Plant, Soil, and Microbial Sciences
Michigan State University
East Lansing, MI 48824-1311

Effective Date: April 15th, 2016
Version Number: 1.2

Submitted by Field Research Director: Mary K. Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-29-16

SOP: 1.1

TITLE: General requirements for Standard Operating Procedures (SOPs).

PURPOSE: To provide guidance in development and use of SOPs for field research studies.

SCOPE: All pesticide residue studies conducted under the direction of Mary K. Hausbeck and requiring GLP compliance.

PROCEDURES:

1. A standard operating procedure, or SOP, is a written document that describes how routine activities are to be performed. The IR-4 Field Research Director will develop standard operating procedures (SOPs) for all phases of research conducted in support of chemical registration.

2. The protocol always takes precedence over any SOP.

3. An SOP should exist for all routine procedures and regularly used equipment. Individuals completely familiar with the process or equipment should write the SOPs. The SOP should include enough detail so that someone with the appropriate education, training, and experience can perform the procedure correctly.

4. The individual SOPs, together with an index and SOP Approval Log, will constitute SOPs for IR-4 chemical residue studies at the Department of Plant, Soil, and Microbial Sciences, Michigan State University.

5. SOPs will be reviewed annually and revised as needed. The review and revisions will be recorded on the SOP APPROVAL LOG, which will be maintained as part of SOPs. All earlier versions of SOPs must be retained in an archive file.

6. Each individual SOP will be approved, initialed/signed, and dated by the Field Research Director and the Regional Field Coordinator.

7. Any deviations from the SOPs that would affect the results of a study must be documented in writing and signed by the Study Director.

8. The Regional Field Coordinator and other study personnel will receive copies of the SOPs on request. The original SOPs will be kept in a secure file in the Field Research Director's office.

9. The original copy of the previous years SOPs will be shipped to IR-4 Headquarters for archiving within one year of the updated version being signed by the Field Research Director. A true copy of all previous SOPs will be archived in the IR-4 document cabinet in the office of the Field Research Director.

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Effective Date: March 1, 2018
Version Number: 1.3

Submitted by Field Research Director: Mary K. Hausbeck MH Date: 2-27-18

Approved by Regional Field Coordinator: John Wise JW Date: 4/10/18

SOP: 1.2

TITLE: Annual review and revisions.

PURPOSE: Document annual review and revision procedures.

SCOPE: All related SOPs.

PROCEDURES:

1. The "Standard Operating Procedures, Field Residue Trials" (THE SOP) will be reviewed annually by the Field Research Director and revised as needed. When a SOP is edited, it must be listed under the "Approval of Revisions in the Current Year of Standard Operating Procedures" which should then be submitted for approval by the Regional Field Research Coordinator.

2. Major revisions of SOPs will be designated by whole numbers (e.g., 1.0, 2.0, 3.0).

3. Revisions of individual SOPs will be designated by decimal numbers (e.g., 2.0, 2.1, 2.2, 2.3).

4. When many individual SOPs have been revised, a major revision of a SOP will be instituted and designated as the succeeding whole number.

Department of Plant, Soil, and Microbial Sciences
Michigan State University
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Effective Date: March 1, 2018
Version Number: 1.1

Submitted by Field Research Director: Mary K. Hausbeck MH Date: 3-27-18

Approved by Regional Field Coordinator: John Wise JW Date: 4/10/18

SOP: 1.3

TITLE: Definitions for the SOP.

PURPOSE: To define the terms used in this SOP.

SCOPE: For all SOPs developed by Mary K. Hausbeck, Michigan State University.

PROCEDURES:

1. In the SOPs, the following terms will have the meanings specified.
   a. BATCH - a specific quantity or lot of a test substance that has been adequately characterized.
   b. EXPERIMENTAL START DATE - the first date the test substance is applied to the test system (crop).
   c. EXPERIMENTAL TERMINATION DATE - the last date on which data are collected directly from a study.
   d. GOOD LABORATORY PRACTICES (GLP) - a set of guidelines mandated by Congress to which researchers must adhere to assure the integrity of research data. All IR-4 studies are conducted under GLP guidelines.
   e. MASTER TIMETABLE - a list of trials which is maintained by the Field Research Director. It must be indexed by test chemical and crop, and contain type of trial, approximate experimental start dates, and termination dates.
   f. MASTER SCHEDULE – a list, maintained by the quality assurance unit, of all studies conducted at the testing facility indexed by test substance, and containing the test system, nature of study, date study was initiated, current status of each study, identity of the sponsor, and name of the study director.
   g. PROTOCOL - a specific document, provided by the sponsor that contains details for accurate completion of a trial.
   h. QUALITY ASSURANCE UNIT (QAU) - any person or organizational element, as per 40CFR 160.35, who is designated to perform the monitoring duties to assure that the research is conducted according to Standard Operating Procedures and GLP. Regional IR-4 representatives will designate the Quality Assurance Officer (QAO) for IR-4 trials.
   i. RAW DATA - worksheets, records, memoranda, notes, etc., that are the results of original observations and activities of a study. This includes photographs and computer printouts.
   j. SPONSOR - the individual, corporation, association, scientific or academic establishment, government agency or other organizational unit who initiates and supports, by provision of financial or other resources, a study.
   k. STANDARD OPERATING PROCEDURES (SOP) - written documentation of routine activities utilized in research studies.
   l. TRIAL - an experiment in which a test substance (pesticide) is applied to as test system to determine or help predict its effect, metabolism, environmental and chemical fate, or other characteristics.
   m. FREQUENTLY USED ACRONYMS –
      Michigan State University (MSU)
      Plant Biology Building (PBL)
      Center for Integrated Plant Systems (CIPS)
      Plant and Soil Greenhouses (PSG)
      Plant Pathology Farm (PPF)

Department of Plant, Soil, and Microbial Sciences
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Effective Date: April 15th, 2016
Version Number: 1.1

Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP: 1.4

TITLE: Numbering system for the SOPs.

PURPOSE: To provide the numbering system used to index SOPs.

SCOPE: For all SOPs developed by Mary K. Hausbeck, Michigan State University.

PROCEDURES:

1. A numbering system will be used for SOPs. Its format is as follows: X.Y, where X=section number, Y=specific SOP number in the category.

2. SOPs will be organized using these major section headings:

    1. General rules and requirements for Standard Operating Procedures (SOPs)
    2. Personnel
    3. Study protocol and experimental design
    4. Site selection and plot establishment
    5. Agronomic practices for field experiments
    6. Application equipment calibration, maintenance and use
    7. Test chemical receipt, storage, handling and disposal
    8. Safe handling of pesticides and use of safety equipment
    9. Data recording.
    10. Residue sample collection, storage, and shipping
    11. Trial reports, archives, and record storage
    12. Quality Assurance
    13. EPA inspection

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Effective Date: March 1, 2018
Version Number: 1.3

Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 2-27-18

Approved by Regional Field Coordinator: John Wise JW Date: 4/10/18

SOP: 1.5

TITLE: Format guidelines for SOPs.

PURPOSE: To provide a uniform format for development of SOPs.

SCOPE: For all SOPs developed by Mary K. Hausbeck, Michigan State University.

PROCEDURES:

1. The following header will be used on each SOP:

   Department of Plant, Soil, and Microbial Sciences
   Michigan State University
   East Lansing, MI 48824-1311

   Effective Date:
   Version Number:

   Submitted by Field Research Director: Mary K. Hausbeck_____ Date:_____

   Approved by Regional Field Coordinator: John Wise_____ Date:_____

2. Margins:
   Top 0.5", Bottom 1.0", Left and Right 1.0"

3. Font:
   Times New Roman at 10 pt size.

4. Description header of SOP:

   SOP: SOP number

   TITLE: Concise description of procedure

   PURPOSE: Expansion of title and reason for SOP

   SCOPE: Describe limits of SOP.

   PROCEDURES: Describe procedures in detail, but avoid being overly restrictive. Trained personal should be able to replicate procedure from SOP.

5. Line spacing shall be single spaced within paragraphs with a double space between paragraphs.

6. If text of the SOP requires more than one page, the successive pages will have the top header included.

7. Page numbering will be at the bottom center of each page.

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Effective Date: April 15th, 2016
Version Number: 1.0

Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP: 2.1

TITLE: Personnel.

PURPOSE: To identify personnel involved in chemical registration trials and their responsibilities under GLP and ensure proper personnel records are maintained.

SCOPE: All individuals directly involved in trials.

PROCEDURES:

1. Personnel involved in pesticide registration research will be designated as Study Director, Field Research Director, or other personnel.

2. Field Research Director will ensure that a sufficient number of personnel are available and properly trained to conduct the trials.

3. Each individual engaged in the conduct of, or responsible for the supervision of, a trial shall have education, training, and experience to enable that person to perform the assigned functions.

4. The facility will maintain a current summary (a resume, curriculum vitae, or similar document) of training and experience of each person involved with trials. A job description will be maintained for all permanent employees involved with the study.

5. Individual personnel will maintain their own training records.

6. Personnel applying pesticides must be properly certified.

7. Temporary student helpers and farm managers who provide non-technical labors are not considered as study participants/GLP personnel.

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Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP: 2.2

TITLE: Field Research Director

PURPOSE: To describe responsibilities of the Field Research Director.

SCOPE: All IR-4 field research studies.

PROCEDURES:

1. A Field Research Director (FRD) for each trial will be designated by the Regional Field Coordinator. The Field Research Director has immediate and personal responsibility for conduct of a trial, including supervision of all other personnel involved in the trial.

2. The Field Research Director will:

    a. Assure that the trial is carried out according to the protocol and good laboratory practices.
    b. Report deviations from protocol or SOP to Study Director.
    c. Assure that personnel, resources, facilities, equipment, materials and methods are available as scheduled for the conduct of the trial.
    d. Assure that all personnel conducting the trial understand the protocol and SOPs for the trial.
    e. Respond to all deviations reported by the Quality Assurance Unit (QAU) in writing.
    f. Maintain a Master Timetable for all IR-4 field research trials under his control.
    g. Submit a copy of the Master Timetable to the QAO within 30 days of receiving all trial assignments.
    h. Complete all report forms and transfer all reports and raw data (except farm records, temperature logs and soil test reports) to the IR-4 Regional Field Coordinator upon completion of the trial.
    i. Retain copies of raw data from each trial for a minimum of 5 years or until otherwise directed by Study Director.

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Effective Date: April 15th, 2016
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Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP: 2.3

TITLE: Other Personnel

PURPOSE: To provide information concerning requirements under GLP for other personnel involved in pesticide trials.

SCOPE: All IR-4 field research studies.

PROCEDURES:

1. The Field Research Director will have on file a current vita for all personnel actively engaged in the conduct of a trial.

2. Incidental or temporary help involved in field preparation, planting, weeding, application of fertilizer and/or maintenance chemicals or harvesting for yield purposes are not considered actively engaged in the conduct of a trial. However, their activities will always be supervised and monitored by personnel who are actively engaged in the conduct of the trial.

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Effective Date: April 15th, 2016
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Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP: 2.4

TITLE: Organizational Chart.

PURPOSE: To describe requirements for an organizational chart.

SCOPE: All IR-4 field research studies.

PROCEDURES:

1. An organizational chart which reflects the management of the facility and the reporting lines of the personnel engaged in IR-4 trials will be developed and kept on file by the Field Research Director.

2. Each entry in the chart should show the name and title of the person filling that position.

3. The chart must show how the Field Research Director reports to the Study Director and the National Director, and the relationship between the QAU and the other parts of the organization.

4. The Field Research Director will annually review the organizational chart and revise it as needed. Initial and date the newly revised chart. The outdated chart will be archived.

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Effective Date: April 15th, 2016
Version Number: 1.1

Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP: 2.5

TITLE: Documentation of training.

PURPOSE: To ensure training is properly documented.

SCOPE: All personnel actively involved in IR-4 field trials.

PROCEDURES:

1. Personnel will be responsible for maintaining their own training records.
2. Training record entries should include the names and dates of classes, seminars, workshops, and conferences. If appropriate, include the names of the people performing the training and a brief description of the training.
3. Individuals should annually review their training record, resume or curriculum vitae and position description and revise as needed. Sign and date the documents when they are reviewed or revised.
4. A current summary of training and experience for individuals involved in trial activities will be maintained in the facility files.
5. Personnel records for those people no longer employed at the facility will be archived.

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Effective Date: April 15th, 2016
Version Number: 1.0

Submitted by Field Research Director: Mary K. Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP: 2.6

TITLE: Archiving.

PURPOSE: To ensure documents and data are properly archived.

SCOPE: All documents and data in IR-4 field trials subject to archiving for GLP compliance.

PROCEDURES:

1. A single person will be responsible for all archiving responsibilities.
2. A separate filing cabinet, with a limited access, for all archived data should be maintained within the Field Research Directors office.
3. A log for all items entering or leaving the archive will be maintained.
4. All data must be archived for a minimum of 5 years or until Study Director determines that the documents can be destroyed.

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Effective Date: April 15th, 2016
Version Number: 1.0

Submitted by Field Research Director: Mary Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-29-16

SOP 3.1

TITLE: Study protocol and experimental design.

PURPOSE: To describe the normal methodology for obtaining a study protocol and planning an IR-4 field research trial.

SCOPE: All pesticide registration trials.

PROCEDURES:

1. The Study Director will provide a copy of the protocol to the Field Research Director.

2. The protocol is an official document and must be followed completely to assure that the data generated will be useful in the registration process.

3. The Field Research Director is responsible for meeting all requirements of the protocol.

4. The Field Research Director will prepare a Master Timetable including all major functions of all trials to be conducted at his/her location in a given year. The Field Research Director will submit the Master Timetable to the Quality Assurance Officer and the Regional Field Coordinator as early as possible and before trials are established in the field. The Field Research Director will maintain a file of annual Master Timetables.

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Effective Date: April 15th, 2016
Version Number: 1.0

Submitted by Field Research Director: Mary Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP 3.2

TITLE: Deviations from protocol.

PURPOSE: To provide methodology for dealing with unforeseen changes in protocols or field conditions that preclude complete compliance with the protocol.

SCOPE: Changes or deviations initiated by Sponsor, Study Director, or Field Research Director.

PROCEDURES:

1. For changes in protocols initiated by the Sponsor or Study Director, the Study Director will send a copy of the protocol change form listing specific changes in the protocol to the Field Research Director. The protocol change form should be sent to the Field Research Director before the experimental start date. A copy of the protocol change form will be attached to the protocol and a copy will be filed with the trial report.

2. For deviations from protocol initiated by the Field Research Director, the Field Research Director will prepare a protocol deviation form and submit it to the Study Director. If the study deviation form is approved by the Study Director, he can continue the trial. If the Study Director does not approve the study deviation form, the Field Research Director must either follow the protocol or discontinue the trial.

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Effective Date: April 15th, 2016
Version Number: 1.4

Submitted by Field Research Director: Mary Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP 3.3

TITLE: Experimental design.

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

SCOPE: All IR-4 field research trials to support pesticide registration.

PROCEDURES:

1. The experimental design as specified by the protocol should be used. Residue trials are normally not randomized. The experimental design used should be documented in the Field Data Book.

2. Draw a plot map showing the location of each plot and any surrounding landmarks. Retain the plot map in the Field Data Book.

3. Plot permanent reference points may be collected with a handheld Global Positioning System (GPS), or by measuring the distance to a permanent landmark in the vicinity of the plot with a drag tape or a laser rangefinder.

4. IR-4 does not normally require Field Research Director to analyze data.

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Effective Date: April 15th, 2016
Version Number: 1.2

Submitted by Field Research Director: Mary Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP 4.1

TITLE: Site selection and plot establishment.

PURPOSE: To assure that plots will provide the required data and samples with sufficient uniformity to meet EPA requirements.

SCOPE: All IR-4 field research trials to support pesticide registration.

PROCEDURES:

1. Sites will be selected that are representative of normal production areas for the commodity.

2. Sites will be large enough to accommodate the number of treatments, required buffer zones and the sample sizes required by the protocol. Refer to the protocol for required distances between treated and untreated plots.

3. Where possible establish site so that treated plots are downwind of untreated plot.

4. If possible, locate the site with sufficient isolation to minimize contamination from external sources such as commercial operations or other research studies.

5. Obtain a soil test for the site as per sop 4.2.

6. If the trial is to be conducted on perennial crops on a commercial farm, select a reliable grower with a planting in good health, managed by accepted horticultural practices and with a previous pesticide history that meets protocol requirements.

7. If the site is on a commercial farm, arrangements should be made with the grower for crop destruction and remuneration if a non-registered pesticide is used on his crop. Record date crop destroyed.

8. If the commodity is already established, select a site that has a uniform stand.

9. Prepare a plot map, before the first application, showing the location of each plot on the site, the North azimuth, dominant wind direction, and any significant slope. The plot map should contain permanent reference points so that the plots can be relocated after the trial is terminated. Alternatively, the site may be identified by Global Positioning System giving coordinates for the beginning and end of the treated plot.

10. If plot is located on sloped ground, place the untreated control plot uphill of the treated plot.

11. Place a stake at each plot identifying the trial and treatment number. Assign each treatment and subplot a number. Number stakes with sufficient information to identify the replicate and treatment assigned to each plot.

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

13. Using an average yield as a baseline, it is best to make the plot large enough to provide at least 4 times the total quantity of sample requested. No calculations need to be provided for these estimates.

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Effective Date: March 1, 2018
Version Number: 1.2

Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 3-27-18

Approved by Regional Field Coordinator: John Wise JW Date: 4/10/18

SOP 4.2

TITLE: Soil sampling and characterization.

PURPOSE: To describe procedures for taking soil samples for field trial sites.

SCOPE: All IR-4 field trials.

PROCEDURES:

1. If the site has had a soil test within the last 5 years, use the results of that test for soil characterization information.

2. If no current soil test is available, take a soil sample during the experimental period using the following methods:

   a. Using a clean soil probe and clean bucket take 15 to 20 random soil samples at a 6- to 8-inch depth in the field and place samples in the clean bucket.

   b. Once all samples are taken, mix the soil thoroughly in a bag or bucket. Fill a clean soil container with soil. Label the container with the field number and send it to the Michigan State University Soil Testing Laboratory for analysis.

3. To determine soil type and series for the site, look up the field location in the appropriate county soil survey report. Include a soil survey map in the FDN and mark the location of the test plot on the map. Also include any keys or information on the soil series into the FDN.

4. Soil must be delivered to the testing facility within four days of sampling.

5. If the soil test results are delivered to the Study Director over email, a printout should be added to the FDN and initial and dated. A printout of the soil test results will also be archived with the Study Director.

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Submitted by Field Research Director: Mary Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP 5.1

TITLE: Agronomic practices for field experiments.

PURPOSE: To assure that crops grown for pesticide trials are produced following good agronomic practices.

SCOPE: All IR-4 pesticide trials to support registration.

PROCEDURES:

1. Determine and document good horticultural practices for the commodity to be grown.

2. Perform and record all cultural practices (e.g. plowing, disking, dragging, cultivating) carried out before and after planting. Fertilize as needed to produce the crop.

3. Apply pesticides that will not interfere with the outcome of the trial, as needed, to grow the crop. Record all pesticides applied in the Field Data Notebook.

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Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 3-27-18

Approved by Regional Field Coordinator: John Wise JW Date: 4/10/18

SOP 5.2

TITLE: Greenhouse and shadehouse facilities.

PURPOSE: To assure that greenhouse and shadehouse facilities are adequate for growing transplants for IR-4 field trials.

SCOPE: All IR-4 field trials in which greenhouse or shadehouse facilities are used for growing transplants.

PROCEDURES:

1. Each greenhouse or shadehouse must be sufficiently large enough to contain the plants for a trial and for separation of treated and untreated plants.

2. Plants should be grown under good horticulture procedures to provide acceptable plants for the trials.

3. Greenhouse temperature will be monitored by a portable thermographic device for projects that require transplants to be treated during greenhouse production or if any part of the project is located in the greenhouse. The thermographic device will be maintained in accordance with SOP 9.5 and 9.6.

4. Untreated plants/plant materials will be placed on the upexhaust part of the greenhouse.

5. Greenhouse benches will be cleaned with a minimum of 10% bleach solution before any material will be placed onto the benches.

6. To prevent the contamination of the untreated plants during a residue study, if the test substance applications are made to plants in the greenhouse, the untreated control plants must be moved to a separate greenhouse just prior to the application and must remain in a separate greenhouse for a minimum of 12 hours and must be returned to the greenhouse with the treated plants within 24 hours of the application.

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SOP 5.3

TITLE: Seeding and transplanting.

PURPOSE: To assure that commodities are established following good agricultural practices.

SCOPE: All IR-4 trials.

PROCEDURES:

1. Select a suitable cultivar for the trial. Note cultivar and source in Field Data Notebook.

2. Seed or transplant to establish the crop according to accepted horticultural practices.

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SOP 5.4

TITLE: Crop maintenance.

PURPOSE: To assure that commodities are grown under good horticultural practices and to provide a uniform crop for observation and harvest.

SCOPE: All IR-4 field trials.

PROCEDURES:

1. Maintain the crop as well as possible throughout the duration of the trial.

2. Apply labeled pesticides as needed to prevent losses due to pests. Do not apply pesticides that may interfere with the chemical analysis of the pesticide under study. If in doubt, call the analytical chemist or laboratory, identified in the protocol, that will receive the residue samples.

3. Keep a record of all pesticide applications, including product and rate.

4. Apply irrigation as needed to produce the crop. Keep a record of all irrigation and rainfall.

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SOP: 6.1

TITLE: Application equipment calibration, maintenance and use.

PURPOSE: To describe the calibration, maintenance, and use of pesticide application equipment used in IR-4 research trials.

SCOPE: All application equipment used in experiments to support pesticide registration.

PROCEDURES:

1. All sprayers and applicators used in pesticide trials done under GLP should be inspected, cleaned and maintained each year. The information from these routine, annual, pre-season calibrations will be recorded on the Equipment Maintenance, Repair and Calibration form for each sprayer.

2. Sprayers should be calibrated daily before use for each trial, or as required by protocols.

3. Written records on the Equipment Maintenance, Repair and Calibration form will designate activities as routine or non-routine. Inspection, cleaning, maintenance, testing and calibration are routine procedures. In the case of equipment failure or malfunction, written records describing the event will be recorded. Any repair as a result of malfunction is non-routine.

4. In the case of equipment failure, malfunction or damage, the remedial action plan will be followed and alternate equipment will be obtained to complete trial requirements.

5. The Field Research Director is responsible for equipment inspection, calibration and maintenance. Other personnel may conduct the operations.

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SOP: 6.2

TITLE: Calibration of a hand carried compressed gas pressurized boom sprayer.

PURPOSE: To set the delivery rate of the sprayer to ensure accurate application of the pesticide.

SCOPE: All IR-4 sponsored trials where a hand carried compressed gas pressurized sprayer is used in the application of pesticides.

PROCEDURES:

1. Visually inspect equipment for obvious wear or potential leaks and repair or replace as necessary.

2. Choose the appropriate nozzle tips to deliver the volume, pressure, and spray pattern required.

3. Pressurize the spray tank with sufficient CO2 to maintain the desired pressure throughout the application. Set the pressure regulator on the CO2 tank to deliver the desired volume and pressure at the nozzle.

4. To determine whether all nozzles are discharging uniformly, place each nozzle in a graduated cylinder and open the trigger valve for a given length of time. Replace nozzle tips that vary more than 5% from the average. Repeat the above procedure until all nozzles are discharging relatively uniformly.

5. When spraying with a single nozzle boom, use even spray nozzles and measure the desired band width before spraying the treatment plots.

6. When spraying with a multiple nozzle boom, hold boom at desired height over target to obtain an approximate 30% overlap on each side of each nozzle.

7. Calibrate the boom as follows:

   a. Calibration calculations are based on information provided in the Spraying Systems Co. Catalog. All fluid delivery calculations are based on the assumption that any XX02 nozzle delivers 0.2000 gallons of water a minute at 40 psi, any XX04 nozzle delivers 0.4000 gallons of water a minute at 40 psi and XX06 nozzle delivers 0.6 gallon of water a minute at 40 psi. 3785.3 ml/gal x 0.1734 gpm = 656.4 ml/min = 10.94 ml/sec per nozzle @ 30 psi. The delivery of an XX04 nozzle is double the delivery of an XX02 and the delivery of XX10 is five times the delivery of XX02. Therefore, in this formula, an XX04 nozzle would deliver 21.88 ml/sec and an XX10 would deliver 54.70 ml/sec.

   b. To calibrate the boom and CO2 pressure regulator, the tank is filled with water and pressurized. The nozzles are placed into graduated cylinders and the trigger valve is opened for 30 seconds. At 40 psi 8006 and 11006 nozzles should deliver 1135.6 ml in 30 seconds. If delivered volume is not correct, the CO2 regulator is adjusted and the process repeated until the delivery is within 2% of the desired amount. The reading on the CO2 regulator and spray boom are recorded for each test. When the correct amount has been delivered twice in a row, the boom is considered calibrated. Record pressure readings and nozzle volumes for the annual, preseason

calibration in the Sprayer Log Book. Calibration results for pesticide application are recorded in the Field Data Book.

8. Calculate walking speed as follows:

a. Walking speed is calibrated by marking out a 50 foot strip and walking it with a full sprayer and timing each trip with a stopwatch until the correct time for 50 feet is achieved. Time for 50 ft is calculated by the following formula:

$$\frac{a/12 \times 50 \text{ ft}}{43{,}560 \text{ ft}^2/\text{acre}} \; H \; b \; GPA \; H \; \frac{3785.3 \text{ ml/gal}}{c \text{ ml/sec}} = \text{time in sec for 50 ft}$$

a = effective band width in inches
b = desired volume per acre in gallons (normally 100)
c = volume of boom in ml/sec at desired pressure

b. Our four nozzle boom consists of four nozzles mounted 18 inches apart with a band width of 72 inches. With 8006 nozzles it delivers 151.4 ml/sec (four 8006 nozzles x 37.85 ml/sec). Thus:

$$\frac{72/12 \times 50 \text{ ft}}{43{,}560 \text{ ft}^2/\text{acre}} \; H \; 100 \; GPA \; H \; \frac{3785.3 \text{ ml/gal}}{151.40 \text{ ml/sec}} = \underline{\textbf{17.2 sec for 50 ft}}$$

This will deliver 2606.9 ml per 300 ft^2

c. Wider or narrower bands can be obtained by adding or deleting nozzles from a boom and recalibrating as above.

d. Speed in miles per hour is calculated as follows:

$$\frac{5{,}280 \text{ ft/mile}}{50 \text{ feet}} = 105.6 \times 17.2 \text{ sec} = 1816.32 \text{ sec/mile}$$

$$\frac{1816.32 \text{ sec/mile}}{3600 \text{ sec/hour}} = 0.504 \text{ hr/mile}$$

$$\frac{1}{0.504 \text{ hr/mile}} = 1.98 \text{ mile/hr}$$

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SOP: 6.3

TITLE: Cleanup of application equipment.

PURPOSE: To ensure that pesticide application equipment is clean to avoid contamination of succeeding trials.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. Apply any excess pesticide mix to an approved area.

2. Triple-rinse the spray tank in the field. Tank rinses should be in this order; water, soap + water, water. Boom should be sprayed through with soap + water, water, and finally remove all liquid from boom with air. Rinses after the triple rinse in the field are non-toxic and can be dumped anywhere.

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

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

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SOP 6.4

TITLE: Calibration of instruments and gauges.

PURPOSE: To assure that all instruments and gauges used in a GLP study are accurate and in good working order.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1.Each guage or instrument used in a GLP study (i.e. sprayer pressure gauge, temperature and humidity gauges, photometers etc.) should be tested annually prior to the start of any trials and determined that it is reasonably accurate. If the item is used continuously, it should be tested frequently enough to assure its continued accuracy (i.e. monthly, after every 10 hrs use etc.). If the item is used infrequently, it should be tested before it is first used in a GLP study and as often thereafter as necessary to assure its accuracy.

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

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

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

5. At the beginning of each season, the GPS unit will be tested against known geological markings and must be within the accuracy noted in the instruction manual (<10 meters).

6. Plot end reference points may also be collected using a handheld laser range finder to measure distance to permanent landmarks. This device must be calibrated yearly, prior to use, by testing the accuracy against a premeasured distance. The device must be accurate within one meter for every 50 meters distance measured. The Study Director must also update a maintenance log for the range finder on a yearly basis.

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SOP 6.5

TITLE: Remedial action in case of equipment failure.

PURPOSE: To ensure that a prescribed course of action is followed in case of equipment failure.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. Equipment is identified as all instruments, apparatus and implements used to complete measurement, application and assessment for IR-4 field trials. Equipment includes, but is not limited to, sprayers, applicators and nozzles, tanks and pressure regulators, temperature monitoring systems, weather monitoring equipment and meters, soil probes and samplers, freezers, balances used for weighing test substance and sample weights, graduated cylinders, tape measure and wheel.

2. In the case of equipment failure, malfunction or damage, equipment will be repaired, alternate equipment will be secured, or service personnel will be contacted to perform non-routine maintenance to return equipment to working order and complete trial requirements.

3. If a trial is not affected or impacted in any way, it does not have to be noted in the Field Data Book. If a residue trial is impacted by equipment failure, it should be noted in the Field Data Books of all affected trials. At a minimum, documentation of these actions should include the nature of the failure, date of first notice, remedial actions taken and an assessment of potential impacts on trial.

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SOP 7.1

TITLE: Test chemical request, receipt, and disposal.

PURPOSE: To ensure that chemicals used in residue tests are properly received, logged, stored, and disposed of upon completion of the trial.

SCOPE: All test chemicals used in IR-4 field research trials.

PROCEDURES:

Test Substance Request and Receipt

1. Upon receiving study protocols for the current year, the Study Director will contact the manufacturers of the required pesticides to obtain characterized samples of the pesticides of sufficient quantity to complete the trial. If the pesticides were not ordered by the Study Director the Field Research Director will contact the manufacturer to obtain characterized samples.

2. Upon receipt of test chemicals, record the information required by the protocol or sponsor. Place shipping documents, information from the container label or product documentation in the respective IR-4 Field Data Books. If no directions are given in the protocol or by the sponsor, record the following:
    a. Date received and person who received the shipment.
    b. Test substance name and formulation.
    c. Lot/batch number.
    d. Expiration date.
    e. Number of containers and their condition on arrival.
    f. Amount received (listed on container label), and gross weight of container and sample.
    g. Evidence of GLP characterization.
    h. Storage location and date placed in storage unit.
    i. Courier and bill of lading or tracking number.

3. Label test substance container with Field ID #, date received, CAS or code number, batch or lot number, expiration date, storage requirement conditions and initials. If there is more than one container for a single trial or more than one location using the same test substance, each container should be labeled with a unique number such as 1 of X, 2 of X, etc. An additional label may be attached to container to document this information.

4. Contact Study Director immediately if upon receipt of test substance any registrant documentation, information (name, CAS or code number, batch/lot number, expiration date and storage conditions) is missing.

5. If the test material container is damaged or leaking, the sponsor should be notified and a new shipment should be arranged if necessary. Appropriate steps should be taken to ensure that the leak has been contained and properly cleaned up. Dispose of waste and contaminated materials in a legal safe manner.

6. All arriving test substances should be placed in a locked box labeled "GLP Only" in the chemical storage closet located at the Plant Pathology Farm on North College Road. If the test substance is being stored at a facility closer to the field plots, temperature monitoring and 'GLP Only" labeling must continue in the same manner as the chemical storage cabinet at the Plant Pathology Farm. Monitor temperature during transfer of the test substance to the offsite storage facility.

Submitted by Field Research Director: Mary K. Hausbeck MKH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

Test Substance Disposal

1. Test chemical substance containers or empty containers must be stored until instructed to dispose of them by the Study Director.

2. Upon completion of a trial, extra test chemical may remain in the possession of the Field Research Director, or disposed of in a legal manner; e.g., crop maintenance. Material also can be returned to the registrant, sponsor, or other designated entity. Record all use or transfer in the IR-4 Field Data Book. Retain all test substance disposition shipping information as raw data.

3. When documenting test substance/container disposition, include the following information:
    a. Study or trial number
    b. Address of recipient
    c. Date shipped
    d. Courier name, bill of lading or tracking number
    e. Person shipping material

4. When shipping package substance containers to meet DOT shipping requirements. Include a copy of the test substance MSDS with the shipment. Secure bottle and jar lids with tape. If needed, include adequate absorbent in the package in case of a spill. If transferring the test substance with a university owned vehicle, follow all DOT requirements and monitor temperatures during transfer.

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SOP 7.2

TITLE: Test chemical storage, use and handling, and records.

PURPOSE: To describe procedures related to storage and handling test chemicals and documenting all uses.

SCOPE: All test chemicals used in IR-4 field research trials.

PROCEDURES:

1. All personnel involved in the handling or transportation of test chemicals should be properly trained in their use and wear appropriate protective equipment as described on the chemical label or MSDS when handling chemicals. An emergency contact list, with phone numbers and addresses, should be readily available where pesticides are stored, handled, and transported. Inspect personal protective equipment before each use for leaks or damage. All personal equipment should be cleaned or disposed after each use.

2. First aid kits and eye flush kits should be available and accessible in work vehicles and at work sites.

3. All test chemical applications must be recorded in the facility Pesticide Log Book or the respective IR-4 Field Data Book. Record the information required by the protocol or sponsor. If no directions are given, record the following information:
    a. Name of test substance
    b. Number of containers removed
    b. Date of removal and initials of person responsible for removal
    c. Lot/batch number
    d. Purpose of removal
    e. Amount used
    f. Date test material container returned to storage unit and initials of person returning container

4. Test materials may be pre-weighed or measured and placed in a suitable mixing container (spray bottle or ziplock bag) at the storage facility and transported to the trial site in a weather resistant container. If placed in spray bottle, the bottle should contain half the amount of fluid needed for a complete application mix. Transportation container should be kept outside the passenger compartment of any vehicle and protected from direct sunlight.

5. Test chemicals will be stored in a secured facility in a manner to ensure the chemicals integrity: see SOP 7.3.

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SOP 7.3

TITLE: Test chemical storage and monitoring

PURPOSE: To describe acceptable storage facilities and procedures for storage of test chemicals.

SCOPE: Storage facilities for test chemicals used in IR-4 field research trials.

PROCEDURES:

1. The chemical storage facility should have limited access and a sign on the door indicating the hazardous nature of the contents. The area should be secure, dry, and well ventilated with adequate fire protection and should be separate from equipment and sample storage areas, offices and laboratories. When the test substance is being stored at a facility without a separate chemical storage area, it may be kept with the application equipment as long as the test substance is in a locked container separate from the equipment and the cabinet is marked as containing pesticides or hazardous materials.

2. All test chemicals should be stored in a locked cabinet or container. Container should be of sufficient size to contain any possible spills of test chemicals. Monitor storage temperature with a data logger (primary device), or a min/max thermometer (backup system) and monitor temperatures for as long as these chemicals are used in active trials. Keep pesticides from freezing. Check logger or min/max thermometer monthly and recorded checks on a log. Maintain storage temperature records in a permanent file. If the temperature of the test substance storage exceeds the stability range for any stored test substance, notify the Study Director or other designated contact immediately.

3. Have a spill kit and personal protection equipment accessible (minimal of nitrile gloves) in the pesticide storage facility. Personnel involved in handling test substances should be familiar with the principles of containing spills and decontamination.

4. Test substances used in residue trials will be kept separate from other pesticides.

5. Test substance containers shall be retained until the final study report is completed and permission for disposition is given by the Study Director.

6. Test substances should be kept in their original, labeled containers and checked regularly for corrosion and leaks. Damaged containers should be replaced. If this is not possible, the contents may be transferred to a suitable container. Label the container with the test substance name, batch/lot number, CAS or code number, storage conditions requirements, and expiration date. If necessary, the container and its contents may be disposed of properly.

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SOP 7.4

TITLE: Calculating and measuring liquid pesticides.

PURPOSE: To ensure and accurate dosage of liquid pesticides in the IR-4 field research trial.

SCOPE: All IR-4 field trials using liquid formulations of pesticides.

PROCEDURES:

1. Apply all applications as directed by the protocol. Mix a minimum of 1 gallon of spray mix for each treatment.

2. Calculate the amount of liquid pesticide needed per volume of spray mix by the following formula:

$$\frac{\text{rate lbs ai/acre}}{\text{lbs ai/gal}} \times \frac{3785.3 \text{ ml/gal.}}{\text{gal. spray volume/acre}} = \text{ml pesticide/gal. of spray mix}$$

ml. pesticide/gal. of spray mix x gallons needed = total ml. of pesticide required.

3. Before removing any test substance from the container, vigorously shake the container in case product has settled.

4. Use a clean plastic syringe to remove the test substance from the container. For all liquid test substances, use a syringe with a minimum measurable amount of 0.1 ml.

5. Eject the test substance from the syringe into a flask with a minimum 200 ml of water and poor the mixture into the spray tank. Triple rinse the flask with all of the rinsate being pored into the spray tank.

6. Record in the Test Substance Records Use Log of the IR-4 Field Data Book the amount of pesticide removed from the container, the purpose, initial and date.

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SOP 7.5

TITLE: Calculating and measuring dry pesticides.

PURPOSE: To ensure an accurate dosage in the application of dry pesticides in IR-4 field research.

SCOPE: All IR-4 field trials using dry formulations of pesticides.

PROCEDURES:

1. Apply all applications as directed by the protocol. Mix a minimum of 1 gallon of spray mix for each treatment.

2. Calculate the amount of dry pesticide needed per volume of spray mix by the following formula:

$$\frac{\text{rate lbs ai/acre}}{\text{\% ai (as a decimal)}} \times \frac{453.6 \text{ g/lb}}{\text{gal. of spray volume/acre}} = \text{grams of pesticide/gal of spray mix}$$

grams of pesticide/gal. of spray mix x gallons needed = total grams of pesticide required.

3. Calibrate the balance according to SOP 7.6.

4. Place a clean container on the balance and tare it. Transfer the dry pesticide from its original container to the clean transfer container with a clean spatula, spoon, or other appropriate device. Measure the pesticide as accurately as possible to the nearest 0.01 gram. When the desired amount of pesticide has been transferred, seal both containers.

5. Mark the transfer container with the name of the chemical, trial ID# that chemical will be applied to, and amount.

6. Wash transfer devices and reusable containers with soap and water after each use.

7. Record in the Test Substance Records Use Log of the IR-4 Field Data Book the amount of pesticide removed from the container, the purpose, initial and date.

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SOP 7.6

TITLE: Calibration of an electronic balance for dry pesticide measurement.

PURPOSE: To ensure an accurate dosage in the application of dry pesticides.

SCOPE: IR-4 trials where a dry material is weighed for use in field research.

PROCEDURES:

1. At a minimum of every two years, test weights will be weighed on a certified scale and then used within 90-days to calibrate scale. If the scale is moved, repeat instructions in the previously mentioned sentence.

2. Prior to use visually inspect electronic balance for cleanliness. Wipe with clean moist cloth as needed.

3. Prior to weighing the pesticide for use in a trial, calibrate the balance by first establishing zero followed by weighing standard weights that bracket the amount of test substance needed. Record in the Field Data Book the date, standard weights used and recorded weights and initials of person performing this task. If the balance does not weigh within one percent of the standard weight, service will be called and another balance used. Tare balance with weighing receptacle in place and weigh test substance. Record amount of test substance removed in Field Data Book.

4. The Field Research Director is responsible for inspection, maintenance and calibration of the electronic balance.

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SOP 7.7

TITLE: Adding the pesticides concentrate to the water carrier in the spray tank of a sprayer.

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

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. Deionized or distilled water will be used as a carrier for all applications.

2. Measure the amount of the water needed to dilute the measure amount of concentrate into a separate container. Make sure the spray mix will be enough to cover at least one treatment plot (preferably all plots in one treatment). Make sure the spray tank will hold this amount and the concentrate. If the tank cannot hold the calculated spray mix, split the application into two tanks. Add the water to the spray tank.

3. If needed (i.e. wettable powder formulation) make slurry mix first by adding the concentrate to a small volume of water in a separate, reasonably clean container. Add the pesticide concentrate or a slurry to the water in the spray tank. Triple rinse the container holding the pesticide concentrate (and slurry) with the appropriate water and add the wash to the spray tank.

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

5. Agitate the spray mix before application to insure an even mix of the pesticide and water.

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SOP 7.8

TITLE: Application of pesticides in the field.

PURPOSE: To apply pesticides accurately in the field to meet requirements for GLP.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. Apply the material beginning with the lowest concentration and continuing with higher concentrations up to the highest concentration if more than one trial is located at the same field site.
2. Proceed at the correct speed toward the plot and turn on the sprayer as you enter the plot. Maintain the correct speed throughout the plot.
3. Turn off the sprayer at the end of the plot.
4. Record pass times in the Field Data Book.
5. Spray out excess pesticide mix on an approved location, preferably near the experimental area.
6. Record weather data at time of application, including air temperature, soil temperature, wind direction and velocity, relative humidity, sky conditions, soil surface moisture, and plant surface moisture. Keep a record of all precipitation, irrigation, and air temperatures for the duration of the trial.

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SOP 7.9

TITLE: Procedures for problems during application of test chemicals.

PURPOSE: To explain the procedures to handle problems that may occur during the application of the test chemical that could effect the integrity of the trial.

SCOPE: All IR-4 field research trials..

PROCEDURES:

1. The applicator should observe all aspects of the application to make sure that the test chemical is distributed accurately.

2. If there is a problem, stop the application and mark the spot the problem first occurred. The operator should take immediate action to correct the situation. If the problem can be remedied (clearing of a clog nozzle, etc) in the field the application should continue starting at the flagged area.

3. If a portion of the plot has been compromised, clearly mark off affected area. This portion should not be used for obtaining residue samples. If the unaffected area is too small to obtain the samples required for analysis, the trial should be discontinued.

4. The Regional Field Coordinator and the Study Director should be notified by phone, fax or e-mail and in writing of the incident and details should be recorded in the IR-4 Field Data Book.

Department of Plant, Soil, and Microbial Sciences
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East Lansing, MI 48824-1311

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Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP 7.10

TITLE: Storing and maintaining adjuvants for use in field studies.

PURPOSE: Define the expectations for maintaining spray additives, along with appropriate supporting data and documentation as part of IR-4 GLP residue studies.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. The use of adjuvants in a test study must be approved in the protocol.

2. Adjuvants are considered to be reagents and not a test substance.

3. Although GLP characterization is not expected, the following must be documented:

   a.) The purchase date of the adjuvant.
   b.) The recommended storage conditions.
   c.) Identity and concentration of the adjuvant.
   d.) The expiration date from label or assigned by field personnel (<5 years from purchase date).
   e.) Record the date the adjuvant is initially opened after purchasing.

4. Whenever possible, purchase new adjuvants each year.

5. Secondary containers can be used but should be avoided. Secondary containers should be fully labeled with test substance name, active ingredients, storage requirements, and expiration date.

6. Spray adjuvants will be stored in a location that has limited access and is temperature monitored.

7. To avoid contamination, do not place a measuring device (syringe, etc.) back into the adjuvant container after being placed into the test substance solution.

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SOP 8.1

TITLE: Safe handling of pesticides and use of safety equipment.

PURPOSE: To ensure that pesticides are handled safely and that personnel are protected by adequate safety equipment.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. All personnel involved in field pesticide experiments will be properly trained in the safe use of spray equipment and pesticides.
2. When applying or mixing pesticides wear protective equipment as described and required on the pesticide label or the MSDS. This equipment may include rubber gloves, boots, coverall, goggles, hat, and respirator.
3. Do not apply pesticides when winds exceed 10 MPH. Apply pesticides with the wind to your back or side whenever possible.
4. Always have clean water available while handling pesticides to wash off pesticides that contact exposed skin or eyes.
5. Upon completion of the application wash all exposed skin with soap and water.
6. Clean safety equipment and let it dry before placing it into storage.
7. Clothing worn during pesticide application should be washed at the end of the day.
8. Whenever a fumigant is present, a respirator must be worn by all field personal for the duration of the calibration, application, and cleaning processes.

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SOP 9.1

TITLE: Recording of raw data.

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

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. All raw data will be recorded directly and promptly in an IR-4 Field Data Book in indelible ink. All entries must be dated and initialed.

2. Corrections will be made by crossing through the item with one line, initialing and dating it, and writing a short explanation for the change. Error codes in the IR-4 Field Data Book should be used whenever possible to explain the change.

3. Pages containing data should not be removed from the Field Data Book. Copies of blank forms may be added to the book as needed. Verified copies of data from other trials may be added following requirements on the form.

4. Give the location of the original data if true copies are included in the Field Data Book.

5. Make sure that all data required by the study protocol or by the forms provided is collected and recorded. Carefully review the forms provided with the protocol to make sure that all the required data is being collected.

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SOP 9.2

TITLE: Collection of pesticide data, weather data, and other information.

PURPOSE: To obtain accurate and complete data for pesticide registration.

SCOPE: All IR-4 field trials for pesticide registration.

PROCEDURES:

1. Collect and record all data per protocol/SOPs requirements.

2. Whenever possible, data collected or transcribed from other farm operations should be verified for accuracy.

3. NOAA Weather data may be cited/used in the reports.

4. Pesticide data, weather data, and other information that are not part of the study may be exempt from the GLP requirements.

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SOP 9.3

TITLE: Collecting phytotoxicity data.

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

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. Consult the protocol to determine the method and timing of the phytotoxicity data. If no method is cited proceed as directed in this SOP.

2. Rate all the crop plants in the treatment at one time. Record phytotoxicity on a 1-10 scale: 1 = no injury; 10 = complete kill.

3. Rate phytotoxicity upon first observation. Take pictures of phytotoxicty symptoms and send to Study Director immediately.

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SOP 9.4

TITLE: Determining yield and quality.

PURPOSE: To accurately assess yield and quality, if required by the protocol.

SCOPE: All IR-4 trials in which the protocol requires or encourages yield data.

PROCEDURES:

1. Check the protocol for information on time of harvest. Note the number of days between treatment and harvest (Pre harvest interval - PHI). If none, follow commercial practices in the area for the time of harvest of the commodity.

2. Harvest a sufficient area of the plot to give a good estimation of the effects of the treatments. Harvest by hand or machine using accepted harvest procedures for the commodity. Weigh and record the yield.

3. If quality grading is required by the protocol, grade according to USDA or other accepted grading standards and record weight of each portion. Record the grading standard.

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SOP 9.5

TITLE: Collection and recording of data from monitoring devices.

PURPOSE: To describe methods for handling data from temperature and weather collection and recording devices.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. All automatic data collecting and/or recording devices (except the Automated Weather Station, scale used to measure sample weight, and the freezer) should be inspected and calibrated as described in SOP 9.6.

2. Printouts or charts from these devices must be legible to persons with normal vision. The original print-outs of the downloaded data from a device is the original document. A printout of a data file is an original document and copies of these documents must be marked as copied.

3. Hard copies of computerized data and/or other written, typed, or plotted data sheets must be retained in the file folder of the trial or if applicable to more than one trial, in a separate file in the Field Research Director's archives.

4. Each chart or data sheet from a monitoring device should be marked in ink with location, dates (month, day, year) of the period measured, and initialed by the individual who removes the chart from the device. Units should be indicated on the charts. Charts will be changed weekly.

5. Automated weather monitoring equipment is preferred when available. Hard copies of weather data from automated monitoring equipment should be included in trial reports.

6. If automated equipment is not available, weather data should be recorded manually in an experiment station log book and maintained on file at the station. The data can be transferred from there to the final report.

7. The Field Research Director is responsible for inspection, maintenance, and calibration of this equipment.

8. Since the Field Research Director does not control weather monitoring devices, weather data may not meet requirements for GLP.

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SOP 9.6

TITLE: Calibration of temperature monitoring devices.

PURPOSE: To assure that temperature monitoring devices under the control of the Field Research Director are reasonably accurate and in good working order.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. All temperature monitoring devices (not including the Automated Weather Station) under the Field Research Director's control used to gather data in field or storage should be periodically tested to determine that they are reasonably accurate. Temperature monitoring devices should be tested annually by comparison to a high quality thermometer.

2. A written record should be kept of the dates and results of the tests.

3. Temperature monitoring devices that give inconsistent results or are not accurate to within ten percent should be repaired or replaced.

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SOP 9.7

TITLE: Data storage during a field trial.

PURPOSE: To assure that data collected from a field trial is maintained securely until transferred to the Field Research Coordinator.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. The Study Director has the responsibility to store all raw data produced in IR-4 field trials. The Study Director will maintain permanent archives.

2. The Field Research Director will maintain in a secure location all raw data under his/her control during the field phase of a trial. He/she will return the IR-4 Field Data Books containing raw data and other information to the Regional Field Coordinator for transmission to Study Director as soon as possible after completion of the trial.

3. The Field Research Director should maintain a file with a copy of all information related to and all data generated from a trial, including all information in the IR-4 Field Data Book.

4. A hard copy of electronic data and charts from monitoring devices should be placed in the file as soon as possible after the information is generated. The printout of data from monitoring devices is considered an original document from which exact copies may be made.

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

6. IR-4 Field Data Books will be placed in permanent archives at IR-4 Headquarters upon completion of a trial.

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SOP 9.8

TITLE: Equipment not requiring SOPs.

PURPOSE: To identify what type of equipment will not require an SOP.

SCOPE: Equipment used in IR-4 Research Trials.

PROCEDURES:

1.The equipment used in trials that require a SOP are:
- a. Application Equipment (i.e. boom sprayers).
- b. Electronic Balance For Measuring Test Substance.
- c. Automated Data Collection/Recording Devices (except the weather station), SOP 9.6.

2. All other equipment used to conduct an IR-4 field trial will not require a SOP.

3. As new equipment is acquired, the Field Research Director will determine whether it requires a SOP.

4. In certain trials, the use of equipment not owned by the lab can be used to complete the requirements of the protocol. In particular, specialty dryers are used on ginseng samples in accordance with standard industry. If using these dryers, record the temperatures with the labs tested GLP monitoring equipment. If small dryers are used, place the untreated and treated samples in separate dryers. Always clean off the dryer racks/trays prior to use by wiping off all ginseng residue with a clean wet cloth. A sign should be placed on the outside of the dryer and/or on the drying rack stating that a GLP IR-4 trial is in progress and that samples should not be moved or touched. The labs/FRDs contact information should be placed on the sign.

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SOP 10.2

TITLE: Collecting residue samples.

PURPOSE: To assure that representative residue samples are collected and that their integrity is maintained.

SCOPE: All IR-4 field trials from which residue samples are required.

PROCEDURES:

1. At the beginning of each growing season, the Field Research Director will prepare a Master Timetable for all projects. The Master Timetable will include projected planting, treatment, and sample collection dates. The Master Timetable will also indicate the type of trial: residue (R) or phytotoxicity and efficacy (P-E). Depending on the protocol, samples may be required at a specific number of days after treatment, at the earliest commercial harvest, or at some other predetermined time. Sample collection dates may be adjusted as the season progresses, and the projected dates modified on the Master Timetable. The Quality Assurance Officer (QAO) will be kept informed of changes in projected sampling dates.

2. Avoid collecting samples during rain, unless it is necessary to do so to meet a time requirement in the protocol (e.g., specified days after treatment).

3. Collect samples from untreated plots first, then proceed to the lowest and sequentially higher dosages. Collect all samples from each subplot of a treatment before proceeding to the next treatment.

4. Consult the protocol to determine sample size and sampling method. If no instructions are given, collect 2 to 4 lb of good quality product from each subplot.

5. If 2 lb of product is not available collect as much as possible, and describe in the IR-4 Field Data Book any reasons for the small sample size.

6. Do not wash or clean sample product unless directed to do so by the protocol.

7. Avoid cross-contamination of samples during harvesting and handling by wearing clean gloves and changing the gloves between samples.

8. Post harvest interval (PHI), prompted in Part 7A of the Field Data Book, is calculated by counting the day after the last application up to and including the day of harvest.

9. After harvest, treated rows of annual crops will be mowed, disked, or otherwise destroyed. Date of this action will be noted in the Field Data Book.

10. Treated rows or trees of perennial crops will be marked and signs will indicate that fruit should not be eaten.

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SOP 10.3

TITLE: Sample containers and identification.

PURPOSE: To specify how samples are to be identified.

SCOPE: All IR-4 field trials requiring residue samples.

PROCEDURES:

1. Prior to sample collection, obtain from the IR-4 Regional Field Coordinator a sufficient number of IR-4 plastic lined cloth bags for the trial. If these bags are not available use any clean sampling bag suitable to protect the integrity of the samples. Double plastic bags are usually sufficient. Plastic, food type bags (supplied by the Field Research Director) may be used for sample collection. These are placed inside of cloth bags after sampling is completed.

2. Before sampling, label each IR-4 plastic lined cloth bag with waterproof ink with the following information:
   a. Field trial identification number
   b. Commodity (crop)
   c. Chemical (common name) and application rate (lb ai/a)
   d. Sample number
   e. Sampling date
   f. Investigator: Name/Address/Phone

3. Each plastic, food type bag should be labeled with the following information:
   a. Field trial identification number
   b. Commodity (crop)
   c. Chemical (common name) and application rate (lb ai/a)
   d. Sample number
   e. Sampling date

4. Place the plastic bag with the harvested sample in the IR-4 plastic lined cloth bag and tie the bag closed securely.

5. Upon completion of the sampling, fill out the Residue Sample Shipping sheet (Form 8B) from the IR-4 Field Data Book.

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SOP 10.4

TITLE: Sample packing and storage.

PURPOSE: To assure the integrity of the samples after collection in the field.

SCOPE: All IR-4 field research trials where residue samples are collected.

PROCEDURES:

1. Place the sample as it is collected into the sample bag marked for that sample.

2. Close the sample bag and expel excess air. Tie the bag securely closed, to ensure that treated and untreated samples are kept separate.

3. Place filled sample bags into a clean shipping container. Keep the samples cool with ice or shade if time between sample collection and placing time in cooler containing dry ice will exceed 2 hours. If samples will be analyzed at the Michigan State University Residue Laboratory, deliver the samples directly there from the field.

4. Currently, our lab does not have a freezer for sample storage. For all samples collected, immediately place the sample bags in cooler containing dry ice for shipping to the testing lab. Place enough dry ice in the cooler to keep the samples frozen for a minimum of 24 hrs. Always ship the samples next day air morning delivery.

5. Treated and untreated samples should always be shipped in separate coolers.

6. For ginseng samples, collect the roots directly from the drying racks and place into the labeled IR-4 sample bags. After filling the sample bags, place the closed bags directly into the shipping coolers containing dry ice.

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SOP 10.5

TITLE: Sample shipping procedures.

PURPOSE: To assure that residue samples are shipped to the residue laboratory with no loss of integrity.

SCOPE: All residue samples from IR-4 field trials.

PROCEDURES:

1. Ship samples by ACDS freezer truck or by air freight. If shipping by freezer truck, contact ACDS concerning the next opportunity for sample pickup. ACDS will then contact the Field Research Director when a truck will be here. When the truck arrives, deliver the samples directly to the truck. If it is not possible to deliver the sample directly to the truck, deliver the samples to the MSU IR-4 Pesticide Residue Freezer and the laboratory personnel will deliver the samples to the truck.

2. If shipping with next day delivery. Ship air freight on Monday through Wednesday to avoid weekend arrivals. If possible, schedule for AM delivery.

3. Place a copy of the completed IR-4 Residue Sample Chain of Custody Form into each shipping container.

4. Pack the boxes securely so that they can withstand stacking in the truck. Tape the lids shut. No dry ice is needed for shipping by freezer truck. Add dry ice if shipping by air freight in a 3:1 ratio to sample weight (3# dry ice to 1# sample).

5. Label each shipping carton with the following information:
   a. Name and address of the sender
   b. Name, address, and phone no. of the residue lab receiving the samples.
   c. Number of the container if more than one is used (e.g., 1 of 2, 2 of 2).
   d. Where used, affix "Dry Ice" on two sides of the container.
   e. Field ID number.
   f. Commodity and pesticide.

6. After samples have been shipped, send a copy of the Residue Sample Chain of Custody Form to the Regional Field Coordinator and Study Director.

7. Contact lab personal prior to the shipping of samples as per protocol.

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Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 9-7-16

SOP 10.6

TITLE: Sample Drying Procedures.

PURPOSE: To assure that samples are dried correctly and in accordance to industry standard and dryers are maintained to produce consistent results.

SCOPE: All residue samples from IR-4 field trials that require drying as per protocol.

PROCEDURES:

1. Each dryer will have a unique identifier number that will be recorded in the Equipment Log Book. When possible, serial numbers will be recorded in the equipment log.

2. Each year, for a particular test substance, dryers will be assigned only treated or untreated samples. Dryers will be labeled at the beginning of each season as either 'treated' or 'untreated'.

3. Temperatures will be monitored and recorded in a minimum of one treated and one untreated dryer.

4. To keep samples from falling through the racks inside the dryers, aluminum foil will be placed over each rack. The aluminum foil will be removed and replaced after each use.

5. All samples in the dryers will be labeled with the study #, sample ID, harvest date and date placed into the dryer.

6. At the beginning of each season, prior to use, each dryer will be tested for a minimum of 12 hours.

7. Any dryer that produces inconsistent temperatures will be fixed or replaced.

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SOP 11.1

TITLE: Trial reports.

PURPOSE: To describe the format for reporting trial activities and results.

SCOPE: All IR-4 field research trials.

PROCEDURES:

1. All data and information from a trial will be included in the IR-4 Field Data Book, which will be returned to the Regional Field Coordinator by the Field Research Director upon completion of the field portion of the trial.

2. Additional forms, data, or information that may be needed to comply with standard operating procedures and GLP include:

   a. Organizational chart
   b. Current vita and position descriptions
   c. Chemical min/max storage recorder.
   d. Balance/equipment calibration form
   e. Balance weights verification
   f. Master timetable
   g. Freezer/equipment form
   h. Freezer inventory log
   i. Study deviation forms
   j. Protocol change forms
   k. Correspondence concerning the trial or project
   l. Sprayer/equipment calibration form
   m. Global positioning system (GPS) report
   n. Soil analysis report
   o. Weather records
   p. Maps

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SOP 11.2

TITLE: Completion of the IR-4 Field Data Book.

PURPOSE: To explain the use of report forms contained in the IR-4 Field Data Book.

SCOPE: All IR-4 field research trials..

PROCEDURES:

1. All forms should be filled out legibly in indelible ink or typewritten. Mistakes should be crossed out with one line, initialed, and dated. Include the reason for the change. Use error codes where appropriate.

2. Use forms provided in the IR-4 Field Data Book. Blank forms may be photocopied as needed.

3. The appropriate forms should be filled out as completely as possible within a reasonable period of time after the information is available. Data should be recorded directly into the IR-4 Field Data Book.

4. If a form or section of a form does not require a response, draw a diagonal line from the top of the page or section to the bottom. Initial and date on the diagonal line and sign and date at the bottom of the page. If the requested data are not applicable, give an explanation.

5. Paginate each form within each section of the IR-4 Field Data Book.

6. If raw data applies to more than one trial (e.g., weather data, field history, or personnel information), the original will be kept in temporary storage by the Field Research Director until the end of the field season. Copies placed in the Field Data Books will be marked as a "True Copy of the Original", signed and dated, and the location of the original noted. The original may also reside in the permanent archives/record storage facility and "True Copies" signed, dated and indicating this location may be used in the Field Data Book. At the end of the field season the original documents will be transferred to Headquarters to the archives.

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SOP 11.3

TITLE: Disposition of IR-4 Field Data Books and other documentation.

PURPOSE: To assure that completed IR-4 Field Data Books reach the Study Director as soon as possible.

SCOPE: All IR-4 field research trials..

PROCEDURES:

1. The original of the completed IR-4 Field Data Books and any accompanying documentation should be forwarded to the IR-4 Field Research Coordinator within a reasonable period of time after the trial is completed.

2. The IR-4 Regional Field Coordinator will review the IR-4 Field Data Books for completeness and accuracy. Incomplete IR-4 Field Data Books will be returned to the Field Research Director with comments on deficiencies and suggestions for completion. The IR-4 Regional Coordinator may elect to amend the Field Data Book upon contacting the Field Research Director via e-mail, fax, or telephone.

3. The Field Research Director will place a copy of the completed reports and related correspondence in his trial folder.

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SOP 12.1

TITLE: Quality Assurance.

PURPOSE: To meet IR-4 requirements for Quality Assurance.

SCOPE: All IR-4 field trials.

PROCEDURES:

1. A Quality Assurance Unit (QAU) will monitor research trials to assure that good scientific methods are being employed and that the Field Research Director is complying with his SOP and with study protocols. Quality Assurance Unit SOP's are maintained by IR-4 Headquarters and all QAU activities are coordinated by the IR-4 QAU.

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SOP 13.1

TITLE: Procedures to follow prior to an EPA inspection.

PURPOSE: To provide guidance to MSU personnel in responding to a request for an EPA audit, inspection or review.

SCOPE: All IR-4 research trials in the Department of Horticulture at Michigan State University.

PROCEDURES:

1. Notify the Study Director and Regional Field Coordinator of the pending audit or review as soon as possible.

2. Have available as much as possible all personnel directly involved in IR-4 trials in Plant Pathology, MSU.

3. Make sure that someone who is authorized to accept the Notice of Inspection will be present at the start and finish of the inspection.

4. Prepare trial and/or facility personnel for the inspection.

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

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

c. Instruct personnel to respond specifically to the questions asked and not to provide extraneous information. Do not provide any information unless asked.

d. Be certain that all documents relevant to the trial and facilities are available, including:

1.) Master schedule for the field research director.

2.) Study Protocol and current and historical Standard Operating Procedures.

3.) Raw data, correspondence and logs.

4.) Training records and CVs of personnel involved in the audited trial.

5.) Documentation of test substance characterization, receipt and handling.

6.) Maintenance/Calibration logs on equipment.

5. Have available the organizational charts and a map of the facility.

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Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-29-16

SOP 13.2

TITLE: Procedures to follow during an EPA inspection.

PURPOSE: To provide guidance to trial personnel in responding to a request for an EPA audit/inspection or review.

SCOPE: All IR-4 research trials in the Department of Horticulture at Michigan State University.

PROCEDURES:

1. Greet the inspection team and follow any institutional procedures for signing in. Escort the entire group to the meeting room.

2. At the opening of the conference ask the lead inspector for his/her credentials and for any opening statements.

3. Introduce the facility personnel present and state their function in the facility or trial. Identify the person who will accept the Notice of Inspection.

4. Ask the lead inspector for his/her agenda for the inspection.

5. Proceed with the inspection.

   a. Provide documents requested and provide explanations as needed.

   b. Keep notes of observations and of all interviews.

   c. Keep IR-4 management informed of the progress of the inspection and the findings.

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Submitted by Field Research Director: Mary Hausbeck MH Date: 4-15-16

Approved by Regional Field Coordinator: Satoru Miyazaki SM Date: 4-27-16

SOP 13.3

TITLE: Procedures to follow after the EPA inspection.

PURPOSE: To provide guidance to trial personnel in responding to a request for an EPA audit/inspection or review.

SCOPE: All IR-4 research trials in the Department of Horticulture at Michigan State University.

PROCEDURES:

1. The Field Research Director or designated representative must be present for the close-out conference.

2. If the inspector's comments are in error, call this to the inspector's attention.

3. If you have corrected any problems during the inspection make sure that the corrections are so noted in the inspector's logbook.

4. Have someone take accurate notes or record the close-out conference on tape if taping is acceptable to the inspectors.

5. Be sure you obtain copies of documents or other materials that may be taken as exhibits by the inspectors.

6. Inform site personnel, Regional Field Coordinator, and the Study Director(s) of any problems found.

7. Respond to deficiencies as required.

8. Keep Study Director(s) informed of any activity related to the inspection.