SOP Log

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FRD/LRD: Shawn May
Submitter

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S: Facility Files- SOP Cover Sheet

7/31/14 JT

MICHIGAN STATE
UNIVERSITY

TO: Dr. Sharon A. Clay
Plant Science Department
South Dakota State University (SDSU)
Box 2207a
Room 247 Biostress Lab
Brookings, SD 57007

FROM: Satoru Miyazaki, IR-4 Regional Field Coordinator

SUBJECT: STANDARD OPERATING PROCEDURE (3.1) APPROVAL

DATE: April 27, 2015 (Effective Date)

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

SOP	Rev. #	Last Revision Date	SOP	Rev. #	Last Revision Date	SOP	Rev. #	Last Revision Date
1.1	3.0	3-27-14	5.4	3.0	3-27-14	9.1	3.0	3-27-14
1.2	3.0	3-27-14	6.1	3.0	3-27-14	9.2	3.0	3-27-14
1.3	3.0	3-27-14	6.2	3.0	3-27-14	9.3	3.0	3-27-14
1.4	3.0	3-27-14	6.3	3.0	3-27-14	9.4	3.0	3-27-14
2.1	3.0	3-27-14	6.4	3.0	3-27-14	10.1	3.0	3-27-14
2.2	3.0	3-27-14	6.5	3.0	3-27-14	10.2	3.0	3-27-14
2.3	3.0	3-27-14	6.6	3.0	3-27-14	10.3	3.0	3-27-14
3.1	3.1	3-25-15	6.7	3.0	3-27-14	11.1	retired	3-27-14
3.2	3.1	3-25-15	7.1	3.0	3-27-14	11.2	retired	3-27-14
3.3	3.1	3-25-15	7.2	3.0	3-27-14	11.3	retired	3-27-14
3.4	3.0	3-27-14	7.3	3.0	3-27-14	11.4	retired	3-27-14
3.5	3.0	3-27-14	7.4	3.0	3-27-14	11.5	retired	3-27-14
4.1	3.1	3-25-15	7.5	3.0	3-27-14	11.6	retired	3-27-14
4.2	3.0	3-27-14	7.6	3.0	3-27-14	12.1	3.0	3-27-14
4.3	3.0	3-27-14	7.7	3.0	3-27-14	12.2	3.0	3-27-14
4.4	3.0	3-27-14	8.1	3.0	3-27-14	13.1	3.0	3-27-14
4.5	3.0	3-27-14	8.2	3.0	3-27-14	13.2	3.0	3-27-14
4.6	3.1	3-25-15	8.3	3.0	3-27-14	14.1	3.0	3-27-14
5.1	3.0	3-27-14	8.4	3.0	3-27-14	14.2	3.0	3-27-14
5.2	3.0	3-27-14	8.5	3.1	3-25-15	14.3	3.0	3-27-14
5.3	3.0	3-27-14	8.6	3.1	3-25-15			

IR-4 NORTH CENTRAL REGION RESEARCH CENTER
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STANDARD OPERATING PROCEDURES

FOR

MAGNITUDE OF THE RESIDUE-FIELD STUDIES

Revision 3.1
March 25, 2015

Weed Stress / Environmental Herbicide Fate Laboratory
South Dakota State University
Brookings, SD 57007

Dr. Sharon A. Clay SD Field Research Director	Sharon A. Clay (Signature)	SAC (Initials)	3/30/15 (Date)
Dr. S. Miyazaki Regional Field Research Coordinator	Satoru Miyazaki (Signature)	SM (Initials)	4/27/15 (Date)

Contents- Standard Operating Procedures

Weed Stress/Environmental Herbicide Fate Laboratory
South Dakota State University
Brookings, SD 57007

Revision Number: 3.0

Submitted by: Kalyn Brix-Davis — Submitted Date: May 1, 1993
Revised by: Graig Reicks — Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director Sam 3/27/14
S. Miyazaki, Regional Field Research Coordinator Sam 4/8/14

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

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

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

Procedures: 1 Each facility where studies are conducted in the support of the registration of pesticides will develop an SOP for each phase of the research. A Quality Assurance Research Officer (QARO) will periodically inspect and audit the research to determine if the studies are being conducted under the GLP's.

2 The typed name and title of both S.A. Clay and the Regional Field Coordinator will appear immediately following "Approved by" on each individual SOP. S.A. Clay and the Regional Field Coordinator will initial and date each individual SOP next to their typed name and title upon approval.

3 Each individual SOP will be reviewed annually and revised as needed. The revision number of each individual SOP should begin with 2.0 and increase sequentially (i.e. 2.1) with each revision. All earlier revisions will be retained in an Archives file.

4 The ENTIRE SET of SOP's will also be assigned a revision number starting at 2.0. This number will appear on the Cover Page along with the date it that it was submitted to the Regional Field Coordinator. If a revision is made that affects the entire set of SOP's, then the revision number of the entire set will increase by one whole number (3.0, 4.0, etc.). A decimal number (i.e. 2.1) will not be used to name the entire set.

5 The effective date of an entire set of SOP's will be the date that the Regional Field Coordinator signs, initials, and dates his approval on the Cover Page. This will be done on an annual basis prior to the start of GLP trials.

6 Any deviations from the SOPs should be noted in the raw data notebook and approved by the Field Research Director.

7 The SOP's developed for field studies also apply to greenhouse studies unless an SOP has been specifically developed for the greenhouse.

Weed Stress/Environmental Herbicide Fate Laboratory
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Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

Submitted Date: May 1, 1993
Revised Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director SAC 3/27/14
S. Miyazaki, Regional Field Research Coordinator GMR 4/8/14

Title: 1.2 Numbering system for SOPs

Purpose: To provide a general outline for SOP's via a numbering system.

Scope: All SOP's should follow the numbering system to provide uniformity in the system.

Procedures: The numbering system for SOP's is as follows:

1. General
2. Personnel
3. Facilities
4. Equipment
5. Agronomic Practices
6. Pesticides
7. Data Handling
8. Residue Sample Handling
9. Reporting
10. Archives
11. Quality Assurance...retired as of 3/17/06, supplied by HQ
12. Disposal of Pesticides
13. Safety and Health Procedures
14. Procedures to Handle an EPA Audit or Inspection

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Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director SAC 3/27/14
S. Miyazaki, Regional Field Research Coordinator GM 4/8/14

Title: 1.3 Format for use in developing SOPs

Purpose: To assure an uniform format in the development of SOPs

Scope: Applies to all SOPs developed by scientists for use in the conduct of studies under GLPs in Weed Stress/Environmental Herbicide Fate Laboratory.

Procedures: The following is the format to be used for each Standard Operating Procedure (SOP):

Name of Laboratory (centered)
Address (centered)
1 space
Revision Number: (Left) Effective Date:(Right)
1 space
Submitted by: (Left) Submitted Date: (Right)
Revised by: (left)(if revised) Revision Date: (Right)(if revised)
1 space
Approved by: (Name and title of Approving official)
1 Space
Title: (SOP section number as a decimal and title)
1 space
Purpose: (Brief description of the purpose of the SOP.)
1 space
Scope: (Determines where and when the SOP is applicable)
1 space
Procedures: (Describe the operating procedures in numerical order from beginning to end so that a reasonably intelligent person with some knowledge of the process can carry out the procedures without any verbal input from other sources.)

Weed Stress/Environmental Herbicide Fate Laboratory
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Revision Number: 3.0

Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

Submitted Date: May 1, 1993
Revised Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director sac 3/27/14
S. Miyazaki, Regional Field Research Coordinator GM 4/8/14

Title: 1.4 Designation of Field Research Director and responsibilities.

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

Scope: All field personnel involved in the GLP program where a single study is not controlled by a single study director and more than one field and/or laboratory sites are involved.

Procedures:

1 S. A. Clay (Professor, Plant Science) is appointed as Field Research Director by an institutional authority at the research testing facility to conduct the study. The Field Research Director should be a scientist with appropriate training and experience to conduct the study.

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

a Assure that the study is carried out according to an approved protocol (See example in Appendix 1).

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

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

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

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

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

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

h Designate the location for the Archives if necessary.

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Revised Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 2.1 Personnel

Purpose: To provide information to locations concerning requirements under Good Laboratory Practices for Personnel.

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

Procedures:

1. The laboratory will have on file a current summary of the training and experience and a brief description of duties or responsibilities for each person supervising any aspect the study.

2. The Field Research Director will determine that the person or persons conducting the study are of sufficient number to carry out the study to its completion and are sufficiently trained to conduct their portion of the study.

3. The laboratory will have a supply of safety equipment in reasonable working order and sufficiently clean to protect the health and safety of the personnel connected with the project as required by the EPA Worker Protection Standards for Agricultural Pesticides, regulations, other institution regulations, pesticide labels or the study protocol.

4. Where the application of restricted use pesticides is required in the study, the applicator must be certified or under the direct supervision of a certified applicator.

5. Personnel handling pesticides should be trained in accordance with the current policies and guidelines of their institution.

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Revised Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 2.2 Organizational Chart

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

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

Procedures:

1 An organizational chart should be developed which reflects the management of the facility and the reporting lines of the personnel engaged in the GLP studies.

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

3 At the top of the chart, show the head of the unit (i.e. Department Chair, Director etc.). This person should be the one who appoints the Field Research Director.

4 The chart should then show how the Field Research Director (FRD) and the Quality Assurance Research Officer (QARO) independently report to the head of the unit.

5 Supervisors engaged in the conduct of the studies should then be shown on the chart with lines of supervision indicated.

6 A Quality Assurance Unit (QAU) will monitor several trials to assure that good scientific methods are employed and that the FRD is complying with SOPs and study protocols. QAU SOP's are maintained by IR4 headquarters and all QAU activities are coordinated by the IR4 QAU.

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Revision Number: 3.0

Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

Submitted Date: May 1,1993
Revised Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director Sac 3/27/14
S. Miyazaki, Regional Field Research Coordinator S.M 4/8/14

Title: 2.3 Documentation of Training

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

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

Procedures:

1. A supervisor will oversee every aspect of GLP studies. This supervisor will be either S.A. Clay or another person that she assigns as a substitute.

2. Supervisors will complete a CV to be included in the field data book. At a minimum, this CV will summarize the education; work experience; and special training, qualifications, or accomplishments relevant to the work involved with GLP studies.

3. Supervisors will annually review and understand the standard operating procedures and the sections of the protocol that pertain to their responsibilities. These reviews will be considered training, and will be documented in the personnel files at the location.

4. Training received from workshops, conferences, etc. should be noted as to the name of the event and dates of attendance. A copy of any type of training certificates issued should be retained in the personnel files at the location.

5. A document will be inserted into the field data book that outlines training offered to non-supervisors. This will include the non-supervisor's name, summary of the training received, name of supervisor who provided the training, and the non-supervisor's initials if he/she is to make entries into the field databook.

Weed Stress/Environmental Herbicide Fate Laboratory
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Revision Number: 3.1

Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

Submitted Date: May 1,1993
Revised Date: March 25, 2015

Approved by: S. A. Clay, SD Field Research Director SAC 3/30/15
S. Miyazaki, Regional Field Research Coordinator SM 4/27/15

Title: 3.1 Guidelines for pesticide storage

Purpose: To assure that all pesticides are stored in a manner consistent with GLP requirements.

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

Procedures:

1 Pesticides will be stored in accordance with current policies and guidelines of the testing facility institution (attach to SOPs).

2 Store pesticides in a dry, climate-controlled, well-ventilated room (Ag Hall 201A) which is separate from offices and laboratories and where fire protection is provided. A highly visible warning sign should be placed on the door.

3 The storage facility should have limited access, with only trained personnel wearing protective equipment handling the pesticides. The names and telephone numbers of these personnel should be posted outside the door of the storage facility.

4 All pesticides containers in storage must be properly labeled. A current inventory of all pesticides in the storage unit in an inside location accessible and visible to study personnel. This inventory will include the name of the pesticide as it appears on the container label, the number of containers received, the date received, the expiration date, and the trial ID number.

5 Check containers of pesticides regularly for corrosion and leaks. If such is found, the contents should be transferred to a sound, suitable container and be properly labeled, or the container and its contents should be properly disposed of.

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

7 All pesticide containers should be retained until the study is completed and the QARO has made a final inspection of the study and permission is given by the Study Director to dispose of the container and its contents.

8 GLP pesticides should be stored separate from other pesticides and chemicals in the room.

9 Storage facility temperature will be monitored weekly using maximum/minimum thermometers calibrated yearly.

Weed Stress/Environmental Herbicide Fate Laboratory
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Revision Number: 3.1
Submitted by: Graig Reicks

Submitted Date: May 1, 1993
Revised Date: March 25, 2015

Approved by: S. A. Clay, SD Field Research Director 4/16/15
S. Miyazaki, Regional Field Research Coordinator 4/27/15

Title: 3.2 Guidelines for spray additives in GLP trials

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

Scope: All field and laboratory studies conducted under the direction of S.A. Clay where of spray additives must meet GLP compliance.

Procedures:

1 Store in a location that has limited access and is temperature monitored.

2 If the additive has a suspicious appearance or smell, discontinue its use in GLP trials.

3 Proper labeling will include: name, concentration, required storage conditions (from the label or Safety Data Sheet), and expiration date.

4 Spray additives can be subdivided into secondary containers. These secondary containers must meet the same labeling requirements as stated in this SOP. Labeling of secondary containers must be done as soon as possible after pouring from the original container.

5 Always use a newly-opened measuring device (i.e. syringe or pipette tip) when dipping into the additive container. These devices can only be re-dipped into the container if there's been no potential for contamination, as determined by the best judgment of the mixer.

6 Adding additive back to the container is acceptable only when a newly-opened measuring device is being used. Again, the device can have no potential for contamination.

7 Spray additives purchased prior to 2015 with no previous temperature monitoring are still acceptable, as long as temperature monitoring begins prior to use in 2015 GLP trials.

8 There must be near certainty that old additives being transitioned into GLP status in 2015 have had no potential for contamination.

9 If no expiration date exists, then spray additives are considered effective for five years past their purchase date.

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Revision Date: March 25, 2015

Approved by: S. A. Clay, SD Field Research Director 3/30/15
S. Miyazaki, Regional Field Research Coordinator 4/27/15

Title: 3.3 Site selection for field studies.

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

Scope: All locations selected for field studies under the direction of S. A. Clay and requiring GLP compliance.

Procedures:

1 Site selection will be made in accordance with the horticultural practices acceptable for the commodity.

2 Site will be large enough to accommodate the required number of replicates, buffer zones and treatments in accordance with an approved study protocol and for the commodity to be grown under simulated commercial conditions yielding samples of sufficient size for analysis where required.

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

4 Where samples for residue studies are required, locate a second site within the same area but with enough isolation to produce untreated, uncontaminated samples.

5 If the commodity is not to be newly established, select a site that has a uniform stand for production.

6 Cultural practices (plowing, planting, etc.) should be performed prior to plot layout and marking.

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 so that the plots can be relocated after the study is terminated.

8 A Global Positioning System (GPS) may be used to determine reference points. It the trial protocol does not specify a minimum number of GPS points to record, then a minimum of 2 opposite corners per plot in the trial shall suffice. GPS accuracy will be checked against a known latitude and longitude reference point before and after taking readings of the GLP trials each year. Three readings will be taken for each GPS accuracy check, with the GPS

unit being fully shut-down and restarted for each reading. Each GPS readings should be within 1 meter of the known point for the unit to be considered functional.

9 Each plot will be identified as detailed in the study protocol. If statistical analysis is to be performed on the data, assign the replicates and treatments to the plot map using a commonly accepted statistical design with sufficient information to identify the replicate and treatment assigned to each plot.

10 Lay out each plot on the site using a string and suitable measuring device to accurately locate the plots on the site.

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

12 Number each stake with a permanent number corresponding to the plot number on the plot map.

13 The plot map (item 7) and a summary of the cultural practices (item 6) should be part of the raw data notebook.

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Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 3.4 Greenhouse/shadehouse facilities

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

Scope: All greenhouse/shadehouse studies performed under the direction of S. A. Clay and requiring GLP compliance.

Procedures:

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

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

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

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

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

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

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

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Revised by: Graig Reicks

Submitted Date: May 1, 1993
Revision Data: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 3.5 Archives facility

Purpose: To assure that each location conducting GLP studies has a designated area for archives of records pertaining to the program.

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

Procedures:

1 A limited access facility (i.e. locked room or cabinet) of sufficient size to contain all records and data generated should be available at or in close proximity to the location of the Field Research Director responsible for the conduct of the studies.

2 The facility should be in a building with adequate fire protection or should contain protection devices within the room.

3 The facility should be under lock and with limited access to the key. File cabinets containing GLP documents in the room should be under lock and key with controlled access.

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Revision Date: March 25, 2015

Approved by: S. A. Clay, SD Field Research Director Sac 3/30/15
S. Miyazaki, Regional Field Research Coordinator SM 4/27/15

Title: 4.1 Calibration and use of an analytical balance.

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

Scope: All field and laboratory studies conducted under the direction of S.A. Clay and requiring GLP compliance. Applicable to those studies where a dry material is weighed for use in a field, greenhouse or shadehouse trial.

Procedures:

1. The balance should be calibrated using certified standard weights immediately prior to weighing the quantity for use in the study by following manufacturer's directions.
2. Do not touch the standard weights with bare hands. Either use clean gloves or some type of clean laboratory wipe.
3. For the scale calibration to be acceptable, the balance must read within +/-1% of the value stated on the standard weight.
4. Recertify standard weights annually by recording the weight of each standard as soon as possible following professional calibration of the balance. Again, this value should read +/-1% of the value stated on the standard weight.
5. Select a clean glass jar with a tight fitting lid or other container suitable to hold the desired amount of pesticide and tare it on the scale following the manufacturer's directions.
6. Select and wear or use appropriate safety equipment while handling pesticide concentrate.
7. Weigh the concentrate in tared container. Return excess to original pesticide container if this procedure does not affect the integrity of the contents or dispose of the excess by using appropriate methods for handling hazardous wastes.
8. Label the container to identify it as to the appropriate treatment or plot numbers.
9. If taring the container is not practical, then record the weight of the container and add the amount of test substance until the container reaches the correct wt.
10. Maintain a written record of the amount of the pesticide removed from the original container in the test substance use log in the Field Notebook.

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 4.2 Measuring a liquid formulation

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

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

Procedures:

1 Obtain a reasonably clean container (preferably glass) large enough to hold the volume of pesticide needed for the treatment and graduated in increments small enough to read to an accuracy within +/- 1% of the total volume required (i.e. if 100 ml is needed the smallest division on the cylinder should be 1 ml or less.)

2 If the opening of the cylinder is too restricted to allow pouring of the pesticide from the original container without danger of spillage, then do one of the following:

a. Use a reasonably clean glass container with a pour lip as an intermediate and fill the cylinder from it or
b. Use a reasonably clean glass funnel that is large enough to allow filling the cylinder with a minimum of spillage.

3 Select and wear or use appropriate safety equipment while handling pesticide concentrate.

4 Measure the liquid in the cylinder. Take the reading of the liquid in the cylinder at the bottom of the meniscus.

5 Pour the liquid into a clean glass jar, fit with a liquid proof lid, and label jar with both the trial and plot ID numbers that will receive the treatment. An alternate method is to pour the liquid directly into the spray tank. Make sure that as much as possible of the liquid is transferred to the jar or spray tank. The container should be triple-rinsed into the spray tank with a portion of the carrier.

6 Maintain a written record of each volume of the pesticide removed from the original container during the study and maintain a master sheet of the pesticide removed for each container used.

7 Containers used to measure or transfer the pesticide concentrate should be triple rinsed into the spray tank and then washed with soap and water after use to ensure that they are reasonably clean and cross-contamination of pesticides will not occur in future use.

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Revision Date: March 27, 2014

GwR 5/6/15

NOTE: A revision date error was discovered and corrected on 5/6/15. GwR 5/6/15

Approved by: S. A. Clay, SD Field Research Director
S. Miyazaki, Regional Field Research Coordinator

SM 5/13/15

Title: 4.3 Calibration of a Sprayer

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where a sprayer is used in the application of pesticides.

Procedures:

1. Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear or potential leaks and repair or replace as necessary. Use a calibration method suitable to the application and the equipment used such as that accompanying the sprayer manual or a TeeJet catalog. If none is available then proceed with the following:

2. Refer to the protocol for any specified application requirements. Select the appropriate type of nozzle, which is based on the pesticide formulation, the application method, the operating speed, the pressure setting and the dilution rate (gal/acre).

3. All sprayers should have a pressure gauge. Select the proper operating pressure.

4. Place the sprayer on level ground. Adjust the boom height and nozzle spacing for the correct application pattern. Determine whether all nozzles are discharging uniformly by spraying water through them at a uniform pressure. Visually evaluate the spray pattern for its complete and symmetrical arrangement. Then catch the discharge from each nozzle in a separate container over a timed interval (usually 20 - 60 seconds, but no less than 15 seconds) beginning after the nozzles are discharging. If the discharge varies by more than 5%, replace all nozzle tips that have a much larger or much smaller discharge. Variation among nozzle tips should be less than 5%. Repeat the above procedure until all nozzles are within 5% of mean discharge. Once within 5% of the mean, this should be verified by collecting discharge 2 more times.

5. Determine the nozzle flow rate over the timed interval chosen above. Calculate the average discharge per nozzle in milliliters per second.

EXAMPLE: Sprayer boom had 6 nozzles on 20 inch spacing; boom width is 10 feet.

Calibration	Nozzle 1	Nozzle 2	Nozzle 3	Nozzle 4	Nozzle 5	Nozzle 6	Average
1 30 sec	332	344	345	347	327	351	341.00
2 30 sec	338	339	342	338	320	345	337.00
3 30 sec	333	342	345	342	325	347	339.00
Average	334.33	341.67	344.00	342.33	324.00	347.67	339.00

Average output for 3 calibrations = 339 ml / 30 second or 11.3 ml / second per nozzle.

6 Calibrate the speed of the sprayer. This can be performed over the same paths that the applicator will traverse when spraying the plot. Alternatively, a course the same length as the plot about to be treated, over similar terrain, and within a similar crop canopy can be used for speed calibration. One person will operate the sprayer. One person will operate a stop clock. Measure the time it takes to transverse the desired distance in seconds. Calculate the miles per hour using the following equations:

EXAMPLE: The course was 75 feet long and it took 18 seconds to transverse the course.

$$\frac{75 \text{ feet}}{18 \text{ sec}} = \frac{4.17 \text{ feet}}{\text{sec}}$$

$$\frac{4.17 \text{ feet}}{1 \text{ sec}} \times 60 \text{ sec} = \frac{250 \text{ feet}}{60 \text{ sec}}$$

1 mph = 88 feet / 60 seconds

$$\frac{250 \text{ feet}}{60 \text{ sec}} \times \frac{60 \text{ sec}}{88 \text{ feet}} = 2.84 \text{ mph}$$

The sprayer should be operated over the course for 3 runs. Calculate the average miles per hour from these three runs. The mph from each run should be within 5% of the average mph.

7 Delivery rate calibration. This is divided into 3 steps; (1) calculate area covered by boom per second; (2) calculate gallons / sec; (3) calculate gallons / acre.

EXAMPLE:

A. Area covered by boom per second:

$$\frac{4.17 \text{ feet}}{1 \text{ sec}} \times 10 \text{ ft boom width} = \frac{41.67 \text{ feet}^2}{1 \text{ sec}}$$

$$\frac{41.67 \text{ feet}^2}{1 \text{ sec}} \times \frac{1 \text{ Acre}}{43560 \text{ feet}^2} = \frac{0.000957 \text{ Acre}}{\text{sec}}$$

B. Calculate gallons / second

$$\frac{11.3 \text{ ml}}{\text{sec}} \times 6 \text{ nozzles} = \frac{67.8 \text{ ml}}{\text{sec}}$$

$$\frac{67.8\ \text{ml}}{\text{sec}} \times \frac{1\ \text{gallon}}{3785\ \text{ml}} = \frac{0.0179\ \text{gallon}}{\text{sec}}$$

C. Calculate gallons / acre

$$\frac{0.0179\ \text{gallon}}{\text{sec}} \times \frac{1\ \text{sec}}{0.000957\ \text{Acre}} = \frac{18.72\ \text{gallon}}{1\ \text{Acre}}$$

8 Volume mixing and dilution calculations. This is divided into 5 steps; (1) calculate plot area in acres; (2) calculate total amount of solution (in milliliters) applied over plot (use gallon / Acre calculated above); (3) determine amount of test chemical to be mixed; (4) determine amount of surfactant (when required); (5) determine amount of water to add to tank mix.

EXAMPLE:

A. Plot area in acres.

50 feet x 100 feet = 5000 feet^2

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

Add a minimum of10% extra area to fill booms before each pass.

- 20% was used in the example.

0.115 Acre x 0.20 = 0.023 Acre; 0.115 + 0.023 = 0.138 Acre

B. Total amount solution applied over plot area.

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

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

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

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

0.005 x 9759 ml = 49 ml surfactant

E. Amount of water to add to tank mix.

Amount of water = Total volume – product – surfactant

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

Weed Stress/Environmental Herbicide Fate Laboratory
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Brookings, SD 57007

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 4.4 Calibration and use of Granulator Applicators

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where a granular applicator is used in the application of pesticides.

Procedures:

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

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

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

4 Measure an area of 0.01 acre or 435.6 square feet in close proximity to the area to be treated. A simple method to calculate the distance is:

$$\frac{435.6\ ft^2}{\text{(width of application in feet)}} = \text{feet to travel}$$

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

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

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

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

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

10 lb A.I.=67 lbs formulation/acre
or 10 lbs A.I./0.15
width of applicator =10 ft

435.6= 44 ft. to travel
or 435.6/10

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

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Title: 4.5 Operation and maintenance of farm equipment.

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

Scope: All field studies conducted under the direction of S. A. Clay and requiring GLP compliance and where the farming operations are performed in GLP studies.

Procedures:

1 Just prior to the initiation of the use of the equipment (tractor, plow, disk, harrow, planters, harvester etc.), the Field Research Director or his/her designated representative will visually inspect the equipment to see that it is in good working order, properly lubricated, and in reasonably good mechanical condition

2 Any necessary repairs or adjustments should be made prior to the use of the equipment in the study.

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

4 Manuals on the operation and maintenance of the equipment and the name, address, and telephone number of a parts supply company should be kept in a place accessible to the operator and the Field Research Director.

5 A written record should be maintained for each piece of equipment used in a GLP study. The record should contain maintenance service dates and what was done and repair dates and type of repair.

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S. Miyazaki, Regional Field Research Coordinator SM 4/25/15

Title: 4.6 Calibration of instruments and gauges.

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

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

Procedures: 1 Measurement instruments used in a GLP study should be periodically tested to confirm reasonable accuracy. Electronic devices have a higher probability of failure and should be tested more frequently (i.e. monthly, after every 10 hrs. use etc.). A non-electronic instrument has a lower probability of failure. Therefore, calibration against a standard once per year will be acceptable.

2 Calibration points will bracket the working ranges of the instrument.

3 Calibration of the thermometers should be done against standard thermometers, in an ice bath, or in boiling water. Water (including ice) should be distilled or nanopure.

4 Each calibration point should be within +/-1°C of the standard thermometer. If an ice bath is used, the thermometer being tested should read within -1 to 1°C. For boiling water, the thermometer should read within 99 to 101°C.

5 The standard thermometers will receive an ASTM or NIST calibration at least biannually.

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

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

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

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S. Miyazaki, Regional Field Research Coordinator 4/3/14

Title: 5.1 Field preparation for seeding or transplanting

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where the seeding or transplanting is prepared.

Procedures:

1 Refer to a reasonably up-to-date publication on the production of the commodity under study. If no such publication exists, consult with an agricultural specialist familiar with the production practices for the commodity..

2 Determine pH and soil fertility requirements of the commodity. Obtain random samples of soil for testing from the study site. Have the soil tested to determine how well it will meet the requirements of the commodity (specify whether or not the testing was done under GLP in the raw data book.)

3 Lime, fertilize and/or condition the soil at the site as necessary to bring the soil reasonably within the requirements of the commodity.

4 Till the field as specified for the commodity.

5 Apply appropriate pesticides (preplant herbicide, soil insecticide, fungicide drench, soil-incorporated nematicide etc.). Apply and document application of pesticides as specified in other areas of these SOP's.

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 5.2 Method for seeding or transplanting

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where the seeding or transplanting is prepared.

Procedures:

1 Determine the correct species and variety to use as specified by the study protocol. If the variety is not specified, determine the variety most commonly used in the area by commercial producers and use it for the study. If a commercial producer is providing the plants, try to select plants as uniform in growth and color as possible.

2 Plant seeds at the proper population, row spacing, and depth as specified from SOP 5.1. Plant the seed or transplant in reasonably straight lines or rows with fairly accurate measurements to assure the commodity is planted according to specifications.

3 Identify each treatment according to protocol or in such a manner so that it will be visual throughout the life of the study.

4 Irrigate or perform other agricultural practices as necessary to get the commodity started.

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 5.3 Commodity maintenance

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

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

Procedures:

1. Maintain the commodity in a healthy state and good growing condition throughout the life of the study as directed in document obtained from SOP 5.1 or as recommended by agricultural specialist.

2. If pesticides are applied to the commodity to prevent losses due to pests not under study, they should be applied according to the relevant SOP's in this document. If this is a residue study, no pesticide should be applied that would interfere with the chemical analysis of the pesticide under study. If in doubt, call the analytical chemist or analytical laboratory identified in the protocol to receive the residue samples.

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 5.4 Determining yield or quality

Purpose: To assure that a measurement of yield or quality of the various treatments is taken if required to evaluate the effects of the treatments.

Scope: All field and laboratory studies conducted under the direction of S.A. Clay and requiring GLP compliance and requiring yield data. (Sample handling for residues is covered under another SOP).

Procedures:

1 Where possible, obtain a reasonably up-to-date copy of the United States standards for grades of the commodity under study from the Agricultural Marketing Service or other sources. If U.S. grade standards do not exist, then consult other sources and document the plant stage, fruit ripeness, or other characteristics needed to determine quality in the raw data notebook.

2 Check the protocol for information on time of harvest. If none, then follow commercial practices in the area for the appropriate time to harvest the commodity.

3 Where grading standards are known or exist, the commodity should be graded accordingly at harvest to segregate the harvest to measure quality.

4 If specified by protocol, each portion of the commodity, divided as to its quality standard, should be weighed or measured to determine yield. Written records should be kept of each measurement for each plot.

5 The commodity will be harvested according to the method designated in the protocol.

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Title: 6.1 General procedures in the application of pesticides.

Purpose: To describe the practices common to all types of pesticide application.

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

Procedures:

1. All personnel involved in the mixing, application, storage and cleanup of pesticides should be properly trained in accordance with current policies and guidelines of their institution.

2. Equipment used in the application of the pesticides should be inspected and calibrated as indicated under SOP 4.3.

3. Personnel mixing and applying the pesticide should wear appropriate protective clothing as stated on the pesticide label or as indicated under SOP 13.1 and 13.2.

4. The pesticide concentrate should be measured out as indicated under SOP's 4.1 or 4.2 for a dry or liquid concentrate respectively.

5. If the pesticide application is for maintenance of the plots, then apply the pesticide to all the plots in the study according to the directions on the pesticide label.

6. If the pesticide application involves the test substance, then procedures for handling the test substance as indicated in SOP 7.6 should also be followed.

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Title: 6.2 Adding the pesticide concentrate to the water carrier in the spray tank of a sprayer.

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

Scope: All field studies conducted under the direction of S. A. Clay and requiring GLP compliance and where a sprayer is used.

Procedures:

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

2 Add about half the water to the spray tank.

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

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

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

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

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Title: 6.3 Procedures for the application of the study pesticide(s) in the field.

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

Scope: All field studies conducted under the direction of S. A. Clay and requiring GLP compliance and where the study pesticide(s) is applied.

Procedures:

1 Application equipment should be inspected and calibrated as described under SOP 4.3

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

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

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

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

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

7 Record pass times in the Field Data Book.

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Title: 6.4 Application of pesticides in the greenhouse.

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

Scope: All greenhouse studies conducted under the direction of S. A. Clay and requiring GLP compliance and where pesticides are applied.

Procedures:

1 Application equipment should be inspected and calibrated as described under SOP 4.3

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

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

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

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

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Approved by: S. A. Clay, SD Field Research Director
S. Miyazaki, Regional Field Research Coordinator GM 5/13/15

Title: 6.5 Cleanup of application equipment

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

Scope: All field and laboratory studies conducted under the direction of S.A. Clay and requiring GLP compliance and where pesticides application equipments are used.

Procedures:

1 Granules-Remove any excess granules and return them to the original container if this procedure does not affect the integrity of the contents or dispose of the excess by using appropriate methods for handling hazardous wastes. Note in the pesticide log for the chemical, the amount of granular material used in the study.

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

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

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

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

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

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

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Title: 6.6 Handling the Test, Control and Reference Substance.

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance. For the purposes of this SOP, test substance also applies to control and reference substances.

Procedures:

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

2 Each container needs a unique ID. This ID is usually the protocol number, which may already be listed on the container at receipt. If protocol number is not listed, then add this number to the container along with initials and date. Sometimes multiple containers of the same test substance are received with the same ID/protocol number. If this happens, create a unique ID for each container similar to the following format: 12345.67-1, 12345.67-2, and 12345.67-3.

3 The test substance should then be stored in the pesticide storage facility until it is needed for use in the study.

4 Temperatures will be monitored weekly in the pesticide storage facility using maximum/minimum thermometers that are reset weekly after recording.

5 The storage conditions of the test substance should be recorded in the raw data book.

6 All test substance containers must be stored until notification by the study director that the containers may be discarded. Whenever possible, discarded containers should be returned to the registrant or sponsor.

7 Separate areas should be established for mixing and handling of the test substance and for storage of test substance mixtures.

8 Test substances and mixtures should be stored in a manner to prevent any possibility of contamination, deterioration, or damage during the conduct of the study. The test substance label should be consulted and followed for storage conditions.

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

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and when pesticides are used. For the purposes of this SOP, test substance also applies to control and reference substances.

Procedures:

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

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

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

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

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Title: 7.1 Collection of raw data electronically was merged with and became SOP 7.6 Rev. 3.0 in 2014

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Title: 7.2 Recording of raw data.

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

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

Procedures:

1 Under most circumstances, raw data will be recorded into the field data book by supervisors only. Raw data shall be recorded in ink, preferably blue, so copies and originals can easily be distinguished.

2 Date and initial each time an entry is made. Periodically have entries witnessed (signature and date) by another employee. If non-supervisors enter data, then their initials should accompany the data entry along with documentation of their training to perform the task which they are documenting (see SOP 2.3 Rev. 3.0 for further detail).

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

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

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

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

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

True and Exact Copy
Initial_Date____
Original in_____

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

a. Names of all personnel conducting specific research functions
b. Amendments and deviations from protocol and SOPs
c. Test site information
d Plot maps
e. Test substance
f. Test substance receipt, use and container/substance disposition records
g. Test substance storage conditions and temperatures
h. Data regarding calibration and use of application equipment
i. Treatment application data
j. Crop maintenance pesticides and cultural practices, test plot history, and soil information
k. Residue sample identification, collection, storage conditions, and handling
l. Residue sample shipping information
m. Description of crop destruction, or explanation for lack of destruction
n. Meteorological/Irrigation records
o. Pass times (if applicable) to confirm proper amount of material applied to plot
p. Equipment maintenance records with indication of routine vs. non-routine nature of maintenance
q. Other relevant data requested in Field Data Book to confirm study was done in accordance with protocol

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

Weed Stress/Environmental Herbicide Fate Laboratory
South Dakota State University
Brookings, SD 57007

Revision Number: 3.0

Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

Submitted Date: May 1, 1993
Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/3/14

Title: 7.3 Method for collecting efficacy and phytotoxicity data.

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

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

Procedures: A. Phytotoxicity data:

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

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

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

B. Pest data:

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

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

3 Disease data-Record the name of the disease(s) being observed. Record the symptom(s) for each disease. Randomly select 5 plants in the middle row of each plot and record the severity of each disease in a rating system of 0 to 10 for each plant. Zero= plant healthy. Ten= Plant dead. One thru nine= the percentage disease appearing on the plant. If there are less than five plants/row, record data from 5 plants/plot or all the plants in a plot.

4 Insect data-Record the name of the insect(s) being observed. Record the damage symptom(s) for each insect. For damage symptoms-randomly select 5 plants in the middle row of each plot and record the severity of damage for each insect in a rating system of 0

to 10 for each plant. Zero= plant healthy. Ten= Plant dead. One thru nine= the percentage damage appearing on the plant. If there are less than five plants/row, record data from 5 plants/plot or all the plants in a plot.

For insect pest population counts-take a random sample of the pest population (i.e. 5 leaves/plant of 5 plants/plot, 4 3-in diam. soil cores/plot, 100 apples/tree etc.) to insure an accurate reflection of the pest density/unit area.

5 Nematode data-Record the name of the nematode(s) being observed. Record the damage symptom(s) for each nematode. For damage symptoms-randomly select 10 plants in the middle row of each plot and record the severity of damage for each nematode on each plant using one of the rating systems described by the following:

> Barker, K.R., J.L. Townshend, G.W. Bird, I.J. Thomason and D.W. Dickson. 1986. Determining nematode population responses to control agents. In Kickey, K.D. (ed.). Methods for evaluating pesticides for control of Plant Pathogens. Pages 283-296.

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

For nematode population counts-take a random sample of the pest population (i.e. root system of 2 plants/plot, 4 3-in diam. soil cores/plot, etc.) to insure an accurate reflection of the pest density/unit area as described by Barker et. al. cited above. Use a method suitable to extract the nematodes from the soil or plant sample and cite the method here. Count and record the number of nematodes by the various life stages/unit of soil or root.

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

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Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 7.4 Experimental design and data analysis

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

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

Procedures:

1. The experimental design as specified by the protocol should be used. If none is designated, then the researcher should use a commonly accepted experimental design such as a complete randomized block design. The experimental design used should be documented in the raw data notebook.
2. A minimum of 3 replicates should be used (4 is preferred). No replicates or statistical analysis are required where the study is for magnitude of the residue only.
3. Draw a plot map showing the location of each plot in the site selected for testing as described under SOP 3.2.
4. Randomly assign the treatments to the plots using a random number table or random number generator. Note the location of the treatments on the plot map.
5. Retain the plot map in the study folder.
6. Determine the level of significance for the study.
7. Select an appropriate statistical package for data analysis and record sufficient information to identify the statistical package (i.e. Date, Revision no., Title, Authors, Source etc.).
8. When the raw data are available for analysis, utilize the statistical package and follow instructions contained therein to conduct an analysis of variance and mean separation of the data.
9. Record the data as required on the appropriate forms and identify statistically significant differences in the data in the raw data note book.
10. Retain all data, analyses, notes etc. in the study folder with sufficient information to recalculate the data summaries and statistical analyses by another person.

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Revised by: Graig Reicks

Submitted Date: May 1, 1993
Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director WE See 3/27/14 See 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 7.5 Completion of summary forms was merged with and became SOP 7.2 Rev. 3.0 in 2014.

Weed Stress/Environmental Herbicide Fate Laboratory
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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 7.6 Collection and recording of data from electronic devices

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

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

Procedures:

1 All remote sensing and other automatic data collecting and/or recording devices should be inspected and calibrated according to their respective SOP. If not required by GLP, some form of inspection and/or calibration is highly-recommended.

2 Check the power supply on portable units to see that it will be adequate during the data collection and data transfer period.

3 Make sure the correct program for data collection is ready and available for use.

4 Electronic data must be legible to persons with normal vision.

5 Electronic data should be transferred to a storage system and immediately printed out with appropriate identification. The original printout should have an initial and date of the person who performed the printing. If this original printout is inserted into the field data book, a copy should be retained at the field testing location.

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 7.7 Data storage during the active life of the project

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

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

Procedures:

1 The Field Research Director will see that a separate file containing all raw data connected with the study is maintained during the active life of each project for which he/she is responsible.

2 If the raw data was put into the field data book, which will be permanently stored elsewhere, then a copy of the raw data shall be retained in the archives.

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

Weed Stress/Environmental Herbicide Fate Laboratory
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Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

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Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 8.1 When to obtain residue samples.

Purpose: To assure that residue samples are collected in a timely fashion.

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where studies are conducted to obtain residue samples.

Procedures:

1 Consult the study protocol to establish specific dates for the collection of samples. If these dates are based on uncontrolled events (fruit size, spray applications etc.) then tentative dates should be established and refined as necessary. The Study Director and Quality Assurance Research Officer should be kept informed when the dates are changed.

2 Samples should not be taken during periods of inclement weather.

3 Untreated samples should be collected first, followed by the lowest dosage rate and working toward the highest dosage rate.

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 8.2 Method of sample collection

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where studies are conducted to obtain residue samples.

Procedures:

1 Representative samples of the crop in each plot must be taken by a recognized procedure. Cite the procedure used.

2 Consult the study protocol to determine sample size and special instructions for the commodity.

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

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

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

Weed Stress/Environmental Herbicide Fate Laboratory
South Dakota State University
Brookings, SD 57007

Revision Number: 3.0

Submitted by: Zhuojing Liu
Revised by: Graig Reicks

Submitted Date: April 4, 1997
Revision Date: March 27, 2014

Saw 5/6/15 NOTE: A revision date error was discovered and corrected on 5/6/15. GWR 5/6/15

Approved by: S. A. Clay, SD Field Research Director
S. Miyazaki, Regional Field Research Coordinator SM 5/13/15

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

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where studies are conducted to obtain residue samples.

Procedures:

1. In protocols that justify sample collection by a combine, a self-propelled plot combine will be utilized.

2. Make one swath around the perimeter of the untreated plot with the self-propelled combine to remove plot margins, which are often not representative of the plot.

3. Make a single pass diagonally across the plot to collect the first sample, which will constitute combine/thresher clean-out and will be discarded. Make the next pass in the opposite direction. For example, if the first pass ran NE-SW, make the next pass in a NW-SE orientation. Continue collecting the samples in this manner. Avoid harvesting more than half of the plot.

4. If sample collection will consist of delivering plant material to a stationary combine or small bundle thresher operating outside the plot area, follow the same procedures as above. Collect from at least 12 different areas (unless the protocol says differently) while traversing the plot in diagonal patterns.

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

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

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Revised by: Graig Reicks

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/3/14

Title: 8.4 Sample identification and records

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where studies are conducted to obtain residue samples.

Procedures:

1 Plastic-lined cloth sampling bags with an identification tag sewn into the bottom stitching are usually provided to GLP cooperators for sample collection. If these bags have not been provided, a sampling bag suitable to protect the integrity of the sample should be used.

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

3 Before entering the field, use waterproof ink to fill in the label attached to the bottom of the bag and indicate the study ID number and bag number on the tag if more than one is used for the plot sampled. Each sample bag should also contain a card within a waterproof bag or container the following information (same info. as found on outside of bag)::

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

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

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

Weed Stress/Environmental Herbicide Fate Laboratory
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Revision Number: 3.1

Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

Submitted Date: May 1, 1993
Revision Date: March 25, 2015

Approved by: S. A. Clay, SD Field Research Director 3/30/15
S. Miyazaki, Regional Field Research Coordinator 4/25/15

Title: 8.5 Sample storage and freezer malfunction procedures.

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where residue samples are packed and stored.

Procedures:

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

2 Carefully place the sample as it is collected in the sample bag marked for that sample. Make sure that labeling in the bag is enclosed with the sample.

3 Close the sample bag so as to prevent loss of the sample under reasonable storage, handling, and transportation conditions. Excess air should be expelled from the bag.

4 If storing temporarily in a cooler, use separate coolers for treated and untreated samples. As a rule of thumb, a 20 lb. bag of ice will be placed in each 48 qt. cooler. Even if placed in coolers on ice, make every attempt to get the samples in the freezers as soon as possible. Samples should be in coolers no more than 4 hours.

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

6 Most of the time, samples will be stored in a freezer in Northern Plains Biostress Laboratory that is wired to a generator in the event of a power failure. We are limited to only one freezer with backup power. Therefore, treated and untreated samples should be physically separated within the freezer to reduce the chances of contamination. A divider, boxes, bags, etc. could be used for separation

7 The freezer in Northern Plains Biostress Lab is capable of continuous temperature recording and should alert trial personnel via voice and/or text message to their mobile device in the event of a freezer failure.

8 At least one of the people chosen to receive an automated alert must be within reasonable distance of the freezer at any given time. Even if a person receiving an alert is not available to tend to the freezer emergency, that person should still contact others to ensure the message wasn't missed and that someone is able to fix the problem.

9 In the event of a freezer failure, samples will be delivered to the backup freezers in Berg Agricultural Hall. These backup freezers are not wired to a generator nor do they have an alarm system. They will have an automated temperature monitoring system.

10 In the unlikely event that all three freezers should fail, at least twice the total sample weight worth of dry ice will be purchased and put into separate coolers, one for treated and another for untreated samples. A min/max thermometer will also be put in each cooler. If possible, consider shipment of the samples to the analytical lab with dry ice. Otherwise, keep adding dry ice as needed until freezer space is restored.

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

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

12 The backup freezers in Berg Agricultural Hall may also be used during times of high sample volume when the single freezer in Northern Plains Biostress Lab can't accommodate all of the samples. In this case, treated samples should be stored in Northern Plains Biostress Lab (has a backup generator) and untreated samples in Berg Agricultural Hall (no backup generator).

13 All freezers should be under lock and key and only used to store GLP samples. The rooms where the freezers are situated should only have limited access.

14 In addition to the automated temperature monitoring system, each freezer should also contain a min/max thermometer as a backup. The min/max will be reset once per day, if possible. Temperatures will be recorded from the min/max each time it is reset.

15 Regular checks of the alarm system will be performed during the sampling season, but during times when the freezers are empty. The freezers will be unplugged to see if everyone receives an alert on their mobile device. The results of these checks will be documented.

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

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

Weed Stress/Environmental Herbicide Fate Laboratory
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Revised by: Graig Reicks — Revision Date: March 25, 2015

Approved by: S. A. Clay, SD Field Research Director Sac 3/30/15
S. Miyazaki, Regional Field Research Coordinator SM 4/27/15

Title: 8.6 Sample packaging and shipping procedures

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where residue samples are shipped.

Procedures:

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

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

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

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

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

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

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

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 9.1 Raw data report forms was merged with and became SOP 7.2 Rev. 3.0 in 2014

Weed Stress/Environmental Herbicide Fate Laboratory
South Dakota State University
Brookings, SD 57007

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 9.2 Use of report forms for raw data was merged with and became SOP 7.2 Rev. 3.0 in 2014

Weed Stress/Environmental Herbicide Fate Laboratory
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Brookings, SD 57007

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 9.3 Handling completed report forms that transcend two or more studies was merged with and became SOP 7.2 Rev. 3.0 in 2014

Weed Stress/Environmental Herbicide Fate Laboratory
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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 9.4 Disposition of raw data from the study. This SOP was retired in 2014.

Weed Stress/Environmental Herbicide Fate Laboratory
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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 10.1 General procedures - Archives

Purpose: To assure that each location conducting studies establishes and maintains an archives facility for retention of all data and documents connected with the program.

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

Procedures:

1 Each location should designate a site for the archives as described under SOP 3.5.

2 At each location, the Field Research Director is to be responsible for the archives and may designate An Archives Librarian as appropriate.

3 **The Field Research Director will be responsible for seeing that the study file containing the raw data, copies of reports, logs, etc. are submitted to the Study Director for archiving and a true copy placed in the archives.**

4 The Archives Librarian will establish and maintain a log of contents for each section (usually a 3 ring binder) of the archives. Each time a document is removed from and returned to its respective section, the person performing these actions will initial and date the log.

5 The Archives Librarian will also make certain that the contents of each cabinet are clearly marked on the outside of the cabinet. The log should be visible when viewing the cabinet from the front and legible at a normal reading distance.

6 Only the Archives Librarian will file or remove all documents in the archives cabinets. A place card will be used in place of any documents removed from the cabinets.

7 The archives may be used only by those persons so authorized by the Field Research Director. The names of authorized users should be on file with the Archives Librarian.

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S. Miyazaki, Regional Field Research Coordinator 4/3/14

Title: 10.2 Retention times for documents in archives.

Purpose: To assure that GLP documents are retained for the periods as specified in the regulations.

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

Procedures:

1 Data supporting the registration of a pesticide use pattern should be retained for the life of the registration following the date the study is submitted.

2 Data which is not used in the support of the registration of a pesticide use pattern should be held for 2 years following the date of the completion of the study.

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 10.3 Information to be retained in the archives.

Purpose: To assure that all the information is placed in an archive as required under GLP's.

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

Procedures: 1 Items placed in the archives should be identified as to the studies they pertain to or the dates when the items were in use. The following is a list of information to be retained in the archives:

a. True copies of each Field Data Book, which also contains a true copy of its QA Audit.

b. Facility Information (maps, storage temperature records, thermometer record, freezer maintenance)

c. Calibration of scales and standard weights

d. Equipment maintenance records

e. Weather records

f. Test substance information

g. Training records

h. Personnel records

i. Copies of inspections, both field and facility

j. Historical Standard Operating Procedures

k. Cropping, pesticide, and land management records

Weed Stress/Environmental Herbicide Fate Laboratory
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Approved by: S. A. Clay, SD Field Research Director sac 3/27/14
S. Miyazaki, Regional Field Research Coordinator Sm 4/8/14

Title: 11.1-11.6

The above SOPs deal with Quality Assurance and have been retired March 17, 2006 due to the fact that IR-4 headquarters now supplies QA. They have been placed in the SDSU SOP archive folder.

Weed Stress/Environmental Herbicide Fate Laboratory
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Brookings, SD 57007

Revision Number: 3.0

Submitted by: Kalyn Brix-Davis
Revised by: Graig Reicks

Submitted Date: May 1, 1993
Revision Date: March 27, 2014

Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 12.1 Disposal of pesticides.

Purpose: To assure that pesticide concentrate, spray solutions, rinse water, and containers are disposed of with minimal environmental contamination and in accordance with federal, state and local regulations.

Scope: All filed and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where pesticides are disposed.

Procedures:

1 Personnel dealing with pesticides disposal should follow current policies and guidelines of their institution (A copy should by attached to the SOPs as An appendix).

2 Where institutional guidelines do not exist, the procedures under SOP 12.2 should be followed.

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 12.2 Guidelines for the disposal of pesticides.

Purpose: To assure that pesticide concentrate, spray solutions, rinse water, and containers are disposed of with minimal environmental contamination and in accordance with federal, state and local regulations.

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance and where institutional guidelines for disposal of pesticides do not exist.

Procedures:

1 Disposal of pesticide concentrate and/or containers.

a. Follow procedures in the protocol. Generally, the pesticide and/or empty containers cannot be disposed of under GLP until the study is completed and the QA unit has signed off. If it is necessary to dispose of the container prior to the end of the study, the Study Director should be consulted.

b. Where possible, the pesticide concentrate and containers should be returned to the registrant or manufacturer. Transportation must be according to all Federal, State, and local laws and regulations.

c. Follow label directions for disposal of the pesticide if option 1.b is not available.

d. If no label directions exist for disposal, arrangements should be made with a licensed waste disposal firm for pickup and disposal of the pesticide and/or the empty containers.

2 Disposal of pesticide rinse water, unused spray solutions and other dilute pesticide waste.

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

b. Dispose of the dilute pesticide waste in the field by adding to the spray tank and spraying on an overplanting of the crop where this procedure does not violate any laws or regulations. All pesticide solutions should be mixed with the intent of limiting the problem of excess solutions.

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 13.1 Safety and health procedures in handling pesticides

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

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

Procedures:

1 Personnel should follow current policies and guidelines of their institution (A copy should by attached to the SOPs as An appendix).

2 Where institutional guidelines do not exist, the procedures under SOP 13.2 should be followed.

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 13.2 Guidelines for handling pesticides safely.

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

Scope: All field and laboratory studies conducted under the direction of S. A. Clay and requiring GLP compliance where institutional guidelines for handling pesticides do not exist.

Procedures:

1 A supply of soap/detergent and water should be readily accessible for cleanup in the case of an emergency.

2 All personal protective equipment and clothing as required by the label or written SOPs should be worn in the handling of pesticides for storage, mixing and application. Emergency personal protective equipment (e.g. coveralls, self-contained breathing apparatus) must be available when handling hazardous pesticides such as restricted use pesticides.

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

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

5 Prior to application, the equipment should be checked to make sure there are no leaks in the pump or tanks, hose connections, or worn spots in the hoses. All spray tanks should have lids. Filling the spray tank should be done carefully so it does not run over. All machinery should be shut down if necessary to adjust or repair any moving parts. Never blow out nozzles, hoses, or clogged lines by mouth. Inspect all pesticide containers for leaks, tears, or holes before handling. Do not mishandle or abuse containers and thereby create hazards and/or emergencies by carelessness.

6 All pesticides should be mixed in quantities which are adequate for the job to avoid excess dilute solutions after the job is completed. Cleanup procedures should be established whereby excess sprays can be safely discarded preferably by spraying the material on An overplanting of the commodity. The equipment should be washed off both inside and outside and all pesticides and pesticide containers should be returned to a storage area immediately after use.

7 At the end of the working day employees who have applied or mixed pesticides should

take a shower and change clothes. Clothing should be washed after the end of the day. In no case should the same clothing, including shoes, be worn on a second day after it has been worn during a pesticide application.

8 A pesticide-treated area, greenhouse, or field should not be entered until adequate time, as specified on the label of the pesticide, has been allowed for the treated area. For persons who regularly handle organophosphates and/or large quantities of carbamates, a cholinesterase level should be determined at least monthly throughout the pesticide application season.

9 Do not permit unauthorized persons in the pesticide storage area.

10 Do not store pesticides next to food, feed, seed, fertilizer, or other articles intended for consumption or use by humans or animals. Do not store food, beverages, tobacco, clothing, eating utensils, or smoking equipment in any area where pesticides are present.

11 Do not drink, eat food, smoke, apply cosmetics, or use tobacco in areas where pesticides are present.

12 Wear unlined rubber gloves while handling containers and mixing or measuring pesticides.

13 Do not put fingers in mouth or rub eyes while working with pesticides.

14 Wash hand thoroughly with soap and water immediately after handling pesticides and, especially before eating, smoking, or using the toilet.

15 The local fire department should be provided with a floor plan of the pesticide storage area indicating where different pesticide classifications are regularly stored. The fire chief should be furnished with the home telephone of the person responsible for the pesticide storage facility.

16 Treated field should be posted with warning signs.

17 Pesticide storage areas should be properly ventilated.

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S. Miyazaki, Regional Field Research Coordinator 4/8/14

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

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

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

Procedures:

1 Notify the Study Director and other interested personnel of the pending audit or review as soon as possible.

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

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

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

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

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

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

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

e. Be certain that all documents pertaining to the study/facilities inspection are available. This would include:

1) Master schedules for both the field research director, Quality Assurance Research Officer and possibly their counterparts at the region and IR-4 headquarters.

2) Study Protocol and Standard Operating Procedures

3) Raw data, correspondence and logs.

4) Training records, CVs etc. of personnel assigned to the study.

5) Appropriate chain of custody documents for samples and freezer logs and storage temperature documentation.

6) Documentation of the characterization of the test substance, receipt and handling.

7) Calibration logs on equipment such as balances and application equipment.

8) Archives or storage of records and logs indicating removal and replacement of documents.

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

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Approved by: S. A. Clay, SD Field Research Director Sac 3/27/14
S. Miyazaki, Regional Field Research Coordinator Gm - 4/8/14

Title: 14.2 Procedures to follow during an EPA inspection.

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

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

Procedures:

1 Greet the inspection team and follow any institutional procedures for signing in. Provide name tags and escort the entire group to a conference or meeting room.

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

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

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

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

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

7 Proceed with the inspection.

a. Provide documents requested and provide explanations needed.

b. Keep notes of observations and of all interviews.

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

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Approved by: S. A. Clay, SD Field Research Director 3/27/14
S. Miyazaki, Regional Field Research Coordinator 4/8/14

Title: 14.3 Procedures to follow after the EPA inspection.

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

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

Procedures:

1. Make sure that all personnel involved in the inspection are present for the closeout conference.
2. If the inspector's comments are in error, call this to the inspector's attention. Remember the close out conference is not the forum for any debate.
3. If you have corrected any problems during the inspection make sure the corrections are so noted in the inspector's logbook
4. Have someone present during the close-out take accurate notes or record the conference on tape if taping is acceptable to the inspectors.
5. Be sure you obtain a copy of the list of documents or other materials that may be taken as exhibits by the inspectors.
6. Debrief management, staff, and the Study Director with an explanation of any problems found.
7. Assign responsibility for preparation of possible solutions to problems and obtain time estimates for implementation.
8. Prepare any replies to the regulatory agency as necessary within a timely basis and keep interested parties such as management and the Study Director informed.