

Washington State University
Environmental and Agricultural Entomology Laboratory

2023 Standard Operating Procedure for Conduct of Field Trials

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F.01.01.17	Format for Writing Standard Operating Procedures	Revision 17	1-23-23
F.01.02.06	General Requirements for the Development and Use of Standard Operating Procedures	Revision 6	2-10-12
F.02.01.09	Organization and Personnel of the Field Test Facility	Revision 9	1-23-13
F.02.02.11	Personnel Requirements	Revision 11	3-16-17
F.02.03.05	Quality Assurance Unit	Revision 5	2-10-12
F.02.04.16	Testing Facilities	Revision 16	4-27-20
F.02.05.01	Responsibilities of Field Research Director	Revision 1	1-23-13
F.03.01.11	Facility Files Maintenance and Operation	Revision 11	4-15-19
F.03.02.09	Copy Certification	Revision 9	12-1-10
F.04.01.05	General Protocol Procedures	Revision 5	1-23-13
F.04.02.08	Master Schedule Maintenance	Revision 8	4-27-20
F.04.03.08	Deviations from Protocol or SOP(s)	Revision 8	1-24-14
F.04.04.05	Correcting Errors and Using Abbreviations	Revision 5	2-06-09
F.04.06.05	Testing Facility Inspections	Revision 5	1-23-13
F.06.01.10	Management and Use of Material Safety Data Sheets	Revision 10	4-27-20
F.07.01.17	Monitoring and Maintenance of Freezers	Revision 17	4-27-20
F.08.01.06	Field Trial Personnel Records and Responsibilities	Revision 6	1-23-13
F.09.01.11	Safety	Revision 11	09-23-18
F.10.01.05	Equipment Inspection and Maintenance	Revision 5	1-21-04
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F.10.03.05	Equipment Failure	Revision 5	1-18-08
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F.11.01.20	Instrument Calibration	Revision 20	3-2-21
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F.11.04.11	Calibrating Broadcast Spreaders	Revision 11	2-20-17
F.11.05.05	Calibrating, Installing, and Using Rain Gauges	Retired	3-2-21
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F.11.07.03	Global Positioning Satellite (GPS) Devices Calibration	Revision 3	2-20-17
F.12.01.12	Plot Layout and Identification	Revision 12	3-09-18
F.12.02.12	Test Substance Receipt and Storage	Revision 12	3-09-18
F.12.03.02	Storing and maintaining Adjuvants or Spray Additive	Revision 2	4-27-20
F.13.01.04	Test System Establishment and Maintenance	Revision 4	1-21-04
F.13.02.10	Collecting Soil Characterization Samples	Revision 10	3-09-18
F.13.03.04	Observing and Reporting Phytotoxicity	Revision 4	4-15-19
F.14.01.05	Calculating the Volume of Spray Solution and Test Substance	Revision 5	3-09-18
F.14.02.12	Measuring Test Substances and Preparing Solutions for Application	Revision 12	2-21-14
F.14.03.04	Applying the Test Substance to the Test System	Revision 4	2-10-12
F.15.01.18	Sampling Raw Agricultural Commodities	Revision 18	4-4-19
F.15.02.10	Cleaning Sampling and Harvesting Equipment	Revision 10	1-22-15
F.15.03.07	Sample Storage and Maintenance	Revision 7	3-16-17
F.15.04.13	Sample Shipment	Revision 13	3-2-21
F.15.05.13	Determining Percent Moisture	Revision 13	3-2-21
F.16.01.09	Disposal of Excess Test Substance and Containers	Revision 9	4-27-20
F.17.01.05	Using Services Which are Not GLP Compliant	Revision 5	1-19-16
F.17.02.05	Recording Raw Data	Revision 5	2-10-10
F.17.03.06	Using and Analyzing Electronically Collected Raw Data	Revision 6	1-21-04
F.18.01.01	Inventory and Transfer of GLP Raw Data and Records to IR-4 Headquarters	Revision 1	4-4-19
F.19.01.03	Post Sampling Crop Destruction	Revision 3	1-15-16
F.20.01.01	Fire Alarms and Fire Extinguishers	Retired	4-27-20
F.21.01.01	Greenhouse Facilities	Revision 1	3-23-22

*All SOPs have been reviewed prior to signing.
SOPs submitted by*



Field Research Director, WSU-EAEL

01 / 24 / 2023

Date

SOPs approved by



Assistant Regional Field Coordinator

01 / 26 / 2023

Approval Date

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Title: Format for Writing Standard Operating Procedures

SOP No.: F.01.01.17

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1. PURPOSE

To establish a format for developing Standard Operating Procedures (SOP)

2. SCOPE

All SOPs for conducting studies/trials

3. PROCEDURES

- 3.1. Use the most recent SOP file as the basis for the new year's SOPs.
- 3.2. Enter the title of the SOP in the header of the form following "Title:"
- 3.3. Number SOPs F.ZZ.YY.XX, where F = Field, ZZ= category number, YY = SOP number within the category (01...n), and XX= the revision number.
- 3.4. Under 1.0 Purpose, briefly describe the reason for writing the SOP.
- 3.5. Under 2.0 Scope, state the area to be covered by the SOP.
- 3.6. Under 3.0 Procedures, clearly describe the procedures necessary to complete the task so that a competent person with some knowledge of the situation can complete the task independently.
- 3.7. Revisions
 - 3.7.1. Indicate the revision in the heading by adding one (1) to the current revision number, the third double digit number in the SOP number.
 - 3.7.2. Revisions may be completed by any member of the field facility who is knowledgeable of the procedure(s) the SOP is addressing.
 - 3.7.3. The original or previous revision must be placed in the retired SOP archive files and replaced by the current revision in the active SOP file.
 - 3.7.4. Each SOP will be reviewed approximately every two years. Review will be documented on Form F0101374. Completed forms will be retained in the SOP file. It may be useful to retain copies of SOPs with reviewer comments until the next review or SOP revision, but it is not necessary to retain the reviews themselves.
 - 3.7.5. Revisions of SOPs should be completed within one month after review, but a review does not necessarily mean a revision will be done.
 - 3.7.6. A "revision" provides a substantive change in the meaning of the SOP. Correcting spelling and formatting errors along with making minor changes that clarify the intended meaning of the SOP do not constitute a revision.
 - 3.7.7. The effective date of revisions will be the date SOPs are approved by IR-4 management.
 - 3.7.8. Retired SOPs will be listed in the index with "Retired" in the revision date for the year it is retired, and may be removed from the index in subsequent years.
- 3.8. SOP Table of Contents (TOC)
 - 3.8.1. The SOP TOC will be included in the front of SOP collections. This TOC will contain the SOP title, number, and revision date.
 - 3.8.2. The SOP TOC will be updated when a new SOP or revision is submitted for approval.
 - 3.8.3. The original copy of the current TOC will be retained with the original copies of the current SOPs.
- 3.9. Forms
 - 3.9.1. Forms can be created and used for logs and data collection as needed. Before use, modified Field Data Book (FDB) pages must first be approved by the Regional Field Coordinator (RFC) or designee. Those forms that have logs covering several studies need to be maintained in the facility file and true copies can be made and entered into the relevant FDB. When forms are not at hand note paper can be used and entered into the FDB. The record can be transcribed into the facility log noting which FDB has the original record.

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Title: General Requirements for the Development and Use of Standard Operating Procedures

SOP No.: F.01.02.06

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1. PURPOSE

To describe guidelines for the development and use of Standard Operating Procedures (SOPs)

2. SCOPE

All SOPs which are written for procedures used at the field facilities.

3. PROCEDURES

3.1. SOPs will be prepared as written instructions for all procedures which are expected to be performed in the same manner each time and which influence the quality and integrity of data generated during studies.

3.2. The format, approval and requirements for use of forms will conform to SOP F.01.01.XX (XX = revision number). SOPs should be written or revised by persons regularly performing the described task(s).

3.3. SOPs are to be followed as written. Any variation is considered as a deviation, which must be dealt with as discussed in SOP F.04.03.XX. When there is a discrepancy between the protocol and SOP, the protocol takes precedence.

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Title: Organization and Personnel of Field Test Facility

SOP No.: F.02.01.09

Page 1 of 1

1. PURPOSE

To describe organizational structure of WSU/EAEL-IR4 Field Research Center personnel

2. SCOPE

Personnel performing work in any regulated study/trial

3. PROCEDURES

3.1. Organizational charts that are used to define relationships are as follows and can be found in the facility files and field log book:

3.3.1. IR-4 communications Flow chart

3.3.2. Western Region Program Organizational chart

3.3.3. Generic IR-4 Organizational Chart for Research Centers.

3.3.4. Organizational Chart for Washington State University Research Center.

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Title: Personnel Requirements

SOP No.: F.02.02.11

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1. PURPOSE

To ensure there are sufficient personnel to complete the study/trial and they are qualified and capable of performing assigned duties

2. SCOPE

All trials or studies conducted according to EPA's Good Laboratory Practice Standards

3. PROCEDURES

- 3.1. Sufficient numbers of properly trained personnel shall carry out the studies/trials efficiently.
- 3.2. A personnel file will be maintained at WSU/EAEL for each individual engaged in or supervising a study/trial by this testing facility. Records will be retained in the facility files after personnel leave employment at EAEL. The file will include, but not be limited to, the following:
 - A job description
 - A signed and dated Curriculum Vitae (CV), reviewed approximately annually and noted, signed and dated on the CV if no updating is necessary
 - Training records
- 3.3. Training will be recorded promptly on updated and completed forms placed in the individual's personnel file. Additionally, training needs to be recorded and kept in the onsite Facility File. Lastly SOP training needs to be recorded and completed before being able to participate in GLP trials. Training can include, but is not limited to procedures, SOPs, GLP issues or regulations, on the job training, and/or formal classes.
- 3.4. The training record shall contain the following information as appropriate:
 - Employee's name
 - Training description
 - Training date
 - Length of training session
 - Employee's signature or initials
 - Instructor's signature when available
- 3.5. Current employees are responsible for updating their job description, CV and training records approximately annually.

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Title: Quality Assurance Unit

SOP No.: F.02.03.05

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1. PURPOSE

To describe the makeup and duties of the Quality Assurance Unit (QAU)

2. SCOPE

All facility activities conducted under EPA's Good Laboratory Practice Standards

3. PROCEDURE

All QAU (Quality Assurance Unit) procedures/activities will be conducted according to and in compliance with the IR-4 QAU SOPs.

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Title: Testing Facilities

SOP No.: F.02.04.16

Page 1 of 1

1. PURPOSE

To document EAEL testing facilities at WSU-IAREC

2. SCOPE

All EAEL office, laboratory and storage space and equipment for the conduct of field trials

3. PROCEDURES

3.1. Documentation of EAEL office, laboratory, and storage space at WSU-IAREC

3.1.1. Office in Hamilton Hall utilized by the Coordinator Integrated Pest Management

3.1.2. Office space in the North building Research Facility occupied by the Field Research Director and assistants.

3.1.3. An area delineated by an 8 ft high cyclone fence utilized for freezer, archive, field cooler, and miscellaneous supply storage located in the Hop Research Facility.

3.1.4. Work room for storage of supplies and small equipment such as backpack sprayers, located about 100 feet south of the Hop Research Facility and 100 ft west of the North build in building 18 room A.

3.1.5. An open canopy for storage of farm equipment, e.g., tractor with airblast sprayer.

3.1.6. Area for storage of test materials in the Pesticide Storage building and another for storage of maintenance pesticides.

3.2. Documentation of equipment

3.2.1. List of current equipment is kept in the WSU/EAEL Facility file

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Title: Responsibilities of Field Research Director

SOP No.: F.02.05.01

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1. PURPOSE

To provide information on the responsibilities of the Field Research Director

2. SCOPE

All trials or studies conducted according to EPA's Good Laboratory Practice Standards

3. PROCEDURES

- 3.1. Ensure that the study is carried out according to an approved protocol.
- 3.2. Ensure that personnel, resources, facilities, equipment, materials, and methods are available as scheduled for the conduct of the project.
- 3.3. Make sure that all personnel conducting the study understand Good Laboratory Practices (GLPs), the protocols and SOPs for the Project.
- 3.4. All deviations and findings reported by the quality Assurance Unit (QAU) are responded to in writing. All deviations from the protocol or the SOPs are communicated to the Study Director and Western Regional Coordinator as soon as possible.
- 3.5. All original raw data, (e.g. Personnel records, Soil maps, soil test results, and plot maintenance records and any original documentation), supporting data summaries and other items connected with the study are copied, the copy retained in a retention file at the WSU/EAEL facility, and the Original sent to IR-4 Headquarters for archiving. Personnel records, Soil maps, soil test results, and plot maintenance records will remain at the WSU/EAEL facility in a retention file.
- 3.6. Maintain on file a current summary of training education and experience and a job description for all personnel engaged in the study.

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Title: Facility Files Maintenance and Operation

SOP No.: F.03.01.11

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1. PURPOSE

To establish procedures for annual facility files operation

2. SCOPE

Personnel files, SOPs, Master Schedules, annual calibration, maintenance and weather data, and temperature records (freezer, chemical storage) supporting pesticide registration

3. PROCEDURES

- 3.1. Maintain the facility files at the Washington State University, Irrigated Agricultural Research and Extension Center under the following conditions:
 - 3.1.1. Lock the gate at all times when the facility files are unattended
 - 3.1.2. Lock the file cabinets, which are to be accessed only as WSU/EAEL Field Research Director.
 - 3.1.3. The file cabinets shall be fire resistant: for GLP FDB held on site prior to submission and current year's facility files.
- 3.2. Material to be maintained in the facility file will include, but not be limited to, the following:
 - 3.2.1. Any original data or information not sent to a study director, e.g. weather data pertinent to more than one trial, temperature records relevant to more than one trial (original data sent in Field Data Books (FDBs) may be referenced to the original in another FDB if both FDBs go to the same sponsor). In all cases when the original is not maintained in WSU/EAEL field archives, the original's location must be referenced on the copy.
 - 3.2.2. Originals of all SOPs, retired or in effect, including original revisions
 - 3.2.3. Personnel records (CV, job description, training records)
 - 3.2.4. Project schedule
- 3.3. Retain all data for the Archive process F18.01.XX
- 3.4. Unless specified otherwise in these SOPs, store records updated on a continual basis, e.g., maintenance or calibration logs, at EAEL field station in an active file or in the "Equipment Maintenance, Repair, and Calibration Log."

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Title: Copy Certification

SOP No.: F.03.02.09

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1. PURPOSE

To standardize procedures for making copies of original documents

2. SCOPE

Copies of all documents related to research studies/trials regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURES

- 3.1. If the document is an inter-study/trial document, a copy will be certified by stamping in red "Exact Copy of Original, or True copy of Original" dating and initialing. If a stamp is not available the words may be written and initialed and dated. A note must be written on the certified copy(s) near the certification stating the location of the original.
- 3.2. A multi-page document will be certified on the title page by writing or stamping "Exact copy of Original or True Copy," dating and signing or initialing and adding a brief description, e.g., "Entire Field Data Book(FDB)" and sending or archiving the copy(s) as appropriate.

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Title: General Protocol Procedures

SOP No.: F.04.01.05

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1. PURPOSE

To establish procedures to be followed prior to experiment initiation

2. SCOPE

All studies/trials regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURES

- 3.1. Prior to the first application of the test substance to the test system by WSU/EAEL personnel, a copy of the protocol, signed by the study director, must be on site.
- 3.2. Each protocol, whether written in-house or received from outside sources, shall include as a minimum the following information:
 - Descriptive title
 - Statement of the purpose of the study
 - Name, address, and phone number of the sponsor, study director, and testing facility
 - Test substance name, CAS number, or code number
 - Experiment's proposed start and termination date
 - Specific identification and justification for selection of the test system
 - Description of the experimental design
 - Statistical methods
 - Application information (treatments, rates, timing, pre-harvest interval, etc.)
 - Data and/or sample collection(s), and measurements to be made
 - Residue sample inventory and handling
 - Sample identification, preparation, storage, and disposal
 - Protocol amendments
 - Dates and signature of the study director and approval of the sponsor

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Title: Master Schedule Maintenance

SOP No.: F.04.02.08

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1. PURPOSE

To ensure that a master schedule is developed and maintained using current information

2. SCOPE

All studies that involve a commitment of WSU/EAEL facilities or personnel

3. PROCEDURES

- 3.1. The master schedule will be printed from the IR-4 website.
- 3.2. In addition to the project number and test substance, the schedule will include at least the test system, nature of study, initiation date, sponsor identity, study director, and current status.
- 3.3. The master schedule will be reviewed approximately monthly and updated as projects are initiated or completed.
- 3.4. Updates will be made from individual study schedules or by communication with the principal investigators of research not requiring study schedules.
- 3.5. Master schedules may be maintained in an active file and archived either annually or monthly.

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Title: Deviations from Protocol or SOP(s)

SOP No.: F.04.03.08

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1. PURPOSE

To define procedures for documenting deviations from SOPs and protocols

2. SCOPE

All studies/trials regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURES

3.1. Any procedure or event that differs from the approved protocol or SOP(s) is considered a deviation. If there is conflict between the protocols and SOP(s), the protocol takes precedence and is not considered an SOP deviation.

3.2. All deviations must be reported to the study director by phone, Email, or other means of personal communication preferably within 24-48 hours of detection and followed with a deviation report sent to the study director within five working days of discovery.

3.3. A copy of the original or a certified copy of the deviation report shall be entered in the Field Data Book.

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Title: Correcting Errors and Using Abbreviations

SOP No.: F.04.04.05

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1. PURPOSE

To establish procedures for correcting errors or using abbreviations when recording or compiling raw data

2. SCOPE

All information supporting pesticide marketing and registration

3. PROCEDURES

3.1. If an error is made, mark out the incorrect entry with a single line.

3.1.1. Write the correct entry above, adjacent to or below the incorrect entry.

3.1.2. Initial and date the corrected entry

3.1.3. Write the reason for the error or use an error code from the list below and circle the code:

AW Accidental Write over

CE Calculation Error

EE Entry Error

IC Incorrect Comment

IE Illegible Entry

IW Inappropriate Word

LE Late Entry

ME Measurement Error

NA Not Applicable

NR Not Recorded

PE Pagination Error

SP Spelling Error

TE Transcription Error

UE Unnecessary Entry

WE Wrong Entry

FE Footnote Entry (for extended explanations; must appear on the page requiring the explanation). If more than one footnote is needed for a page, number the FE sequentially with a subscript (example FE₂).

3.2. Abbreviations from the following list may be used when appropriate:

XD Missing data (data requested is appropriate but not available)

UNK Unknown

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Title: Testing Facility Inspections

SOP No.: F.04.06.05

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1. PURPOSE

To describe the procedures to be followed before, during and after an on-site testing facility inspection by a sponsor or regulatory agency.

2. SCOPE

All official site inspections at WSU/EAEL except those for human health and safety, animal welfare or use of controlled substances.

3. PROCEDURES

3.1. Notification:

- 3.1.1. WSU/EAEL will usually be notified, orally or in writing, of the intent to inspect. Notification should include the names and phone numbers of inspectors, the date of inspection and the area of inspection.
- 3.1.2. Notify the study director and sponsor of inspections of their studies by outside agencies.
- 3.1.3. The inspection dates must not interfere with the conduct of the study to be inspected or other research activities.
- 3.1.4. Written inspection notifications will be retained.

3.2. Inspection:

- 3.2.1. An WSU/EAEL contact appropriate to the areas to be inspected will be designated. The contact person will schedule the inspection with other WSU/EAEL personnel, escort the inspectors, and keep written records of inspection activities.
- 3.2.2. The nature of information to which inspectors have access is specified by the regulatory authority under which the inspection is held.

3.3. Inspection Completion:

- 3.3.1. At the end of the inspection, the WSU/EAEL contact may request a verbal summary of the inspection, and should request that a written report also be sent to WSU/EAEL.
- 3.3.2. The WSU/EAEL contact will record any pertinent observations, including problems or deficiencies noted recommendations, suggestions, and indications of the need for additional inspections.
- 3.3.3. The WSU/EAEL contact will communicate the inspection information to appropriate personnel at the facility, and develop a plan for implementing appropriate recommendations or suggestions.
- 3.3.4. All records pertaining to the inspection will be retained.

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Title: Management and Use of Material Safety Data Sheets

SOP No.: F.06.01.10

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1. PURPOSE

To describe the management, access, reading and interpretation of Material Safety Data Sheets (MSDSs) aka SDSs.

2. SCOPE

All persons working on field studies/trials; all chemicals and reagents received, stored or used by field personnel.

3. PROCEDURES

3.1. Management of MSDSs/SDSs

- 3.1.1. Upon receipt of all chemicals and reagents, note the presence or absence of the material's MSDS or other documentation such as the COA (certificate of Analysis). If any documentation is not present, notify the SD (study director) right away either by phone or email and request the missing documentation. Note in communications that the request was made of SD.
- 3.1.2. Ensure MSDSs for chemicals and reagents used in the field are available to personnel at the field site in a field notebook.
- 3.1.3. Approximately annually, archive a copy of the MSDSs for chemicals no longer in use by WSU/EAEL personnel nor present at WSU/EAEL facilities or field sites.

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Title: Monitoring and Maintenance of Freezers

SOP No.: F.07.01.17

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1. PURPOSE

To describe procedures to ensure that WSU/EAEL field freezers are operating correctly, and to document difficulties when they occur

2. SCOPE

All freezers at EAEL field facility in which samples are stored

3. PROCEDURES

3.1. Identification and location

3.1.1. Sample Freezers maintained by the WSU/EAEL field facility; one designated as Freezer #3 is for storage of untreated samples. A second designated Freezer #5, is for storage of treated samples. The freezers are properly signed as designated above.

3.1.2. The freezers are located in the Hop Research Facility in a locked cage in the southeast corner of the room labeled Growth Chambers, and/or also located at the basement of the office in the North Building, which is only accessible by the Field Research Director and study personnel, WSU-IAREC, 24106 North Bunn Road, Prosser, WA 99350.

3.2. Freezer maintenance

3.2.1. Maintenance operations, such as repairs, defrosting freezers and decontaminating freezers, will be performed by personnel assigned as necessary by the Field Research Director.

3.2.2. Maintenance of freezers, except as noted in 3.2.1, is to be performed by physical plant or outside service personnel.

3.2.3. Two Hobo Temp Loggers will be placed in each freezer, each set to record 30-60 days.

3.2.4. The Hobo Temp loggers will be offloaded approximately every 30 days but at staggered intervals so the 2 loggers in a freezer will be offloaded at least one week apart.

3.2.5. After offloading a graph of the data will be printed, the graph initialed and dated and placed in the active file.

3.2.6. Freezer temperatures will be maintained at generally less than 0°F (-18 °C). Temporary spikes resulting from addition or packing of samples are acceptable.

3.2.7. The freezers will be locked at all times when not attended.

3.2.8. A tube approximately half full of water, frozen and inverted will be placed in each freezer as an indicator of amount of thawing in the event of a malfunction.

3.2.9. A SensaphoneWEB600 alarm system with data logging capabilities will be used for calling out due to Power shortages or high temperatures and as an additional backup for temperature monitoring and graphing. This unit will be verified approximately annually and monthly backup/downloaded in the same fashion as the hobo units. In the event of any maintenance or updates to the monitoring system, the unit will be re-tested to confirm it is working properly.

3.2.9.1. Turn off one freezer for the cleaning process (Untreated first then Treated). Sensor verification can be done at the same time or at a different time. Freezers are typically empty of samples for several months at this time of the year.

3.2.9.2. Note on freezer maintenance log if the alarm for both main and backup sensors were activated and called out.

3.2.9.3. Note system callout time.

3.2.9.4. Temporarily turn off the alarm activation (call out cycle) and finish cleaning.

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- 3.2.9.5. Restore or reactivate alarm settings once the freezer units have been cleaned. Note that the alarm stops calling out when the temperature is at or below 0°F for a minimum of 30 minutes.
- 3.2.9.6. Once the temperature is restored to both freezer units, the Sensaphone WEB600 will be unplugged from the outlet and allowed to go through a power outage alarm cycle. Again a minimum of 30 minutes is needed for callout.
- 3.2.9.7. Print outs for both graph & recorded sensor Temp/Time/power/battery backup points will be made and placed into the facility file for the Testing of the alarm system for temperature and power outages. Note in Sensaphone log the testing of the alarm system which includes both temperature alarms and power outage alarms. Also include verification that personnel on the alarm notification list received the notifications and record the time and date they were received by each.
- 3.2.9.8. Around the first or second week of each month, the SensaphoneWEB600 system will be downloaded and saved as a file denoting date and month for the unit. The data will be graphed and printed out as well as the recorded sensor temp/time points. The top page of these printouts will be initialed and dated and placed into the facility file. This is a back up to the Hobo temperature units used to monitor the freezer temperature. These sheets will be kept on site and not sent in at the end of the year with the seasonal facility files for calibration and maintenance of sensor and other equipment.

3.3. Malfunctions

- 3.3.1. If the failure is due to the unit not working properly and the delay for repair is lengthy, transfer samples to alternative storage moving the monitoring hobos with samples for temperature tracking.
- 3.3.2. If electrical power failure is the cause, attempt to determine when power will be restored. Monitor temperatures closely and if temperatures reach 0 °F (-18 °C) for over an hour, move the samples to alternative storage or obtain another power source such as a generator.
- 3.3.3. Alternate storage may include ice chests with dry ice, an alternate unit or an off-site storage facility. It is important to keep or run new hobo's with the samples to track temperature range of samples
- 3.3.4. Note the conditions of the contents. Notify appropriate study or research directors if temperatures possibly damaging to the contents have occurred.
- 3.3.5. Update sample tracking logs to indicate changes in sample storage locations and the failure of the prior storage unit.

3.4. Documentation

- 3.4.1. Record all maintenance and other operations on "Equipment Maintenance, Repair and Calibration Log Form." For any defects in operation, indicate the nature of the defect, how and when the defect was discovered, and the remedial action taken in response. Indicate when any of the actions taken did not follow this SOP.
- 3.4.2. Archive maintenance logs along with any temperature printouts or traces from recording devices at least annually according to F18.01.XX. Temporary filing in the facility file or active file is acceptable.

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Field Trial Personnel Records and Responsibilities

SOP No.: F.08.01.06

Page 1 of 1

1. PURPOSE

To define responsibilities for permanent and temporary personnel

2. SCOPE

All personnel who work on field trials

3. PROCEDURES

- 3.1. All personnel who make applications to the test system, record entries in field trial notebooks or records, collect specimens or perform all or part of a critical phase during a field trial will sign the signature page in the field trial notebook.
- 3.2. Temporary or part time employees whose duties are not associated with a critical phase of the trial (e.g., weeding, irrigating) are not required to sign the personnel page. Supervised temporary or part time employees under the direct supervision of qualified personnel (e.g., Principal Field Investigator or designee) need not sign the personnel page.
- 3.3. All personnel who sign the signature page of a Field Data Book are certifying they have read and understand the protocol, SOPs and MSDSs associated with the trial in which they are engaged.
- 3.4. Included in each Field Data Book will be a certified copy of the CVs of personnel listed on the signature page of that Field Data Book. The signed and dated original CVs (including training records) will be retained in the personnel files.

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Safety

SOP No.: F.09.01.11

Page 1 of 1

1. PURPOSE

To define procedures for safely handling pesticides in the field and for safely conducting field trials

2. SCOPE

All field study/trial personnel

3. PROCEDURES

- 3.1. A MSDS/SDS will be included in the "Test Substance" section of a Field Data Book taken to the field.
- 3.2. All personnel who handle pesticides must read and sign a training record for the SOP, "Management and Use of Material Safety Data Sheets."
- 3.3. All personnel who handle pesticides must read, understand, and follow the directions and information found in the MSDS/SDS for the pesticide being used.
- 3.4. By signing the personnel page of a Field Data Book, personnel who handle pesticides are signing a statement that they have read and understand the label, if available, and MSDS for the test substance.
- 3.5. All personnel who handle pesticides (test substance or maintenance) will wear the appropriate personal protection equipment (PPE) (i.e., respirator, impermeable gloves, resistant coveralls, impermeable boots, etc.) as indicated on the label and MSDS/SDS.
- 3.6. A portable eyewash or sufficient clear water will be carried to the field and be readily available when handling pesticides.
- 3.7. Prior to making an application, the user will check all sprayer hoses, connections and fittings for leaks and other conditions that can cause ruptures.
- 3.8. All personnel who handle pesticides will have had training for the safe handling of pesticides or work under the direct supervision of experienced, trained personnel. Training documentation will be retained as specified in the SOP, "Personnel Requirements."

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Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Equipment Inspection and Maintenance

SOP No.: F.10.01.05

Page 1 of 1

1. PURPOSE

To ensure all inspection and maintenance procedures performed are properly recorded

2. SCOPE

All equipment used in field trials

3. PROCEDURES

- 3.1. All equipment inspection and maintenance (e.g., cleaning, oil change, filter replacement, hose replacement, etc.) will be performed by the user or qualified designee and performance will be recorded on an inspection and maintenance form appropriate to that piece of equipment.
- 3.2. If the maintenance performed is a non-routine repair the records shall document the nature of the defect, how and when the defect was discovered and the remedial action taken to repair the defect. If a piece of equipment is found to be unrepairable, it will be disposed of and so noted on the form.
- 3.3. The inspection and maintenance form will include the following:
 - Serial number or other identifying number or characteristic for the piece of equipment.
 - Date
 - Name or initials of person(s) performing inspection and/or maintenance
 - Inspection and/or maintenance performed
- 3.4. Completed inspection and maintenance forms for field equipment will be kept in the Equipment Maintenance, Repair and Calibration file, the contents archived approximately annually.

Washington State University
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Standard Operating Procedure for Conduct of Field Trials

Title: Application Equipment Maintenance

SOP No.: F.10.02.10

Page 1 of 1

1. PURPOSE

To establish procedures for application equipment maintenance

2. SCOPE

Application equipment used in field trials

3. PROCEDURES

3.1 Prior to the start of the field season, application equipment will be inspected, cleaned and tested for proper operation. Such maintenance shall be the responsibility of the field investigator or designee and will be recorded in the appropriate maintenance log.

3.2 Immediately following a pesticide application, it shall be the responsibility of the person making the application to clean the application equipment as follows:

3.2.1. Rinse application equipment and test material reservoir with clean water, disposing of rinsate by applying to crop, or field road, well away from the untreated plots or by other lawful means.

3.2.2. Add sufficient clean water and ammonia, detergent or a commercial cleaning agent to the reservoir, agitate, and operate the application equipment.

3.2.3. Flush the system a third time with enough clean water equivalent to at least 20% of the reservoir volume.

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Standard Operating Procedure for Conduct of Field Trials

Title: Equipment Failure

SOP No.: F.10.03.05

Page 1 of 1

1. PURPOSE

To define procedures to follow in the event of equipment failure

2. SCOPE

Equipment being used on a field study/trial regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURES

3.1. In the event of equipment failure during work on a study/trial take the following actions.

3.1.1. Immediately stop all activity.

3.1.2. Remove the equipment from the plot area doing as little damage to the plot as possible.

3.1.3. Flag the area in the plot affected by the equipment failure.

3.1.4. Determine the extent of damage to the plot.

3.1.5. Contact the study director as soon as possible with all available information to determine if the trial can be salvaged or if action must be taken to initiate the trial in another location.

3.1.7. Show the location of the plot damage on the plot diagram and report details in the Field Data Book if the trial is to be salvaged.

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Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Borrowed or Rented Equipment

SOP No.: F.10.04.03

Page 1 of 1

1. PURPOSE

To describe the proper use and documentation for acquired rental/borrowed equipment for use in GLP studies

2. SCOPE

Applies to all research trials conducted through the Washington State University, IRAEC WSU/EAEL IR-4 Field Research Center.

3. PROCEDURES

- 3.1. Contact the appropriate source for the equipment to be borrowed or rented.
- 3.2. Determine whether there is an operation manual available for the equipment. Request a copy of the manual when it is available.
- 3.3. Document the following information for Borrowed or Rented equipment
 - Owner or source
 - Description of Equipment, E.g. Type, make, model and serial number where possible.
 - Year manufactured and or acquired (when possible).
 - Purpose (what the equipment will be used for).
 - Study Identification (field ID, chemical, crop for trial) FDB page label will work.
 - Condition upon receipt e.g., good, needed repair.
 - Maintenance performed (when applicable)
 - Modifications performed (when applicable)
 - Cleaning/decontamination procedures performed.
 - FRD statement of suitability for use
 - Date of use
 - Time the procedure was initiated and completed
- 3.4. Person entering the information must initial and date entries
- 3.5. The above information is maintained in the Facility Files and a certified copy placed in the FDB. A copy of the manual, when available, will also be kept in the facility files.-.

Title: Instrument Calibration

SOP No.: F.11.01.20

Page 1 of 2

1. PURPOSE

To define procedures for Verifying instruments used in field studies/trials

2. SCOPE

All measurement instruments used in conducting field studies/trials

3. PROCEDURES

- 3.1. Instrument calibrations shall be the responsibility of the field investigators and staff.
- 3.2. All thermometers and temperature recorders will be verified approximately annually by one of the following methods:
 - Comparing temperature readings from the thermometer or temperature recorder with the temperature registered on a NIST calibrated traceable thermometer at temperatures covering the intended use range of that device (typically freezer low, room temperature and small or large drying oven).
 - For freezer maintenance and alarm thermometer covering the freezer low: Put the thermometers in the freezer and verify freezer thermometer against a NIST calibrated traceable thermometer at the normal operating temperature of approximately (-10F to -20F). Compare these temperatures across at least three different time points.
 - For high temperature: 160°F-180°F, run the hop dryer or either of the dryers used for determining dry matter. Then record any deviations.
- 3.3. Any thermometer will be discarded and replaced if it is inaccurate by more than 5 °F (2.8 °C) and cannot be adjusted.
- 3.4. Record thermometer verifications in "Thermometer Verification Log (TVL)" or other lined or unlined paper and transferred to the TVL, noting and verifying the transcribed data and keeping the original record in the facility file.
- 3.5. Electronic temperature recording devices, e.g., Hobo Temp electronic recorders, will be verified as thermometers.
- 3.6. Wind gauges will be purchased new every year and verified as needed by comparing readings to those of previous wind gauges to check that the readings are reasonably similar. Verification checks may be recorded on, "Equipment Maintenance, Repair and Verification" or other paper.
- 3.7. Hydrion paper or other pH paper will be used to conduct application carrier pH test.
- 3.8. The field balance weight set FBW-3, will be verified approximately annually by one of the following methods:
 - Using a balance with a traceable calibration less than one year old, record the nominal weight and weight reading from the balance in the calibration file. If available, copy and retain the certification records
 - Sending to the manufacturer, vendor, or other qualified business for verification and entering the certification in the calibration file
 - Comparing with other standardized weight sets with traceable records
- 3.9. Balances and scales used for weighing will be calibrated approximately annually by one of the following methods:
 - Using a set of field weights with a traceable verification less than one year old. Record the nominal weight reading from the balance in the calibration file. If available, copy and retain the certification records.
 - Have balances certified approximately annually by third party service.
 - Sending in to manufacture for calibration and certification.
- 3.10. Balances used for weighing test material shall be calibrated immediately prior to each use as follows:
 - Obtain the target weight from the calibration calculations

Title: Instrument Calibration

SOP No.: F.11.01.20

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- Select the proper container for the material being weighed, place it on the balance and, using the tare feature, zero the balance or weigh the container and subtract from the balance reading
- Using calibration weights above and below the target weight, check and record the balance reading
- Make adjustment for any inaccurate readings
- If a balance is found to be off by more than 1% and cannot be adjusted, it must be repaired by competent personnel, adjusted by the manufacturer, or discarded and replaced
- Test substance weights of less than 1.0 grams use a scale with 0.01g accuracy or mix enough solution to require over 1.0 grams of test substance
- Record on the appropriate "Balance Calibration Log," or other paper.

Title: Sprayer Calibration

SOP No.: F.11.02.11

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1. PURPOSE

To establish procedures for calibrating sprayers

2. SCOPE

All applications of a test material using a sprayer

3. PROCEDURES

3.1. The sprayer calibration shall be the responsibility of the field investigators or GLP trained staff.

3.2. The sprayer will be calibrated prior to each test substance application. If an application of a test substance is repeated one or more times within the same working day, and none of the equipment or settings is changed, repeating the entire calibration procedure (see below) is not necessary. However, a verification must be run by catching one output from the nozzles and verifying the rate is within $\pm 5\%$ of the full calibration. Remember to clean the unit thoroughly according to SOP F10.02.xx before catching output. These data entries must be recorded in the Field Data Book

3.3. Check equipment in the following manner prior to the calibration routine:

- The equipment is appropriate for the job
- There is no visual damage such as broken hoses or lines, loose or broken connections, broken pressure gauges, etc.
- There are no leaking connections.
- Sprayer attachments are correctly mounted on the vehicle (if applicable)
- Pressure is constant
- Nozzles and screens are appropriate for the delivery of the desired spray volume, droplet size and spray pattern

3.4. Adjust line pressure to desired setting using the pressure regulator or bypass valve depending on the type of sprayer pressure source.

3.5. Using the following steps, determine the average nozzle output:

3.6. Catch the output from each nozzle in a cup for a minimum of 15 seconds (preferably 30 seconds) and measure in a graduated cylinder

- Repeat three times and record the amounts in the field trial notebook
- Compute the average volume for each nozzle, for each replication, and an overall average
- Using the overall average, calculate the values for $\pm 5\%$
- If all averages do not fall within this range, rearrange, replace, clean or unplug the nozzles and/or screens, or if the inconsistency is in the replications, repeat above steps until all nozzle outputs are within $\pm 5\%$ of the overall average.
- If calibrating airblast sprayers by nozzle output: note range for nozzle sizes $\pm 5\%$ of the average of multiple sized nozzles. Single nozzle outputs do not require $\pm 5\%$ average records.

- Example: 200 psi at 2300 rpm nozzle 15 sec discharge and average

- Top

- Nozzle 1 size 4 630 ml

- Nozzle 2 size 4 600 ml avg 615ml ($\pm 5\%$) Range 584ml to 646ml

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Title: Sprayer Calibration

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- Nozzle 3 size 3 480 ml
- Nozzle 4 size 3 460 ml avg 470ml (\pm 5%) Range 447ml to 494ml
- Nozzle 5 size 2 270 ml
- Nozzle 6 size 2 270 ml avg 270ml (\pm 5%) Range 257ml to 284ml
- Nozzle 7 size 1 190 ml no average necessary
- Bottom
- This shows if nozzles are in line with each range and the intended spray pattern for airblast sprayer is correct.(more detail is better.)

3.7.Using the following steps, determine the ground speed:

- Walk or drive the spray vehicle over a known distance (usually the length of the plot to be sprayed) in the same conditions as for the actual spraying, while measuring the elapsed time with a stop watch. If using an engine driven spray vehicle (e.g. tractor) note the tachometer and/or throttle setting and gears and record in the field trial notebook.
- Repeat above three times and determine the average to use for calculations. All passes must be within \pm 5% of the average. If not within this range, take corrective action and repeat as above. Corrective action may be anything from practicing walking until consistency is obtained to changing spray vehicles.

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Standard Operating Procedure for Conduct of Field Trials

Title: Calibrating Drop Spreaders

SOP No.: F.11.03.08

Page 1 of 1

1. PURPOSE

To establish procedures for calibrating drop spreaders

2. SCOPE

All applications of test material using a drop spreader

3. PROCEDURES

- 3.1. The drop spreader will be calibrated by the field investigators or the GLP trained staff making the application prior to each test material application. If an application of a test substance is immediately repeated (same day) one or more times, repeating the entire calibration procedure (see below) is not necessary. However, the calibration must be verified by catching output from the spreader and verifying the rate as being within $\pm 5\%$ of the full calibration. These data must be shown in the Field Data Book where complete calibration information is normally found.
- 3.2. Check equipment in the following manner prior to the calibration routine:
 - Wheels rotate freely without obstruction
 - Ensure the agitator rotates freely
 - Check that the agitator baffle opens and closes properly
- 3.3. Adjust the agitator baffle opening to the desired setting.
- 3.4. Determine the average output from the spreader by walking a measured distance (at least 25 feet) while pushing the spreader, catching the dropped granules, and measuring the weight collected on a balance.
- 3.5. Repeat procedure 3.4 three times and calculate the average output from the three tests. If each test is not within $\pm 5\%$ of the average, make adjustments and repeat.
- 3.6. Enter calibration details in the WSU/EAEL Field Data Logbook and enter the calibration on, "Equipment Maintenance, Repair and Calibration Log Form."

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Title: Calibrating Broadcast Spreaders

SOP No.: F.11.04.11

Page 1 of 2

1. PURPOSE

To establish procedures for calibrating broadcast spreaders

2. SCOPE

All applications of test material using a broadcast spreader

3. PROCEDURES

3.1. The broadcast spreader will be calibrated by the field investigators or GLP trained staff prior to each test material application. If an application of a test material is immediately repeated the same date one or more times, repeating the entire calibration procedure (see below) is not necessary. However, the calibration must be verified by catching output from the spreader and verifying the rate as being within $\pm 5\%$ of the full calibration. These data must be shown in the Field Data Book where complete calibration information is normally found.

3.2. Check equipment in the following manner prior to the calibration routine:

- Ensure the impeller rotates freely without obstruction
- Ensure the drop holes are free from obstruction
- Check that the flow gage lever on the flow gage opens and closes properly.

3.3. Adjust the flow gage lever to desired setting.

3.4. Determine the application band width by turning the broadcast spreader crank at the desired revolutions per minute (RPM) and measuring the distance the material is broadcast to the right and left of the applicator. Add the distances to the right and left of the applicator to get the full band width. Complete the procedure three times and average. All tests must be within $\pm 5\%$ of the average.

3.5. Determine the average output from the broadcast spreader by turning the broadcast spreader crank at the desired RPM for 30 seconds, catching the granules in a large plastic bag placed around the broadcast spreader, and measuring on a balance the weight collected. Or, put a known amount of test material or placebo in the spreader, crank at the desired RPM for a known amount of time and weigh the remaining test material or placebo. Subtract the second measurement from the first to determine the rate per unit time. Adjust slide gates on bottom of bin to regulate flow of material until desired rate is obtained. Complete the procedure three times and average. If all tests are not within $\pm 5\%$ of the average, the process must be repeated.

3.6. Using the following steps, determine the ground speed:

- Walk a known distance (usually the length of the plot receiving the application) in the same conditions as the actual spreading, while measuring the elapsed time with a stop watch.
- Complete the above procedure three times and calculate the average for use in succeeding calculations. All passes must be within $\pm 5\%$ of the average. If not within this range, take corrective action and repeat above.
- If the walking speed is too fast for the applicator, reset the lever to a lower setting and repeat all of the above steps for the new setting.
- Record details of the application in the WSU/EAEL Field Data Logbook entering record on, Equipment Maintenance, Repair and Calibration Log Form.

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- 3.7 Alternate method for application would be to weigh out and record between 100-105% of the test substance for the plot. On the lowest setting that the material can be applied through the hand-held broadcast spreader make an application around the perimeter of the plot directing the test substance into the plot. Then make equally spaced passes up and down the length of the plot then back and forth across the width until all test materials are evenly applied. If there is remaining material then repeat the process of going up and down the length and back and forth across the width until all test materials are evenly applied.
- 3.8 For test substance that are large in nature a shaker bottle with holes drilled in the top can be used to sprinkle or scatter the material over the plot.
- When using the shaker, the plot can be divided into 4 quadrants. The test substance is split in to four equal amounts, one for each quadrant to make application of the larger pelleted test substances easier to apply more uniformly.

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Standard Operating Procedure for Conduct of Field Trials

Title: Bucket Calibration

SOP No.: F.11.06.03

Page 1 of 1

1. PURPOSE

To assure proper calibration of plastic buckets or carboys used in measuring carrier

2. SCOPE

All GLP trials that require use of larger volumes of carrier material

3. PROCEDURES

3.1. The calibration of this equipment is the responsibility of the field investigators or GLP trained staff.

3.2. Using a carpenter level, find a level surface for supporting the bucket or carboy to be calibrated, e.g. a floor or table. Place the bucket on the level surface and check across the top of the bucket for level.

3.3. Carefully measure 3,785 ml water (1 gal) into the bucket using graduated cylinders.

3.4. Shine or hang a light inside the bucket to make the water meniscus show clearly from the outside of the bucket.

3.5. Using a Sharpie carefully mark a line over about 2" of the line made by the meniscus on the outside of the bucket or carboy. Put another line on the opposite side of the bucket in the same manner.

3.6. Add 3,785 ml water again and repeat 3.4 at the 2 gal level and so on until the 5 gal level has been marked.

3.7. Empty and dry the bucket then shining a light from the outside mark lines on the inside of the bucket over the outside lines now visible through the sides of the bucket.

3.8. Using the Sharpie, label each line 1 gal, 2 gal, etc.

3.9. Record the date of the calibration on the bucket and initial.

3.10. Assign a number to the bucket or carboy and record the calibration

1. PURPOSE

This standard operating procedure is for use by research personnel to ensure that Global Positioning Satellite (GPS) devices are operated and maintained properly so that they will give an accurate location reading. GPS units are used to mark permanent positions of plot.

2. SCOPE

Applies to use of GPS receivers for mapping plot locations and related features of trials conducted according to Good Laboratory Practice (GLP) Standards

3. PROCEDURES

- 3.1. Standardization: The calibration of this equipment is the responsibility of the field investigators or GLP trained staff.
 - 3.1.1. Check the GPS device accuracy approximately every 12 months and record information in the appropriate maintenance log
 - 3.1.2. Using the GPS Essentials Android App, record and export a waypoint in KML format. Use an outside location with a clear view on Google Earth (such as buildings, water pond, telephone poles, etc.)
 - 3.1.3. Start Google Earth and import the KML sent from the smart phone. Use the Google Earth "Ruler" application to determine the difference between the known Google Earth and your phone's KML waypoint. The difference in these two measurements is the relative accuracy of the phone GPS recorder.
 - 3.1.4. Repeat the same procedure with three different timings in the day and/or three different timing in three different days. Record all the information in the GPS verification Log.
 - 3.1.5. If the measured location varies from the known coordinates of the location by more than 30 feet, the GPS receiver should not be used.
- 3.1. Use:
 - 3.2.1. Use GPS Essentials to record waypoints as needed.
 - 3.2.2. Regularly update Android phone with current operating system version and any updates to GPS Essentials apps when notified by smart phone.
- 3.3. Maintenance:
 - 3.3.1. Routine maintenance is required for the GPS receiver, such as replacing new batteries.
 - 3.3.2. Regularly update Android phone with current operating system version and any updates to GPS Essentials apps when notified by smart phone. If the batteries become weak or dead, replace them per operating instructions.
 - 3.3.3. If the device is damaged or stops, replace it with another smart phone.
- 3.4. Documentation
 - 3.4.1. Record maintenance and accuracy verification procedures in the GPS receiver log. Note the SOP that was followed and if the operation was routine or non-routine. If the operation was non-routine, i.e. done due to a failure or malfunction, describe the nature of the defect, how and when it was discovered, and any corrective actions taken. Remember to initial and date all entries.

1. PURPOSE

To ensure consistent plot identification and to ensure a construction that can be relocated.

2. SCOPE

All field studies/trials

3. PROCEDURES

3.1. Plot construction will conform to the following specifics:

3.1.1. Field Crops plot size The minimum plot size will be sufficient to produce, by best estimates, at least 2X the necessary sample size without sampling within 2 ft. of any edge of the plot.

- Orchard, vineyard crops and bush or cane crops plot size: The minimum plot size will be six(6) trees, vines or plants in a row sufficient to produce, by best estimates, at least 2X the necessary sample size without sampling the plants at either end of the plot.
- The buffer area between the treated and untreated plots will be sufficient to prevent any contamination of the untreated plot with the test substance through movement from the target area.
- The untreated plot will be located uphill and upwind (prevailing according to conventional wisdom of the area) from the treated plot(s) if possible.
- The plots will be tied to a permanent marker, i.e., building corner, power pole or other reasonably permanent marker. A diagram of the plot layout will be entered in the Field Data Book and include at least the plot dimensions, head of the plot (the end of the plot with the field id marker) distances between plots (buffer), row direction, plant and row spacing, slope, distance to permanent marker and north direction.
- The test site location will be entered in the field trial notebook either by a diagram showing the nearest town or other prominent landmark or by a map of the area.

3.2.1. Plot identification shall conform to the following specifics:

- Field crops:
 - Facing the head of the plot as designated on the plot diagram, a metal sign holding stake or other appropriate marker will be driven into the soil at the left hand corner of the plot and two wire stakes with flags will be placed as close as possible to the stake. Any additional signage may be added to further identify the material being trialed and the plots.
 - At minimum a wooden stake and at least one of the flags will be labeled using a permanent marker with the trial number/FID# and plot/subplot designations.
 - Wire flags will be placed at the other three corners of the plot(s)/subplot(s).
 - Plot numbers will consist of two digit treatment number e.g. Trt # 02
 - If any of the replicate numbers, treatment numbers or plot plan do not agree, the plot will not be disturbed until the error is found.
 - If GPS is used to locate plot corners or centers, a minimum two corners or two center points for each plot must be recorded.
- Orchard, vineyard crops, and bush or cane crops:
 - Facing the head of the plot as designated on the plot diagram, At minimum, a metal sign holding stake or other appropriate marker will be driven into the soil

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Title: Plot Layout and Identification

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- at row center of the plot and two wire stakes with flags will be placed as close as possible to the stake.
 - At minimum, a wooden stake and at least one of the flags will be labeled using a permanent marker with the trial number/FID# and plot/subplot designations
 - Two wire flags will be placed at the other row end of the plot(s)/subplot(s).
 - Plot numbers will consist of two digit treatment number e.g. Trt # 02.
 - If any of the replicate numbers, treatment numbers or the plot plan does not agree, the plot will not be disturbed until the error is found.
 - If GPS is used to locate plot corners or centers, a minimum two corners or two center points for each plot must be recorded.
- Greenhouse Facilities
 - When facing the head of the plot on the left hand side a minimum of metal sign holder will be stuck in a pot or the corner of a raised bed. This with two white flags to visually denote the untreated plot and two flags colored other than white will help to visually denote the treated.
 - At a minimum a wooden stake and at least one flag will be labeled using permanent ink and two wire flags will be placed at the other row end of the plot(s)/subplot(s).
 - Two wire flags will be placed at the other row end of the plot(s)/subplot(s).
 - If GPS is used to locate plots, a minimum of one point can be used to designate the greenhouse.

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Standard Operating Procedure for Conduct of Field Trials

Title: Test Substance Receipt and Storage

SOP No.: F.12.02.12

Page 1 of 1

1. PURPOSE

To define procedures for receiving and storing test and reference substances

2. SCOPE

All test and reference substances received for field trials to be conducted under EPA's Good Laboratory Practices Standards

3. PROCEDURES

- 3.1. Test substances arriving will be checked immediately for damage to shipping cartons. If cartons are damaged, immediately inspect for breakage, spillage or leakage. If any spillage or leakage is found, initiate cleanup procedures and notify the study director.
- 3.2. Record the following information on the chemical storage log, other form provided, or other paper
 - Name* (exactly as shown on container label), Chemical abstracts service (CAS) number or code number*
 - Formulation
 - Date received
 - Condition on arrival
 - Source of expiration date, i.e., label, receipt/packing slip, etc.
 - Amount received
 - Storage location
 - Name of shipper
 - Signature of the receiver
 - Date signed
 - Container description
 - MSDS/SDS present or absent (if absent, must obtain)
 - Bill of Lading/Waybill or Tracking # (include certified copy)
 - Number of containers
 - Batch/lot number*
 - Expiration date*
 - Storage instructions*
 - GLP Status from COA* (Certificate of Analysis)
- 3.3. The items in 3.2 of this SOP (marked with an *) must be on the container label. If they are not on the label when received, write the information on the label or add a label with the information to the container.
- 3.4. Storage temperature data recorded by a recording thermometer will be downloaded at approximately monthly intervals and a hard copy original placed in the facility file.
- 3.5. If recording thermometers are used, e.g., Hobo Temps, a main recorder and backup will be used and downloaded at staggered intervals at least one week apart.

Title: Storing and Maintaining Adjuvants or Spray Additive

SOP No.: F.12.03.02

Page 1 of 1

1. PURPOSE

To define procedures for receiving and storing adjuvants or spray additives for use in IR-4 studies

2. SCOPE

All adjuvants or spray additive substances purchased or otherwise received for field trials to be conducted under EPA's Good Laboratory Practices Standards

3. PROCEDURES

- 3.1. All adjuvants or spray additives for IR-4 studies will meet GLP labeling requirements for reagents which shall include but not be limited to: name, concentration, lot or batch number (if shown), storage conditions and expiration date (if known) and purchase date or opened date.
 - 3.1.1. Secondary containers are permitted for storage (e.g. a 1 gallon container subdivided into 30 or 60 mL containers for ease of use and transport to remote sites) but must be properly labeled as per the original container and now take on all the requirements and properties of an "original container".
 - 3.1.2. Items to be included in labeling are: name, concentration, lot or batch number (if known), storage conditions and expiration date (if known) and purchase date or opened date.
- 3.2. Spray additives used in IR-4 studies will be stored in the GLP test substance locker that has limited access and is temperature monitored.
- 3.3. If spray additives do not have an expiration date or one cannot be obtained from the manufacturer, they will be assigned an expiration date 5 years from the date of purchase. Noted on both the main container and all secondary storage containers. (Example: purchased in 2015 has a 2020 expiration date.)
- 3.4. Spray additives will be in good condition prior to use i.e. the physical characteristics of the additive should not have changed from purchase or be compromised (i.e. different color, consistency (cloudy, darkened) or have the appearance of rancidity).
- 3.5. Spray additives must be handled in a manner to prevent cross contamination with test substances and spray additives. Suggested options are provided below,
 - 3.5.1. Spray additives will be dispensed into a labeled container (such as a 30 or 60 mL wide mouth Nalgene bottle) prior to being used in a GLP residue trial. The spray additive once dispensed will not be used for a different trial or returned to the original or secondary container; the contents will be labeled for this trial and application and remain with the test substance. When the test substance is returned or removed from inventory then the adjuvant may be discarded appropriately.
 - 3.5.2. Spray additives are dispensed from the original spray additive container using a factory sealed syringe or by pouring directly into the container. After this syringe is used it is discarded and never used again. This syringe never returns to the spray additive container. The test substance is also dispensed by a different newly opened syringe, discarded after use.
 - 3.5.3. The critical element in both these examples is: No "double-dipping" into an original or secondary container. No measuring device will be placed directly into a spray adjuvant original or secondary container and then directly into a spray tank or container intended to hold GLP test substance and revisit the spray adjuvant container. Other methods that prevent double dipping into the original or secondary containers are also acceptable.

Washington State University
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Standard Operating Procedure for Conduct of Field Trials

Title: Test System Establishment and Maintenance

SOP No.: F.13.01.04

Page 1 of 1

1. PURPOSE

To establish procedures for test system establishment and maintenance

2. SCOPE

All test systems being established and/or maintained for studies/trials

3. PROCEDURES

- 3.1. Consult a reasonably current publication or an expert on production of the test system being studied.
- 3.2. Select regions/sites with soil and environmental characteristics typical of those in commercial production for the test system.
- 3.3. Establish and maintain test systems using practices similar to those of commercial growers, provided these practices are within the parameters of the protocol.
- 3.4. When possible, obtain approval from the study director prior to applying maintenance pesticides. If the study director is not available, contact the sponsor or the residue research director at the performing laboratory.
- 3.5. Pesticide use must be limited to those which will not conflict with chemical analysis for the test substance.

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Collecting Soil Characterization Samples

SOP No.: F.13.02.10

Page 1 of 1

1. PURPOSE

To define procedures for collecting soil characterization samples

2. SCOPE

All studies/trials requiring soil characterization samples

3. PROCEDURES

- 3.1. Soil characterization samples will be collected from trial sites approximately every three years, unless the protocol stipulates otherwise.
- 3.2. A soil probe at least 0.75 in. in diameter, shovel or other tool may be used to take sub-samples of soil to a depth of 12(±2) in.
- 3.3. Collect ten sub-samples from the trial site, preferably five from the untreated plot and five from the treated plot; composite the sub-samples, mix and place the soil in a pre-labeled soil sample bag or similar container.
- 3.4. Send the sample to a qualified soils laboratory for characterization requesting analysis for pH, percent organic matter, percent silt, clay, and sand, texture and cation exchange capacity (CEC).
- 3.5. When the report is received, it becomes part of the raw data and is included in the Field Data Book.
- 3.6. In the event that a soil sample is not collected, a soil characterization report from previous trials may be used (Or the Web Soil Survey from NRCS) if the trial site is the same and samples were collected in the manner described above and analyzed as in 3.4 above. If certified copies are available rather than originals, copies of the copies may be used and certified.

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Observing and Reporting Phytotoxicity

SOP No.: F.13.03.04

Page 1 of 1

1. PURPOSE

To establish procedures for observing and reporting phytotoxicity

2. SCOPE

All trials conducted according to Good Laboratory Practice (GLP) standards

3. PROCEDURES

- 3.1. Within three weeks after an application of the test substance to a test system, unless otherwise detailed in the protocol, look closely at the test system paying close attention to any distortion (twisting, turning, curling, drooping), any discoloration or burning, or spotting of leaves or any other unusual appearance of the plants. If in question, contact a crop expert and ask their opinion.
- 3.2. In the event that an aberration is noted that may be related to the test substance, carefully write a description of the appearance including part(s) of the plant affected, color and distortion(s). Compare the affected plants with healthy plants in the non-treated plots.
- 3.3. If possible, photograph the affected parts of the plants and healthy plants for comparison. Submit the photos with the field data notebook.
- 3.4. Contact the study director, sending photos (if available) and a description via email.
- 3.5. If the trial goes to completion, attempt to estimate the effects on yield if possible.
- 3.6. If ratings are required by the protocol:
 - 3.6.1. Review the protocol to determine the method and timing of collecting phytotoxicity data. If no method is cited, follow the procedure below:
 - 3.6.2. When possible, record phytotoxicity data at an appropriate time after the application
 - 3.6.3. Observe all plots and rate the phytotoxicity on a scale of 0 to 100%. Zero percent = no phytotoxicity and 100% = completely dead. The rating between 0 and 100% indicates the degree of injury expressed as stunting, necrosis, chlorosis, leaf deformation, etc.
 - 3.6.4. Document the use of other rating scales.

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Standard Operating Procedure for Conduct of Field Trials

Title: Calculating the Volume of Spray Solution and Test Substance

SOP No.: F.14.01.05

Page 1 of 1

1. **PURPOSE**

To ensure accurate calculations of spray volume and test material

2. **SCOPE**

All field studies/trials

3. **PROCEDURES**

3.1. The exact method of calculating the volumes of carrier, adjuvant and test substance will be the choice of the field investigator. However, whichever method is selected, all calculations must be shown clearly in the Field Data Book. To minimize errors, have the calculations verified by other personnel if possible.

3.2. The number of significant figures will be determined by the measuring device; answers can be no more accurate than the least accurate figure.

3.3. When rounding numbers, 1, 2, 3, and 4 will be rounded down; 6, 7, 8, and 9 will be rounded up; 5 will be rounded to the nearest even number.

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Standard Operating Procedure for Conduct of Field Trials

Title: Measuring Test Substances and Preparing Solutions for Applications

SOP No.: F.14.02.12

Page 1 of 1

1. PURPOSE

To ensure accurate and consistent measurement of test substances and preparation and mixing of the spray solutions

2. SCOPE

All applications of test substances

3. PROCEDURES

- 3.1. Wear protective clothing and safety apparatus as indicated in the MSDS/SDS or on the label of the test substance.
- 3.2. The graduations of all measuring devices should be accurate within 1% of the total volume or weight capacity of the measuring device.
- 3.3. Preparing spray solutions using granular, powdered, or viscous liquid preparations of the test substance (all preparations of test substances may be measured using this procedure):
 - Calibrate a balance according to SOP F.11.01.XX (XX=revision number) paragraph 3.10, "Instrument Calibration."
 - Tare the selected sample container, e.g., small cup, bottle, or bag, and measure the desired amount of test substance into the container
 - Measure the carrier (usually water) into the spray tank or mixing bucket, retaining sufficient carrier to rinse the sample container three times
 - Add the test substance, rinse the sample container and pour rinsate into the spray tank or mixing bucket
 - Add the remaining carrier to the spray tank or mixing bucket
 - If using an adjuvant, follow the mixing instructions (if any) on the adjuvant label.
 - Agitate or stir as necessary
 - If using a mixing bucket, pour into the spray tank
- 3.4. Preparing spray solutions using emulsifiable concentrates or liquid preparations of the test substance:
 - Measure the carrier (usually water) and pour 50% to 95% into the spray tank retaining a sufficient amount in a separate container to rinse the measuring device three times (syringes need not be rinsed)
 - Using a graduated cylinder, pipette, micropipette, or syringe, measure and dispense the test substance into the spray tank or mixing bucket.
 - If using an adjuvant, follow the mixing instructions (if any) on the adjuvant label.
 - Rinse the measuring device three times (graduated cylinders, pipettes and micropipettes need to be rinsed three times as well, syringes need not be rinsed) with some of the retained carrier material, add all the carrier material to the spray tank or mixing bucket and agitate as necessary
- 3.5. Proceed with the application according to SOP F.14.03.XX (XX=revision number), "Applying the Test Substance to the Test System."

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Standard Operating Procedure for Conduct of Field Trials

Title: Applying Test Substance to the Test System

SOP No.: F.14.03.04

Page 1 of 1

1. PURPOSE

To ensure consistent applications of the test substances

2. SCOPE

All applications of test substances

3. PROCEDURES

- 3.1. Consult the MSDS/SDS and/or label for the test substance and wear the appropriate protective clothing and apparatus.
- 3.2. Use a properly calibrated applicator (e.g., sprayers, spreaders, etc.):
 - Ensure that the holding tank contains sufficient material to make at least one pass on the treated plot
 - Check all connections, hoses, clamps and apparatus to ensure proper functioning of the applicator
 - When using sprayers, charge the lines with solution by spraying outside the plot area until all nozzles are functioning properly and the spray pattern is correct
 - Make sure the applicator is properly aligned with the plot, the height is correct, and begin traveling toward the boundary of the plot
 - Arrive at the boundary of the plot receiving the application at the appropriate speed
 - If using a sprayer or drop spreader, activate the applicator as it crosses the plot boundary. When using a broadcast spreader activate the spreader prior to crossing the plot boundary to ensure the edge of the plot receives the proper amount of test material
 - Continue to operate the applicator until crossing the boundary line on the other end of the plot
 - Stop applying test substance.
 - Verification of the application rate can be accomplished by timing each pass with a stop watch, record in the field trial notebook and calculate the rate from calibration data.

Title: Sampling Raw Agricultural Commodities

SOP No.: F.15.01.18

Page 1 of 2

1. PURPOSE

To ensure contamination-free sampling techniques

2. SCOPE

All field studies/trials in which agricultural commodities are collected according to EPA's Good Laboratory Practice Standards

3. PROCEDURES

- 3.1. Protective clothing and equipment, e.g., coveralls, respirator, gloves, will be worn as indicated by the MSDS/SDS, label, or protocol.
- 3.2. The untreated plot(s) will be sampled before the treated plot(s). Treated plots will be sampled from lowest rate(s) progressively to highest rate(s).
- 3.3. Disposable gloves will be changed between treatments. If it is not practical to wear gloves while sampling, hands must be washed with soap and water or alcohol prior to sampling each treatment.
- 3.4. Tools and equipment used to collect samples must be cleaned according to SOP F.15.02.XX prior to sampling and between treatments.
- 3.5. Collected specimens will be placed in pre-labeled residue bags (if more than one residue bag is used per sample, the bags must be marked 1 of n...x of n).
- 3.6. In addition, with a sharpie or similar pen, write the following on the outside of the bag: Field ID Number, TRT Number, and Sample ID in case the tag is lost during shipping.
- 3.7. For crops that are dried prior to sampling, e.g., seed crops, one of the following procedures may be used.
 - 3.7.1. The crop may be mowed or windrowed and left in the field to dry or if allowed by the protocol, the crop plant material may be collected, spread on clean tarps or placed in pre-labeled burlap bags or laid out on screens, in a well ventilated heated or unheated, protected area for drying. An estimate of moisture content needs to be done before sampling.
 - 3.7.2. If the samples are for hay, the dried plant material will be sampled for moisture content and then placed in residue bags. If the plant material is for seed, it may be combined or thrashed and winnowed by hand, then cleaned by screening or by using a seed cleaner. Fractions, as requested by the protocol, may then be placed in residue bags.
 - 3.7.3. When sampling hop cones:
 - Prior to harvesting and drying, a hand or mechanically picked test sample will be run in the dryer to estimate the dry matter present in cones. Divide the dry sample weight by 0.9 to give you a weight with a 10% moisture content. Use this as a target weight ($\pm 2\%$) for the day of harvest. If doing multiple trials with different varieties, each variety needs to have an estimate done.
 - The day of harvest spread a new tarp on the ground or trailer by the bines to be taken. Use tarp for Trt#01 and collect control first, then collect Trt#02. New tarps will be used for each collection if there are additional treatments or decline trials with several harvest dates except the day of the control harvest in which a single tarp can be used to harvest the untreated plot then the treated plot.
 - Using a large knife or machete, sever the base of the bines and pull it from the overhead cable so that it lands on the tarp, doing the best you can to make sure that the bine lands on the tarp.
 - Move the tarp to the next selected bines and repeat the procedure until the required numbers of bines have been collected for the plot, being careful to keep edges up and not walking on the tarp.

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Title: Sampling Raw Agricultural Commodities

SOP No.: F.15.01.18

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- Wrap the tarp and tie tarp edges around the bines and load them on a trailer for transport to the WSU-IAREC commercial style hop (cone) picker.
 - Run the picker for several minutes while blowing out any debris from previous pickings. Making sure all screens and belts are clean, proceed with running the bines through the picker to dislodge the cones.
 - Collect the cones into clean 55 gallon or comparable containers. To collect multiple samples from one set of bines (for example, samples A and B from the untreated hops), use separate containers. Alternate collecting into each container several times while the hop picker is running to collect representative samples.
 - Once the cones have been collected, run the picker for several minutes more and blow out all loose material and making sure all screens and belts are as debris free as possible.
 - Take the bag of hop cones to the hop kiln at WSU-IAREC and, after mixing the cones in the bag(s), place the cones to be dried into the properly cleaned screen bottom boxes (untreated and treated samples may be dried concurrently using the down draft or drawn through kiln (the heated air is drawn through the samples and expelled outside). It is preferable to dry the samples at different times whenever possible running the untreated samples first then the treated.
 - Start the kiln by:
 - 1) turning on the propane at the tank near the southwest corner of the hop kiln building
 - 2) throwing the main electrical switch on the electrical panel on the kiln to the ON position
 - 3) turning the blue handled ball valve on the gas line near the burner parallel with the gas line
 - 4) turning the switch on the Burner Control panel to ON and pressing the red button
 - 5) the temperature should oscillate between 140°F and 160°F
 - 6) setting the timer for about 2 ½-3 hours depending on test samples.
 - In the case of equipment failure in both single harvest and decline studies on hops the cones may also be removed by hand from the bines collecting sufficient cones from inside, outside, upper and lower portions of the bines directly into collection bags or drying bins giving a good homogenous sampling of the cones present to meet the sampling size.
 - In the case of decline studies: The plot size needs to be increased by the addition of six harvestable hills of hops for every sampling date to have enough bines so the hop picker can be used for each sampling to eliminate the difference in harvest method.
 - When running decline treatments make at least one run with 6 or more hop bines that have not been treated with the test substance between samplings to help clean the belts. Then remove loose debris by running the picker for five minutes and blowing out before next sampling.
- 3.8. Samples will be put into field coolers containing ice, blue ice, or dry ice for transport to frozen storage pending shipment. Samples may be carried directly to the freezers if the elapsed time from collection site to freezers is less than 60 minutes. All samples will be placed in frozen storage within four hours of collection. When anticipating more than one hour of transport time a hobo needs to be included to monitor temperature.
- 3.9. Enter the following in the Field Data Book: A description of the sampling procedures, sample numbers, sample type, approximate weight or quantity of units (e.g., fruits, spears, tubers, etc.), and the elapsed time between sampling and placing the samples in frozen storage.

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Standard Operating Procedure for Conduct of Field Trials

Title: Cleaning Sampling and Harvesting Equipment

SOP No.: F.15.02.10

Page 1 of 1

1. PURPOSE

To describe procedures for cleaning harvesting and sampling equipment

2. SCOPE

All field studies/trials

3. PROCEDURES

- 3.1. Hand tools shall be cleaned prior to sampling and between treatments.
 - Tools may be cleaned by spraying with acetone, allowing runoff and then air drying or wiping dry with a clean paper towel.
 - Tools may be cleaned by washing in a solution of water and cleaning agent, rinsing and then air drying or wiping dry with a clean paper towel.
- 3.2. Cleaning larger equipment prior to harvest such as combines, tractors, Wolf hop picker, belt thresher, etc. may be accomplished by vacuuming or using air blower or compressed CO₂ to remove loose debris and/or washing with cleaning agent and water or with a pressure washer using water and cleaning agent.
- 3.3. Cleaning in between harvest of treated materials on large equipment like the Wolf hop picker or the belt thresher may be done by blowing out unit using air or compressed CO₂ and keeping it dry for the harvest of the next sample. Running additional clean material through the unit maybe done as well prior to blowing the unit out.
- 3.4. When using larger equipment such as combines or potato harvester it is not necessary to clean the equipment between samples if:
 - Moving from non-treated to treated samples
 - Sufficient non-treated crop material is run through the machine prior to collecting the sample to serve as a cleaning agent (an estimate by visual observation or actual measurement of this amount of non-treated crop must be reported in the Field Data Book).

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Title: Sample Storage and Maintenance

SOP No.: F.15.03.07

Page 1 of 1

1. PURPOSE

To state the procedures for sample storage and maintenance

2. SCOPE

All field samples stored

3. PROCEDURES

- 3.1. When possible, all samples will be placed in frozen storage pending shipment within four (4) hours of collection. If transport to the freezers is over one hour, sufficient blue ice will be carried to the field to cool the samples in the field coolers until they can be placed in frozen storage. If samples cannot be placed in the freezer within approximately one hour, a temperature monitoring device should be included in the cooler, and temperatures recorded in the field data notebook.
- 3.2. All samples placed in or removed from the freezer shall be noted in the freezer log. Recording samples removed for short periods of time, e.g. packing for shipment, is not necessary.
- 3.3. Untreated and treated samples will be physically separated.
- 3.4. A tube approximately half full of water will be frozen, then inverted and maintained in the freezer to indicate the amount of thawing in the event of freezer failure.
- 3.5. The freezer maintenance and monitoring shall conform to SOP F.07.01.XX.

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Standard Operating Procedure for Conduct of Field Trials

Title: Sample Shipment

SOP No.: F.15.04.13

Page 1 of 1

1. PURPOSE

To establish guidelines for shipping samples

2. SCOPE

All samples to be shipped

3. PROCEDURES

- 3.1. Packaging shall be sturdy and sufficiently insulated to maintain sample integrity throughout the shipping process.
- 3.2. Individual samples will be placed in plastic bags, preferably plastic lined cloth residue bags or equivalent.
- 3.3. If dry ice is used, it should not touch plastic bags during shipping because the bags may become brittle and rupture. Please follow the IR-4 advisory #2007-01 for calculating dry ice necessary to ship treatment.
- 3.4. Untreated and treated samples are typically packed in separate shipping containers. If the samples are small enough that both the control and treated samples will fit in a single shipping container then:
 - Untreated and treated samples will be double bagged and individually labeled.
 - Place treated double bagged samples in the bottom of the shipping container.
 - Place untreated samples in the upper portion of the shipping container.
 - Multiple trials shipping together will be packed in separate shipping containers unless otherwise approved by the SD in writing, or in the protocol.
 - Multiple treatments may be double bagged and placed in the same shipping container unless otherwise directed by SD in writing or the protocol.
- 3.5. When shipping samples to IR-4 labs, a single box should not weigh more than approximately 50 pounds.
- 3.6. As soon as possible after collection, samples will be shipped by the most expedient means to the facility designated in the protocol.
- 3.7. Frozen samples will be shipped overnight by a traceable courier service, e.g., Federal Express, or by a freezer truck, e.g., ACDS, or by hand if the location is convenient.
- 3.8. If overnight shipment is used, follow these steps to avoid problems if samples are lost:
 - Pack samples in sufficient dry ice to keep them frozen for at least two days.
 - Ship samples on a Monday or Tuesday so they may be recovered before the weekend.
- 3.9. Samples may be shipped directly from the field to the testing facility provided the samples are packed in sufficient dry ice to freeze the samples and maintain them frozen until they arrive at the destination following the IR-4 advisory #2007-01.
- 3.10. Transmittal forms must be sent with each sample shipment. A certified copy of the transmittal form must be sent to the study director and the original placed in the field trial notebook.
- 3.11. The study director, laboratory director or other designated person will be notified of the shipment preferably by email or phone call and noted in the comments log for that trial.

Title: Determining Percent Moisture

SOP No.: F.15.05.13

Page 1 of 2

1. PURPOSE

To establish procedures for determining moisture percentage in plant material

2. SCOPE

When required by protocol

3. PROCEDURES

- 3.1. Equipment: air dry oven, calibrated balance capable of reading 0.1 gram,
 - If using air dry oven, calibrated balance with the reading 0.1 grams, foil plate or container, use 3 - 100 gram sample minimums and set the oven for 160-180°F. Run for 1 hour and check sample weight, repeat process till there is less than a 1 % (0.01) change in the sample weight.
 - If determining percent dry matter. Test samples can be put in the drying oven overnight at approximately 150-180°F. Actual temperatures during drying may fluctuate. Weigh the samples before placing in the oven and record the weight. Measure the sample weight 30 mins -1 hour after starting, then allow to dry overnight. Weigh the sample in the morning and record the weight. Take one additional weight at minimum of 30 minutes to prove stability of sample weight. Samples typically only need 4 hours to completely dry down. The test samples do not need to be dried for additional periods to see if there is further weight change.
- 3.2. When anticipating the harvesting of hop cones, collect at least one- 5 lb sample (preferably four- 5 lb samples for averaging estimated dry wt.) at 0-5 days prior to scheduled harvest. Clean the drying box and weigh the sample(s) to be dried in a drying box(es). Weigh the sample box and record its weight and box number, tare the scale to that box and add sample to ~5 lbs weight and record. Set the box and sample in the WSU building #60 hop dryer for a normal drying period of approximately 2 hours. Start noting sample weight approximately every 15-30 minutes till there is less than a 1 % change in weight (0.8 oz). This will give a reasonably accurate estimate of dry matter present for the future harvested samples. Divide the dry sample weight by 0.92 to calculate an 8% moisture content target weight, divide the dry sample weight by 0.88 to calculate a 12% moisture content target weight. Hops need to be dried to ~10% ($\pm 2\%$) moisture content to simulate commercially acceptable dried hops. A typical harvest uses four- 5 lb samples of which two are selected for the trial sampling. It will probably be necessary when weighing the final sample to temporarily place the sample into a new 13 gallon trash bag or sample bag. Then weigh the empty drying box to get the final wt for calculating the actual sample wt since the wood drying boxes lose some moisture weight during the drying process. When drying additional samples at the designated time points for decline studies, dry at least one of the additional two samples down to approximately 0% moisture content. This parallel sample will be used to verify the estimated % moisture content of the current samples. If this sample's dry matter percentage has changed more than 5% from the original pre-sample, use this sample's dry matter percentage to calculate the moisture content target weight range for the next designated sample time point.
- 3.3. If a sample has been dried down past the 8% moisture content or if the sample needs to be removed from the dryer prior to cooling, it can be left in the temporary new 13 gallon trash bag with the top open. This is to allow the dried hops to absorb moisture from the surrounding air to bring up the sample wt. to the appropriate target range, or to allow it to cool to ambient temperature. Over dried samples may need to equilibrate for up to 24 hours. An alternate means would be to run a second set of samples either from materials

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Title: Determining Percent Moisture

SOP No.: F.15.05.13

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left from the day's picking or collect a new set of bins for that day or the next and rerun the samples, checking the weights more often.

For example:

A 5lb.(80 oz., or ~2268g) sample, dries down to 17.6 oz.(520.4g) dry matter, thus the % moisture and % dry matter present is as follows:

$$(80\text{oz} - 17.6\text{oz})/80\text{oz} * 100 = 78\% \text{ starting moisture content}$$
$$17.6\text{oz}/80 \text{ oz.} * 100 = 22\% \text{ dry matter}$$

We are looking for a sample end weight that has a range of 8-12% moisture content. The easiest method for calculating this is by dividing the dried sample by 0.92 this will give you an estimated wt. of the sample at 8% moisture content. For the other end of the range divide by 0.88 this will give you an estimated wt. of the sample at 12% moisture content

If we have a dry wt. of 17.6oz.divided by 0.92 (for 8% moisture content target wt.) this will equal a target sample wt. of 19.13 oz.

$17.6\text{oz} / 0.92 = 19.13 \text{ oz. target weight for } 8\% \text{ moisture content.}$

If we have a dry wt. of 17.6 oz. divided by 0.88 (for 12% moisture content target wt.) this will equal a target sample wt. of 20.0 oz.

$17.6\text{oz} / 0.92 = 20.0 \text{ oz. target weight for } 12\% \text{ moisture content.}$

This will give us a target range of 19.13 oz.(565.6g) @8% moisture content to 20.0 oz. (591.4g) @ 12%moisture content.

Record pre-sample target weight range and study sample weights to verify procedure and that RAC samples reached within sample target weight moisture content range.

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Title: Disposal of Excess Test Substance and Containers

SOP No.: F.16.01.09

Page 1 of 1

1. PURPOSE

To ensure proper disposal of excess test substance, reference substance (including solutions) and containers

2. SCOPE

All test substances, reference substances and containers used in the field trials

3. PROCEDURES

3.1. When practical, return all excess test substances or reference substances to the sponsor in their original containers.

- Return test substances, reference substances and/or containers only at the discretion of the study director
- Retain the test material containers in the chemical storage at least until the study completion date (date the final report is signed by the study director) unless otherwise directed by the study director. Study completion can be confirmed by contacting the Study Director or the Regional Field Coordinator, or by searching the IR-4 web site Test Substance Container Disposal Approval link under the Food Crops Database Search Options.

3.2. Apply excess test or reference substances according to label directions. Dispose of excess test or reference substances according to Federal/State laws.

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Title: Using Services Which Are Not GLP Compliant

SOP No.: F.17.01.05

Page 1 of 1

1. PURPOSE

To ensure documentation of use of non-GLP compliant services

2. SCOPE

All field studies/trials regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURES

3.1. In the Field Data Book, document use of non-GLP compliant services, e.g., weather data from NOAA or Washington Public Agricultural Weather System, field and chemical use history obtained from a grower, etc.

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Title: Recording Raw Data

SOP No.: F.17.02.05

Page 1 of 1

1. PURPOSE

To establish procedures for recording raw data

2. SCOPE

All field studies/trials regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURES

- 3.1. All raw data shall be recorded in ink, preferably in permanent blue ink.
- 3.2. Entries in the Field Data Book will be recorded within 24 hours of the activity or noted as late entry.
- 3.3. Any data, copies, or originals, added to the Field Data Book shall be marked with the study/trial number to guard against misfiling loose pages.
- 3.4. Date and sign each page of the Field Data Book when entries are made. If the page is not completed, each entry must be dated and initialed at the time of the entry.

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Using and Analyzing Electronically Collected Raw Data

SOP No.: F.17.03.06

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1. PURPOSE

To establish procedures for using data from electronic devices

2. SCOPE

All field trials/studies regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURES

- 3.1. Ensure the correct program is available and workable for the application desired.
- 3.2. Test and calibrate or verify sensing and data collecting devices according to manufacturer instructions and/or SOP F.11.01.XX and record the procedure(s)/data in the calibration log.
- 3.3. Record all instrument parameters at the beginning of the run.
- 3.4. Download and generate hard copies of all data as soon as possible after collection.
- 3.5. Transfer all data to archives as soon as practical (hard copies).
- 3.6. Each hard copy must be signed or initialed and dated by the operator.
- 3.7. The first printout of the data is defined as the original, raw data.

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Inventory and Transfer of GLP Raw Data and Records to IR-4 Headquarters

SOP No.: F.18.01.01

Page 1 of 1

1. PURPOSE

To ensure that all original raw data generated under GLP are safely maintained at the IR-4 Headquarters Archive.

2. SCOPE

All field trials/studies regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURE

3.1. The official archive will be located at IR-4 Headquarters. Copies of raw data will be made and retained at the EAEL facility.

3.2. The procedure for transferring current raw data to the archive at Headquarters will be as follows:

3.2.1. *As Field Data Books are completed, they will be copied and the originals forwarded to the IR-4 Western Region Office.*

3.2.2. At the end of the current field season, after the last study has been completed, the originals that have been used for all current studies (forms that go in all field data notebooks or apply to multiple studies) will be compiled. Original items, such as freezer check-in/check-out log, scale calibration, yearly verification for chemical storage and freezer thermometers, and yearly verification for instruments that record environmental information will be included. This may also contain any other original supporting data, or other documentation, generated during the course of studies for the current season. These originals will then be sent to IR-4 Headquarters for archiving approximately every 1-2 years as time is available. Data required in the Field Data Books will be copies from the originals. A chain of custody form will be generated and an inventory of all data pages sent to headquarters. On the form, document the identity of the courier and the tracking number. The transfer should be by certifiable mail or some other traceable mechanism (e.g. FedEx). A copy of this Archive Inventory will be forwarded to the Western Region IR4 office at UC Davis.

3.3. A "retention file" will be maintained at EAEL. Access to this file is restricted to personnel involved in IR-4 studies. The EAEL facility retention file may contain the following:

3.3.1. Copies of all Field Data Books from the immediate past year. (Note: notebooks from previous years are stored in a safe, dry area at the facility.)

3.3.2. True copies of original data.

3.3.3. Signed original SOP

3.3.4. Supporting data associated with the facility

3.3.5. Personnel records and training information

3.3.6. QA facility inspection reports

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Post Sampling Crop Destruction

SOP No.: F.19.01.03

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1. PURPOSE

To ensure that a crop sprayed with an unregistered pesticide (test substance) does not enter the channels of trade and is inaccessible for human and animal consumption.

2. SCOPE

All field trials/studies regulated under EPA's Good Laboratory Practice Standards

3. PROCEDURE

- 3.1. Research personnel will decide, on a case-by-case basis, the best method for crop destruction. The criteria considered includes: accessibility of the crop to the public, wildlife, and/or livestock; the grower/cooperator involved in the trial; the feasibility of pursuing other options; and time/labor/funding issues.
- 3.2. When a study is undertaken at a grower/cooperator site, the grower/cooperator and the researcher must have an understanding about what will be done with the treated crop for which no tolerance has been established. In most cases, the grower will harvest and dispose of the contents of the treated crop in a manner that ensures it will not be consumed. In addition, at the discretion of the Field Research Director, the treated crop may be harvested and destroyed by IR-4 personnel or, possibly, just rendered unharvestable.
- 3.3. For trials conducted at EAEL or other University property, a different set of circumstances exists. These plots are considered off-limits and non-accessible to uninformed personnel or the public. They are under close supervision of IR-4 personnel; flagging and signage clearly mark the trial plot locations. At EAEL and other University sites, it will be up to the discretion of the FRD to determine how best to dispose of excess treated crop.
- 3.4. Whether a plot is at a grower field or on university property, include in the Field Data Book an explanation of how the crop was destroyed and the date of destruction or why no crop destruction was necessary.
- 3.5. In either case if a crop destruct fee is assessed by the University or a grower, a P.O. number must be acquired from the accounting office prior to the crop destruct being billed. This must accompany the invoice for the crop destruct.

Washington State University
Environmental and Agricultural Entomological Laboratory
Standard Operating Procedure for Conduct of Field Trials

Title: Greenhouse Facilities

SOP No.: F21.01.01

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1. PURPOSE:

Greenhouse facilities for trials

SCOPE: All greenhouse studies/trials regulated under EPA's Good Laboratory Practice Standards

2. PROCEDURES:

3.1. Greenhouse Facilities

- 3.1.1. Greenhouses should be large enough to contain an entire trial or portion of a trial with sufficient distance between the UTC and TRT plots to prevent contamination. To further prevent contamination when untreated and treated plots are in the same greenhouse, a plastic curtain can be installed up to the eaves of the greenhouse dividing the greenhouse in half.
- 3.1.2. If conducting more than one trial at a time in the greenhouse there should be adequate distance between the TRT plots to prevent contamination. When two treated plots are in close proximity to each other, the plot not receiving an application can be tented with clear plastic that is a minimum of 2 mil in thickness. The plastic will be removed from the plants once the spray solution has dried on the plot receiving the application. Usually this is a period of approximately 30 minutes.
- 3.1.3. Temperature and humidity should be uniform at the trial sites within the greenhouse(s) to allow for uniform plant growth throughout the greenhouse. This is especially important if the greenhouse is divided in half with a barrier. When the greenhouse is divided in half with a barrier, electronic temperature and humidity monitoring devices will be placed in each half of the greenhouse to record daily temperature and humidity levels inside the greenhouse. These devices will be contained inside an aspirated box. Monitoring device data will be downloaded approximately once a month when trials are being conducted in the greenhouse.
- 3.1.4. Greenhouse should be equipped to allow temperature, humidity, moisture and fertilization to be maintained that closely simulates commercial greenhouse production conditions.
- 3.1.5. The walls, floors and ceilings of the greenhouse should be maintained in good condition. The floors and aisles should be well drained and kept clear of any plant debris, weeds or unused equipment.
- 3.1.6. At the time of study applications, all fans/coolers will be shut off and all vents closed. These items will remain in this condition until the spray application has dried on the plants that were sprayed. Usually this is a period of approximately 30 minutes.
- 3.1.7. Once an application has been made to the treated plot, further activity within the greenhouse(s) shall proceed from the UTC plot to the TRT plot.
- 3.1.8. After each test substance application in the greenhouse, signs will be posted at entry points to the greenhouse and any shared plenums with the following information: pesticide, rate, date applied, re-entry interval, contact name and contact phone number. Any personnel entering during the re-entry interval must wear the appropriate personal protective equipment.
- 3.1.9. At the conclusion of each study the remaining treated crop plants will be cut off near the soil level in pots. The cut off plants, remaining treated crop, root systems, and soil will be placed into the crop destruct area at the IAREC vegetative matter composting area.