# 2024 Standard Operating Procedures

for

Magnitude of Residue Field Studies

IR-4 Program

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**Effective Date** = Approval Signature Date

*All SOPs have been reviewed prior to signing

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Pesticide Registration Research Program, Oregon State University

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## SOP: 10-1.8 Standard Operating Procedures

PURPOSE: To provide guidelines for the utilization of indexed Standard Operating Procedures.

PROCEDURES:

1. All Standard Operating Procedures (SOPs) will be reviewed approximately every 2 years by the Field Research Directors and OSU IR-4 Field Center Director then approved by the Western Regional Field Coordinator or designate, prior to the initiation of studies for that year. Management (Western Region Field Research Coordinator) and Field Research Directors need only sign the title page which will indicate the effective date of all the SOPs, since SOPs are treated as a package. The index page includes the date that an individual SOP was revised. If SOPs are not revised, documentation (i.e. initial and date) that they were reviewed should be sent to Western Regional Field Coordinator. Retired SOPs will be listed on the index page under the "Retired" heading in the year they are retired and can be removed from the index in the following year.

2. The SOP title page will contain an effective date for the entire package of SOPs. "20-2.1" is an example of the SOP numbering system. The first number ("20") indicates the general category (e.g. data); the second number ("2") indicates the SOP number within that category (e.g. 20-2 is recording raw data); the third number (".1") is the version number. If a particular SOP is revised, the version number changes (e.g. from 20-3.1 to 20-3.2). The index page indicates the revision date of each SOP. 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. These SOPs are designed to be general guidelines for procedures and instructions to follow if procedures have become a standard set of actions. This cannot be all-inclusive. These SOPs are intended to describe procedures to perform tasks related to the conduct of a Good Laboratory Practice (GLP) study. Actions specific to unique activities noted in the study protocol will be explained and documented in the Field Data Book (FDB).

## SOP 10-2.10 Responsibilities of the Field Research Director (FRD)

PURPOSE: To provide information on the responsibilities of the Field Research Director

PROCEDURES:

1. The Field Research Director has the responsibility for the following:
    a. Review draft protocols and inform the Study Director of any potential issues.
    b. Read and understand the signed protocol prior to initiation of a GLP trial.
    c. Ensure that each study is carried out according to an approved protocol.
    d. Assess personnel needs, facilities, equipment, and materials required to adequately conduct each study.
    e. Maintain and calibrate/verify (if applicable) all equipment, instruments, and gauges according to GLPs, and SOPs and communicate needs to management.
    f. Ensure that all personnel conducting each study understand Good Laboratory Practices (GLPs), the protocol and SOPs.
    g. Respond to all deviations and findings reported by the Quality Assurance Unit (QAU) through the eQA system.
    h. Archive data for each trial:
        a. For paper only FDBs, scan completed books for each study prior to shipping the FDB to Western Region.
        b. For electronic data books (eFDB) scan all original raw data, supporting data, summaries and other items connected with the study that were recorded on paper. Electronic originals of these records are acceptable and archived copies should be stored with any scanned archives. Saving a pdf copy of electronic original data for archiving is preferred.
        c. Original raw data recorded in the eFDB needs no additional archiving.
    i. Ship paper-only FDBs to Western Regional office billing recipient. Paper accompanying the eFDB should be shipped to IR-4 Headquarters when the eFDB is completed prior to QA review.Maintain a current record of GLP training, education and experience, and a job description for all personnel engaged in any study, excluding directly-supervised temporary personnel. These records will be reviewed and updated as necessary (generally annually) before the initiation of the studies for the current year.

## SOP: 10-3.9Field Research Personnel

PURPOSE: To provide information concerning requirements under GLPs.

PROCEDURES:

1. The field research personnel involved in these studies will review and update as necessary (generally annually) their personnel file before the initiation of the studies for the current year. CV's will be updated approximately annually.

2. Personnel will be supplied with all necessary safety equipment as required by the pesticide label and/or protocol.

3. The applicator of any pesticide required in the study must be certified or be in the direct supervision of a certified applicator. A copy of this certification will be kept in the training file records. Personnel conducting critical phases of the study must have completed, at a minimum, one full Basic GLP training course. Field personnel should also observe/assist on several critical phases with GLP-qualified personnel, before conducting critical phases alone.

4. Temporary or summer field personnel will be noted in Part 2C of FDB for any trials in which they participated. Temporary/summer personnel will not have responsibilities in critical phases beyond assistance with timing (stopwatch), harvesting, and other directly supervised work.

5. Temporary work crews may occasionally be necessary for supervised tasks associated with a particular trial. Relevant information concerning these persons will be entered in Part 2C (Temporary/Seasonal Personnel Involved in Trial) of the FDB.

6. GLP training or related technical training for full-time IR-4 field personnel will take place at a minimum of once every three years. Training can be from a private GLP trainer or from any GLP training session organized or offered by IR-4. Informal or formal training related to field and/or greenhouse research (non-GLP) may be included with a CV, but generally is not included on the GLP Training Summary. Quarterly GLP online training offered by IR-4 will be attended, if possible.

## SOP: 10-4.7 FRD Responsibilities Relating to Quality Assurance and Quality Control Audits

PURPOSE: To ensure that data generated during the study meets the requirement of EPA's Good Laboratory Practices (GLPs).

PROCEDURES:
1. All QA (Quality Assurance) procedures/activities will be conducted according to, and in compliance with, SOPs of the Quality Assurance Unit (QAU).

a. QAU picks certain trials each year for a critical phase inspection. The FRD will respond through the eQA system.
b. For paper only FDBs sent to the Western Region, the WR Field Research Coordinator or a designee performs a quality control review of the FDB. The FRD will respond to the QC audit through e-mail to make any necessary changes. eFDBs may also go through a QC audit with a similar procedure.
c. After QC the FDB goes to QA. The FRD will respond to any findings through the eQA system, making sure to use the most current version of the FDB (available on the Western Region website).
d. Responses to Facility Inspections findings will also be made through the eQA system.

## SOP: 20-1.12 Archives and Retention Files

PURPOSE: To ensure that all original raw data generated under GLP are safely maintained at the IR-4 Headquarters Archive, and at Oregon State University North Willamette Research & Extension Center (OSU-NWREC) retention file.

PROCEDURE:

1. The official archive for original raw data will be located at IR-4 Headquarters. Completed original raw data on paper for the current season will be kept in the "FireKing" file cabinet at NWREC. Original raw data retained electronically in the eFDB does not require further compilation or archiving. True copies of original, raw data from IR-4 trials will be saved in the NWREC retention file.

2. The procedure for transferring raw data to the archive at IR-4 Headquarters will be as follows:
   - At the end of a field season, after the last FDB QA audit has been completed, remaining original raw data that have been generated will be compiled and shipped to IR-4 Headquarters for archiving. Original raw data may include: annual standardization of temperature monitors for test substance storage and freezers, standardization of instruments that record environmental data, and any other supporting original raw data or documentation that applies to residue studies.
   - These paper originals will be sent to IR-4 Headquarters for archiving. A chain-of-custody form will contain an inventory of all documents being sent to headquarters. The chain-of-custody form will include the name of the courier and the tracking number. The transfer should be by certifiable mail or some other traceable mechanism (e.g. FedEx). A scanned copy of the completed chain-of-custody form will be kept in the NWREC retention file, and also will be sent to the IR-4 Western Region Field Office. HQ retains the original, signed chain-of-custody form.

3. Any paper documents in the retention file maintained at NWREC will be stored in the "FireKing" file cabinet. Retention files that are electronic originals or scanned true copies will be stored on a cloud-based remote server at Box.com. Access to all retention files is restricted to personnel involved in IR-4 studies. The NWREC facility retention files may contain the following:
   a. True copies of original raw data and signed and dated SOPs;
   b. True copies of supporting data associated with the facility such as freezer and balance maintenance;
   c. Personnel and GLP training records; and
   d. QA facility inspection reports.

4. Copies of older field data books may be discarded if no longer needed. This can be determined by reviewing the project status, as listed in "Test Substance Container Disposal Approval" database on the IR-4 Headquarters website, for individual field researcher directors. Field Data Book retention copies can be discarded if the project status indicates: Use Registered; Tolerance Established; Request Withdrawn; Mfg. Will Not Support; or Use Cancelled.

## SOP: 20-2.10 Recording of Raw Data

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

PROCEDURES:

It is the responsibility of the Field Research Director to see that all data and other items connected with the study are stored in a clean, secure place and are accessible by authorized study personnel. Raw data may be located in the following areas:

a. FDBs/eFDBs are the preferred location to store original raw data when feasible.
b. Freezer activity logs: door of the freezer.
c. Test Substance activity logs: within each individual FDB. For multiple trials using the same test substance container, a true copy of the use log will be placed in the appropriate notebooks
d. Field and Equipment maintenance logs: Electronic logs will be stored on a cloud-based remote server at Box.com. Access will be limited to personnel involved in IR-4 trials.
e. Paper FDBs: with field personnel or IR-4 office.

1. All raw data written on paper will be recorded in dark ink. Blue ink is preferred as it distinguishes the original from true copies. Initial and date each activity as it occurs.

2. Forms generated to replace or supplement the forms in the IR-4 FDB must contain all information on the original form and will be included in the FDB.

3. Corrections to raw data written on paper will be made by crossing through the item, initialing, dating and giving reason or code for the correction. Blank spaces in the paper FDB will be lined out.

4. All notebooks, data sheets, summaries, etc. should be clearly marked with the name of the project, field ID number, date and initials of the person responsible for the data, and other information that may be needed to understand the data and its source to aid in reconstructing of activities associated with the GLP study.

5. Make sure that all data required in the study protocol is collected, recorded and retained. Carefully review the protocol to ensure that all required data is collected.

6. Transcribed data in the paper FDB must indicate location of the original.

7. Copies or scans of original documents or data will be indicated as such with a stamp or annotation that reads: “True copy of the original” (or similar) with the location of the original provided and the entry dated and initialed.

8. Pagination and the application of Field ID# labels in the FDB are not considered raw data and do not require a date and initial; these components are considered data organization.

## SOP: 20-3.9 Method for Collecting Additional Data

PURPOSE: To describe the procedure used for noting phytotoxicity and describing soil information data.

PROCEDURES:

1. Phytotoxicity Data
   Consult the protocol to determine the method and necessity of the phytotoxicity data. In general, the crop should be checked periodically for evidence of phytotoxicity and crop vigor. Include the rating scale or other reference guide used to assess phytotoxicity and crop vigor.

2. Soils Data
   Soils data can be found at a USDA Web Soil Survey internet site (http://websoilsurvey.sc.egov.usda.gov/App/HomePage.htm), and data downloaded for inclusion in the FDB.

3. Weather Data
   Weather data for trials conducted at NWREC and at the OSU Corvallis-based farms (Hyslop, Lewis Brown Farm, Veg Farm) may be downloaded from US Bureau of Reclamation, Agrimet stations (https://www.usbr.gov/pn/agrimet/). Use station ID 'arao' for NWREC trials and station ID 'crvo' for Corvallis trials. For trials placed with grower cooperators, use the closest weather station, which may be located at other sites such as airports, and can be searched for and downloaded from NOAA Climate Data Online. (https://www.ncdc.noaa.gov/cdo-web/).

## SOP: 20-5.5 Significant Figures and Rounding

PURPOSE: To provide guidelines for determining significant figures in a measurement and to provide guidelines and ensure consistency in rounding numbers in a calculation.

1. When reporting a measurement, record only to the degree of accuracy of the equipment or instrument or device used to make the measurement. For example, if a scale can weigh to a tenth of a gram, record 3.2 grams, not 3.20 grams. Remember, however, that conventional scientific notation allows that four tenths of a gram (.4) is commonly written as "0.4".

2. Rounding: When rounding, look at the digit following (i.e. to the right of) the digit that is to be the last value recorded:
   If it is less than 5, drop it and all the digits to the right of it.
   If it is greater than 5, increase by 1 the digit to be rounded.
   If it is 5, round in the direction that will result in an even number (i.e. if already even, drop the 5; if odd, round up by one to make an even number).

| | | |
|---|---|---|
| Examples (rounding to 4 places): | 64.534 = 64.53 | 64.575 = 64.58 |
| | 64.538 = 64.54 | 64.505 = 64.50 |
| | 64.585 = 64.58 | 64.495 = 64.50 |

3. In calculations involving significant figures, the answer is reported with significant figures that reflects the reliability of the least precise measured value or operation. Calculations performed within the eFDB will use validated internal settings for calculating significant figures.

## SOP 30-1.9 Site Selection and Design of Experimental Plots

PURPOSE: To ensure plots will meet all protocol-specified requirements for a specific crop.

PROCEDURES:

1. A trial site may be located in a commercial production field, at NWREC, or at other University or private agricultural grower sites.

2. The test plots will be large enough to accommodate the required number of replicates, buffer zones, sample size, and treatments in accordance with the protocol.

3. Locate plots with sufficient isolation to minimize contamination from external sources such as commercial operations or other research activities. Untreated plots should be located upwind of the treated plots (or at a minimum, not directly downwind). Placing the untreated plot up-slope from the treated plots, if possible, is also desirable.

4. If the crop is established, select a site that has a uniform stand density.

5. Follow the experimental design specified by the protocol. Individual plots should be large enough so that no more than 50% of the harvestable area will be needed to provide samples.

6. Distance from the permanent reference point(s) to at least two corners of each plot should be marked on the plot map to facilitate location of the plots. Distances may be measured directly from the reference point to the plot corners using a measuring tape or measuring wheel.

7. Measure the dimensions of each plot on the site using a suitable measuring device. Identify each plot with an individual designation specific to that plot (e.g. untreated plot is TRT 01; treated plot is TRT 02) and the Trial Identification Number. Not all flags in plot will have identification numbers as some flags are used to mark quadrants, etc.

8. Prepare a plot map showing the location of each plot and any other research plots at the site. The plot map should include the dimensions and locations of treated and untreated plots; the plant and row spacing, the number of rows and/or beds and their direction; the amount and direction of slope, the North azimuth, and the dimensions and locations of buffer zones. Reference points are usually power poles, fence lines, irrigation hydrants, roads, etc.

## SOP: 30-2.3 Commodity Establishment and Maintenance

PURPOSE: To ensure that crops are grown under common and current agricultural practices and to provide a uniform crop for the study/trial.

PROCEDURES:

1. Follow the practices required to produce the commodity under standard commercial conditions.

2. Determine that the soil type and fertility of the trial site are suitable for the commodity.

3. When field trials are conducted in commercial production fields, accepted grower practices will be considered as standard procedures unless the protocol states differently. Communicate clearly to the cooperating commercial grower the scope of the trial and the data required from the plots; encourage him/her to call before conducting an activity in the plots that may affect the integrity of the GLP trial. When trials are conducted at NWREC or other University agricultural facilities, accepted commercial practices should be performed as necessary to bring the soil reasonably within the requirements of the commodity. If necessary, prepare the soil as is commonly defined as "accepted grower practices" in the region where the commodity is being grown.

4. Determine the correct species and variety to use as specified by the study protocol. If the variety is not specified, use one that is representative or commonly planted in the area by commercial producers. If a commercial producer is providing the plants, try to select plants as uniform in growth and color as possible.

5. If seeded or transplanted, determine the within and between row spacing and seed depth as specified. Plant the seed or transplant in straight rows with accurate measurements to assure the commodity is planted according to commonly accepted grower practices.

6. Irrigate, fertilize, or perform other agricultural practices, as necessary, to get the commodity established and maintain growth and health of the crop throughout the life of the trial.

7. Before application of maintenance pesticides to the test crop, take into consideration any conflicts with the test substance to ensure that they will not interfere with residue analysis in the laboratory. If in doubt, contact the Study Directory identified in the protocol for approval.

8. When temporary labor is hired for plot maintenance, limit activity to untreated plots first, finishing untreated completely before proceeding to a treated plot. This also applies to any other field activities during the season.

## SOP: 40-1.18 Test Substance and Adjuvant Maintenance

PURPOSE: To ensure that all test substances and adjuvants are received and maintained according to GLP standards.

PROCEDURES:

1. Upon receiving a test substance, fill out all information required on the appropriate forms in the field data book. Contact the study director in case of any discrepancies from the protocol or missing information with the shipment or on the test substance label.

2. Be sure each test substance container has the following information written on it:
    - Chemical name
    - Expiration date
    - Batch or lot number
    - Storage conditions
    - "GLP" (if GLP status is confirmed with COA or by SD)
    - IR-4 Study ID number

3. Document the following information for adjuvants:
    - Date received at the field facility (usually the purchase date)
    - Identity and concentration of the adjuvant
    - Recommended storage conditions
    - Expiration date

        If no expiration date is supplied by the manufacturer, assign an expiration date up to 5 years from the date of purchase (unless other information supporting a different expiration date is available).

    The original container and secondary containers used to store an adjuvant must be labeled as indicated above. These requirements do not apply to temporary containers used for measuring, but they should be adequately labeled to uniquely identify the product.

    If there are any questions or concerns about the integrity or condition of the spray additive (e.g. contamination, color change, consistency) it should be removed from use for GLP residue trials.

4. All test substances and adjuvants for GLP residue studies will be securely stored in the IR-4 test substance storage unit located in the General Pesticide Storage Building at NWREC. Access to test substance will be limited to authorized personnel only.

5. If a test substance requires unusual or abnormal storage conditions, it may be maintained separately according to those conditions. If that is the case, document the storage location and record storage temperatures. All test substances will be put into the temperature-monitored storage unit within 48 hours of test substance receipt.

6. A temperature monitor and backup recording device (e.g. HOBO) will be maintained within the test substance storage unit. The HOBO is programmed to record every hour.

7. All test substance use or removal shall be recorded on the use-log form found in the eFDB/FDB. This form will be individual to each test substance bottle and will be included in the FDB upon completion of the study.

8. After mixing, the test substance and adjuvant will be put back into storage as soon as possible. When the test substance is relocated off-Station for mixing or extended periods of time, include a temperature monitor and an ice substitute such as blue ice in the transport container if weather dictates.

9. If a secondary container is used to transport the test substance, label the transport container with trial ID, chemical name, expiration date, batch or lot number and storage conditions plus the date and the amount of test substance measured.

10. Adjuvants must be handled in a manner to prevent contamination with test substances and other spray additives. Dispense adjuvant into a temporary container for measuring and discard any remaining adjuvant.

11. After completion of the current field trial the test substance can be used for general farm pest management on registered crops. The container, whether empty or full, must be retained until permission to discard is received from IR-4 Headquarters (https://ir4app.cals.ncsu.edu/Ir4FoodPub/SubstanceDispoSch). Triple rinse empty test substance containers if they are to be stored.

12. If required, test substances or test substance containers will be sent back to the manufacturer/distributor/chemical company after the completion of the field trial. In this case, personnel shall coordinate and follow direction from the OSU EH&S Hazardous Material Shipping Office (https://ehs.oregonstate.edu/shipping-hazardous-materials). Retain shipment tracking information.

## SOP: 50-1.16 Residue Sample Collection

PURPOSE: To ensure residue crop samples are collected according to protocol and within GLP guidelines.

PROCEDURES:

1. Consult the study protocol to establish specific dates for the collection of crop samples. If these dates are based on uncontrolled events (plant size, fruit maturity, etc.) then tentative dates should be established and refined as necessary. The Regional Field Coordinator should be kept informed when the dates are significantly changed (e.g. greater than 1 month from the tentative dates). Quality Assurance should be kept informed of date changes for targeted trials.

2. Depending on the Raw Agricultural Commodity ( RAC) and protocol, samples may be placed directly into the laminated sample bag, into clean buckets then transferred into sample bags, or into zip-top bags which are then placed into the laminated sample bag.

3. Prior to sample collection, obtain a sufficient number of sample bags to collect all the samples. Label each sample bag with an identification tag on the outside of the bag. If collecting samples into a bucket or other temporary container, ensure each container is appropriately labeled with the information required in the protocol.

4. Before entering the plots, place cloth sample bag in a plastic bag or bucket to protect it from dirt and contamination. If tools or other devices are needed for harvest, be mindful of decontamination of the tools. Wash any sampling device with soap and water, or baby wipes or disinfectant wipes, prior to and after use for each treatment.

5. Sample plots in order of ascending test substance rates, starting with the untreated rate, unless treatments are sampled by different people.

6. Take special care to do the following in the sample collection process:
   - Change gloves between samples
   - Avoid contamination of the field sample with the test substance during the sampling, labeling, storage, and shipping processes.
   - Avoid taking diseased or undersized crop parts.
   - Take care to not remove surface residues during handling, packing or preparation; do not wash commodity unless specified in the protocol.
   - Do not remove any soil or plant parts or trim the commodity unless it is specified in the study protocol.

7. After collecting each sample, tie and secure the laminated cloth sample bag, then place into a larger plastic bag (if not already in one) and secure the opening.

8. If the samples cannot be placed into a freezer within one hour, place them into clean coolers containing blue ice and a temperature monitoring device in each cooler. Separate coolers are used for treated and untreated samples.

9. Considerations for drying samples
   a. Hay Sampling

- Transport the harvested plant material to a covered building and spread on clean paper. Label the paper where each sample is placed with the trial ID number and sample ID number.
- At time of harvest, collect approximately a 5-gal bucketful of extra material to help determine percent moisture content of the RAC samples after drying. Dry under similar conditions as the RAC samples.
- Turn the samples periodically to aid in drying, beginning with untreated then treated samples, using gloves at all times. When, by feel, it is estimated that the samples are hay, place in cloth residue bags, weigh, label and freeze as usual.
- After sampling, determine the dry matter/moisture content of hay samples using the extra collected plant material per section 10 (below).

   b. Hop cone sampling

- Collect enough hop cones until the fresh weight is more than five pounds for each sample
- At the time of harvest for RAC samples, collect additional plant material from the same bines for dry matter and percent moisture prediction and verification. Collect approximately half a 5-gal bucketful.
- Transfer RAC samples to individual mesh drying bags, tie off, and add to dryer.
- Determine target dry down weights as per section 11, below.
- After samples have reached target drying weight, allow to cool at room temperature for up to one hour, then place in cloth residue bags, weigh, label and freeze as usual.

   c. Hazelnut sampling

- At the time of harvest for RAC samples, collect additional nuts from the tarps for dry matter and percent moisture prediction and verification. Collect approximately three pounds of additional nuts.
- Determine target dry down weights as per section 11.

10. Determining percent moisture content or percent dry matter

- From the additional material collected, determine the initial weight of four small samples, approximately 100 to 200 grams.
- Place samples in paper bags and into a drying oven until there is no change (within +/- 1%) in weight following successive weighing at least one hour apart. This indicates 0% moisture.
- Use the initial and final weights to calculate % moisture content or % dry matter. If appropriate, report the average of small samples as the percent dry matter or moisture content of RAC samples.

11. Determining target dry-down weights

- From the additional material collected, determine the initial weight of four small samples, approximately 100 to 200 grams.
- Place samples in paper bags and into a drying oven until there is no change (within +/-

1%) in weight following successive weighing at least one hour apart. This indicates 0% moisture.

- Use the initial and final weights to calculate % moisture content or % dry matter of the RAC sample.
- Calculate target weights for each RAC using the formula:
  Target weight = (% dry matter of fresh sample * fresh weight of RAC sample)/reciprocal of required moisture fraction

## SOP: 50-2.5 Post-Sampling Crop Destruction

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.

PROCEDURES:

1. Field Research Director will decide the method for crop destruction. The criteria to consider 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.

2. When a study is undertaken at a grower/cooperator site, the grower/cooperator and the researcher must have an agreement and understanding about what will be done with the treated crop for which no tolerance has been established. In most cases, the FRD will harvest and dispose of the contents of the treated crop in a manner that ensures it will not be consumed (e.g. compost, burn, trash bin).

3. Trials conducted at NWREC or other University property are considered off-limits and non-accessible to un-informed personnel or the public. At NWREC and other University sites, it will be up to the discretion of the FRD to determine how best to dispose of the treated crop.

4. For all trials, include in the FDB an explanation of when and how the crop was destroyed or why no crop destruction was necessary.

## SOP: 50-3.17 Sample Storage and Freezer Use

PURPOSE: To ensure the integrity of the samples while in storage.

PROCEDURES:

1. There are two upright freezers. One freezer, marked "FRZ1" is the main freezer. The second freezer, marked "FRZ2" will be used in case of catastrophic failure of the main freezer or if there is not enough space for samples in FRZ1. A third freezer for backup, marked "BKP" is available if needed.

2. All maintenance, repairs and cleaning for the freezers will be documented in the freezer maintenance log (maintained electronically). The freezers will be cleaned approximately once per year by wiping/washing the racks and entire interior, and rinsing well. The area around the outside of freezers should be kept free of debris.

3. Maintain freezers preferably at or below 0°F (-18°C). Regular, auto-defrost cycles of the freezers are normal. If the thermometer on the outside of the freezer is above 0°F, ascertain whether it is due to a freezer malfunction or due to a scheduled defrost cycle. To do so, remove the grate on the outside of the freezer, below the doors, and locate two lights within; the light labeled "defrost mode" will be on if the freezer is in the defrost mode. Keep the thermometer probe in ethylene glycol or sand to prevent display of large temperature spikes from the defrost cycle.

4. When placing samples in the freezer, record the time and date samples were placed in the freezer, the field ID number, the number of samples and their sampling code, and the initials of the person placing the samples in the freezer on the appropriate log form on the freezer door. Removal of samples should be recorded on the log form and include: time and date removed, and person's initials.

5. Storage temperature of the samples will be recorded automatically with the use of HOBO U-12 loggers. The freezer contains two HOBOs, one primary and one backup. Set temperature monitors to record every 30 minutes. Place loggers in the middle shelf of the freezer. Keep temperature monitors or probes in a buffer (ethylene glycol or sand) to prevent temperature fluctuations from the defrost cycle.

6. Keep bottom racks in the freezer 2-3" from the bottom of each unit to allow good air circulation in the freezers. No samples should be stored on the bottom of the freezer.

7. When samples are in the freezer, freezer doors are to remain locked with keys on wall near light switch.

8. In the event of a prolonged (> 1 hour) spike or cycle above 5°F, which could indicate freezer failure or malfunction, an alarm on the freezer will automatically sound. The names and phone numbers of IR-4 personnel are listed on the freezer. An OSU employee lives on-site and has been instructed to call IR-4 personnel if the alarm sounds. This system also has an automatic dialer that is engaged when the temperature rises above 5ºF for one hour. It will phone IR-4 personnel and the NWREC Farm Manager.

9. In the event of a power outage, a back-up generator, powered by natural gas, will start automatically and provide power to the freezers.

10. Testing the Alarm and Natural Gas Generator: Test the alarm system and backup generator at the beginning of each season, and in-season if possible when freezers are empty. To simulate a freezer malfunction and test the alarm system, unplug the freezer and prop door open to allow temperature to rise above 5°F for more than one hour. The alarm (horn) should sound, and the phone dialer will engage and phone appropriate personnel. The FRD will check to make sure that everyone on the phone list has received the recorded message. To simulate a power outage, the electrical circuit is turned off at the power box; if the system is working properly, the gas generator will engage and power the freezers. The gas generator automatically runs for about 30 minutes once a week.
    a. Document the testing with information including the date and time of the test, time(s) messages were received, and personnel contacted.
    If the system did not work properly in the test, complete repairs as soon as possible, document, and conduct a subsequent test.

11. What to do if a freezer malfunctions: If the freezer alarm sounds, remove samples from the malfunctioning freezer and place them and the datalogger in FRZ2 or BKP, keeping treated and untreated samples separate. Document in sample storage log. The FRZ2 or BKP will be used if/until the FRZ1 can be fixed.

## SOP: 50-4.13 Sample Shipping Procedures

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

PROCEDURES:

1. The preference is to ship samples as soon as possible; check the protocol to ascertain if there are storage or shipping requirements specific to the project. Shipment can be via overnight air express with the use of dry ice, or via a freezer truck service, such as ACDS.

2. Make arrangements with the carrier for pick-up and shipment of the samples. ACDS usually calls about one week in advance to inform us they will be in the area for a pick-up. Dry ice shipments need dropped off at the FedEx facility in Lake Oswego.

3. Contact the residue laboratory by e-mail and notify them of the shipment dates and method of shipment. Notify lab of shipment within the timeframe specified in the protocol. Overnight air shipments should be made on Monday through Wednesday to avoid potential weekend layovers.

4. Shipping boxes should match the size of the shipment. TRT and UTC samples shipped in the same box must be double-bagged. TRT samples should be placed in the box first, with UTC samples on top. Completely packed boxes should weigh less than 75 lbs each.

5. Include the Sample Arrival Check Sheet and a copy of the signed Residue Chain of Custody form in a zip-top bag in each shipping container; retain the original form in the FDB.

6. Apply tape over any stickers and labels to secure them to the container. Label each shipping container with the following information:
    - Name and address of the sender
    - Name and address of the reside laboratory receiving the samples
    - Study ID for all samples included in the box

7. For overnight shipments with dry ice, pack the containers with enough dry ice and distribute throughout the container, to ensure integrity of the samples. A 4:1 weight ratio of dry ice to commodity for every 24 hours in transit is preferred; if feasible, a 6:1 ratio is desirable in the off chance the samples might get lost and are not delivered within 24 hours. Samples should already be hard-frozen prior to packing with dry ice, unless permission is given by the Study Director to do otherwise.

8. Labels don't stick to plastic coolers because of condensation caused by cold samples. Place plastic coolers in a cardboard box and tape shipping labels to the box. Affix official dry ice sticker to each box and fill in with appropriate information. Leave gaps in the container to allow dry ice gas to escape.

## SOP: 60-1.17 Standardization and Maintenance of Instruments and Gauges

PURPOSE: To ensure that instruments crucial to GLP data requirements are standardized and maintained to provide accurate readings.

PROCEDURES:

1. Instruments used in GLP field research should be tested and standardized approximately once per year. Testing should be completed under conditions similar to those under which the instrument will be used.

2. Standardize instruments by comparing like instruments. If instruments are not within the specified average, that instrument will not be used; it should be re-calibrated or replaced. Standardizing instruments against known constants, (e.g. thermometers in ice water slurry, stainless steel weights, etc.) or through certification may also be used for testing accuracy of instruments and gauges.

3. Document the date of standardization, the equipment identification number of the instrument involved, and the results of the tests. Unless otherwise noted, instruments that have been standardized are considered to have had adequate maintenance. New or professionally serviced instruments are considered calibrated for the period of one year unless the manufacturer specifies another date.

4. Batteries will be changed when the battery indicator reads 40%. Record battery changes in the maintenance log.

5. When a new instrument is purchased, issue a new ID number for it and mark this number on the instrument. If the new instrument is replacing an old instrument of the same type, a new ID number needs to be assigned. Document this change on the maintenance log specific to that instrument.

6. All documentation that has been produced by the standardization and maintenance of these instruments will be saved in facility records and archived at IR-4 Headquarters.

7. Instrument specific notes
   a. pH: pH of the water used as carrier for the test substance is determined via pH paper test strips. Ensure the pH strips have not passed the manufacturer's expiration date.

   b. National Institute of Standards and Technology (NIST) Thermometer: The NIST is used for comparison only and is not used when calculating the average of the temperature recording devices. A new NIST thermometer will be purchased with certification, or the one currently being used will be re-certified every 3 years. Maintain the NIST certificate in the facility records.

c. Soil and Water Thermometers: Standardization is conducted by immersing the probes of at least three digital thermometers in the same container of water and recording temperatures after about two minutes. Include a NIST-certified thermometer, for comparison. Results should be within 5% of the average of all thermometers. The NIST thermometer reading is not used when calculating the average; it is used for comparison only, but must be within 5% of the average of the digital thermometers. Standardize the thermometers in both cold water (e.g. 35 to 45° F) and in warm water (e.g. 65 - 75° F). Record the results in the maintenance log.

d. Kestrel 3000: Standardization is accomplished by comparing readings from three instruments while under the same conditions. To standardize for air temperature and relative humidity, allow 15 minutes for the Kestrels to stabilize, then take readings from all instruments, for each environmental parameter (i.e. air temp, RH). Results should be within 5% of the average of all three instruments. To standardize for wind speed, direct a fan towards the meters which are aligned next to each other on the same surface. Read all three meters concurrently and document. Results should be within 10% of the average of all three instruments. Standardize the wind function of the Kestrels using two wind speeds. Record the results in the maintenance log.

e. Freezer HOBO temperature-recording devices: launch loggers and set them to record every minute for 1 hour. Place loggers and a NIST thermometer in a freezer set at less than 0° F (-18° C). Allow several hours for HOBO devices to reach the ambient temperature. After one hour of recording, immediately read and record the temperature of the NIST thermometer, download all loggers, and print out a graph for each logger. The NIST thermometer reading is not used when calculating the average of the HOBOS; it is used for comparison only, but must be within 5°F of the average of all the loggers. The reading of each unit should be within 5°F of the average of all instruments. If results differ more than 5°F, replace a battery and restandardize, use a different temperature recording instrument, or send logger back to manufacturer for repair or replacement.

f. HOBO temperature-recording devices: launch HOBO loggers and set them to record every minute for 1 hour. Place all the temperature-recording devices in the test substance storage unit. Include a NIST-certified thermometer for comparison. Allow several hours for HOBO devices to reach the ambient temperature. After approximately one hour of recording, immediately read and record the temperature of the NIST thermometer, then download all the loggers and print out a graph for each. The reading of each unit must be within 5ºF of the average of all the units. The NIST thermometer reading is not used when calculating the average of the HOBOS; it is used for comparison only, but also must be within 5ºF of the average of the HOBOS. If results differ more than 5°F, replace a battery and restandardize, use a different temperature recording instrument, or send logger back to manufacturer for repair or replacement.

g. Balance Calibration: professional calibration and maintenance on these balances will be performed every two years. Standard weights used for balance bracketing and validation will also be checked at this time.

h. Mobile device for eFDB use: record verification of app and any major updates and maintenance. Typically, a major update would involve calling OSU Information and Technology or another outside party for assistance. Minor updates, such as automatic software updates, do not need to be recorded in the log.

## SOP: 60-2.11 Application and Harvesting Equipment Maintenance

PURPOSE: To ensure application equipment used in GLP studies is functioning and in good condition.

1. Record routine and non-routine equipment maintenance in the IR-4 Equipment Maintenance Log for that equipment.

2. Non-routine maintenance is when something breaks and needs to be fixed. When describing non-routine maintenance, record:
    - Nature of the defect
    - How and when discovered
    - Any remedial actions taken

3. Routine maintenance includes changing screens, nozzles, etc. When describing routine maintenance, record:
    - SOP followed
    - Date of occurrence
    - Clear indication that this was routine

4. Spray booms are cleaned during post-application cleanup, which is documented in the field data book and does not need to be documented additionally on the equipment maintenance form. All other equipment maintenance will be recorded on the equipment maintenance log.

5. Inspect nozzle tips and screens for wear. Check spray pattern from each nozzle. Nozzle tips or screens will be replaced when it fails to stay within 5% of the average of all nozzles on the boom during discharge calibration. If the spray pattern from a nozzle is noticed to be different than expected, the affected nozzle should be replaced.

6. Pressurize the system and listen carefully along length of boom and hoses for hissing, feel for escaping gas, and watch for bubbling. Replace hose, thread tape or sealant if leaks are found.

7. For the gas-powered mist blower, visually check the unit for condition or leaks before each use. Any other maintenance will be performed by the dealer on an "as needed" basis. At the end of each season completely drain the fuel tank; to accomplish this, run the engine until the carburetor is dry.

8. The farm crew is responsible for winterization of the airblast sprayer and over the row sprayer. Verify that they have completed this task ahead of hard freezes each autumn.

## SOP: 60-3.15 Sprayer Calibration

PURPOSE: To determine the delivery rate of a sprayer and make adjustments, as necessary, to ensure accurate application of the test substance according to the protocol.

PROCEDURES:

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

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

3. The eFDB provides automated, validated calculations for discharge and speed calibrations, and these are preferred to manual calculations when feasible.

4. Calibration for boom sprayers: Charge the $CO_2$ system, then begin timing the discharge. Collect the discharge from each nozzle for at least 30 seconds. Start and stop the spray system and the stopwatch simultaneously. Measure the discharge. Replicate three times. Each nozzle discharge should not vary more than 5% from the average discharge of all the nozzles. If so, check and clean nozzles or screens, as necessary, or adjust diaphragm check valves. If an individual nozzle discharge still varies more than 5% from the average discharge of all nozzles, replace questionable nozzle tip or screen.

5. Calibration for airblast sprayer: Park the sprayer on level ground. Prime the system by partially filling the tank and turning on the sprayer. Turn off sprayer, empty tank completely, then fill tank again with a known amount of water. Start the sprayer and spray for at least 30 seconds, then shut off. Collect the water remaining in the tank and measure. Repeat three times. Total discharge of each run should not vary more than 5% from average discharge of all three runs.

6. Calibration for the mist blower: Add a known amount of water to the tank. Start the engine and begin timing discharge as you simultaneously turn on the mist blower flow control valve. Run for at least 30 seconds, then close the valve. Allow remaining water in tank to flow into a catch jug and measure. Repeat three times. Total discharge of each run should not vary more than 5% from average discharge of all three runs.

7. Calibration rechecks: a recheck may be used when protocol requirements have been satisfied. Calculate the discharge from a single run, then ensure the discharge is within 5% of the original replicated calibration. If the re-check discharge is not within 5% of the original calibration, do a complete recalibration of the equipment.

8. All calculations used to determine amount of test substance, carrier volume and overage are left to the discretion of the GLP-trained applicator and will be recorded in the FDB.

9. Speed calibrations: the applicator or tractor speed should be timed over a known distance three consecutive times. A metronome may be used to aid pacing. Tractor gear and RPM

should be recorded in the FDB. Each individual pass time should not vary more than 5% from the average of the three measurements.

10. Speed calibration rechecks: if protocol requirements are satisfied, a speed recheck may be used. Set the walking speed or tractor speed as per the original replicated calibration, and time the duration to traverse the same distance. If the speed recheck is not within 5% of the original calibration, do a new complete speed calibration.

## SOP: 60-4.14 Measuring a Test Substance Formulation

PURPOSE: To ensure an accurate dosage in the application of test substances in field research.

PROCEDURES:

1. Wear appropriate PPE, per the label and/or SDS.

2. Procedures for measuring and mixing liquid test substance:
   - Use clean graduated cylinders or syringes. Do not estimate volume. Report the actual volume level to the nearest increment. Readings of volume of test substance should be made at the bottom of the meniscus.
   - If the opening of the cylinder is too restricted to allow pouring of the test substance from the original container, then do one of the following:
     - Use a clean glass container with a pour lip as an intermediate and fill the cylinder from it.
     - Use a clean funnel large enough to allow filling the cylinder with a minimum of spillage.
     - Use an unused syringe with appropriate gradations to extract the test substance from the container.
   - To mix, add some pre-measured carrier to the spray container. Pour the liquid test substance directly into the spray tank. If a graduated cylinder is used to measure or transfer the test substance, triple-rinse the cylinder with some pre-measured carrier; add rinsate to the spray container.

3. Procedures for measuring and mixing dry formulated test substance:
   - Select a clean container suitable to hold the desired amount of pesticide and tare it on the scale. Do not lean on or bump the weighing table when using scales.
   - Verify the scale accuracy prior to weighing the test substance for use in the study. With the empty container tared on the scale, bracket the weight as appropriate and record in the FDB.
   - Add the test substance into the tared container and record the weight in the FDB. When transferring the test substance from the weighing container to the spray tank, be sure to remove all test substance. If necessary, rinse the container with some of the pre-measured carrier and add the rinsate to the spray tank.
   - With some difficult-to-dissolve dry formulations, create a slurry before adding the test substance to the spray container. Use a stirring rod to mix the weighed test substance into a slurry with some pre-measured carrier. Pour the slurry into the spray container, then triple rinse the slurry container with pre-measured carrier and add rinsate to the spray tank. Add remaining carrier and agitate to mix.

4. Procedures for measuring granular formulations:
   - Weigh appropriate amount as for dry formulations, but pour test substance directly into application container without dilution.

## SOP: 60-5.5 Procedure for the Application of a Test Substance

PURPOSE: To describe the procedures used in the application of a test substance.

PROCEDURES:

1. All personnel involved in the mixing, application, transportation, storage and cleanup of test substance should be properly trained and wear appropriate PPE.

2. Equipment used in the application of a test substance should be inspected and calibrated prior to application. Calibrate the equipment as described in SOP 60-3.14. If performing a calibration re-check, check that all settings are the same as the previously performed calibration.

3. Charge the $CO_2$ system away from the test plots to eliminate risk of contamination to the plots. Agitate the spray mix before and during application to ensure an even mix of the test substance and water. Apply the test substance spray solution beginning with the lowest concentration and work up to the highest concentration.

4. Apply the test substance solution while being timed with a stopwatch or other similar device. Begin timing as engaged spray boom enters the plot and stop timing at the end of the plot.

5. Based on pass times and discharge rate of the boom, perform a back calculation to determine whether application is within protocol requirements (usually between –5% and +10%). If a back calculation shows a deviation beyond that allowed in the protocol, notify the Study Director.

6. For granular formulations of test substance, application can be made with a spreader, which can be calibrated, designed for granular or other dry formulations. If given prior approval by the Study Director, test substance can be delivered with a gloved hand or a hand-held shaker can, making several passes over the test plot to ensure uniform distribution. Plot can also be divided into quadrants, and the test substance weighed out in four separate aliquots for more precise application of dry formulations.

## SOP: 60-6.3 Problems During Application of a Test Substance

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

PROCDURES:

1. During application, the operator should observe the delivery pattern to ensure that the test substance is being evenly distributed to the entire plot.

2. If something goes wrong (e.g. a plugged nozzle, a hose leak, a clogged hopper), then the operator should take immediate action to correct the situation.

3. The Study Director, OSU IR-4 Field Center Director and the Regional Field Coordinator should be notified of the incident immediately, details recorded in the field data book, and any actions taken recorded in the appropriate equipment maintenance log and FDB.

4. The affected portion of the plot should be carefully marked off and staked to indicate the area affected. This portion should not be used for obtaining samples for residue analysis unless approved by the Study Director.

5. In the event of using up solution prior to the end of the spray plot, the following procedure should be applied:
    - Mark and calculate area actually sprayed with the test substance.
    - Calculate rate applied. If rate applied is within acceptable levels as specified by protocol then note changes in area applied within the field data book and sample from within the marked area at the appropriate time.
    - If acceptable levels are exceeded, contact the Study Director for next steps.

## SOP: 60-7.3 Clean-up of Application Equipment

PURPOSE: To ensure that test substance application equipment is clean and ready for use.

PROCEDURES:

1. After application, and away from the test plot, spray out all test substance solution to empty tank, hoses, and boom. Triple rinse inside of spray tank with clean water; then add clean water and spray out through boom to ensure that hoses and nozzles are free of test substance residues. In the case of oily residues from adjuvants a diluted tank cleaner may be used according to instructions.

2. Check nozzles and screens and clean if necessary.

3. Use clean water to rinse the outside of the spray tank, boom, hoses, etc., and return clean equipment to the IR-4 equipment storage area.

4. For cleaning after airblast sprayer application, drain the spray tank contents in a non-crop area far from any plots. Rinse inside of tank and let drain. Clean any in-line hoses, nozzles and filters by refilling tank, charging system and spraying out completely. Rinse outside of equipment, nozzles, pump and all exposed parts.

5. To clean the shaker bottle and bucket used for granular applications, wash the bottle, lid and bucket in warm, soapy water then rinse with clean water.

6. To clean the backpack mist blower tank, especially if an adjuvant has been used, a solution of tank cleaner may be necessary to remove leftover oily residues. Rinse 2-3 more times with clean hot water to remove any traces of the tank cleaner. Re-attach the outlet hoses and replace lid and screen on top of tank. Fill tank with more water and start motor then spray out to clean hoses.

## SOP: 60-8.1 Borrowed Equipment

PURPOSE: To describe procedures for equipment that is borrowed or rented for use in GLP studies.

PROCEDURES:

1. Become familiar with equipment manuals that describe the use, calibration, or maintenance of the borrowed equipment. If no manual accompanies the equipment, it may be available online.

2. Record the following information on a new sheet in the Equipment Maintenance Log:
   - Equipment identifier (assign an identifier that includes owner of the equipment)
   - Equipment description
   - Date of acquisition (list in the activity table)
   - Date and description of cleaning (list in the activity table)
   - Any maintenance performed & indication if it is routine

3. Clean any borrowed harvest or application equipment prior to use in a study to minimize risk of contamination. Follow steps in the equipment manual, as appropriate. Cleaning may include: cleaning spray tank with tank cleaner, replacing screens and nozzles, hosing off exterior of equipment, wiping down with hot soapy water, or similar steps, as appropriate for the type of borrowed equipment. Record in the Equipment Maintenance Log.

4. Calibrate any borrowed equipment prior to use in a study to ensure it is performing as per expectations. Record in the Equipment Maintenance Log.

5. A copy of the equipment maintenance log may be included in the FDB. Retain the original copy with the facility records.