2020 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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10-1.7
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 annually 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 Notebook (FDN).

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10-2.9
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 the study is carried out according to an approved protocol.
    d. Assess personnel needs, facilities, equipment, and materials required to adequately conduct a trial with a project. It is also the duty of the FRD to maintain all equipment, instruments, and gauges according to GLPs, and schedules and practices outlined in these SOPs and communicate needs to management.
    e. Make sure that all personnel conducting the study understand Good Laboratory Practices (GLPs), the protocol and SOPs.
    f. All deviations and findings reported by the Quality Assurance Unit (QAU) are responded to

through the e-QA system.

g. Create electronic copy of the following:

1) Completed Field Data Notebooks for all studies prior to shipping the books to Western Region.

2) All original raw data, supporting data, summaries and other items connected with the study.

g. Ship FDNs to Western Regional office billing recipient. Ship no more than two FDNs per box.

Maintain on file a current record of training (including webinars), education and experience, and a job description for all personnel engaged in the study. These records will be reviewed and updated as necessary (generally annually) before the initiation of the studies for the current year.

10-3.8
Field 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, to be placed in FDN'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 FDN 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 FDN.

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. GLP webinar trainings offered by the Western Region will be attended, if possible.

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 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. After the FDN is sent to the Western Region, the WR Field Research Coordinator or a designee performs a quality control review of the notebook. The FRD will respond to the QC audit through e-mail to make any necessary changes.
   c. After QC the FDN goes to QA. The FRD will respond to any findings through the eQA system, making sure to use the most current version of the Field Data Notebook (available on the Western Region website).
   d. Responses to Facility Inspections findings will also be made through the eQA system.

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20-1.10
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. Original raw data for the current season will be kept in the "FireKing" file cabinet at NWREC. True copies of original, raw data will be made and retained at the NWREC facility.

2. The procedure for transferring raw data to the archive at IR-4 Headquarters will be as follows:

   At the end of the current field season, after the last study has been completed, original raw data that have been generated for all current studies will be compiled in preparation for shipping to IR-4 Headquarters for archiving. These original items, such as annual standardization of temperature monitors for test substance storage and freezers, and standardization of instruments that record environmental data, will be included, as well as any other supporting original raw data or documentation that apply to the studies. These originals will then be sent to IR-4 Headquarters for archiving and a copy of the originals inventory list will be provided to the IR-4 Western Region office. A chain-of-custody form will be generated and include 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). Also, a scanned copy of the completed chain-of-custody form is kept at NWREC retention file, and is also sent to the IR-4 Western Region Field Office. HQ retains the original, signed chain-of-custody form.

3. Retention Files of facility records and documents will be maintained at NWREC (located in a "FireKing", or similar file cabinet, with a one-hour fire rating). 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; true copies of signed and dated SOPs; copies of supporting data associated with the facility such as work orders for freezers and balance maintenance; personnel and GLP training records; and QA facility inspection reports.

   b. All retention files will be maintained as digital copies on a backed up remote access server at OSU-Corvallis.

4. Copies of older field data notebooks may be discarded if no longer needed. This will 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 Notebook retention copies can be discarded if the project status indicates: Use Registered; Tolerance Established; Request Withdrawn; Mfg. Will Not Support; or Use Cancelled.

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20-2.8
Recording of Raw Data

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

PROCEDURES:

1. 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.

   During the active season, raw data may be located in the following areas:
   a. Freezer activity logs are located in each individual FDN.
   b. Test Substance activity logs, indicating amount removed with date and user initials will be located in each individual FDN. For multiple trials using the same test substance container, a true copy of the use log will be placed in the appropriate notebooks with the original in one of the FDNs.
   c. Equipment maintenance will be recorded directly into the FDN.
   d. All supporting data forms (e.g. field maintenance activity, etc) will be located on the desk in the IR-4 modular building or in the office of the Field Research Director.
   e. The field data notebooks will remain in the custody of the field personnel responsible for the study.

2. All raw data will be recorded in dark ink. Blue ink is preferred as it distinguishes the original from true copies.

3. If forms are generated to replace or supplement the forms in the IR-4 Field Data Notebook (e.g. freezer logs, chemical storage log), the forms must contain all information on the original form and, once completed, will be included in the field data notebook. Before use, modified FDN pages must first be approved by OSU IR-4 Field Center Director in consultation with the Western Region Regional Field Coordinator or designee.

4. Corrections will be made by crossing through the item, initialing, dating and giving reason or code for

the correction and circling code for ease of reference. Blank spaces in the field data notebook will be lined out; if there is no reason given for the line-out, it means the space was not needed.

5. Initial and date each activity as it occurs.

6. Electronic data, computerized summaries, etc., can be used in individual field data notebooks.

7. 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

8. 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.

9. Transcribed data in the field data notebook must indicate location of the original, and be verified by someone other than the transcriber with date and initials.

10. Pagination and the application of Field ID# labels in the FDN are not considered raw data and do not require a date and initial; these components are considered data organization. Copies of original documents or data will be indicated as such with a stamp or written statement that reads: "This is a true copy of the original"; with the location of the original provided/noted.

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20-3.7
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 (consult Study Director if evaluation methods are not included in protocol). If this data is optional, collection may or may not be done; however, 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 and printed for inclusion in the FDN.

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20-5.4
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: Do not round any numbers within a calculation; round only the final result. 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.

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30-1.7
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 other University or private agricultural grower sites. For FDN Part 5A, indicate the name and location (street, town, state) of the test site, the county, and provide directions from the nearest city or town to test site. If multiple trials of the same test substance are being conducted by the same FRD, considerations to FDN Part 6L will need to be addressed.

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. Determine prevailing wind direction. Placing the plots at the trial site is determined on a case-by-case basis but wind direction and slope should be considered. 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. The minimum buffer distance between treated and untreated plots that is listed in the protocol will be followed. However, when practical, the greater the buffer, the better. For FDN Part 5B the location of the irrigation source and meteorological equipment, if they are on site, need to be provided.

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

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

6. For FDN Part 5C prepare a plot map showing the location of each plot at the site. If other trials (IR-4 or other) are in close proximity to the study plot according to the instructions in the FDN, the location of these plots should also be included on the same map. 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. 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.

7. Measure the dimensions of each plot on the site using a suitable measuring device. Use stakes or flags to accurately indicate the plots on the site.

8. Identify each plot with an individual designation specific to that plot (e.g. untreated plot is TRT 01; treated plot is TRT 02). In addition, each stake, flag or card shall contain the Trial Identification Number and the name of the test substance. Not all flags in plot will have identification numbers as some flags are used to mark quadrants, etc.

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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.

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40-1.15
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 notebook. Check information in the Safety Data Sheet (SDS) and container label, and compare to information listed in the protocol; if discrepancies occur, contact the Study Director. Be sure test substance has been characterized for GLP studies; if not, contact Study Director immediately. When checking a test substance into the storage unit, be sure each container has the following information written on it: chemical name, expiration date, batch or lot number and storage conditions. If no expiration date is given, contact the Study Director. Check to be certain the name of the product listed in the protocol is the same as the name on the container label and the certificate of analysis (COA). COAs are either sent with the test substance, or e-mailed by the registrant or study director. Verify that the % active ingredient and formulation is the same as in the protocol. Alert Study Director to any discrepancies. If container label does not include storage conditions, such information may be found in the SDS and should then be written on the container. Field ID number, NWREC container ID number and name of study (test substance/crop) should also be marked on the container. The condition of test substance and its container, quantity received, date received and date entered into IR-4 storage unit should be documented in the field data notebook. If EPA Reg. No. is given in protocol, check to see if the test substance received has the same EPA Reg. No.

2. Each container of test substances for all IR-4 residue trials to be conducted at NWREC will be given a unique, NWREC container identification number in the format of the last two digits for the trial year/and the next two digits assigned sequentially in the order test substance is checked in. A list that includes the NWREC container ID number, the test substance chemical name, crop, field ID #, and field researcher's name, will be located in an FRD's office in a secure cabinet and completed as test substances arrive.

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, odor) 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, and include a

temperature recording device (e.g. HOBO). The HOBO is programmed to record every hour. Access to test substance will be limited to authorized personnel only. 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.

5. A temperature monitor will be maintained within the test substance storage unit. The data retrieved from this device should include the range of temperatures from the date when the t.s. was checked in until the last application. Maintain a backup temperature recording device, to be included in the test substance storage unit, in case of failure of the primary temperature recording device.

6. All test substance activity (amount removed from container, etc.) shall be recorded on the use-log form found in the FDN (Part 4B). This form will be individual to each test substance and will be included in the Field Data Notebook upon completion of the study.

7. Mixing will take place in the IR-4 modular building or at the offsite location of the trial. After mixing, the test substance and adjuvant (if used) will be put back into storage as soon as possible. When the test substance is checked out of storage for off-Station mixing or for extended periods of time, include a temperature monitor and an ice substitute such as blue ice in the transport container if weather dictates (be sure temperature monitor does not come in direct contact with the blue ice). 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. When returning the test substance back to storage unit, document the temperature during transit in the field data notebook.

8. Adjuvants must be handled in a manner to prevent contamination with test substances and other spray additives, e.g. dispense into a temporary container for measuring (and discard any remaining adjuvant in the temporary container), use a new disposable measuring device to measure adjuvant directly from the original container (and discard after single use), etc.

9. After completion of the current field trial (when no further applications will be made) 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 (test substance disposal is permitted per the National IR-4 Website database). Empty test substance containers should be triple rinsed if they are to be stored.

10. 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, substances will be packaged according to Department of Transportation and shipping company's chemical shipping policies, following instructions given with the SDS and tracking information.

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50-1.14
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 and OSU IR-4 Field Center Director 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. Prior to sample collection, obtain a sufficient number of sample bags to collect all the samples. Plastic-laminated cloth bags are preferred. Label each sample bag with an identification tag on the outside of the bag (and preferably attached) and include information requested in the protocol. Before entering the plots, place cloth sample bag in a plastic bag or other suitable container to protect it from dirt and possible contamination.

3. Collect and place samples directly into sample bags, if possible. Samples can also be collected directly into clean buckets and then transferred to laminated cloth sample bags, or into labeled zip-lock plastic bags which are then placed in the laminated cloth sample bag. If tools or other devices are needed for harvest, be mindful of decontamination of the tools; also, change gloves between treatments. Wash any sampling device with soap and water, or disinfectant wipes, prior to and after use for each treatment.

4. Read the protocol carefully to determine sample size and whether there are any special instructions for sampling the commodity.

5. Representative samples of the crop in each plot must be taken in an impartial manner usually stated in protocol. If none is stated in the protocol, contact the Study Director.

6. Each duplicate sample should be collected individually, with a separate "run" through the plot for each sample, beginning with the untreated plots and working up to the highest treatment rate. After collecting each sample, tie and secure the laminated cloth sample bag that has already been placed into a large plastic bag, then tie off the plastic bag with a twist-tie. If the samples cannot be placed into a freezer within the time allowed in protocol (Sec. 19), generally within about one hour, place them into coolers containing ice or an ice substitute (e.g. blue ice) and a temperature monitoring device in each cooler. Separate coolers are used for treated and untreated samples. Be certain both coolers are clean before and after each sampling event. For convenience at time of shipping, place duplicate samples (e.g. A and B, or C and D) that are already enclosed in an individual plastic bag, together into another plastic bag and secured with a twist tie.

7. For trials with multiple harvest dates (decline trials), collect samples from the untreated plot on the first sample date or before if allowed by the protocol. It is <u>not</u> necessary to collect untreated samples on the later harvest dates unless otherwise specifically requested in the protocol.

8. Take special care to do the following in the sample collection process:
    a. Avoid contamination of the field sample with the test substance during the sampling, labeling,

storage, and shipping processes.

b. Avoid taking diseased or undersized crop parts.

c. Take care to not remove surface residues during handling, packing or preparation; do not wash commodity unless specified in the protocol.

d. Be certain that tools are clean as sampling commences. Clean tools after each sample, or at least after each treatment. Wear clean gloves when sampling and change gloves between sampling different treatments.

e. Do not remove any soil or plant parts or trim the commodity unless it is specified in the study protocol.

10. Grass and Clover Hay Sampling

*For crop portions to be dried prior to sampling for **hay,** use the following procedure:*

a. Cut the crop at the correct crop stage and/or pre-harvest interval as per protocol and allow it to dry in the field. If the weather is unsuitable for drying to hay, if allowed in the protocol or allowed with approval from the Study Director, harvest the crop portions to be sampled and place in a covered building and allow them to dry to the correct moisture content as per protocol.

b. After cutting the plant material for each sample in the field, place the sample in a labeled paper bag or clean bucket (labeled with the field ID number and sample ID number), and be sure to keep the treated and untreated samples physically separated. Transport the plant material to a covered building, remove the material from the paper bags or buckets, and spread on clean paper. Label the paper on which the plant material for each sample is placed with the trial ID number and sample ID number.

c. Collect additional plant material at the same time the plant material for the RAC samples is cut. This additional plant material will be used later to help determine percent moisture content of the RAC samples after drying. Transport this plant material to a covered building to dry under similar conditions with the RAC samples. About 5-gal bucketful should be enough material to collect for drying to 0% moisture.

d. Under western Oregon conditions, it usually takes a few days for samples in a covered building to dry down to hay (hay will have approximately 10 to 20% moisture content). It helps to turn the samples periodically to aid in drying, beginning with untreated then treated samples using gloves at all times. After drying, estimate the % moisture by feel; if it feels moist, continue drying. When, by feel, it is estimated that the samples are hay, starting with untreated samples then moving to treated samples, place hay in cloth residue bags and sample as usual.

e. After sampling the RAC hay, the dry matter/moisture content of hay samples needs to be determined. Return to the additional dried plant material that was collected at harvest that has been drying concurrently with RAC samples (as per ‘c’ above).

   From this material, determine the initial weight of four small samples. Place these samples in drying boats and dry in an oven until there is no change (or within +/- 1%) in weight following successive weighing at least an hour apart, which indicates 0% moisture.

The values obtained in this procedure are used to determine percent moisture and percent dry matter of these samples using the following equations.

Determining Percent Moisture Content and Percent Dry Matter:

% Moisture Content= (initial hay weight – dry weight)/initial hay weight*100
% Dry Matter = 100 - % moisture

Report average of small samples as percent dry matter/moisture content of RAC samples.

11. Hop Cone Sampling
    a. Spread a clean tarp on the ground or in the back of a truck next to the hop hill to be harvested.

    b. Cut the bines at the base of the hill and cut the training string at the top of the trellis system; then pull the bines down from the overhead cable so that they land on the tarp. Move the tarp to the next selected hill and repeat the procedure until the required number of bines from the required number of hills within the plot have been cut down.

    c. Collect hop cones into a clean and labeled 5-gallon bucket, collecting from all portions of all the bines that were cut for sampling (making sure to take cones from the inside and outside portions of the bine, and all along the length of the bine). Cones for duplicate samples may be collected from the same set of cut bines as long as they are collected during a separate run through the entire set of cut bines.

    d. Collect enough hop cones until the fresh weight is more than five pounds for each sample, changing gloves between each sample. Keep the untreated and treated buckets separated during transport back to the station.

    e. At the time of harvest for RAC samples, additional plant material from the same area is taken for dry matter and percent moisture prediction and verification. About half a 5-gal bucketful should be enough material to collect for drying to 0% moisture.

12. Preparation of Dried Hop Samples:
    a. The two dryers in use are VWR Shellab Horizontal Flow Model 1685. Generally, there isn't regular maintenance performed on the dryers, but the yearly cleaning performed prior to placing samples in the dryers should be documented. The area around the outside of the dryers should be kept free of debris. Prior to the field season, or prior to placing samples in dryer, place a temperature recording device in each dryer to verify that the dryers are running at the set temperature. A copy of this pre-season temperature check may be placed in the FDN. This data may be maintained as a facility record.

    b. Starting with the untreated samples, carefully transfer fresh hop cones from 5 gallon buckets to labeled mesh drying bags. Tie off drying bags, weigh and record weight, date and time for each bag and place them on shelves in the dryer for untreated samples with the temperature set at 140 °F (±12 °F).

    c. Once all untreated samples have been placed in the dryer, drive the short distance to the treated sample dryer and load the treated samples into the dryer using the same method as already described for untreated samples.

d. Taking precautions not to cross contaminate samples, periodically monitor and record weights to determine when the target weight has been reached that will correspond to the proper range of % moisture content for the RAC samples (see below).

e. At the end of drying, hop cones will be warm in the drying bags and should be set on a clean surface to cool, generally not more than an hour, to ambient air temperature. Once the samples have equilibrated, and starting with the samples in the untreated dryer, carefully transfer the dried hops from the drying bags to appropriately labeled residue sample bags. Weigh each bag and record weight, date and time before placing sample bags in the freezer. Record the time that has elapsed between sample collection and placement in the freezer. Once the untreated RAC samples are in the freezer repeat this procedure for the treated samples.

13. Drying RAC samples to protocol-specified moisture contents

Some protocols require RAC samples to be placed in the freezer at a certain % dry matter or moisture content. As these RAC samples are never dried to zero moisture it is necessary to have a method of accurately predicting and confirming the dry matter/moisture content of the RAC samples so that they can be removed from the drying oven at the correct % moisture.

The following are methods used to predict RAC sample dry matter content and predict weight of RAC samples that will meet percent moisture content limits stated in the protocol.

a. Method used to predict RAC sample weight at specified moisture content:
At the time of harvest for RAC samples, additional plant material from the same area is taken for dry matter and percent moisture prediction and verification.

From this material four small samples (~100 g) are weighed, placed in drying boats (pie pans) and dried in an oven until there is no change in weight (or within +/- 1%) following successive weighing at least an hour apart, which indicates 0% moisture. The BA3 scale is used to measure material.

The values obtained in this procedure are used to determine percent moisture content and percent dry matter of these samples using the following equations.

b. Determining Percent Moisture Content and Percent Dry Matter:
% Moisture Content = (Initial fresh weight – dry weight)/Initial fresh weight*100
% Dry Matter = 100 - % moisture

Record the average of the values for the small samples as the % dry matter/moisture content for the RAC samples

c. Determining target post-drying weight of RAC samples

Target weight of RAC sample = (% dry matter of fresh sample * fresh weight of RAC sample)/reciprocal of required moisture fraction

Example for Hops:
Calculated % dry matter = 27%
Fresh weight of RAC sample = 5.0 lbs

Protocol requirement for RAC sample moisture content = 8 – 12% which corresponds to 92 to 88% dry matter content.

(0.27*5.0 lbs) = 1.35 lbs/0.92 = **1.47 lbs** (to achieve 8% moisture)
(0.27*5.0 lbs) = 1.35 lbs/0.88 = **1.53 lbs** (to achieve 12% moisture)

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. For trials conducted at NWREC or other University property, a different set of circumstances exists. These plots are considered off-limits and non-accessible to un-informed personnel or the public. They are under close supervision of IR-4 personnel; flagging and signage clearly mark the trial plot locations. 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 understanding that the treated crop cannot be consumed by humans and/or animals

4. Whether a plot is at a grower field or on university property, include in the field data notebook an explanation of when (e.g. the date) and how the crop was destroyed or why no crop destruction was necessary.

50-3.14
Sample Storage and Freezer Use

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

PROCEDURES:

1. 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 in the field data notebook. Removal of samples should be recorded on the log form and include: time and date removed, Field ID #, person's initials. There are two upright freezers. One freezer, marked "FRZ1" is the main freezer. The second freezer, marked "FRZ2" will be used only in case of catastrophic failure of the main freezer. For 2020 we will primarily be using the "FRZ1" freezer. There is also a BKP (back-up freezer) available if needed (See item #10 below).

2. 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.

3. Storage temperature of the samples will be recorded automatically with the use of HOBO "U-12 Stainless Steel Temp Loggers" that will be kept in the freezer to ensure that the freezer temperature is maintained to preserve the commodity as stated in the protocol. The freezer contains two HOBOs, designated as Recorder C and D (one primary and one backup). Set temperature monitors to record every 30 minutes. Be certain temperature monitors are placed in the middle shelf of the freezer at all times. Temperature monitors or probes may be placed in a buffer (ethylene glycol) to prevent temperature fluctuations. It is imperative that the bottom racks in the freezer are always at least 2-3" from the bottom of each unit allowing good air circulation in the freezers.

4. The freezers where the samples are stored will have limited access in a dedicated temperature controlled room. Freezer doors will remain locked with keys on wall near light switch.
5. All maintenance, repairs and cleaning for the freezers will be documented in the freezer maintenance log. The freezers will be cleaned approximately once per year, before the active season, wiping/washing the racks and entire interior, and rinsing well. The area around the outside of freezers should be kept free of debris.

6. Maintain freezers preferably at or below 0°F (-18°C), or at temperatures stated in the protocol. Regular, auto-defrost cycles of the freezers are normal. Temperatures may rise for a short period due to the defrost cycle (spikes) and is not cause for alarm. 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. If the light labeled "regular mode" is on and the freezer temperature is rising, then a malfunction has occurred (see #10 below for actions to be taken in such an event.)

7. 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 and, whoever hears the alarm is instructed to contact one or all the names on the list. An OSU employee lives on-site and has been instructed to call IR-4 personnel if the alarm sounds; he will hear the alarm and make contact whether during work hours, after work hours, during the night, or on the weekend. This system also has an automatic dialer that is engaged when the

temperature rises above 5ºF for one hour. It will phone the on-site OSU employee and IR-4 personnel, informing them, via a recorded message, of the rise in freezer temperatures.

8. In the event of a power outage and electricity is not reaching the freezers, a back-up generator, powered by natural gas, will start automatically and provide power to the freezers.

9. Testing the Alarm and Natural Gas Generator: To be certain that the alarm system and the gas generator back-up system are working properly, a test is performed at the beginning of each season and several times during the season (generally after the freezer truck comes and the freezers are empty). To simulate a freezer malfunction and test the alarm systems (horn and phone dialer), 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, if the alarm and phone dialer are working properly. 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 to cut the electricity going to the freezers; 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 to ensure that it is working properly. The automatic alarm will be tested prior to use for the field season with the details of the test result (i.e. pass/fail) recorded in the freezer log. Should the test fail, corrective actions will occur until the test provides a pass outcome.

    a. Thoroughly document the testing in the freezer maintenance log with information including the date and time of the test, time(s) messages were received, and personnel contacted. (See item #10 below for further details.)

    b. If the system did not work properly in the test, complete repairs as soon as possible, document corrective actions and conduct a subsequent test to ensure the system is working.

10. What to do if a freezer malfunctions: If the freezer alarm sounds, indicating a rise in temperature above 5°F for greater than one hour (due to a freezer malfunction and not due to a power outage), remove samples from the malfunctioning freezer and place in large, plastic bags, labeled with ID numbers, then place them and the datalogger in the BKP, keeping treated and untreated samples separate (e.g. different shelves). The second upright freezer "FRZ2" will be turned on and utilized until the freezer can be fixed. Be sure to include a temperature recording device with samples no matter where samples are temporarily stored.

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50-4.12
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. As per protocol, contact the residue laboratory by phone, fax or e-mail, and notify them of the shipment dates and method of shipment. Overnight air shipments should be made on Monday, Tuesday, or Wednesday, to avoid potential weekend layovers.

2. Complete Form 8A ("Residue Sample Shipping Information") and Form 8B ("Residue Sample Chain of Custody"). Include a true copy of 8B and a blank copy of 8C ("Sample Arrival Check Sheet") in each shipping container (preferably within a plastic bag). Retain the original of Form 8A and Form 8B

in the field data notebook.

3. 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.

   For overnight air shipping:
   a. Obtain insulated containers or coolers of sufficient size and strength to hold the residue samples plus dry ice (where required). Using one container for UTC samples and another one for TRT samples is preferable, but shipping UTC samples and TRT samples from the same trial in the same container is possible if physically separated by at least 2 additional plastic bags or other suitable method.
   b. Individual boxes should weigh no more than 75 lbs each.
   c. Containers should approximate the size of the samples plus dry ice; pack so shifting of samples and ice does not occur during transit. (Fill any voids with wadded paper, bubble wrap or Styrofoam to minimize sample movement as the dry ice sublimes.)
   d. Samples should already be hard-frozen prior to packing with dry ice, unless permission is given by the Study Director to do otherwise. Pack the containers with enough dry ice (just prior to shipment) 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.
   e. Fill out documentation, as needed. Place plastic bag containing a true copy of Form 8B ("Residue Sample Chain of Custody") and a blank Form 8C ("Sample Arrival Check Sheet") within each container and seal the container with tape, leaving gaps in the seal to allow dry ice gas to escape.
   f. Labels don't stick very well to plastic coolers due to condensation on the outside caused by the cold samples; hence it is preferable to place the plastic cooler in a cardboard box and affix shipping labels to the box. Be sure the official dry ice sticker (black and white diamond) is affixed to each box and is filled in with appropriate information.

   For ACDS (freezer truck) shipping:
   a. Obtain sturdy boxes large enough to contain samples to be shipped. Using one box for UTC samples and a separate box for TRT samples is preferable. Shipping UTC and TRT samples from the same trial in the same box is acceptable if they are physically separated by placing the frozen TRT samples in bottom of box then placing the already double bagged UTC samples in an additional plastic bag and placing on top of TRT samples.
   b. Individual boxes should weigh no more than 75 lbs each
   c. Fill out all required shipping forms. Place a plastic bag containing a true copy of Form 8B ("Residue Sample Chain of Custody") and a blank Form 8C ("Sample Arrival Check Sheet") within the box and seal the box with tape.
   d. Once the ACDS driver arrives, the samples are removed from the freezer and placed in the appropriate box. Boxes taped shut, weighed then placed in freezer truck.

5. Label each shipping container with the following information:
   a. Name and address of the sender
   b. Name and address of the residue laboratory receiving the samples
   c. Apply tape over any stickers and labels to secure them to the container

6. For the carrier, the phone number of the laboratory receiving the samples is recorded on the shipping form.

7. Once the samples are picked up by the carrier, fax or scan a true copy of Form 8A ("Residue Sample

Shipping Information") and Form 8B ("Residue Sample Chain of Custody") to the Study Director, OSU IR-4 Field Center Director and the Western Region Regional Field Coordinator. Retain all originals in the field data notebook.

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60-1.14
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.

EQUIPMENT LIST: See the Equipment Log located in the Facility Files for a list of equipment currently in use.

PROCEDURES:

1. Instruments used in GLP field research should be tested to determine that they are accurate to meet the data-recording requirements of professional scientific methods and, specifically, what is required for GLP or the protocol. At a minimum, standardize each environmental testing instrument once per year. Testing should be completed under conditions similar to those under which the instrument will be used. Testing can include standardizing instruments with comparison to two other like instruments; they should test out at ±5% of the average of all (except wind speed indicators, which will be at ±10%). If one is more than 5% different (or 10% for wind meters) than the average, that instrument will not be used; it should be 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.

   New instruments are considered calibrated for the period of one year unless the manufacturer specifies another date (pressure gauges are handled differently, as explained below). Regardless of the accuracy-testing method, 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. Any instrument that uses batteries will have them changed prior to annual calibration/standardization and recorded in the maintenance log. 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.

2. 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.

3. Pressure Gauges:
   Pressure gauges can be tested for accuracy at the beginning of each season.

4. pH: pH of the water used as carrier for the test substance is determined via pH paper test strips. Dip the pH paper strip in the water, wait for the amount of time indicated in the instructions, then compare the strip colorimetrically to the chart provided with the strips. Record the pH as the closest in matching color.

5. National Institute of Standards and Technology (NIST) Thermometer

A NIST thermometer is used when standardizing temperature recording devices used in GLP trials. The NIST is used for comparison only and is not used when calculating the average of the temperature recording devices. Either a new NIST thermometer will be purchased with certification that it is accurate or the one currently being used will be re-certified every 3 years. The NIST thermometer will be replaced before the 3-year expiry period. Record the results in the maintenance log.

6. Soil and Water Thermometers:
   Soil and water temperature is measured using a digital thermometer. 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. It is recommended to standardize the thermometers under conditions that generally reflect the conditions in which the thermometers will be used. For example, 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.

7. Kestrel 3000 for air temperature, relative humidity, and wind speed:
   The primary instrument used to measure air temperature, relative humidity, and wind speed is a digital Kestrel 3000 by Nielsen-Kellerman. Prior to standardization change the battery. Standardization is accomplished by comparing readings from the three instruments while under the same conditions.

   - To standardize for air temperature and relative humidity, line up the instruments on the same surface, at the same level, under the same conditions. After about 15 minutes, 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. Instruments should be standardized in the approximate range of the temperatures likely to occur in the unit. Record the results in the maintenance log.

   - To standardize for wind speed, align the instruments next to each other on the same surface, aiming them all in the same direction. Conducting the standardization indoors with a fan directed towards the meters facilitates the standardization process. Read all three meters concurrently and document. Results should be within 10% of the average of all three instruments. It is recommended to standardize the wind function of the Kestrels under a range of wind speeds. For example, for a given standardization, altering the distance from the fan that the instruments are placed will give different wind speeds. Record the results in the maintenance log.

8. Temperature-recording devices for Freezers, Test Substance monitors:

   For Freezers: U-12 Stainless Steel Temp Loggers are used to record temperature in the freezers. Each freezer will contain two Temp Loggers. Check battery status prior to standardization.

   To standardize: Using the computer software, launch the four loggers and set them to record for a short span of time (about every minute for approx. 1 hour). Place all four loggers and a NIST-certified thermometer for comparison, in a freezer that is maintaining at approximately a temperature less than 0° F (-18° C). After approximately one hour, immediately read and record the temperature of the NIST thermometer, download all loggers, and print out a graph for each logger which can be combined into a single graph showing the temperature readings of all loggers. (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 graph should be "zoomed-in" to show the stabilized temperatures (i.e. where the approx. lowest readings become constant), and then printed. 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, use a different temperature recording instrument, or send logger back to manufacturer for repair or replacement.

For Test Substance: For monitoring temperature of the test substance in the facility storage unit or during transport, UX100-003 temperature loggers are used. Check battery status prior to standardization.

To standardize: Using the computer software, launch HOBO loggers, and set them to record about every minute for approx. 1 hour. Place all the temperature-recording devices in the storage unit. Include a NIST-certified thermometer for comparison. After approximately one hour, immediately read and record the temperature of the NIST thermometer, then download all the Temp Loggers and print out a graph for each. The graph should be "zoomed-in" to show the stabilized temperature (i.e. where the readings become constant), and then printed. The reading of each unit must be within 5% 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 5% of the average of the HOBOS. If results of the standardization are greater than 5% of the average, replace a battery, use another Temperature Recorder, or send the unit back to the manufacturer for repair or replacement. Instruments should be standardized in the approximate range of the temperatures likely to occur in the storage unit.

9. Balance Calibration:
BALANCE-1 (OHAUS scale) and BALANCE-2 (Sartorius scale) are balances used for weighing test substances. It is recommended that professional calibration and maintenance on these balances be performed approximately once every two years. Standard weights used for balance bracketing and validation will also be checked at this time.

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60-2.10
Application and Harvesting Equipment Maintenance

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

1. Routine and non-routine equipment maintenance is recorded in Part 6M of FDN. Non-routine maintenance is when something breaks and needs to be fixed. When describing non-routine maintenance, state the nature of the defect, how and when it was discovered, and any remedial actions taken. Routine maintenance includes changing screens, nozzles, etc. When describing routine maintenance add the SOP followed, the date of occurrence and a clear indication that this was routine.

2. Spray booms are cleaned during post-application cleanup, which is documented in the field data notebook 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.

3. Inspect nozzle tips and screens for wear. Check spray pattern from each nozzle. Nozzle tips 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.

4. 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.

5. For the gas-powered mist blower (S_3), 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.

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60-3.12
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. Determine whether all nozzles are discharging uniformly by spraying water through them at a uniform pressure and catching the discharge from each nozzle in a separate container for at least 30 seconds. Begin timing the discharge after the system is primed or charged. Start the spray system and the stopwatch simultaneously, timing the discharge. 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, and adjust diaphragm check valves, if such valves are being used. If an individual nozzle discharge still varies more than 5% from the average discharge of all nozzles, replace questionable nozzle tip or screen.

4. **DISCHARGE RATE CALIBRATION FOR SPRAYERS**

a**. Boom Sprayer**: Average discharge rate of the spray equipment can be accomplished by collecting discharge from each nozzle for at least 30 seconds. Charge system and then begin timing the discharge. Collect and measure discharge from each nozzle. Replicate this procedure three times. The total boom discharge rate (ml/sec) is determined by dividing the average boom output (ml) by the average time (sec).

b. **Airblast Sprayer:** Park the airblast sprayer on level ground. From the protocol, determine the appropriate nozzle placement to be used in the application. Prime the system by filling the tank part way with water and turn on sprayer to fill the lines. Turn off sprayer and empty the tank completely, via the drain hole. Close the drain hole and fill the tank again with a known amount of water that has been measured with a graduated cylinder. Engage the PTO and bring RPMs to proper level, as determined previously during speed calibration procedure. Start the sprayer and spray for at least 30 seconds. Shut off the sprayer and the PTO. Via the drain hole at the bottom of the sprayer, collect the water that was left in tank and measure with a graduated cylinder. To determine sprayer output, subtract this amount from the known amount initially added to the tank. Replicate this procedure three times. The total discharge rate (ml/sec) is determined by dividing the output (ml) by the average time (sec).

c. **Air Mist Sprayer (Mist Blower):** Add a measured amount of water to the spray tank then start the mist blower motor. Begin timing discharge as you simultaneously turn the flow control valve to the open position. Allow the mist blower to run at least 30 seconds then close the flow control valve and stop the motor. Carefully disconnect the liquid delivery tube from the aspirator jet and place it in a catch jug. Open the flow control valve and allow all the remaining water to drain into that jug. Measure the drained water with a graduated cylinder and subtract this volume from the initial volume placed in the tank. Record the time and volume discharged. Repeat two more times. Divide the total volume discharged by the total time for the three runs to get the output in mls/sec.

5. **DISCHARGE RATE <u>RE-CHECK</u> for boom sprayer, air-blast sprayer, and mist blower:** For subsequent applications in a specific field trial where spray applications parameters have not changed (i.e. pressure, nozzle type, volume, etc.), a single recheck calibration will be made prior to the application to verify that the discharge rate has not changed more than 5% from the original, replicated calibration. When doing a calibration recheck for subsequent applications, calculate the discharge rate in ml/sec and then divide the discharge rate of the recheck by the original discharge rate (as determined previously after three replications). The calibration recheck should be ±5% of the original calibration. If not, check nozzle(s) for possible cause of discrepancy, and clean if necessary. Recheck the discharge rate and compare again to the original discharge rate. If the recheck discharge rate still differs by more than 5% of the original discharge rate, the spray equipment must be recalibrated again by collecting output three times and taking the average. This discharge rate now becomes the "original" calibration discharge rate, or target rate, used in future calibration rechecks. If there is a change in discharge rate as the result of a new full calibration it is necessary to perform a new full speed calibration.

   If application equipment is transported offsite after the most recent calibration or recheck, another recheck is prudent and may be required by the protocol.

6. **AMOUNT OF TEST SUBSTANCE NEEDED PER PLOT:** The amount of test substance needed per plot is determined by multiplying the plot acreage (which is determined by dividing plot size, expressed in ft^2, by 43,560 ft^2/acre) by test substance rate per acre, as stated in the protocol.

Occasionally, the protocol will indicate that the amount of test substance required is a concentration; in this case, follow directions in the protocol to make a dilution that will achieve the correct concentration needed for the trial.

7. **AMOUNT OF CARRIER NEEDED PER PLOT FOR HAND-HELD BOOM AND AIR MIST SPRAYERS:** First, divide the area of the test plot to be sprayed (expressed as ft^2) by 43,560 ft^2/acre to determine the percentage of an acre the test plot occupies. Then multiply the plot acreage by the gallon per acre rate indicated in the protocol; this will indicate the amount of carrier (in most cases, water) in gallons needed to make an application of the test substance to the test plot (to determine rate of carrier in milliliters, multiply gallon per plot by 3785 ml/gal).

   For example: Test plot = 10' long by 30' wide; 10 x 30 = 300 sq. ft.; 300 sq. ft. per plot divided by 43,560 sq. ft. per acre = 0.0069 acre per plot. If rate for delivery of test substance in the protocol is stated to be 100 gallons per acre, then: 0.0069 Ac/plot multiplied by 100 gallons/Ac = 0.69 gallons per plot; (0.69 gallons/plot multiplied by 3785 ml/gallon = 2612 ml/plot).

   Once the amount of carrier per plot has been determined, multiply that amount by a percentage (such as 25-50%), to allow for enough carrier to charge the system and to allow for a few seconds of spraying before and after the plot begins and ends (this is referred to as "overage"). Take into consideration the number of nozzles and the target pass time to ensure there will be enough spray solution for the application. For smaller plots, with a low GPA, overage may need to be as much as 100-200% to be certain there is enough solution. Whatever percentage is used for overage of the carrier, be sure to use that same amount of overage for the test substance (and adjuvant, if used).

   To determine if the gallon per acre requirement listed in the protocol is being met: after the application, when the amount of spray solution delivered per plot has been determined, convert ml/plot to gallons per acre (e.g. milliliters of solution delivered per plot divided by 3785 ml/gal = gallons of solution per plot; divide gallons per plot by plot acreage to determine gallons per acre). Record this actual delivered GPA in the FDN.

8. **AMOUNT OF CARRIER NEEDED PER PLOT FOR AN APPLICATION WITH A TRACTOR BOOM SPRAYER OR AIRBLAST SPRAYER MOUNTED ON A TRACTOR:** The amount of carrier (ml/plot) is determined by knowing the discharge rate (as determined above in Item #4), the tractor speed (as determined below in Item #11), the boom width, and the plot size.

   For example, if the tractor speed is 5 feet per second, the plot size is 50 ft long by 30 feet wide, the boom width is 15 feet, and the boom discharge rate is 150 ml/sec, the calculation can be made as follows:

   50ft plot length divided by 5ft per second tractor speed = 10 sec to drive length of plot; 10 sec per length of plot times 150 ml/sec boom output = 1500 ml per length of plot; 30 ft wide plot divided by 15ft boom width = 2 passes to cover entire plot; 2 passes times 1500 ml per pass = 3,000 ml/plot.

   To determine if the gallon per acre requirement listed in the protocol is being met, convert ml/plot to Gallons per Acre (For example, a plot size of 50 ft x 30 ft = 1500 sq ft = 0.0344 acre; 3000 ml = 0.793 gal; 0.793 gal/0.0344 acre = 23 gal per acre).

9. **AMOUNT OF TIME NEEDED TO DELIVER TEST SUBSTANCE TO THE PLOT:** Determine the amount of time required to deliver the required amount of spray solution required to the

test plot (water carrier and, if applicable, adjuvant and liquid formulation of the test substance) by dividing amount of solution needed per plot (ml/plot) by the boom discharge rate (ml/sec), which gives you a target time (seconds) per plot. Determine how many passes are needed to deliver all of the required test substance to the test plot. If more than one pass is required, divide the total amount of time needed for the entire plot by the number of passes, to determine the time needed per pass.

10. **SPEED CALIBRATION FOR A BACKPACK SPRAYER OR MIST BLOWER:** Prior to the initial application of the test substance in a trial, practice pacing to achieve the target pass time (as determined above in #9) needed to deliver the proper amount of test substance to test plot (as determined above in #6). In the plot, adjacent to the plot, or on a surface similar to the actual plot and with a similar slope, and a length equivalent to the treated plot, walk at a comfortable pace keeping track of time with a stopwatch or wristwatch. A metronome can be used to help keep a steady, even pace. Once a pace is achieved that will ensure the proper delivery rate of the test substance, repeat three times, calculate average speed, and record in the Field Data Notebook.

11. **SPEED CALIBRATION FOR A TRACTOR:** Prior to the initial application of the test substance in a trial, determine the speed (gear, settings, or RPMs) of the tractor that is necessary to deliver the correct amount of test substance to the plot (the speed should be reasonable; not too fast and not too slow). To perform a speed calibration, flag the length of the actual plot (or the plot length on an area outside the plot), or a known distance (e.g. 100 feet), as long as the surface and driving conditions are similar to those of the actual plot). Set the tractor speed and make a minimum of three passes to get an average. Speed will be expressed as ft/sec. Be certain to adjust speed, if necessary, to coincide with protocol parameters for GPA. Document the three run pass times for initial calibration in the FDN.

12. **SPEED CALIBRATION RE-CHECK:** For subsequent applications, a speed calibration recheck is required prior to the application to ensure that the proper amount of test substance will be delivered during the application. Set the walking speed (pace) or driving speed (gear, settings, or RPMs) as per the original replicated speed calibration and walk/drive the length of the plot or predetermined distance. If results of the speed calibration recheck are not within 5% of the original speed calibration, another complete speed calibration must be done

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60-4.13
Measuring a Test Substance Formulation

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

PROCEDURES:

I. Liquid Formulation

1. Obtain a clean container large enough to hold the volume of test substance needed and graduated in increments small enough to ensure accurate measurement. For example, if amount needed is less than 10 ml, use a pipette, graduated cylinder or syringe with 0.1 ml increments. If amount needed is 10 to 25 ml, increments should be 0.2 ml; if 25-100 ml, increments should be 1.0 ml; if 100-250 ml, increments should be 2.0 ml. If greater than 250 ml, a cylinder with 5 ml increments will be adequate. Carrier (e.g. water) is usually needed in amounts greater than 1,000 ml and using a cylinders with 10, 20 or 50 ml increments is adequate. Readings of the liquid test substance or water must be made at the bottom of the meniscus.

2. If the opening of the cylinder is too restricted to allow pouring of the test substance from the original container into a graduated cylinder without danger of spillage, then do one of the following:
   a. Use a clean glass container with a pour lip as an intermediate and fill the cylinder from it.
   b. Use a clean funnel large enough to allow filling the cylinder with a minimum of spillage.
   c. Use a pipette with appropriate gradations and a squeeze bulb to extract the test substance directly from the test substance container and inject it into the spray tank.
   d. Use a syringe with appropriate gradations to extract the test substance from the container then into the spray tank.

3. 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.

4. Amount of test substance and water needed for the application is determined by knowing the rate of test substance per acre and plot size. Once dosage and water for the individual plot is determined, (from SOP# 60-3 "Sprayer Calibration"), allow for and calculate overage, to ensure there is enough test substance spray solution to prime the boom and to reduce risk of running out of solution before exiting the plot. See SOP# 60-3, Item #7, for details.

5. If application of test substance for a given trial is to be made some distance from the test substance storage unit, the test substance can be transported in a clean, stable container (preferably a cooler) separate from the application equipment. When a secondary test substance container is used, label the container with trial ID, chemical name, expiration date, batch or lot number and storage conditions, plus the date and amount of test substance measured. During transport, ensure the integrity of the test substance by including blue or wet ice in the transport container and document the temperature during transport of the test substance with a Temperature Monitor placed in the transport container.

6. Use appropriate safety equipment while handling pesticide concentrate and spray solution.

II. Dry Formulation

1. 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.

2. The scale should be verified immediately prior to weighing the quantity of test substance for use in the study. A set of certified stainless steel weights are used for scale verification. Verification can be accomplished as follows: Place on the scale the container into/onto which the test substance will be weighed and tare the scale. Then, bracket the weight, as appropriate (e.g. if 7 grams of test substance is required, calibrate with certified weights of 5 grams and 10 grams) and record the verification in the FDN. Do not use the scale if the scale reading varies more than 1% from the certified weight. If greater than 1% variance, then use a back-up scale that has been properly calibrated. The OHAUS scale (BALANCE-1) can be used for weighing any amount of test substance within the range of the scale (0.001 g to 150.0 g). The Sartorius scale (BALANCE-2) however, due to the 1% variance requirement, can be used only for weighing test substances between 10 g and 1200 g.

3. Use appropriate safety equipment while handling the test substance.

4. Add the test substance into/onto a tared container and record the weight in the FDN. 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.) The test substance can be added directly to the spray container, especially if using plastic spray bottles, as follows: add enough pre-measured water to the bottle to dissolve the test substance when the bottle is shaken or swirled, then add remaining pre-measured water and swirl to mix.

5. Alternately, with some difficult-to-dissolve dry formulations, it may be desirable to create a slurry before adding the test substance to the spray container. If so, with the use of a stirring rod, mix the test substance into a slurry with some pre-measured carrier. Pour the slurry into the spray container (into which some pre-measured carrier has been added). Rinse the slurry container at least three times with additional pre-measured carrier and pour each rinsate into the spray container. Add remainder of the pre-measured carrier to the spray tank and attach the cap or lid. Agitate or shake carefully to additionally mix the contents.

6. Amount of test substance and water needed for the application is determined by knowing the rate of test substance per acre and plot size. Once dosage and water for the plot is determined, calculate overage to ensure there is enough test substance solution to prime the boom and to reduce risk of running out of solution before exiting the plot. See SOP# 60-3, Item #7, for details.

7. If application of test substance for a given trial is to be made some distance from the test substance storage area, it is acceptable to pre-weigh the required amount of dry formulation needed for the application (rather than transporting the entire original container of test substance to the test site). The amount needed can be pre-measured, including the option of a second back-up test substance quantity (following procedures outlined in steps 1 through 5, above) into an appropriate container and label properly with trial ID, chemical name, expiration date, batch or lot number and storage conditions plus the date and amount of test substance weighed. The test substance should be transported in a clean, secure container (preferably a cooler) separate from the application equipment. During transport of the test substance, document the temperature with a temperature monitor included in the transport container.

III. Granular Formulation

Follow the protocol for the method of measuring and applying a granular formulation to the plot without dilution. Generally, the method will be to weigh as for dry formulation in Part II of this SOP but instead of mixing with a carrier, pour test substance directly into application container. Or several packets/containers may be used to hold pre-weighed quantity of test substance for direct application (for example in-furrow, or at planting application).

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60-5.4
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 protective clothing.

2. Equipment used in the application of a test substance should be inspected and calibrated prior to application. Check that all settings are the same as the previously performed calibration.

3. For the initial 3-run output calibration put an adequate amount of clean water in the tank and charge the system by spraying out enough water to fill hoses and spray boom. Set up collection buckets and collect discharge from each nozzle for at least 30 seconds. The average output of the three runs will be the original discharge calibration (ml/sec) that will be used for comparison in trials with more than one application. After the original output calibration is performed, as long as parameters have not changed and the equipment has not been used for another trial then a single output discharge is adequate for future applications if the discharge collected during the recheck is within 5% of the discharge determined during initial, 3-run calibration. If not within 5%, then check application equipment (especially nozzle tips for possible blockage). If calibration re-check is not within 5% of the original 3-run calibration, then a new 3-run calibration must be performed. The average boom output from the new, 3-run calibration now becomes the "target output" when performing the back calculation after each subsequent application. At this point a new 3-run speed calibration will be necessary.

4. After mixing test substance and driving to plot, charge the system far 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. 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.

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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 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.

4. 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 notebook, and any actions taken recorded in the appropriate equipment maintenance log and FDN.

5. In the event of using up solution prior to the end of the spray plot, the following procedure should be applied:
   a. Mark and calculate area actually sprayed with the test substance.
   b. Calculate rate applied. If rate applied is within acceptable levels as specified by protocol then note changes in area applied within the field data notebook and sample from within the marked area at the appropriate time. If acceptable levels are exceeded and it is the first scheduled application, contact the Study Director, then mark a new area for the treated plot and re-initiate the trial.

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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 S_3 tank of the backpack mist blower, especially if an adjuvant has been used, a solution of tank cleaner may be necessary to remove leftover oily residues. Remove the tank lid and screen filter on top of the tank. Then unscrew the outlet hoses that the solution empties into during an application. Once this hole in the bottom of tank is open the solution of tank cleaner mixed with warm water can be easily flushed throughout the tank. 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. There are no nozzles associated with this piece of equipment.