

UNIVERSITY OF IDAHO MINOR USE PESTICIDE DEVELOPMENT PROGRAM

315 FALLS AVE EVERGREEN BLDG TWIN FALLS IDAHO 83301

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SOPs reviewed by: Will Mecke 3/10/17
Date review completed

SOPs submitted by: Will Mecke 3/10/17
Field Research Director Date

SOPs approved by: A. R. Flanagan 3/14/17
Stephen Flanagan, Assistant Regional Field Coordinator Approval Date

Approval Date is Effective Date

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STANDARD OPERATING PROCEDURES

CREATION, MAINTENANCE, AND USE OF STANDARD OPERATING PROCEDURES

SOP NUMBER: 0100

REVISION NUMBER: 6

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 4-7-15

Purpose and scope

This standard operating procedure (SOP) is to ensure that SOPs are written and formatted properly, and used when required.

This SOP is for use by U of I research personnel when creating original or revising existing SOPs, and for use by quality assurance personnel when auditing SOPs and facility records for GLP compliance.

Formatting

SOPs should be written by individuals who are completely familiar with the process described and include enough detail so that the procedure can be performed and completed by someone with appropriate education, training and background. SOPs should function as an adjunct to training in a procedure or function. Prior to performing any procedure, the SOP must be read and understood by the person that will perform the procedure.

An SOP must consist of:

1. A cover page, which should include:

the university name and address

the SOP title

the SOP number

the revision number

by whom it was written or edited

the revision date

The following pages should include;

1. a running header with the SOP number, revision number, and page number
2. A Purpose and Scope section, which explains the justification for the SOP and identifies personnel to whom it applies and when it is applicable.
3. A definitions section (optional), which includes any terminology used in the SOP that is specific to the SOP or that the author feels needs to be clarified.
4. The body, which elaborates on the purpose and scope section. The goal of this section is to give instructions on how the operation is to be performed, such that the least experienced person whose job description includes that procedure can execute the task using the SOP alone and achieve accurate, repeatable results.
5. Where appropriate, a section designating personnel (primary and backup) to carry out the procedure or ensure that it is done.
6. Where appropriate, a section showing both required and suggested documentation that may be generated by using the SOP.
7. Where appropriate, a Contingencies section outlining possible contingency plans in case of equipment failure or other unexpected circumstances.

Numbering of SOP

Each new SOP shall be placed in the appropriate category of similar type SOPs and given a number fitting within the category.

0000-Administrational procedures.

1000-Raw Data procedures.

2000-Test System procedures.

3000-Test Substance and Measuring procedures.

4000-Equipment procedures.

5000-Sampling procedures.

Review and revision

Each SOP shall be reviewed and, where appropriate, revised at least every 16 months. Upon revision, the existing SOPs shall be collected and replaced by the revised edition. This is to be performed by the personnel in charge of revising the existing SOPs.

Correcting spelling and formatting errors along with making minor changes that clarify the intended meaning of the SOP do not constitute a revision.

When to use an SOP

An SOP should exist that describes any procedure regularly performed that is not specifically described in a protocol. Some protocols will require, or the sponsor may request, that the sponsor's SOPs be followed. The protocol always takes precedence over any SOP. The data should reflect what procedure was followed, by whom it was performed, and when.

Retired SOPs

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 subsequent years.

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STANDARD OPERATING PROCEDURES

ARCHIVING OF HISTORICAL RAW AND FACILITY DATA

SOP NUMBER: 0150

REVISION NUMBER: 8

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-10-09

Purpose and scope

This standard operating procedure (SOP) is to ensure that historical, raw and facility data are properly archived for future use.

This SOP is for use by U of I MUPDP archivist, and for use by quality assurance personnel when auditing the archiving facility.

Definitions

Archive: A limited access facility (i.e. locked room or cabinet) of sufficient size to contain all records and data generated providing environmental conditions to ensure the preservation of the data.

Archivist: Person in charge of placing and retrieval of archives. This position is to be held by the Research Tech, with the FRD designated as the alternate archivist. If no individual holds this title, the Field Research Director assumes the duties of the archivist.

Raw Data: All data generated by a GLP study.

Historical Data: All historical data pertinent to a GLP study.

Facility Data: Any data pertaining to the facilities used during a GLP study.

Procedures

Raw Data

All raw data from an IR-4 study is to be kept with the study notebook or pertinent logbook until the end of the study at which time the notebook is sent to the Western Region IR-4 Coordinator for final inspection before being sent to IR-4 headquarters. All raw data from any U of I study is to be kept with the study notebook or pertinent logbook until the end of the study at which time the notebook is archived by the archivist if not sent to the sponsor.

Historical and Facility Data

All historical data is to be archived by the archivist in the archive facility provided by U of I. Historical data include but are not limited to historic CVs, historic organizational charts, historical SOPs, historical thermograph and hobo logger charts and historical equipment log books. All historical data is to be maintained for 5 years or the life of the registration, whichever is longer. The historical classification is decided upon by the archivist, example, the log books are considered as a working document until placed in the archive classification. The archiving facility should be available at or in close proximity to the location of the Field Research Director responsible for the conduct of the studies. The facility should be in a building with adequate fire protection.

When archiving data or documents, sign into the Archive Log Book. Date and initials of the person placing the document will be noted and if the document is new, this information will be noted. New data or documents will be assigned a specific document number, using a document # stamp placed in the right hand corner of the document. The doc# will be assigned by beginning with the year and ending with the sequential numerals of the document being placed. For example, the document number of the first document placed for the year of 2007 would be doc#20071, the second would be doc#20072 and so on. When logging in new data the file# in which the document was placed will also be noted. Refer to the Archive Index to locate the proper file in which to place the document. If the proper file does not exist, a new file must be created. At this time, the file will be created, placed in the most appropriate place within the archive, and the Archive Index must be updated, noting all actions taken in the Archive Logbook.

When making copies, reviewing data, or for any reason removing data from the archive, the action taken should be noted in the Archive Logbook. Initials, date, document number, file number, and if the doc was removed and replaced should all be noted in the log book.

On an annual basis, after the trial season for the year is completed and all field databooks are sent to the Western Region, true copies, (stamped as such), of all non-notebook archived data from the last year will be sent to IR-4 H.Q. A copy of the data inventory sent to IR-4 H.Q. will also be sent to the Western Region office.

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STANDARD OPERATING PROCEDURES

ENSURING SAFETY IN THE FIELD

SOP NUMBER: 0200

REVISION NUMBER: 6

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and scope

This standard operating procedure (SOP) is to indicate the minimum procedure that must be followed to ensure the safe handling and application of pesticides in residue testing operations.

Procedures

Health Hazard and Pesticide Spill Notification

Review and understand the actions required in case of pesticide spills and chemical exposure. Keep contact list where pesticides are stored, measured, transported, mixed and applied. All personnel involved in these activities should be aware of these procedures.

A phone list of emergency assistance should be posted within chemical storage area. This list should contain the names and phone numbers of those who could render emergency assistance in case of accidental pesticide exposure and spillage. Those personnel and phone numbers should include the following:

1. Local hospital emergency rooms / ambulance service / police emergency response numbers.
2. Local physicians.
3. Nearest poison control center.
4. Supervisor's home and work numbers.

The above list should be placed where pesticides are stored, measured, transported, mixed and applied. All personnel involved in the above activities should be aware of the location of the emergency phone numbers.

Personal Protective Equipment

Refer to the MSDS, label or technical bulletin for the pesticides one is working with to determine the protective clothing necessary to safely handle and apply the product. All clothing should be washed according to safety requirements after use.

Equipment

Inspect spray equipment annually before the spraying season. Remove and replace inoperable relief (safety) valves, on-off valves and cracked or leaking couplings and cap seals. Inspect and check pressure gauges of CO₂ operated sprayers. Check spray booms and nozzles for leaks prior to each day of spraying. This is generally performed during calibration.

When operating trucks and pulling trailers, tractors, self propelled sprayer or combines, make sure all personnel are familiar with the safe operation of these items. When loading and unloading tractors and miscellaneous equipment, an additional person should be present in the case of accidents and to guide drivers in the loading and unloading process. Make sure all equipment is properly secured before transporting. Fire extinguishers should be available and easily accessible in work vehicles and sites.

Safety Kits

First aid kits should be available and accessible in work vehicles and sites. Eye flush kits and clean water should be available at the site of storage, measuring, mixing and application to minimize the risk of pesticide exposure in the event of an accident.

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STANDARD OPERATING PROCEDURES

FIELD RESEARCH DIRECTOR JOB DESCRIPTION

SOP NUMBER: 0525

REVISION NUMBER: 5

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-10-09

Purpose and Scope

This standard operating procedure is to outline and identify the responsibilities of a Field Research Director and is for use by U of I research personnel in understanding and clarifying the responsibilities of the Field Research Director and/or when writing a job description for hiring purposes.

Field Research Director

The Field Research Director is designated by the Study Director based on the recommendation of the Field Coordinator to conduct the trial. The Field Research Director should be a scientist with appropriate training and experience to conduct a trial.

The Field Research Director shall be responsible for carrying out all field portions of the study throughout its duration as directed by the Study Director. These responsibilities may include but are not limited to:

1. Accepting protocol with consideration of ability to perform tasks required as well as ability to grow crop in designated area.
2. Receipt documentation and proper storage of test substances.
3. Receipt and proper storage of test system sampling materials (bags, boxes, tubes, etc.).
4. The establishment of field study plot locations.
5. Assure proper site preparation and planting of test systems. If no specific instructions are given, use normal planting practices for the area.

6. Maintain on file a current curriculum vitae and job description for all key people engaged in the study. Curriculum vitae (CVs), will be updated on an annual basis reflecting training from throughout the past year. Training records and CVs will be kept current by the individual the document pertains to.
7. Collection and documentation of raw data in appropriate study notebook or log.
8. Sampling of soils and/or plant material according to techniques outlined in the protocol and appropriate SOP.
9. Transportation of samples to storage facilities and proper storage of such samples according to protocol. If no method is specified, use methods according to appropriate SOP.
10. Shipment of samples to appropriate location according to protocol. If no method is specified, use methods according to appropriate SOP.
11. Assure the study is carried out according to an approved protocol. Follow appropriate SOP for items not specified in protocol.
12. Maintenance of all field, test, and laboratory equipment.
13. Assume the duties of archivist by assuring all raw data, historical data and other items connected with the study that need to be retained are stored in archives if no individual holds the title of archivist.
14. Maintain a schedule for all active GLP trials under his/her direction.
15. Retain true copies of raw data from each trial for a reasonable period of time to insure that raw data is not lost prior to archiving by the Study Director.

Also, in addition to the above responsibilities the Field Research Director shall be responsible for the adequate training and supervision of Research Technician(s) and Irregular Help under him/her who will assist in field trials.

The Field Research Director shall report in a timely manner any deviations to the protocol to the Study Director. He/she shall also report to the Study Director anything that may jeopardize the integrity of the trial.

Every U of I employee related to a GLP study is responsible for reading and understanding all current revisions of any SOPs that relate to their duties. Any confusion pertaining to an SOP shall be discussed with a supervisor before performing the procedure. Each individual is also responsible for accurate and timely training log entries.

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STANDARD OPERATING PROCEDURES

RESEARCH TECHNICIAN JOB DESCRIPTION

SOP NUMBER: 0527

REVISION NUMBER: 1

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-10-12

Purpose and Scope

This standard operating procedure is to outline and identify the responsibilities of the Research Technician working with a GLP study and is for use by U of I research personnel in understanding and clarifying the responsibilities of the Research Technician and/or when writing a job description for hiring purposes.

Research Technician

The Research Technician shall be responsible for assisting the Field Research Director in carrying out all field portions of the study throughout its duration. These responsibilities may include but are not limited to:

1. Manage and coordinate chemical residue field research to aid with the U.S. Environmental Protection Agency, (EPA) registration of agricultural chemicals for minor use crops in the state of Idaho and Western U.S.
2. Assist with establishment and culture of Field Research Plots.
3. Edit and compile data manually or by computer program for Good Laboratory Practices, (GLP), field data notebooks.
4. Maintain technical knowledge and certifications as well as keeping up with adequate GLP training as decided by Field Research Director.
5. Coordinate Worker Protection Safety training and equipment for Minor Use Pesticide Development Center.

6. Coordinate efficacy and phytotoxicity trials for Minor Use Pesticide Development Center.
7. Supervise temporary help.
8. Archivist.

Every U of I employee related to a GLP study is responsible for reading and understanding all current revisions of any SOPs that relate to their duties. Any confusion pertaining to an SOP shall be discussed with a supervisor before performing the procedure. Each individual is also responsible for accurate and timely training log entries.

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STANDARD OPERATING PROCEDURES

IRREGULAR HELP JOB DESCRIPTION

SOP NUMBER: 0530

REVISION NUMBER: 3

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and Scope

This standard operating procedure is to outline and identify the responsibilities of any Irregular Help working with a GLP study and is for use by U of I research personnel in understanding and clarifying the responsibilities of the Irregular Help and/or when writing a job description for hiring purposes.

Irregular Help

Irregular Help shall be responsible for assisting the Field Research Director in carrying out all field portions of the study throughout its duration. These responsibilities may include but are not limited to:

1. Receiving training provided by University of Idaho personnel pertaining to farm and pesticide safety as required by the University of Idaho.
2. Receiving GLP training from Field Research Director necessary for field residue work.

No CV or personnel short form will be required for this work. However, documentation should be added to the field data notebook with the names of the workers, a short description of how they were trained and supervised, and the work they performed in the trial.

Every U of I employee related to a GLP study is responsible for reading and understanding all current revisions of any SOPs that relate to their duties. Any confusion pertaining to an SOP shall be discussed with a supervisor before performing the procedure. Each individual is also responsible for accurate and timely training log entries.

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STANDARD OPERATING PROCEDURES

QUALITY ASSURANCE AUDITS

SOP NUMBER: 0535

REVISION NUMBER: 3

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-9-17

Purpose and Scope

This standard operating procedure is to identify the Quality Assurance Officer for studies performed by the MUPDP of the University Of Idaho, and is for use by U of I research personnel in understanding and clarifying their responsibilities during a QA audit.

Definitions

QAO- Quality Assurance Officer.

QAU- Quality Assurance Unit.

QA audit- Inspection of facility, data, or critical phase by QAO.

Facility Audit- Inspection of all equipment, and data pertaining to a particular study.

In Life Audit- Inspection of a particular procedure of a study (ex. application, sampling, shipping).

QAO Designation

The QAO is designated by the Study Director in studies not under the IR-4 heading. IR-4 studies will be QA'd by the appropriate personnel designated by the IR-4 QAU.

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STANDARD OPERATING PROCEDURES

PROCEDURES TO FOLLOW FOR AN EPA AUDIT

SOP NUMBER: 0550

REVISION NUMBER: 3

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 4-14-06

Purpose and Scope

This standard operating procedure is to provide guidance to study personnel in responding to a request for an EPA audit or review by Office of Compliance Management (OCM). This SOP is for use by U of I research personnel in understanding and clarifying their responsibilities during an EPA audit.

Upon Notification of EPA audit

Procedures:

1. Notify the Study Director, Quality Assurance Officer, Regional Field Coordinator, and other interested personnel of the pending audit or review as soon as possible.
2. Arrange to have available the personnel who may be associated with the trial(s) or facilities audit.
3. Make sure someone who is authorized to accept the Notice of Inspection is going to be present at the start and finish of the inspection.
4. Prepare trial(s) and/or facilities personnel for the inspection.
 - a. Review position descriptions with technical personnel so they understand and can explain their role in the trial(s).
 - b. Discuss possible questions that may likely come up about the trial(s) or facility and make sure everyone understands what to expect.

- c. Request personnel to answer the questions only to the extent needed and not to provide extraneous information. Do not volunteer information; keep answers direct and to the point.
- d. Make certain that technical personnel know the safety precautions needed for the work area.
- e. Be certain that all documents pertaining to the trial(s)/facilities inspection are available. This would include:
 - 1.) Master schedules for both the field research director, Quality Assurance Research Officer and possibly their counterparts at the region and IR-4 headquarters.
 - 2.) Study Protocol and Standard Operating Procedures.
 - 3.) Raw data, correspondence and logs.
 - 4.) Training records, CVs, job descriptions, etc. of personnel assigned to the trial(s).
 - 5.) Appropriate chain of custody documents for samples and freezer logs and storage temperature documentation.
 - 6.) Documentation of the characterization of the test substance, receipt, and handling, and storage records.
 - 7.) Calibration logs on equipment such as balances and application equipment.
 - 8.) Archives or storage of records and logs indicating removal and replacement of documents.
 - 9.) Have accessible organizational charts, a map of the facility and any information specific to the facility or area that will make the inspection go smoother.

During an EPA Inspection

Procedures:

- 1. Greet the inspection team and follow any institutional procedures for signing in. Provide name tags and escort the entire group to a conference or meeting room.
- 2. At the opening of the conference ask the lead inspector for his credentials and for any opening statements.
- 3. Introduce the facility personnel present and state their function in the facility or trial(s). Identify the person responsible who will accept the Notice of Inspection.

4. Distribute organizational charts, map of the facility and any other information previously prepared to make the inspection go smoother.
5. Ask the lead inspector for his/her agenda for the inspection as well as a list, if possible, of personnel for interview during the inspection.
6. Explain any housekeeping rules such as the use of safety equipment in work areas etc. to avoid any possible misunderstandings.
7. Proceed with the inspection.
 - a. Provide documents requested and provide explanations needed.
 - b. Keep notes of observations and of all interviews.
 - c. Keep management informed of the progress of the inspection and the findings.

After an EPA Inspection

Procedures:

1. Make sure that all personnel involved in the inspection are present for the closeout conference.
2. Clarify any discrepancies brought up during the exit interview and offer supporting documentation for clarification.
3. If you have corrected any problems during the inspection make sure the corrections are so noted in the inspector's logbook.
4. Have someone present during the close-out to take accurate notes.
5. Be sure you obtain a copy of the list of documents or other materials that may be taken as exhibits by the inspectors.
6. Debrief management, staff, and the Study Director with an explanation of any problems found.
7. Assign responsibility for preparation of possible solutions to problems and obtain time estimates for implementation.
8. Prepare any replies to the regulatory agency as necessary within a timely basis and keep interested parties such as management and the Study Director informed.

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STANDARD OPERATING PROCEDURES

PROCEDURES FOR RECORDING RAW DATA

SOP NUMBER: 1005

REVISION NUMBER: 8

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose

The purpose of this standard operating procedure (SOP) is to ensure the correct recording of raw data and to protect it through proper storage and, ultimately, transfer to the appropriate party.

Scope

This standard operating procedure is for use by research personnel when recording raw data that are generated during the course of a study.

Definition

Raw data: Any worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study.

Recording raw data

Record all raw data legibly in ink.

Raw data that are entered into the trial notebook should be recorded promptly as they are generated.

Whenever possible, raw data should not be transcribed from notes, scrap paper, etc. If it is necessary to do so, the original must be retained and its location noted on the transcribed copy.

When an error has been made in the entry of the data, that original entry must never be obscured. To change an entry, draw one line through the original entry and make the necessary correction, and initial and date the correction. Note the reason for the change, i.e., spelling error or transcription error. The sponsor may designate a code to be followed.

All entries must be initialed and dated by the person making the entry. If entries on one page are made on different days, each must be initialed and dated. If the date the data were generated is different than the date they were entered, this must be noted. Line out unused portions of pages when 2 or more lines of any data entry space are left blank or when entire page is unused, include initials and date the lineout.

A hard copy of electronic data, computerized summaries, etc. should be placed in the study folder and referenced as soon as possible after the information is generated. All pages should be identified with FID # and should be numbered by part__pg __ for page placement purposes.

Make sure that all data required by the study protocol is collected and recorded. The following is a list of information to be retained in the study file if required:

- Raw data including pest counts, yield, phytotoxicity, weather records, logs of instrument calibration and pesticide, etc.
- Copies of summaries including original calculations and citations of sources of information used.
- Copies of reports including correspondence related to reports.
- Copies of completed forms used during the study and for summaries of study data.
- Effective Standard Operating Procedures.

Entering calculations in the raw data

Enter calculations in a logical manner showing all steps and conversions so that they can be easily followed and reconstructed.

Calculations should be completed in a timely manner so that any deviations needed can be sent and appropriate action taken.

Rounding numbers

The rule for rounding numbers is to round down (i.e. drop the last digit) if it is less than 5. Round up, if the last digit is 5 or greater.

Significant figures in data calculations

When rounding numbers, retain enough decimal places to accurately reflect the measurement and measuring equipment.

e.g. $\frac{13.3 \text{ ft} \times 100 \text{ ft}}{43,560 \text{ ft}^2/\text{acre}} = 0.0305326 \text{ acres}$ (not 0.03 acres)

e.g. $0.0305326 \text{ acres} \times \frac{2 \text{ lb ai}}{\text{acre}} \frac{453.6 \text{ gms}}{\text{lb}} = 27.70 \text{ gms ai}$ (not 27.6991747)

IR-4 Field Study Notebooks

Field study notebooks produced for an IR-4 study are to be sent to the Western Region Office for final inspection. Copies of the field study notebook are to be kept for a period of five years or until study completion in case of later occurring questions.

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STANDARD OPERATING PROCEDURES

COLLECTING PHYTOTOXICITY DATA

SOP NUMBER: 1010

REVISION NUMBER: 0

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-12-14

Purpose and scope

The purpose of this standard operating procedure is to describe procedures for recording phytotoxicity data. This SOP applies to all studies specifically asking for Phytotoxicity data as well as any study where phytotoxicity is seen but not specifically asked for.

Procedures

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

Where possible, take phytotoxicity data within 24 hours before the initial pesticide treatment and within one to two weeks after the treatment. If symptoms occur during or after this period that warrant an additional reading, then phytotoxicity data should be taken as necessary. Take pictures of phytotoxicity symptoms whenever possible and notify the Study Director.

Select five representative plants in the middle row of each subplot and record a phytotoxicity rating of 0 to 10 for each subplot. 0 = no effect, 1-3 slight effect, 4-6 moderate effect, 7-9 severe effect and 10 complete effect. If there is one plant or tree per subplot, record data from each plant or tree in the plot.

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STANDARD OPERATING PROCEDURES

TRIAL DESIGN AND IDENTIFICATION

SOP NUMBER: 2005

REVISION NUMBER: 5

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 1-31-13

Purpose and scope

The purpose of this standard operating procedure is to outline procedures on how to design and post field trials for studies. This should be used as a guideline by all research personnel when initially laying out plots for a study unless superseded by specific protocol instructions.

Field trial design

The purpose of field trials is to provide representative samples (crop, soil, water, etc.) that have been treated according to protocol. All activities associated with planning and designing the trial should occur with this purpose in mind. When studying a test substance for residue levels, the target organism (pest) need not be present. Normal agricultural practices should be practiced, including good cultural practices, to maintain a healthy crop.

Location

Choosing an exact location including city and county is usually the U of I Field Research Director's choice. The location should be one where the crop could be grown commercially.

Be sure the pesticide history of the site is in accordance with the protocol.

Size

Consult the protocol for fixed dimensions, or minimum, or maximum plot sizes.

Plot size is normally dependent on the sample size, the number of samples requested in the protocol, amount of test substance available for the trial, and the application method. Plots must be large enough to accommodate all samples requested (consider sample replications and multiple sampling dates) and also provide representative samples. Plots should also be large enough to apply the pesticide accurately with the appropriate equipment.

Figuring from no more than an average yield, it is best to make the plot large enough to provide at least four times the total quantity of sample requested. This can be an estimate at best since field situations are dynamic. No calculations need be provided for these estimates.

Arrangement

Plots should be arranged to avoid drift to or from neighboring plots, or nearby commercially farmed fields. A buffer zone must be provided between treated plots and may include guard rows for field and vegetable crops or alleyways for tree crops. The width of the buffer zone must be large enough to prevent drift of the test substance that could interfere with the purpose of the study, and will be determined by the U of I Field Research Director or as specified in the protocol.

Exercise care when designing the experiment to avoid contamination of control plots. Use buffer zones and avoid drift, volatilization, leaching, or runoff to the control plots. If possible, place the control plots upwind and separated from treated plots. If runoff or flooding could be a problem, control plots should be at a higher elevation. Similarly, plots to be treated with lower rates should be upwind and upslope of higher rate treatments.

Whenever possible ensure that the test system is free of any diseases or pest infestations that could interfere with the purpose or conduct of the study.

Neighboring plots

Care should be taken when selecting a location for the trial plots to take into account what types of chemical applications are going to be made to neighboring fields or plots. Although the untreated checks and the different treatments may have been adequately separated to eliminate the risk of contamination within each trial, other trials in the vicinity may be treated with conflicting chemistries either as their test substance or as a maintenance chemical. This applies to other, non-research, farming activities in the area, as well. Adequate precautions should be taken against contamination from these sources, also.

Identification of test system

Plots for trials must be clearly posted and identified. A wooden stake with the trial number and plot treatment number should be pounded into at least one corner of the plot. Flags should be placed at all four corners of the plots, or other flagging such as caution tape may be used to prevent farm crews from entering accidentally. Steel "T" posts with "NO ENTRANCE" signs may be used for additional marking purposes.

When choosing flags, keep in mind the eventual height of the crop and try to use flags that will stay visible above the canopy. If possible, choose a flag color that will stand out against the plants' vegetative and reproductive structures.

For tree and vine crops, it is helpful to tag each individual plant in the plot with surveying tape. In commercial situations, try to use tape of a different color pattern than may already be in use. Also attempt to place flags where they will not interfere with cultural practices being conducted by farm crew personnel. Aluminum ID tags should be used in long-term studies, or whenever it is possible that future studies may be done in the same field or block. This way pesticide histories can be known for future studies that may be done on the same plants.

To honor agreements of confidentiality, avoid identifying any research plots with the sponsor's name or test substance name, unless specifically requested to do so.

Site and plot maps

When entering a site or plot map into the raw data, try to take the perspective of someone who has never been to the plot and has to find it using these maps. The maps should be drawn as soon as possible after the plot is laid out.

Include on the site map, compass direction, and a major junction or city that can be found in most atlases. From there, it is helpful to include the distances to each labeled turn-off and show, or describe, obvious landmarks.

The plot map should include compass direction, the dimensions of the plots, location and dimension of the buffer(s), the treatment number, direction of rows, and direction of slope. Measure and record the distance to permanent landmarks from at least two plot corners, the location of which is identified on the plot map, or take G.P.S. readings according to SOP#2715; in case plot markers need to be temporarily removed for cultural practices. The relation of the plot to the permanent markers should be in sufficient detail so that the plot can be relocated without flags or other identification.

Consult the protocol and sponsor's field study notebook (and sponsor SOPs if using them) for further requested information.

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STANDARD OPERATING PROCEDURES

TEST SYSTEM ESTABLISHMENT AND MAINTENANCE

SOP NUMBER: 2505

REVISION NUMBER: 3

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-03

Purpose and scope:

This standard operating procedure is for use by research personnel and farm managers to ensure that the test system is established and maintained in a manner consistent with commercial practice while still allowing the requirements of each protocol to be fulfilled.

Protocol requirements

Prior to establishing the test system, the Field Research Director should discuss the goals of the study with the farm manager. The discussion should cover crop timing, possible varieties, ground preparation, layouts for the planting of the crop (row width, plant spacing, plot size), desirable locations, etc. The protocol is always the last word on how the test system should be established and maintained. The Field Research Director should make every effort to ensure that the requirements of the protocol are met. When the protocol specifies practices that are significantly different from the established commercial practices in the area, the study director should be notified. Any time the specifications in the protocol are not followed, these deviations must be documented and reported to the study director immediately.

Site Selection

Of primary consideration when choosing a location for a study is the ability to control as many factors as possible. Keeping in mind the requirements of the protocol, the following order of preference should generally be followed:

1. U of I's research farms

2. Other research farms
3. Commercial farms on which U of I has had successful studies in the past.
4. Commercial farms that have shown receptivity to cooperating in the conduct of studies.

Seed Source

The source of the test system (seed source) is required raw data and shall be documented by variety and planting date. Any tags or labels, or copies thereof, included with seed used in plot establishment shall be retained as raw data. A germination test may be necessary for a seed source that does not have such information furnished on the tag or label, or for seed that may have been compromised by time or environmental conditions.

Establishment and maintenance of the test system should be done according to generally accepted commercial practice for the particular crop in that area. This can be determined by consulting with local producers, consultants, university extension agents and other agricultural professionals. Publications in the form of text books, pamphlets and fliers can be very useful in obtaining the needed information. Some information that can be found using these resources includes:

- Soil salinity tolerance
- Suitable varieties for the area
- Soil preparation
- Pre-plant fertility
- Fumigation
- Planting techniques including row spacing and plant populations
- Weed control, chemical and cultural
- Cultural practices
- Local pest pressures and accepted methods of control
- Irrigation types, quantity and timing
- Harvesting practices

Documentation

The following is a list of some of the information and procedures that should be included, when applicable, in the research farm log. Try to include the date when the practice was done.

- preparatory cultural practices, i.e, plow, disc, bed formation
- planting, and thinning
- fertilizer- type, analysis, method of application
- herbicide or pesticide use
- growth hormones
- dormant oils, traps, or predator releases
- cultivation- mechanical or hand

irrigation-types, quantity and/or timing

frost control-i.e. smudge pots or irrigation

desiccation for harvest preparation

Chemical treatments must be authorized and fully documented.

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STANDARD OPERATING PROCEDURES

TEST SYSTEM MONITORING

SOP NUMBER: 2507

REVISION NUMBER: 3

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-03

Purpose

To provide guidelines on monitoring test systems. The goal is to ensure healthy test systems are maintained to preserve the integrity of studies.

Monitoring the test system:

The Field Research Director will ensure that the test system is monitored prior to the application of a test substance and at regular intervals thereafter. These intervals should take into consideration the type of crop grown and anticipated problems that are inherent to the area and time of year.

When monitoring a test system, the untreated check plot should always be inspected first and then the treated plots in the order of low to high rate if more than one exists. This will avoid accidental contamination of the control plot.

Books, pamphlets, bulletins or other reliable scientific sources should be consulted to identify pest species and establish proper management practices for crops grown in a particular test location.

Areas to Monitor:

1. Soil is considered an integral part of the test system. It shall be monitored to minimize moisture stress and to ensure normal crop growth. Irrigation should be scheduled as needed.
2. Crops shall be checked for signs of nutrient deficiencies. Crop tissue or soil samples may need to be taken and analyzed to determine fertility needs.

3. Weed infestation and weed pressures shall be monitored to minimize competition. Specific attention should be given to the identification and size of weed species and the best method of control, either chemical or mechanical.
4. Any disease that is present or is in sufficient quantity so as to affect the overall health of the test system shall be identified and documented.
5. Monitoring and control of insects is important in maintaining a healthy test system. Insects can vector diseases and heavy infestations can jeopardize the integrity of the trial. Specific attention shall be given to the identification of harmful insects and to infestation levels.
6. Damage from other pests, such as nematodes, rodents and other mammals shall also be monitored.
7. Whenever the number of dead or damaged plants reaches a proportion where it could have an effect on the conduct or integrity of the trial, the Study Director and Research Director should be notified immediately.

Documentation

If the test system contracts a disease or condition that could interfere with the purpose or conduct of a study, this shall be documented and, where possible, the test systems should be isolated and/or remedial treatments applied provided they will not interfere with the study.

Maintenance pesticides

Before any maintenance pesticides are applied, consideration should be given to the impact they may have on nearby plots. Buffer zones should be present to protect neighboring plots from drift of damaging or conflicting chemistries.

Application of maintenance pesticides should always be made in accordance with the label. If in doubt as to whether a maintenance pesticide could interfere with the test substance or conflict with accurate analysis of the residues, contact the Study Director (or designated contact) for approval. Note the approval in the raw data.

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STANDARD OPERATING PROCEDURES

OPERATION, MAINTENANCE, AND STANDARDIZATION OF ENVIRONMENTAL MONITORING DEVICES

SOP NUMBER: 2710

REVISION NUMBER: 12

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-17

Purpose

This standard operating procedure is for use by research personnel to ensure that temperature-recording devices (e.g. min/max thermometers, and data loggers) are operated and maintained properly so that they accurately monitor the environments where they are placed.

General use

Temperature and humidity recorders are rated for use in a specified range of environmental temperatures. It is important to make sure that the appropriate data logger is used in the proper temperature range.

If required, fresh batteries should be installed to ensure continued operation of the device.

Instrument placement:

The recording device should be placed on a stable, relatively level surface or magnetically held in place to ensure it does not fall and become damaged.

When using to monitor freezers, avoid placing the device near the back wall, the defroster coils, or light bulb. Do not place it in the door rack or near warm samples recently placed in the freezer. See also, U of I SOP 5010.

For use in test substance storage areas, do not place the device on chemical containers, by the ventilation system, or in areas that may not accurately reflect storage conditions. See also, U of I SOP 3005, "Test Substance Receipt, Use, Handling, Storage, And Return".

Procedure for exchanging Hobo Loggers:

1. Launch the Hobo data logger using the software provided by the manufacturing company, being certain the parameters to be specified are appropriate for the necessary data and time period.
2. Download the data from the logger using a laptop or other computer, which also re-launches the logger with the same parameters as before. This can be performed where the Hobo is located or in the office if no mobile computer is available, by removing the Hobo logger and driving it to the office and then returning it to its position as quickly as possible.
3. Download data from the Shuttle using the Hobo software. Save a copy of the data downloaded to disk or hard drive and print one copy of the data. This printout is original raw data and should be archived as such.

Charts and printouts are critical raw data and should be handled as such. Exact copies of each chart or printout should be made and placed with the raw data for each study to which it applies. The original should be placed in the archives (see Contingencies).

Contingencies:

Min/Max thermometers will serve as backup temperature monitoring devices.

If the batteries become weak or dead, replace them per operating instructions.

If the device is damaged or stops, replace it with another system if possible. Otherwise, use a minimum-maximum thermometer backup log where possible.

Standardization

Recording devices should be checked annually. To standardize, place devices including appropriate min/max devices, Hobo Loggers, and NIST Traceable Hobo loggers to be standardized in an area that reflects a temperature range that is representative of its actual use. Hobo loggers that are used to monitor freezers should be placed in a freezer together. Hobo loggers that monitor chemical storage areas should be placed in the chemical storage area or in an adjacent room with the same temperature ranges. Devices should be set to collect data for a time period long enough to reflect a full cycle of temperature ranges. For freezer monitors, at least a 24 hour period should be used to collect data from

full defrost cycles and/or normal temperature fluctuations. For chemical storage areas, at least 24 hours of data should be collected to reflect p.m. temperature drop and daytime temperature increases. A NIST traceable device should be placed with the Hobo loggers during standardization period.

Temperature ranges of Hobos must be recorded on the appropriate log (e.g. Environmental Monitoring Device Standardization Log). Spreadsheets of Hobo data should be printed and archived as raw data. If any Hobo logger's temperature range and/or min/max device range differs from the NIST traceable device by more than +/- 5 degrees, the device must be discarded and replaced. Document the date the device was discarded/replaced in the appropriate equipment log.

The NIST traceable Hobo logger used for standardizations must be within 3 years of its certification date. Certification date can be found on the NIST certification letter. To keep NIST certification current, purchase a new logger when needed or send current NIST certified logger to manufacturer for updated certification.

Documentation

All standardization and repair actions should be documented in the appropriate log (e.g. Environmental Monitoring Device Log).

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STANDARD OPERATING PROCEDURES

**OPERATION, MAINTENANCE, AND
STANDARDIZATION OF GLOBAL POSITIONING
SYSTEM, (GPS) SYSTEM**

SOP NUMBER: 2715

REVISION NUMBER: 2

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-17

Purpose

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

General use

If required, fresh batteries should be installed to ensure continued operation of the device.

Instrument placement:

The GPS unit should be placed on a stable, relatively level surface with as much access to clear sky as possible.

Procedure for getting location coordinates:

Power up the GPS unit by pressing the power button which is the lower button on the right side of the unit and positioning unit with full view of sky. As soon as unit has tracked enough satellites to get a location reading, the satellite page appears on the screen of the GPS unit. Place GPS unit in the spot that you want to take the readings (such as on plot stake or near flag for plot corners), and read the location (given in degrees and minutes) at the bottom of the screen and include these in the plot map. Do this for each

corner of the plot and include them in the plot map. Accuracy is listed on the top of this screen and should be listed on plot map as well.

Contingencies:

If the batteries become weak or dead, replace them per operating instructions.

If the device is damaged or stops, replace it with another system if possible.

Standardization

Recording devices should be checked annually.

1. Select a location with known coordinates (e.g., survey control station, geodetic survey point, well surveyed location). Go to that location and operate the GPS receiver to mark the waypoint.
2. Record the waypoint of the known location and the waypoint displayed on the GPS unit in the appropriate equipment log.
3. Compare the waypoint recorded on the GPS receiver and the known waypoint for accuracy, by entering the recorded waypoints into Google Earth and utilizing the ruler tool.
4. If the measured waypoint varies significantly from the known waypoint ($>\pm 50$ ft, latitude or longitude), the GPS receiver should not be used. Repair or replace the device.

Documentation

All standardization and repair actions should be documented in the appropriate log (e.g. Global Positioning Satellite Log).

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STANDARD OPERATING PROCEDURES

TECHNIQUES FOR TAKING ENVIRONMENTAL DATA

SOP NUMBER: 2720

REVISION NUMBER: 9

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-29-16

Purpose and scope

This standard operating procedure is for use by research personnel and outlines what environmental and climatic conditions should be monitored during the course of a study, and how to collect that data.

Environmental data required

The protocol will usually list what environmental data the sponsor requires for a particular study. Some conditions, such as air temperature and rainfall data, must be monitored every day for the duration of the study. Further, at each application additional data will usually be requested. Make sure that the protocol is consulted prior to application so that monitoring of "on-site" conditions can begin.

Obtaining weather data

Daily high and low temperatures and precipitation data can usually be obtained from the nearest NOAA station, Bureau of Reclamations via Agrimet, or other station such as a university research station. Make sure that the protocol does not require "on-site" data before relying on an outside station.

Data required for a test substance application usually include, but are not limited to:

- air temperature
- relative humidity
- soil temperature(s)

soil moisture
wind speed
cloud cover
dew - presence or absence
crop stage

IR-4 studies require min/max temperatures and precipitation plus data needed at time of application.

Air temperature: When measuring air temperatures be sure that the thermometer is in a representative place. For example, do not hold it over the hood of a hot truck.

Relative humidity: Use a digital psychrometer such as a Kestrel unit to measure relative humidity. Facility will keep two working units at all times and units will be standardized annually to assure that they are in proper working conditions. If a unit does not pass standardization, discard and replace with new unit.

Soil temperatures: Take the soil temperature in the plot itself (or as close as possible) rather than in an area by the edge of the field, which may not be representative. Be sure to leave the thermometer or probe in the soil long enough to stabilize.

Approximate wind speed: Hold the anemometer into the wind, avoiding areas where eddies could form. If the wind is gusting, hold it long enough to get an average wind speed. Also note the direction the wind is coming from.

Crop stage: Look at enough plants in the plot to get a representative sample to determine the crop stage. Be as specific as you can and as specific as the protocol requires.

pH of mix water: Use pH paper.

Device Verification

The Field Research Director will ensure that verifications are performed and documented on a regular basis for that equipment which requires it.

To verify thermometers compare readings with a calibration thermometer, preferably over a range of temperatures. Well-mixed water provides a more constant temperature medium in which to verify, if it won't damage the equipment.

To standardize the Kestrel, compare three or more in a 1 to 10 mph wind to determine if they are reading the same. As there is no calibration for these devices if one is more than 5% different from the average reading, it should be replaced.

Documentation

It is helpful to write down in the raw data, the U of I equipment numbers of the measuring devices used that have a logbook.

Any standardization, repairs and/or calibrations of environmental data measuring devices should be documented in the appropriate log(s).

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STANDARD OPERATING PROCEDURES

**TEST SUBSTANCE USE, STORAGE, AND CHAIN
OF CUSTODY DOCUMENTATION**

SOP NUMBER: 3005

REVISION NUMBER: 12

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-29-16

Purpose and scope

This Standard Operating Procedure is for use by research personnel when receiving test substances, documenting their use and detailing how they should be stored and returned. It is intended to ensure proper documentation regarding the receipt, accountability, and return of test substances, and to provide guidelines for their safe use.

Test substance receipt procedure

Upon receipt of a test substance the following steps should be taken.

It is recommended to write your initials, the trial number, and the date received on the carton in which the test substance was shipped. If the carton has a shipping receipt attached, place that with the raw data for the trial. If shipping receipt is not available make a copy of the bill of lading on the box and place it in the notebook as raw data.

All documents filled out in conjunction with this SOP are raw data and should be treated as such. To facilitate complete documentation when logging in test substances, a Test Substance Receipt form such as Part 4A of FDN will be used.

Wherever the log-in information is documented (form, field trial notebook, or elsewhere), the following information will be recorded, then initialed and dated:

U of I or IR-4 study number if known

Date received

Courier

Shipping receipt/freight Number

Quantity received as given on label
Batch/Lot Number
Test substance name
Test substance formulation
Expiration date, if no expiration date, contact study director immediately and include correspondence in field data notebook
Date test substance placed into storage
Storage location
Special storage conditions from protocol, MSDS, etc. if given
Condition of container on arrival

When recording the amount received it is neither necessary, nor advisable, to pour liquid test substances into graduated cylinders, nor remove dry formulations from their containers. Rather, record the quantity given on the label. Multiple containers must be numbered and referenced separately in the logs.

If the container is leaking, the sponsor should be notified immediately as a new shipment may be needed. Take the appropriate steps to ensure that the leak has been contained and properly cleaned up. Dispose of waste and contaminated materials in a legal and safe manner.

All shipping documents, particularly those provided by the carrier delivering the shipment, are critical raw data and must be retained with the study data.

Test substance storage

Upon receipt of a test substance it shall be placed into an appropriate storage area, generally a monitored, dedicated chemical storage area with limited access.

Check the MSDS, Certificate of Analysis (COA), and/or protocol for storage conditions. If storage conditions are not shown on label, write them on label so they are readily available. If storage conditions are not on the label or MSDS, contact the Study Director to obtain this information.

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 COA. Verify that the % active ingredient and formulation is the same as in the protocol. Alert Study Director to any discrepancies.

The test substance should, whenever possible, be kept in its original, accurately labeled container. When this is not feasible, the intermediate container must be labeled properly with the test substance name, batch number, CAS number or code number, special storage conditions, and expiration date, if given. Containers of test substances should be checked regularly to ensure that the lids are tight and there are no leaks or spills.

The storage area should be kept clean and dry and should have adequate lighting to easily read the labels. Care should be taken that all containers are placed on a secure surface and any stacked containers are stable and will not fall. Heavy containers should not be placed on a high shelf or on a shelf without adequate support.

Ventilation must be provided in all areas where test substances are stored or handled. Protective clothing and provisions for cleaning up spills should be readily accessible and any personnel involved in handling test substances should be familiar with the principles of containing spills and decontamination.

Test substance monitoring

The environment of the test substance storage area must be monitored and documented when test substances are present. A constant recording thermometer, Hobo data logger, is ideal. (See SOP #2710 for operation and calibration of recording devices) A minimum and maximum registering thermometer is acceptable. The minimum and maximum temperatures should be recorded and the thermometer reset periodically. The Field Research Director will ensure that charts are replaced, or min/max thermometers are read and reset, and calibrated on a regular basis.

Environmental monitoring records are raw data and should be treated as such.

If the temperature of the test substance storage area exceeds the stability range for any stored test substance, notify the Study Director or his designated contact immediately.

If there is any reason to suspect pests are adversely affecting the storage of test substances, appropriate non-conflicting control measures should be taken.

Documenting test substance use

Test substance use must be documented. The Test Substance Use log or the appropriate log in the field trial notebook can facilitate this.

At each application, the following should be recorded:

- Date of use, initials
- Name of test substance
- Lot/batch number
- Purpose of removal (application, retainer sample, return)
- Amount removed
- Date container returned to storage, initials

**If the container is to be removed from the test substance storage area for more than four hours, document in the log or the trial notebook the storage conditions from the time of removal to the time of return. If the entire container is to be taken to the study site, prevent it from being exposed to temperature extremes.

Test substance handling

General Safety Procedures

All test substances should be regarded as being potentially hazardous to the handler and to the environment, and these procedures shall be followed to minimize risk to both.

Any person in contact with a test substance shall wear appropriate protective clothing. As many test substances have known tolerance levels, the label and/or MSDS should be consulted as to the degree of precautions necessary.

Proper sanitation and decontamination practices shall be observed to ensure personal safety and study integrity.

Measuring of Test Substances

Liquids and emulsions: Refer to SOP #3515

Dry substances: Refer to the appropriate SOP that applies to the type of balance being used. Always use a weighing pan or other container to avoid contamination of the balance and thoroughly clean up the area after weighing. Remember to record the entry in Balance Log.

Mixing Of Test Substances

Mixing should be done as close as possible to the time of application. The pH and temperature of the carrier and time of mixing may be requested by the protocol or field trial notebook. Appropriate measuring devices for the quantity desired should be used.

The exact quantities of all components of the mixture must be documented in the study data.

Mixtures should be adequately agitated to ensure an even distribution of concentration of test substance throughout. Some formulations require more agitation than others. The method of agitation should be addressed in the study data.

Sampling test substances and mixtures

Clean bottles of an appropriate composition for the formulations involved should be used for taking and storing samples of test substances and mixtures.

The requested quantity of test substance or mix should be placed in the properly labeled container. The sample should be stored as soon as possible per instructions from the sponsor. The conditions, times from sampling to storage, and freezer equipment number (if applicable) should be recorded in the raw data.

Spray samples should be taken from the spray tank after adequate agitation. Generally, one sample is taken from the tank before the application and a second sample taken from the mix remaining after the application. The samples should be put into properly labeled containers and stored as soon as possible per sponsor's instructions. The times of sampling, storage, and freezer ID number (if applicable) should be recorded in the raw data.

Extreme care should be taken to avoid contamination when these samples are stored or shipped with crop, soil, or other samples for the study.

Test substance/empty container return

The test substance and/or empty container may be returned to the sponsor or other entity designated by the Study Director or its agent if they will accept it. To ensure acceptance always contact recipient prior to shipping.

Wherever the return is documented, the following information should be included, dated and initialed:

- Study number
- Date returned
- Courier
- Shipping receipt/freight number
- Quantity returned as calculated (Start with label amt. and subtract amts used. Try to keep units consistent.)
- Batch/lot number
- Test substance name
- Test substance formulation
- Sender's address
- Recipient's address

Be sure that the test substance is properly packaged so that it is legal, safe, and meets DOT shipping requirements. It is advisable to wrap tape around bottle and jar lids and be sure there is adequate absorbent in case of spill.

Shipping receipts generated during test substance return are critical raw data and should be treated as such.

If the sponsor will not accept the chemical and/or chemical container for archiving, or sponsor does not have a GLP compliant facility, we will archive the chemical and/or chemical container at our facility until approval for release of chemical and/or container is obtained via the IR-4 Food Request Database Test Substance Container Disposal Approval at http://www.ir4.rutgers.edu/FoodUse/Food_UseSimple3.cfm. Upon approval, chemical and/or chemical container can be turned over to Kimberly Research and Extension Center research facility for registered use on crops, and legal disposal of the chemical and/or chemical container.

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STANDARD OPERATING PROCEDURES

ADJUVANT LABELING, USE, AND STORAGE

SOP NUMBER: 3006

REVISION NUMBER: 2

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-17

Purpose and scope

This Standard Operating Procedure is for use by research personnel when receiving test substances, documenting their use and detailing how they should be stored and returned. It is intended to ensure proper documentation regarding the receipt, accountability, and return of test substances, and to provide guidelines for their safe use.

A. Labeling

- a. The primary container label should include, at minimum: 1) purchase date or date received at the field facility and initials 2) identity and concentration of the adjuvant 3) storage requirements and 4) expiration date. If any of this information is missing, add it to the adjuvant container and initial/date the entry. If the expiration date is not given, assign 2 years from purchase date.
- b. If material is transferred to a secondary container (e.g, from 2.5 gal. jug to 1 pt. bottle), label the secondary container with all information as previously described. These requirements do not apply to temporary containers used for measuring, but they should be adequately labeled to uniquely identify the product.

B. Storage

- a. Store adjuvants in a location that has limited access and is temperature monitored.
- b. Ensure adjuvants are in good condition prior to use. Discard any adjuvant which has undergone physical change (color, precipitation, viscosity etc.)

C. Use

- a. Transfer adjuvant using a method that prevents a measuring device from entering the primary or secondary container more than once. Following are suggested methods. Other methods may be used.
 - i. Dispense a small amount of material into a temporary container (cup, beaker etc.) for measurement into mix. After adding to mix, discard any remaining material; do not use in another trial or return to adjuvant container.
 - ii. Use a sealed, disposable syringe or pipette to transfer required amount of adjuvant from primary or secondary container. Discard syringe or pipette after single use; device should not enter container again.
- b. Any previously opened container of adjuvant that does not have a known history of use as described cannot be used in a GLP trial.

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STANDARD OPERATING PROCEDURES

**OPERATION, CALIBRATION AND
MAINTENANCE OF BALANCES**

SOP NUMBER: 3505

REVISION NUMBER: 10

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and scope

This standard operating procedure is for use by research personnel and establishes procedures for obtaining optimal accuracy in the weighing of chemicals, when using triple-beam, and Ohaus Digital Scout balances.

Equipment operation

The balance should be level, free from any vibration, on a solid support, and away from direct exposure to sun and drafts. Keep the pan clean. Never weigh chemicals directly onto pan. Do not attempt to weigh something which exceeds the capacity of the scale.

Definitions

Standardization: Weighing standard weights to verify the accuracy of the balance.

Calibration: Adjusting the balance to correct an inaccuracy.

Required procedure before weighing test material

The accuracy of the balance must be checked each day the balance is used. The following standardization procedure will be performed before the measuring of a test substance:

A. Weigh two standard weights in the weight range of the total weight of the chemical samples and the weigh boat. (Ex. - if the chemical samples are from 1.5 to 4.5 g, and the

weigh boat is 5.5g, the total weight range is 7g to 10g, weigh the 5 and 15 g standard weights).

B. In the use and maintenance log, record the test number(s) for which the chemical and weigh boat are being weighed, the standard weight sizes, and the measured weights of these standard weights.

1. If the measured weights of both standard weights are within $\pm 2\%$ of the standard weight sizes, proceed with weighing your chemical samples.

2. If the measured weight of either standard weight differs by more than $\pm 2\%$ from the standard weight size, calibrate the balance using the instructions found in the appropriate manual.

a. If, after calibration, the measured weights of both standard weights are within $\pm 2\%$ of the standard weight sizes, again record the measured weights in the use and maintenance log, and proceed with weighing your chemical samples.

b. If, after recalibration, the measured weight of either standard weight still differs by more than $\pm 2\%$ from the standard weight size, weigh a third standard weight (one as close as possible to the weight of the "problem" standard weight) to determine if the problem is the standard weight rather than the balance. If the measured weights of the third standard weight and at least one of the two original standard weights are within $\pm 2\%$ of the standard weight sizes, again record the measured weights in the use and maintenance log, and proceed with weighing your chemical samples. In addition, request a new standard weight and discard the defective weight.

c. If, after recalibration, the measured weights of two of the three standard weights differ more than $\pm 2\%$ from the standard weight sizes, record the measured weights in the use and maintenance log and do not use this balance for weighing until it is professionally serviced.

Calibration for the triple beam balance

1. To zero the balance, dial in the beams to weigh zero, push the end of the beam down and then let it return to zero on its own. If it doesn't return exactly to zero, adjust the silver knob on the balance and repeat the test.
2. Continue to adjust the knob until the beam returns to zero when you push it down and release it.
3. When the released beam returns to zero several consecutive times, lift the beam up and release it. If it returns to zero, the balance is working properly. If it does not, your balance is not working properly and you need to have it repaired.
4. Secure the beam to the balance whenever you move the balance.

Calibration for the Ohaus Digital Scout:

Standardize the balance before weighing chemicals for use on test plot. Refer to manufacturer's directions for calibration and use of balance. Keep manufacturer's directions with balance at all times.

Equipment maintenance

Balance should be cleaned before and after each use. If repair of the balance is necessary, report this to Field Research Director immediately. Record all information called for on the use and maintenance log each time the balance is used.

Weights can be checked and certified by a state agency or other certifying agent for a nominal fee. This should be done on a regular basis. Biennially is recommended for regular use.

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STANDARD OPERATING PROCEDURES

TECHNIQUES FOR MEASURING LIQUIDS

SOP NUMBER: 3515

REVISION NUMBER: 8

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and scope

This standard operating procedure is for use by research personnel to ensure accurate measuring of liquid test substances, using a variety of measuring devices.

General guidelines for measuring liquids

Always wear appropriate protective clothing when measuring liquids.

All liquids should be measured in equipment that is accurate to the fractional increment required. For example, if 10.9 ml of a substance is required, the device used should be marked in increments of tenths of a ml or better.

Use equipment that is of a volume appropriate to the quantity required. For example, use a 50 ml graduated cylinder to measure a 20 ml volume, rather than a 500 ml beaker.

Always try to work on a flat, even surface.

Read all volumes within the measuring device from the bottom of the meniscus, and at eye level.

Guidelines for using specific measuring devices

Graduated Cylinders / Beakers

Pour test substance into cylinder or beaker, keeping device below eye level, while pouring, to minimize risk of splashing into eyes.

When measuring critical quantities, it is advisable to use a syringe or pipette to bring the volume up to the desired level.

Rinse device thoroughly.

Pipette

Use a pipetting bulb or pipette pump. **Never** pipette anything by mouth.

Place tip of pipette into test substance container and draw out slightly more than the desired amount. Allow liquid to settle and bubbles to pop, then allow volume to drop to the desired level. Finally, evacuate the pipette into the proper container or tank.

Syringe

This device is especially desirable when measuring viscous fluids, as the plunger scrapes out the full volume of liquid.

Place tip of syringe into test substance container and draw out slightly more than the desired amount. If the syringe won't fit in the original container it may be necessary to transfer some test substance to an **unused disposable plastic container, clean stainless steel** container, or clean beaker for dispensing purposes. The remainder can be transferred back to the original container. Allow liquid in the syringe to settle, then depress the plunger slightly to drop volume to the desired level. When desired level is obtained, transfer test substance to spray tank, ensuring that some of the carrier water has been added to the tank first. **DO NOT** rinse exterior or interior of syringe and add to tank.

Decontamination

Be sure to clean the measuring vessel using an appropriate solvent, such as water, soap, acetone, etc. Be careful that the solvent you choose will not dissolve the measuring device. If device is disposable, render it useless before discarding it.

Documentation

The volumes of test substance, carriers, and adjuvants in the total mix are critical raw data and must be documented. Record actual amounts measured, which may be different from target amounts, and make sure all data entries are consistent with figures used in calculations.

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STANDARD OPERATING PROCEDURES

**OPERATION AND MAINTENANCE OF CO₂-
POWERED BACKPACK SPRAYERS**

SOP NUMBER: 4005

REVISION NUMBER: 4

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-03

Purpose and scope

This standard operating procedure is for use by research personnel to ensure accurate operation and maintenance of the CO₂ backpack sprayers.

Instructions for proper handling and use of CO₂ cylinders

CO₂ cylinders are manufactured to hold CO₂ under high pressure. A sudden release of this pressure could create dangerous forces, therefore, CO₂ cylinders should be treated with care whether they are full (charged) or empty.

Store, transport, and use in an upright position. Liquid CO₂ will seek a level like water. If a full cylinder is placed on its side, the liquid CO₂ will flow to the valve. This creates a "head pressure" on the side of the cylinder instead of on the valve, and could cause an explosion. Also, the liquid CO₂ can be pulled directly from the cylinder to the regulator. This can cause malfunction of the regulator possibly resulting in dangerous pressures being created in down-stream lines and dispensing valves.

CO₂ cylinders can be damaged through careless handling. One should never be tossed onto a vehicle or allowed to roll around loose during transit. Keep cylinders as cool as practical to avoid excess pressure buildup. Whether empty or full, transport and store with valve closed. Never transport cylinder in passenger compartment of vehicle.

In case of a ruptured safety disc in a CO₂ valve, replace both the safety disc and the copper gasket backing the disc. Use only authorized safety discs, and only one disc at a time. Before replacing, however, be sure to completely remove any pieces of the old disc and gasket remaining in the safety nut.

Never overfill cylinders beyond rated capacity. Fill by actual weight.

Do not weld on cylinder walls.

Cylinders should be inspected by the appropriate officials on a regular basis to assure safety.

Sprayer preparation and maintenance

Inspect the entire unit for cleanliness, broken or worn parts and blocked nozzles. Clean and/or repair the unit as necessary to ensure proper performance. If the unit is non-functional, contact U of I Project Manager to report the problem. All maintenance and repair should be documented in the Use and Maintenance Log.

All tubing attached to the sprayer assembly should be inspected before each use to be sure it is all in safe operating condition. The system should be pressurized with water only in the tank and all connections inspected for leaks. (Normally done during a calibration run.)

Gauges should be checked to make sure they are in operational condition.

Operation

1. Open the CO₂ valve all the way.
2. Adjust the set screw on the regulator to the desired pressure. Double check the pressure after pressurizing spray tank or bottle, as it may change.
3. It is important to attach the hoses in the following order:
 1. Attach the boom to hose and attach the boom hose to the bottle or tank.
 2. Connect the pressure hose.
4. When disassembling it is important to use the reverse order:
 1. Close the CO₂ valve.
 2. Use the pressure release valve on spray tanks to release pressure in tank. When using 2 liter bottles, release the pressure by sliding back the sleeve and slightly disengaging the connector, covering it with a gloved hand to contain the spray.
 3. Disconnect the pressure hose.
 4. Disconnect the boom hose.

Turn off the CO₂ and release pressure off the regulator and the hose during transport and after use.

Sprayer decontamination

Clean the sprayer after each period of use. Many of the chemicals used are corrosive and will damage parts of the sprayer if allowed to stand for even short periods of time.

After an application, mix a small amount of appropriate solvent (e.g. water, detergent, ammonia, acetone) in the tank. Agitate the solution, then flush the solution through the boom. Flush with clean water. Remove and clean all screens and nozzles by washing them in an appropriate solution with a soft brush. A boom without spray nozzles attached gives assurance that it has been decontaminated, however nozzles may be reinstalled into boom after cleaning to prevent loss.

Each use should be documented in the use and maintenance log.

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STANDARD OPERATING PROCEDURES

**OPERATION AND MAINTENANCE OF CO₂-
POWERED 4-WHEELER MOUNTED SPRAYERS**

SOP NUMBER: 4015

REVISION NUMBER: 5

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 1-31-13

Purpose and scope

This standard operating procedure is for use by research personnel to ensure accurate operation and maintenance of the CO₂ 4-wheeler mounted sprayers.

Instructions for proper handling and use of CO₂ cylinders

CO₂ cylinders are manufactured to hold CO₂ under high pressure. A sudden release of this pressure could create dangerous forces, therefore, CO₂ cylinders should be treated with care whether they are full (charged) or empty.

Store, transport, and use in an upright position. Liquid CO₂ will seek a level like water. If a full cylinder is placed on its side, the liquid CO₂ will flow to the valve. This creates a "head pressure" on the side of the cylinder instead of on the valve, and could cause an explosion. Also, the liquid CO₂ can be pulled directly from the cylinder to the regulator. This can cause malfunction of the regulator possibly resulting in dangerous pressures being created in down-stream lines and dispensing valves. When mounted on the 4-wheeler, the mounting rack is at an angle such that the operation is safe.

CO₂ cylinders can be damaged through careless handling. One should never be tossed onto a vehicle or allowed to roll around loose during transit. Keep cylinders as cool as practical to avoid excess pressure buildup. Whether empty or full, transport and store with valve closed. Never transport cylinder in passenger compartment of vehicle.

In case of a ruptured safety disc in a CO₂ valve, replace both the safety disc and the copper gasket backing the disc. Use only authorized safety discs, and only one disc at a

time. Before replacing, however, be sure to completely remove any pieces of the old disc and gasket remaining in the safety nut.

Never overfill cylinders beyond rated capacity. Fill by actual weight.

Do not weld on cylinder walls.

Cylinders should be inspected by the appropriate officials on a regular basis to assure safety.

Sprayer preparation and maintenance

Inspect the entire unit for cleanliness, broken or worn parts and blocked nozzles. Clean and/or repair the unit as necessary to ensure proper performance. If the unit is non-functional, contact U of I Project Manager to report the problem. All maintenance and repair should be documented in the use and maintenance log.

All tubing attached to the sprayer assembly should be inspected before each use to be sure it is all in safe operating condition. The system should be pressurized with water only in the tank and all connections inspected for leaks. (Normally done during a calibration run.)

Gauges should be checked to make sure they are in operational condition.

Operation

1. Open the CO₂ valve all the way.
2. Adjust the set screw on the regulator to the desired pressure. Double check the pressure after pressurizing spray tank or bottle, as it may change.
3. It is important to attach the hoses in the following order:
 1. Attach the boom to hose and attach the boom hose to the bottle or tank.
 2. Connect the pressure hose.
4. When disassembling it is important to use the reverse order:
 1. Close the CO₂ valve.
 2. Use the pressure release valve on spray tanks to release pressure in tank. When using 2 liter bottles, release the pressure by sliding back the sleeve and slightly disengaging the connector, covering it with a gloved hand to contain the spray.

3. Disconnect the pressure hose.

4. Disconnect the boom hose.

Turn off the CO₂ and release pressure off the regulator and the hose during transport and after use.

Sprayer decontamination

Clean the sprayer after each period of use. Many of the chemicals used are corrosive and will damage parts of the sprayer if allowed to stand for even short periods of time. After appropriately emptying spray and lines onto trap crop etc., run water from “water only” tank through system for decontamination. Hose off entire system to prevent contact with any chemical that might have drifted onto equipment.

After an application, mix a small amount of appropriate solvent (e.g. water, detergent, ammonia, acetone) in the tank. Agitate the solution, then flush the solution through the boom. Flush with clean water. Remove and clean all screens and nozzles by washing them in an appropriate solution with a soft brush. A boom without spray nozzles attached gives assurance that it has been decontaminated, however nozzles may be reinstalled into boom after cleaning to prevent loss. Each use should be documented in the use and maintenance log.

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STANDARD OPERATING PROCEDURES

**OPERATION AND MAINTENANCE OF HAND-
CRANKED GRANULAR APPLICATOR**

SOP NUMBER: 4020

REVISION NUMBER: 2

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-03

Purpose and scope

This standard operating procedure is for use by research personnel to ensure accurate operation and maintenance of the hand-cranked granular applicator.

Granular applicator preparation and maintenance

Inspect the entire unit for cleanliness and broken or worn parts. Clean and/or repair the unit as necessary to ensure proper performance. If the unit is non-functional, contact U of I Project Manager to report the problem. All maintenance and repair should be documented in the Use and Maintenance Log. This should include the type of maintenance performed, routine or non-routine, who performed, and date and initialed.

If available, blanks should be used for practice runs as well as calibration of unit.

Operation

1. Measure off an appropriate distance for a test run in terrain that is similar to the test plot. Walk the distance cranking the handle of the applicator at constant intervals, (ex. one turn for one step with steps being held constant by metronome).
2. After the number of cranks are known for the plot distance, set the applicator to apply the known amount of product in the given number of turns on the handle.
3. Weigh out the appropriate amount of test substance and place in hopper. Open outlet at beginning of plot and apply.

Applicator decontamination

Clean the applicator after each period of use. Many of the chemicals used are corrosive and will damage parts of the applicator if allowed to stand for even short periods of time.

Each use and decontamination should be documented in the use and maintenance log.

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STANDARD OPERATING PROCEDURES

**OPERATION, MAINTENANCE, CALIBRATION,
AND APPLICATION TECHNIQUES FOR HAND
GUN SPRAYERS**

SOP NUMBER: 4025

REVISION NUMBER: 6

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and scope

This standard operating procedure is for use by research personnel to ensure accurate operation, maintenance, calibration and application of hand gun type sprayers.

Sprayer preparation and maintenance

Inspect the entire unit for cleanliness, broken or worn parts and blocked nozzles. Clean and/or repair the unit as necessary to ensure proper performance. If the unit is non-functional, contact U of I Project Manager to report the problem.

All hoses and fittings attached to the sprayer assembly should be inspected before each use to be sure it is all in safe operating condition. The system should be pressurized with water only in the tank and all connections inspected for leaks. (Normally done during a calibration run.) The in-line filter should be rinsed out and replaced if necessary.

Operation of 25 gallon sprayer with electric pump

1. Fill tank with sufficient water to run system for several minutes.
2. With handgun in off position, start pump so pressure builds.
3. Squeeze handgun trigger and set operating pressure with pressure regulator valve.

Operation of 50 gallon sprayer with small engine pump

1. Place in-line valve in off position.
2. Be sure handgun is in off position.
3. Start pump engine.
4. Open in-line valve.
5. After ensuring no leaks in line, open hand gun being sure to point hand gun in a safe direction.

Calibration

If protocol lists spray to drip from tree, calibration may not be necessary if the protocol allows other than to confirm that sprayer will place spray where needed and the standard dilution calculations necessary to reach the proper concentration.

If protocol lists a certain amount of spray per tree or area, calculation is performed simply by catching spray for three consecutive 30 second runs. The amount of each catch is measured and used in calculations to determine average output per second as well as output consistency.

Application to drip

When spraying trees to drip, with engine running and in-line valve in the on position on the small engine pump, aim gun at tree and open valve on hand gun. Start on one side of the tree, spraying the entire branch. Spray the tree, always spraying toward the center of the tree, until the tree is dripping. Spray one-half of the tree at a time as it is sometimes hard to get between trees while spraying. Spray one side of the trees in the row then come back down the other side of the trees spraying them as well.

Application for a desired amount of time

When spraying for a desired time or amount with the 25 gallon sprayer with electric pump, aim handgun at tree to be sprayed and turn on pump and start stopwatch at same time. Spray one-half of the tree at a time as it is sometimes hard to get between trees while spraying. Spray one side of the trees in the row then come back down the other side of the trees spraying them as well. Stop the pump when the stopwatch reaches the proper time for half of each tree, write the time down in the notebook, then pull forward to the next tree.

When spraying for a desired time or amount with the 50 gallon sprayer with small engine pump, with pump already running, simply start spraying the tree and start the stopwatch

at the same time. When the stopwatch reaches the desired time, stop spraying. Spray one-half of the tree at a time as it is sometimes hard to get between trees while spraying. Spray one side of the trees in the row then come back down the other side of the trees spraying them as well.

Sprayer decontamination

Clean the sprayer after each period of use. Many of the chemicals used are corrosive and will damage parts of the sprayer if allowed to stand for even short periods of time. After appropriately emptying spray and lines onto trap crop etc., rinse tank three times before running water through system for decontamination. Hose off entire system to prevent contact with any chemical that might have drifted onto equipment.

After an application, mix a small amount of appropriate solvent (e.g. water, detergent, ammonia, acetone) in the tank. Agitate the solution, then flush the solution through the hand gun. Flush with clean water.

Each use should be documented in the use and maintenance log as well as listing if the SOP was followed.

CONTINGENCIES

If there is a problem with the sprayer during the application, stop immediately, turn off the sprayer, and stop the stopwatch if being used. Safety of the applicator is the primary concern. If equipment malfunction, such as a burst hose, has exposed the applicator to excess test material, he/she should immediately take action to remove clothing, rinse off the test substance and prevent further injury.

If the applicator is in no danger, it should be noted or flagged where the application ended. If the malfunction did not affect the application to that point, corrective action should be taken and the application continued.

If a hose ruptured or a nozzle plugged such that it is impossible to determine how much test substance was applied, that area should be noted, flagged, and excluded from sampling. Notify the Study Director of the problem as soon as possible.

If the pressure dropped during the application, measure what is left at the end to determine the actual amount applied. If the pressure jumped, such that there is not enough mix to cover the plot, stop when the sprayer begins to spit. Measure the distance sprayed to determine the actual amount applied.

If the wrong plot was sprayed and it is possible to re-flag and remark (e.g. switch plots) without affecting the integrity of the study, do so. Remember to make changes on the plot map.

If the actual amounts applied differ from protocol guidelines, or the malfunction has affected the integrity of the trial, the study director (or designated person) should be notified immediately for further instructions.

In all cases the data should accurately reflect what occurred and what course(s) of action was taken to preserve the integrity of the study.

If any of the above problems occur, or any other situation that may affect the conduct of the study, notify the study director at the earliest possible time.

Documentation

Record all necessary information in enough detail to reconstruct the procedure, including the SOPs used, into the trial notebook. Remember to make the appropriate entries into the sprayer use and maintenance log, and the Test Substance Use Log.

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STANDARD OPERATING PROCEDURES

**OPERATION, MAINTENANCE, CALIBRATION,
AND APPLICATION TECHNIQUES FOR
AIRBLAST SPRAYERS**

SOP NUMBER: 4030

REVISION NUMBER: 1

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-17

Purpose and scope

This standard operating procedure is for use by research personnel to ensure accurate operation, maintenance, calibration and application of Airblast type sprayers.

- 1.0 Operation
- 1.1 Prior to use, a general inspection will be made of the sprayer and tractor for visual damage or potential problems.
- 1.2 Pressure hoses and fittings will be inspected.
- 1.3 The pressure gauge will be checked to ensure it operates properly.
- 1.4 The PTO must be engaged slowly at low RPM and then slowly increased until desired operating speed is reached.
- 1.5 If an in-line flowmeter is in use, it should be re-set to zero before spray application begins.
- 1.6 Actual spray pressure, and speed (pass times) during application will be recorded and should remain unchanged or similar to calibration.

- 1.7 The sprayer will be timed through a test run in the actual test plot prior to the application to verify travel time and the verification pass time will be recorded.
- 1.8 Each pass through the plot during an application will be timed, recorded and used to determine the actual amount of test substance applied.
- 1.9 The actual rate of test substance applied may also be confirmed by an in-line flowmeter, if present.

2.0 Calibration

- 2.1 Calibration shall either be performed or verified prior to each use.
- 2.2 Prior to calibration:
 - 2.2.1 The spray system will be operated to verify that pressure is constant and that the nozzles provide the desired pattern.
 - 2.2.2 Calibration of the in-line flowmeter (if present) will be performed or verified. To verify calibration, liquid shall be metered through the flowmeter into a calibrated container to the 5-gallon mark. If the LCD meter indicates a variation of $\pm 2\%$ (or ~380 mL), the flowmeter must be recalibrated (refer to instrument operation manual).
 - 2.2.3 Constant pressure and RPM will be set.
- 2.3 The spray volume per acre can then be calibrated, and/or verified, by an acceptable method that is accurate and reproducible. These methods will be documented, and may include, but not necessarily be limited to, the following:
 - 2.3.1 Operating and timing the sprayer over a known area while measuring the output of water. The output volume can be measured with an attached in-line flowmeter (if available) or other method that is accurate and reproducible.
 - 2.3.2 Filling the sprayer with an unknown volume of water, then operating the spray system for a pre-determined period of time, usually 30 seconds, and measuring the output volume for that period with an attached in-line flowmeter or other method that is accurate and reproducible; and b) timing the sprayer over a known distance.

- 2.3.3 Filling the sprayer with a known volume of water, then measuring the time required to discharge that volume from the spray system; and b) timing the sprayer over a known distance.
- 2.3.4 Filling the sprayer with a known volume of water, then running sprayer for a set amount of time and measuring the amount of water remaining.
- 2.4 Minimally, the sprayer output will be measured three times and sprayer speed will be measured three times, and the average of each consecutive like measurement will be used in the final calculations made to determine the actual application volume. Each like measurement (volume or speed) used for final calculations, must not vary more than 5% of the mean value of each individual measurement.
- 2.5 Calibration is preferred on the day of application. If not performed on the day of application, calibration could occur on the day prior to application, but sprayer discharge must be verified and documented at least once on the day of use, and must be within $\pm 5\%$ of the calibrated value or the sprayer must be re-calibrated.
- 2.6 The method of mathematical calculations used will be the choice of the Field Research Director and shall be recorded in the raw data.
- 2.7 After calibration and prior to use the sprayer must have the tank and spraying system drained of all water. The following method should be used:
 - 2.7.1 With the PTO off, open the tank drain valve, remove all water and leave drain valve open.
 - 2.7.2 Open bypass valve to the short relief hose. Start the PTO and turn on spray control valve. Shut spray valve off and stop PTO when water no longer is pumped from relief hose. Close relief hose valve.
 - 2.7.3 Drain the filter canister on the side of the sprayer, check the screen and clean if necessary, then return to position.
 - 2.7.4 Verify spray tank is empty, then close drain valve.

3.0 CLEANING

- 3.1 After use, and between treatments with different chemicals or different formulations of the same chemical, the spray system will be thoroughly cleaned by triple rinsing with water with or without the addition of a cleaner or solvent.
- 3.2 When making applications at different treatment rates with the same chemical, if applications are made in order from the lowest to the highest treatment rate, it will not be necessary to clean the spray system between treatments. When making applications at the same treatment rate but at different gallons per acre (GPA), if applications are made in order from the highest (dilute) to the lowest (concentrate) GPA, it will not be necessary to clean the spray system between treatments. In these cases, at a minimum, the spray tank shall be completely drained and the next tank mix prepared.
- 3.3 The sprayer exterior and the tractor will be hosed off with clean water.
- 3.4 Exceptions to these cleaning requirements may include:
 - 3.4.1 Equipment used in applying chemicals that may require specific solvents to remove chemical residues. In this case, cleaning methods must be thoroughly documented in the trial raw data.

4.0 MAINTENANCE

- 4.1 Maintenance required as a result of normal wear defines routine maintenance. The following routine maintenance will be performed at least once every three years during heavy use years or less frequently during light use years.
 - 4.1.1 Pump lubricant changed.
 - 4.1.2 Driveline and shaft bearings (if present) greased.
 - 4.1.3 Drive belts (if present) checked and replaced if necessary.
- 4.2 Maintenance required as a result of equipment malfunction, not normal wear, defines non-routine maintenance. In the event of non-routine repair, a record will be kept which will describe the nature of the defect, how and when the defect was discovered, and remedial action, if any, taken.

5.0 CONTINGENCY PROCEDURES

- 5.1 If during the generation, measurement or assessment of data, an equipment malfunction occurs, the supervising personnel and Study Director shall be

advised of the malfunction. The equipment will be replaced or repaired and, if possible, the study will continue. Documentation of the malfunction shall be described in the study raw data and an appropriate entry made in the equipment log describing the repair.

6.0 RESPONSIBLE PERSONNEL

- 6.1** Equipment inspection, calibration, cleaning and maintenance (routine and non-routine) shall be performed or supervised by the Field Research Director.

7.0 RECORDS

- 7.1** A record of the dates of equipment inspection, calibration, maintenance and cleaning will be kept in an equipment logbook. Calibration results can be accessed by reference of the study number recorded in the Equipment Log at the time of these operations. Historical records will be retained in the archives

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STANDARD OPERATING PROCEDURES

**CALIBRATION AND APPLICATION TECHNIQUES
FOR A CO₂-POWERED SPRAYER**

SOP NUMBER: 4505

REVISION NUMBER: 7

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and Scope

This standard operating procedure is for use by research personnel to ensure accurate calibration and application of the test substance when applied by CO₂ sprayer.

Instructions for Proper Calibration of Backpack Sprayer

General information regarding the operation and maintenance of CO₂ sprayers can be found in U of I SOP #4005 and #4015. The objectives behind calibrating a sprayer are ensuring that the test substance will be applied with even coverage for the desired pattern and that the amount applied can be accurately determined. To meet these goals the following steps should be performed, though it is not necessary to do them in the order given.

1. Select and install the proper nozzles to achieve the required specifications for gallons per acre (GPA) and pounds per square inch pressure (PSI) and desired pattern characteristics.
2. Before each application, determine spray output (volume/time) for each nozzle. This is done by catching the output from each nozzle and measuring the amount caught. The calibration will consist of at least three trial runs lasting at least 20 seconds each. If any nozzle differs in output by more than 5% of the average of all nozzles, clean the screen and nozzle and/or inspect the boom for leaks. If the nozzle is defective, replace it and check to ensure its output is now within the desired range. If you change a nozzle after a calibration run, you will have to calibrate again. If all nozzles are within the +/- 5% criteria mentioned above, total each output and divide by the number of seconds sprayed. This will give you the amount per second of total output.

3. Determine the volume of spray required to cover the calibration area at the desired GPA.
4. Based on the boom output, determine how many seconds it should take to deliver the volume calculated in step 3.
5. Travel across ground with same conditions as the plot until a speed is reached that gives the amount of time necessary to be in the plot to put out the proper amount of spray as determined in #3 above. (Remember to have a consistent speed, one should be dressed and carrying weight similar to the actual spray.)

Calibration Verification

If the sprayer was calibrated at the trial site on the same day as the application, it is recommended, but not necessary, to verify the calibration. If this is not the case, the nozzle output should be rechecked at the trial site or the calibration should be verified by following the same steps for calibration, however only one run catching the spray is necessary. If the calibration re-check is not within 5% of the initial 3-run calibration, then 2 more runs are needed to produce a new full calibration. The total output per second from the new full calibration will be used for all calculations in the application.

All calibration data (equipment used, nozzle output, parameters of all runs, etc.) shall be entered in the raw data in enough detail to reproduce the procedure.

Application

1. Information about handling test substances can be found in SOP #3005.
2. Wear the needed protective clothing that is required for the test substance.
3. Always maintain the same speed and use the same parameters (psi, nozzle configuration, etc.) as in the successful calibration run when making the application.
4. Follow protocol guidelines for maximum wind velocity during spray operation. If no guidelines are given, do not spray in winds greater than 5 MPH, as measured by the anemometer. When making a decision whether or not to spray, keep in mind the buffer size, the wind direction in relation to the UTC, droplet size, etc.
5. Take and record needed environmental conditions that are specified in protocol and trial notebook.

Methods of application

The following method is suggested for applying test substance so the actual amount applied can be determined.

Output/speed

1. Mix more materials than needed for the application.
2. Start and end spray somewhere in the buffer areas.
3. Time the application between the plot markers with a stop watch. Sample formula suggested to calculate applied amounts:

$$\frac{\text{ml (total mix)}}{\text{sec}} \times \frac{\text{seconds (timed)}}{\text{plot area (acres)}} = \text{ml mix/acre}$$

$$\frac{\text{ml mix}}{\text{acre}} \times \frac{\text{ml test substance}}{\text{ml total mix}} = \frac{\text{ml test substance}}{\text{acre}}$$

Remember, calculations should be included for figuring the GPA and the actual rate of test substance applied. The result should be in the same units as specified in the protocol.

Contingencies

If there is a problem with the sprayer during the application, stop immediately, turn off the boom, and stop the stopwatch. Safety of the applicator is the primary concern. If equipment malfunction, such as a burst hose, has exposed the applicator to excess test material, he/she should immediately take action to remove clothing, rinse off the test substance and prevent further injury.

If the applicator is in no danger, it should be noted or flagged where the application ended. If the malfunction did not affect the application to that point, corrective action should be taken and the application continued.

If a hose ruptured or a nozzle plugged such that it is impossible to determine how much test substance was applied, that area should be noted, flagged, and excluded from sampling. Notify the Study Director of the problem as soon as possible.

If the pressure dropped during the application, measure what is left at the end to determine the actual amount applied. If the pressure jumped, such that there is not enough mix to cover the plot, stop when the sprayer begins to spit. Measure the distance sprayed to determine the actual amount applied.

If the wrong plot was sprayed and it is possible to re-flag and remark (e.g. switch plots) without affecting the integrity of the study, do so. Remember to make changes on the plot map.

If the actual amounts applied differ from protocol guidelines, or the malfunction has affected the integrity of the trial, the study director (or designated person) should be notified immediately for further instructions.

In all cases the data should accurately reflect what occurred and what course(s) of action was taken to preserve the integrity of the study.

If any of the above problems occur, or any other situation that may affect the conduct of the study, notify the study director at the earliest possible time.

Documentation

Record all necessary information in enough detail to reconstruct the procedure, including the SOPs used, into the trial notebook. Remember to make the appropriate entries into the sprayer use and maintenance log, and the Test Substance Use Log.

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STANDARD OPERATING PROCEDURES

**CALIBRATION AND APPLICATION TECHNIQUES
FOR BACKPACK MIST BLOWER**

SOP NUMBER: 4510

REVISION NUMBER: 2

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and Scope

This standard operating procedure is for use by research personnel to ensure accurate calibration and application of the test substance when applied by backpack mist blower.

Instructions for Proper Calibration of Blower

- A. Select the proper nozzle setting to achieve the desired spray pattern characteristics.
- B. Select the proper discharge nozzle setting to achieve the required specifications for gallons per acre (GPA).
- C. Add a known volume of water greater than or equal to 3.0 liter into the spray tank. With the engine running at operating speed and the mister arm moving in the same motion as the application will be performed, discharge the tank for a known time period (approx. 30 seconds), without allowing tank to become empty. Shut down the engine and drain the liquid remaining in the tank into a calibrated vessel. Subtract this volume from that initially in the tank to determine the volume sprayed. Repeat this process a total of 3 times. The volume sprayed in any one run should not differ from the average of all 3 runs by more than 5%. If the volume sprayed in any one run differs by more than 5% from the average of the three runs check the sprayer for leaks, air bubbles etc., correct any problems and recalibrate. Spray the plot using the output/speed method described in SOP #4505.

- D. If the sprayer was calibrated for an earlier application of the same study, and all of the protocol requirements are met, a recheck of sprayer output may be performed. A recheck consists of 1- 30 second run that must be within 5% of the original application calibration. The total output per second from the original will be used for all calculations for the application. If the calibration re-check is not within 5% of the initial 3-run calibration, then 2 more runs are needed to produce a new full calibration. The total output per second from the new full calibration will be used for all calculations in the application.

Contingencies

If there is a problem with the sprayer during the application, stop immediately, turn off the engine and spray valve, and stop the stopwatch. Safety of the applicator is the primary concern. If equipment malfunction, such as a burst hose, has exposed the applicator to excess test material, he/she should immediately take action to remove clothing, rinse off the test substance and prevent further injury.

If the applicator is in no danger, it should be noted or flagged where the application ended. If the malfunction did not affect the application to that point, corrective action should be taken and the application continued.

If a hose ruptured or a nozzle plugged such that it is impossible to determine how much test substance was applied, that area should be noted, flagged, and excluded from sampling. Notify the Study Director of the problem as soon as possible.

If the wrong plot was sprayed and it is possible to re-flag and remark (e.g. switch plots) without affecting the integrity of the study, do so. Remember to make changes on the plot map.

If the actual amounts applied differ from protocol guidelines, or the malfunction has affected the integrity of the trial, the study director (or designated person) should be notified immediately for further instructions.

In all cases the data should accurately reflect what occurred and what course(s) of action was taken to preserve the integrity of the study.

If any of the above problems occur, or any other situation that may affect the conduct of the study, notify the study director at the earliest possible time.

Documentation

Record all necessary information in enough detail to reconstruct the procedure into the trial notebook. Remember to make the appropriate entries into the sprayer use and maintenance log, and the Test Substance Use Log.

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STANDARD OPERATING PROCEDURES

**CALIBRATION AND APPLICATION TECHNIQUES
FOR A GRANULAR DROP TYPE APPLICATOR**

SOP NUMBER: 4520

REVISION NUMBER: 7

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-10-12

Purpose and Scope

This standard operating procedure is for use by research personnel to ensure that all personnel are knowledgeable in the general calibration principles applicable to pull behind Gandy Drop granular application equipment. A standard calibration procedure is also given.

Variables Which Influence Application Rate

Variables which influence application rate of granular pesticides include size of metering opening, speed of agitation, travel speed, roughness of field, flowability of granules (which depends on granule size, density, and type), temperature and humidity.

Standard Calibrating Procedures

1. Determine output per second as with a liquid application making 3-30 second runs. This can be performed by lifting spreader on blocks and spinning wheels for 30 seconds over a tarp or similar catch item.
2. Determine how much active ingredient is needed to cover test plot and convert this to the weight of product.
3. Adjust speed of 4-wheeler to obtain the amount of time in the plot needed to deliver the proper amount of test substance.

Decontamination

Following the application, decontaminate the applicator with an appropriate solution and allow to dry.

Contingencies

If there is a malfunction which disrupts the application for a distance which could affect the integrity of the trial, stop, flag the spot in the field, and note in the data where the disruption occurred. Repair the malfunction and continue. Avoid sampling from any questionable areas.

If any of the above problems occur, or any other situation that may affect the conduct of the study, notify the study director at the earliest possible time.

Documentation

Record all necessary information in the appropriate equipment use and maintenance log.

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STANDARD OPERATING PROCEDURES

**CALIBRATION AND APPLICATION TECHNIQUES
FOR ELECTRIC GRANULAR APPLICATOR**

SOP NUMBER: 4525

REVISION NUMBER: 2

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-01-07

Purpose and Scope

This standard operating procedure is for use by research personnel to ensure that all personnel are knowledgeable in the general calibration principles applicable to electric granular application equipment. A standard calibration procedure is also given.

Variables Which Influence Application Rate

Variables which influence application rate of granular pesticides include size of metering opening, speed of agitation, travel speed, roughness of field, flowability of granules (which depends on granule size, density, and type), temperature and humidity.

Standard Calibrating Procedures

1. Disconnect spreader plate from gandy box by removing chain running spreader plate.
2. Catch granules for three consecutive 30 second runs by placing pitcher under drop tubes on top of spreader plate.
3. Weigh each catch to determine output per second and output uniformity.
4. Reattach spreader plate to gandy box drive system with chain. Speed of spreader plate can be changed by changing gear ratio on side of gandy box.

Broadcast applications:

1. Determine how much active ingredient is needed to cover test plot and convert this to the weight of product.
2. Adjust the speed of the four-wheeler to cover the plot in the desired time.

Decontamination

Following the application, decontaminate the applicator with an appropriate solution and allow to dry.

Contingencies

If there is a malfunction which disrupts the application for a distance which could affect the integrity of the trial, stop, flag the spot in the field, and note in the data where the disruption occurred. Repair the malfunction and continue. Avoid sampling from any questionable areas.

If any of the above problems occur, or any other situation that may affect the conduct of the study, notify the study director at the earliest possible time.

Documentation

Record all necessary information in the appropriate equipment use and maintenance log.

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STANDARD OPERATING PROCEDURES

**OPERATION, MAINTENANCE, AND
MONITORING OF RESIDUE SAMPLE STORAGE
FREEZERS**

SOP NUMBER: 5010

REVISION NUMBER: 14

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-9-17

Purpose and scope:

To establish procedures for use by research personnel to guide them in the proper operation, maintenance and monitoring of residue sample storage freezers to ensure integrity of frozen samples.

Personnel:

The Field Research Director or staff at each location where samples are stored shall be responsible for familiarizing themselves with this SOP and ensuring that it is followed.

Placement and Operation:

The freezer should be located in an area secure from the possibility of contamination and separate from test substance storage or areas where equipment used to handle test substances are kept whenever possible.

Make sure that the electrical outlet to be used is grounded and that the circuit is not overloaded. The outlet should not be connected to a wall switch that could accidentally be turned off. There should be unobstructed air flow around the coils and adequate space around the freezers to ensure that sample integrity is protected during placement into the freezers.

The freezer should be capable of maintaining an average temperature that is within the range acceptable for sample storage. This should be ensured prior to its use by placing a constant recording thermometer or a minimum/maximum registering thermometer inside for a period that contains at least one defrost cycle for frost-free freezers and also includes the hottest part of the day. Freezer doors should be sealed tightly and locked at all times when samples are present. Freezers may be unlocked if no samples are present.

Maintenance:

All freezers shall be defrosted and cleaned with an appropriate decontaminating solution when the absence of any samples permits. Decontamination should be noted on the appropriate freezer log. This is considered routine maintenance. Upon restarting after cleaning, min/max readings shall be checked and recorded in the MUPDP freezer maintenance log within a week to verify that freezers are still holding temperatures within proper range.

Any unusual noises from the motor, failure of the freezer or any of its components, or inability by the freezer to maintain required temperatures warrants the services of a qualified repair person. Any repairs performed must be documented in a log designated for the freezer being repaired. This is considered non-routine maintenance.

Monitoring:

Each freezer shall have a designated number that is posted on the freezer and shall be used on any temperature logs.

Each freezer shall have a standardized Hobo data logger, minimum/maximum recording thermometer, or other recording thermometer inside at all times that samples are enclosed. When samples are absent, monitoring devices are not required. Minimum/maximum thermometer ranges shall be recorded on a freezer temperature log and reset when Hobo data is being downloaded. See SOP number 2710 for guidance on Hobo placement and operation.

Freezers will be equipped with fully operational alarm system connected to a phone line. When freezer alarm system is present, it must be checked approximately annually. Alarm should be set to contact appropriate personnel when temperatures rise above normal established cycling ranges of defrost cycles. As these cycles may vary from freezer to freezer, monitoring of freezer temp cycles is recommended prior to setting temp parameters for alarm systems. Temp alarm setting should be no higher than 20 degrees F. When testing alarm prior to placing samples for the upcoming season, take lead from freezer and hold in the palm of a warm hand until temp increases past alarm settings. Observe phone lines while calls are being made. If alarm fails to make appropriate calls, make repairs as needed or replace device. Check the backup battery in the alarm system during environmental monitoring device standardization each spring. Record alarm test results on MUPDP Environmental Monitoring Device Standardization Log form.

A min/max thermometer will serve as the hobo data logger backup.

Equipment failure:

In the event that the freezer fails due to power outage or malfunction, the following procedures should be implemented:

1. Limit the number of times that the freezer door is opened to retain cold in freezer.
2. If failure lasts longer than 24 hours or temperature in freezers exceeds 32 degrees F, proceed with at least one of the following:
 - a. Put enough dry ice in the freezer to last until samples are moved or freezer functioning is restored and open door slightly.
 - b. Arrange for samples to be moved into another freezer. Any samples transported should be kept with their temperature recording device. If time does not allow for pre-use monitoring of the new freezer, check temperature recording device often enough to ensure that freezing temperatures are being maintained. Make note of new sample storage location in the freezer temperature log or on the thermograph chart, and especially in the trial notebook where the chain of custody should be detailed.
 - c. Use Honda Generator (stored beside freezers) to power up freezers if the problem is power outage. One freezer at a time may be plugged into generator and cooled down. By alternating between freezers, temperatures should be kept in the frozen range. Be sure to place generator outside of building in docking area and use supplied extension cord to reach freezers. Monitor temperatures closely to keep freezers within desired temperature range.

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STANDARD OPERATING PROCEDURES

**SAMPLING AND STORING CROPS FOR RESIDUE
ANALYSIS**

SOP NUMBER: 5505

REVISION NUMBER: 10

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-17

Purpose and scope

The purpose of this standard operating procedure is to ensure that samples obtained for residue analysis are appropriately collected and handled to preserve their integrity and the integrity of the resulting data. If samples are improperly identified, defrosted, or are lost at shipping, it can result in the invalidation of the entire study. All IR-4 research personnel involved in collecting residue samples should be familiar with this SOP. Farm Crews should be trained in specifics for the crop to be sampled if they are helping with the actual sampling.

GENERAL SAMPLING PROCEDURES

Specific sample types requested including growth stage, plant part, soil depth, sample size, and PHI should be outlined in the protocol. Provide the crop and substrate as requested in the protocol. The following guidelines should be adhered to:

1. Samples should be taken representatively from the plot area, however, do not sample diseased, stressed or undersized crops at a stage that would not normally be harvested, unless specifically requested in the protocol or amendment(s).
2. Samples should be typical of the commercial commodity.
3. Do not subsample the plants in the field, i.e., do not provide portions of plants unless specified in the protocol or otherwise allowed and documented.
4. Do not collect samples from the outside edges of the plot where there may not have been uniform coverage of the test substance at application.

5. Do not field trim any sample unless requested to do so in the protocol.
6. Do not wash any crop samples unless requested by Sponsor. Surface dirt should be removed by knocking off the majority of dirt or brushing with a clean brush. Avoid removing surface residues during handling or packing.
7. If the time from sampling to placing of the samples in the designated frozen storage is likely to exceed approximately one hour, or if the ambient temperatures are high, it is recommended that the samples be cooled immediately in the field by placing them in an insulated container with blue ice or dry ice. The longer the samples are to be held between field and freezer or the hotter the conditions, the greater the need to take measures to keep them cool. If wet ice is used, care should be exercised to keep the samples from getting wet. Plastic sample bags that have no fiber reinforcement should be protected from direct contact with dry ice since the extreme cold may cause cracking of the bags. Always record in the data the approximate time from sampling to freezer.
8. Determine the moisture content of the sample if asked for in the protocol. If an estimate of moisture content is asked for, describe method used to determine estimate in sufficient detail in the Field Data Notebook, for example, dry weight measurement, commercial practice, or a generally established field practice.

Using non-U of I owned equipment

When equipment is borrowed or rented and no log is in place, document the condition of the equipment and ensure that it is operating properly. Obtain instructions or consult the operator's manual when possible.

Details of the operation and decontamination of equipment for which U of I has no SOP should be addressed in the raw data in enough detail to be considered up to SOP standards (please see SOP 5615).

Sampling with commercial equipment

Some types of samples can be collected from large scale commercial harvesting equipment. Decontamination of this type of equipment is often best handled by running the harvester through a sufficient amount of the untreated crop to adequately clean it. Edges of plots should be avoided and the sample should be collected in a manner that will result in a representative sample without collecting an excessive volume. Small amounts of the requested commodity could be taken from the stream of the harvested commodity at numerous locations throughout the plot. Another method might be to harvest the entire inner area of the plot, then thoroughly mix the sample and extract the requested sample from the mix.

When moving from one treatment into the next, care should be taken to ensure that all of the commodity from the previous plot has been purged from the harvester before beginning the collection of the next sample.

Processing samples

Most of the concepts expressed above under the section on commercial harvesters applies also to processing equipment such as cotton gins or pecan shellers. Generally the equipment will need to be decontaminated by processing some untreated control commodity through the system before starting and when necessary between treatments. As with all sampling methods, the untreated control samples should be handled first, and then progress from the lowest rate of the treatments to the highest. All of the commodity for each treatment must be purged through the equipment before a sample can be obtained from the next treatment. Particular care must be taken to ensure that the identity of each sample is maintained through all of the processing steps.

AVOID CONTAMINATION

Care should be exercised to avoid contamination when sampling crops.

1. Use clean tools that have been appropriately decontaminated with a solution suitable for the chemistry involved. Work on a clean drop cloth, if necessary, to avoid getting the sample in contact with the ground or a truck bed. Clean gloves should be used for each treatment.
2. Use new storage bags only. Avoid torn or worn bags. Double bag samples that could be handled roughly.
3. Do not contaminate samples with hands or clothes that have been in contact with the test substance or other sources of potential contamination.
4. Sample the control samples first, then proceed from the lowest to the highest rate.
5. Avoid transporting residue samples in vehicles carrying pesticides or contaminated equipment. During transport, care should be taken to keep the untreated samples separate from the treated samples. It is recommended that the bags be placed inside clean coolers or boxes to protect the samples from contamination.
6. Avoid packing, storing, or freezing residue samples in areas that may be contaminated with pesticides.
7. The method of decontamination for equipment and personnel should be noted in the raw data.

8. Use Ziplock style bags when needed for juicy or small samples.

Identification of samples

Each sample must have unique identification that is affixed to the sample container in a way that cannot be accidentally removed or obliterated. The identification may be a code that is referenced in the data, or all of the details of the sample (i.e.: sampling date, treatment number, crop part, PHI, etc.) may be written out on the sample bag. Adhesive labels should be stapled onto bags (near the top of the bag to prevent staple holes being a problem) as they may come off in the freezer.

If a code or sample number is used, this must be explained in the data with a key to the meaning of the sample codes. Often the protocol will designate sample numbers in advance, but, if not, a system should be devised to ensure that the samples can be positively identified both at the field site and at the laboratory.

When using IR-4 sample bags, writing the Sample ID on the outside of the bag in large letters with a sharpie or other marker will help with identification when the samples are frozen.

Storage of samples

Avoid storing untreated and treated samples from the same study in the same freezer if possible. If they are to be stored in the same freezer, keep them physically separated by placing samples on separate shelves or placing a physical barrier between treatments (e.g., place sample bags in separate plastic garbage bags).

When residue samples are placed in the freezer, it is helpful to record the freezer equipment number in the raw data for that trial. Alternatively, or in addition to, samples should be logged in and out (even when being moved to another freezer at the same facility) using a sample inventory sheet.

Documentation

A short description of exactly how the samples were taken, especially how the sampling method ensured an impartial, representative sample to help reviewers reconstruct how sampling was performed should be included.

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STANDARD OPERATING PROCEDURES

SAMPLING SOILS FOR CHARACTERIZATION ANALYSIS

SOP NUMBER: 5515

REVISION NUMBER: 5

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-17

Purpose and scope

This standard operating procedure is for use by research personnel to ensure that representative soil samples are obtained and properly handled when gathering information on the general nutrient level and physical properties of the soil. This document outlines a recommended sampling plan, sampling tools, number of sample sites, sampling technique, and mailing instructions, which should be followed unless specific instructions are supplied by the protocol.

Sampling tools

A recommended sampling tool is a soil probe. In cases where very hard or stony soil is encountered and the use of a soil probe is not practical, then a shovel or trowel should be used.

Number of sample sites and techniques

1. If the experimental area appears uniform, then one composite sample should be taken for the experimental plot area. If the area does not appear uniform, then samples should be taken by replication, or compositing of appropriate replications together to give two, three, or more samples.
2. At least 10 cores (about 10 cores if replicates are taken) should be taken to obtain a representative composite sample. Before taking the core sample, scrape off the top debris or surface residues. Sample a field which has been in row crops by sampling between the rows, thus avoiding fertilizer band areas. However, if a

pesticide in question has been applied as a band, then sampling should be made in the row and this should be noted in any report.

3. The sample cores should be placed in a clean sample bag and labeled by study ID number.

Shipping instructions and distribution of results

1. Ship the individual soil samples as soon as practical after you take them. The soil samples do not have to be dried or sieved; however, wet soil samples should not be stored for a long time before they are mailed to the testing lab. Consult the protocol to ensure you request all desired analyses be performed. If having more than one sample analyzed, request separate sheets for each trial to ensure one original for each trial.
2. All copies of the soil test results will normally be sent to the person paying the bill, unless other procedures are requested. File the original of the soil test results with the raw data and fill out appropriate pages in the field trial notebook. Whenever possible, have the results sent to the U of I Field Research Director so that the complete data package can be prepared in house.
3. Along with the results of the analyses, a copy of the general soil description, chemical and physical properties, and location of the field studies as they appear in the Soil Survey should be retained if the information is available.

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STANDARD OPERATING PROCEDURES

**RESIDUE SAMPLE SHIPMENT AND
DOCUMENTATION**

SOP NUMBER: 5520

REVISION NUMBER: 10

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 4-7-15

Purpose and scope

This standard operating procedure is for use by research personnel to ensure proper residue sample shipment and documentation.

Sample packaging

Follow specific instructions found in protocol for packaging residue samples for shipment. If none are given, consult sponsor SOPs or follow the procedure below:

1. All residue samples should be shipped in boxes designated for residue sample shipment whenever possible. Use the boxes sent by the sponsor for use in that trial. If none have been sent by the sponsor, then alternative residue sample boxes may be used.
2. Write clearly on the top of all boxes to be sent: the unique study identification number for that trial and the unique sample identification numbers assigned to the samples that are within that box, or type of sample (e.g. UTC or TRT). It is also helpful to include a description of the samples (e.g. UTC grain, treated straw, etc.) If shipping by ACDS, identify box by trial number and treatment. ACDS will assign specific box number to each box. If dry ice is included then the box must be labeled with the appropriate warnings and codes as required by law. These can be obtained from the shipping company. ALWAYS write on boxes shipped air freight that there are important frozen samples within that must not defrost.
3. If more than one box is sent to a location, the boxes should be marked in a manner to indicate the number of boxes sent in that shipment. For example: 1 of 3, 2 of 3, 3 of 3.

4. If there is sufficient room, both treated and untreated samples may be placed in the same container. Separate samples by sealing treated and untreated samples separately in additional garbage bags (minimum double bagged) or other suitable barriers to prevent contamination. No single box should weigh more than 75 pounds.
5. The appropriate paperwork should be included in each box.
6. After placing samples in the boxes, close and tape all boxes securely shut to prevent accidental opening during shipment.
7. If the samples that are not packed with dry ice are not immediately put into a frozen storage area, then the temperature within the boxes should be monitored. If the temperatures begin to rise above freezing, they must be protected from defrosting by unpacking them back into the freezer, or by the addition of dry ice. All activities must be written into the data record including all observed temperatures and their duration.

Sample shipment by airfreight

1. Follow specific protocol procedures for shipment by airfreight and if possible follow IR-4 advisory listed below.

IR-4 Advisory:

Shipping with dry ice:

Dry Ice – type and amount: Obtain dry ice from a commercial or university producer, when possible. Large volume production dry ice blocks (or slabs) are denser than dry ice pellets or blocks made with a portable dry ice maker. Having less surface area, these denser blocks or slabs last longer, and are preferable to the other types of dry ice. The amount of dry ice needed depends on the type of dry ice used, the shipping distance and the density of the residue samples. Leafy greens and blueberries will thaw much faster than potatoes. A common ratio in the literature is 4 lbs dry ice per pound of sample per 24 hours of travel. Experienced air shippers recommend 40 lbs of block dry ice, regardless of the sample weight, increasing the amount to 50 lbs if there is any concern that the samples might be delayed (i.e. take more than two days).

Freezer Boxes/Coolers: Freezer boxes or thick-walled foam containers are preferable to coolers for shipping with dry ice, as they allow for better venting. IR-4 has used coolers with no adverse issues; just remember to allow for adequate release of the carbon dioxide gas when taping the coolers. Note that non-insulated plastic containers can become brittle under the low temperature conditions generated by dry ice. Shipping boxes can be made by lining good, heavy, strong cardboard boxes with thick sheets of an insulating material cut to size (tight fit). Non-lined cardboard boxes should never be used as they often lose their integrity during shipping, due to condensation and space voids as the dry ice sublimates.

Packing for shipment: Be sure the residue samples are completely frozen before packing. If not, contact the Study Director. Try to “surround” the samples with dry ice and/or place dry ice in the bottom of the container, then samples, then dry ice, then samples, then dry ice. Be sure that there is dry ice on top of the samples as cold air settles. Try to use a container of appropriate size to fit the samples and dry ice snugly. Fill any voids with wadded paper or bubble wrap to minimize sample movement as dry ice sublimates. The labs request that foam peanuts or shredded paper NOT be used, as they make a horrible mess when unpacking.

Ship Monday to Wednesday by next day AM delivery. After Wednesday there is not enough time before the weekend if there are any problems or delays. Always **get confirmation from the lab** that someone will be there to receive the samples before air shipping. There is always a possibility that the lab would not be able to receive your samples on that date due to holidays, vacations, etc. It is advisable that you track your shipment to confirm arrival at the destination.

Sample shipment by freezer truck

1. Dry ice and insulated boxes are not needed for shipment of residue samples by freezer truck.
2. Package samples just prior to freezer truck arrival. This will minimize the time in which the sample is exposed to room temperature and prevent thawing of sample. If it is not immediate, document in the study data, the time of packing and the time that the samples are placed in the freezer truck. If a walk-in type or other large enough freezer is used where packing can be done while the samples are still in the freezer, then these times are not needed. Time of transfer to the freezer truck should still be recorded.
3. Call or email the responsible party at the destination address and inform him/her of the shipment and when to expect it to arrive.

Hand Delivery of Samples

If the laboratory or processing facility is within driving distance and the protocol allows, the samples may be hand delivered, taking measures to ensure sample integrity while in transit.

Documentation

1. Fill out all sample shipping papers as described in protocol.

2. Fill out the proper pages in the field trial notebook at the time of shipment and place copies of proper pages in the proper boxes.

3. The bill of lading from the shipping company is a critical piece of raw data for the study. The study number must be written on the bill and it is recommended that the sample numbers that are in each box be indicated here as well.

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STANDARD OPERATING PROCEDURES

POTATO SAMPLING IN STORAGE BAYS

SOP NUMBER: 5600

REVISION NUMBER: 4

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-7-03

PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to indicate the minimum procedure that must be followed and the documentation that must be retained during the collection of stored potato tubers that will be analyzed for residues. Tests for which this SOP is appropriate are any stored potato tests from which samples for residue analysis are taken, the results of the analysis of which are to be reported to the U.S. Environmental Protection Agency in support of a pesticide or plant growth regulator registration.

In order to ensure accurate generation of residue samples and data, all personnel conducting tests must follow acceptable procedures that document and/or explain how samples were collected and labeled. This SOP outlines the procedures and it indicates the documentation that must be presented in the study records when potato tuber samples are collected for residue analysis.

CONSIDERATIONS

Refer to U of I SOP 5505 (Sampling and Storing Crops for Residue Analysis) and the study protocol for general information on contamination prevention.

PROCEDURE

Sampling Potato Tubers in Storage Bays

The study protocol describes the study site and study schedule (harvest date, storage bay loading, chemical application, sampling times, and sample sizes). Only the samples indicated in the protocol are to be collected.

To facilitate tuber sample collection from a pile of potatoes 12' wide by 20' long by 16' high, 42" diameter corrugated steel pipes were custom made to serve as free standing access ways to the bottom of the pile. The bottom end of the sampling pipes are sealed with a welded steel plate and the top is covered with a removable steel lid. Six sampling ports are located at three levels in the pipes, with two ports opposite one another at each level. The ports are 18" diameter openings in the vertical pipe and can be closed with a sliding steel door.

In each storage bay there are two sampling pipes, each centered over one of the two subfloor air ducts and oriented so that the sampling ports are in line with a duct. The sampling pipes are offset from one another so that potential variations in the amount of chemical aerosol carried by the air in each duct can be accounted for during tuber sampling.

Contamination Prevention During Collection of Tuber Samples

All samples to be collected from the untreated storage bay should be gathered before collecting any treated samples.

When sampling from treated bays, Tyvek coveralls must be worn by all persons entering the sampling pipes. Disposable latex gloves must be worn by the sample collectors within the pipe and all other persons who handle the tubers prior to placement in the residue bags. Gloves will be changed for fresh ones before collecting samples from each port in the sampling pipe (unless samples are being composited between ports at a given sampling depth). Gloves will be changed in a similar manner by all other persons who are in contact with the collected tubers prior to bagging.

Collected tubers are to be placed in a wire basket lined with a disposable plastic garbage bag that will be changed between each sample. Once the tubers are brought up to the top of the pile for bagging, a poly tarp will be spread out so that collected tubers from the two sampling pipes can be composited if mandated by the protocol. The tarps prevent the samples from coming in contact with tubers on the top of the pile. Tarps must be dedicated to specific bays and are not to be used elsewhere during conduct of the study. Once collected tubers are placed in properly identified residue bags, the bags should be tied shut prior to removal from the bay.

All contaminated gloves and basket liners must be discarded in a waste receptacle directly after exiting the bay and only fresh gloves and liners are to be brought into the next bay to be sampled.

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STANDARD OPERATING PROCEDURES

**OPERATION AND MAINTENANCE TECHNIQUES
FOR STATIONARY THRESHER**

SOP NUMBER: 5605

REVISION NUMBER: 3

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 2-13-13

Purpose and scope

This standard operating procedure is for use by research personnel to ensure accurate operation and maintenance of stationary threshers.

Thresher preparation and maintenance

Inspect the entire unit for cleanliness or broken or worn parts. Clean and/or repair the unit as necessary to ensure proper performance. If the unit is non-functional, contact U of I Project Manager to report the problem.

Never place hands inside of Thresher head while it is running.

Electric Powered Thresher

Before plugging machine in, inspect belts to ensure they are in working condition. Inspect thresher head for any blockage or old plant debris. Check grain catcher for cleanliness and proper placement. Plug in and start thresher and check for airflow through screen. If available, thresh small amount of untreated product to check for proper airflow speed. Too high of a speed and RAC will be blown out, too low speed and trash will gather with RAC.

Gasoline Powered Thresher

Inspect fuel level and oil level in engine prior to use. Before starting machine, inspect belts to ensure they are in working condition and inspect thresher head for any blockage or old plant debris. Check grain catcher for cleanliness and proper placement. Turn

switch to run and start motor with pull. Check for airflow through screen. If available, thresh small amount of untreated product to check for proper airflow speed. Too high of a speed and RAC will be blown out, too low speed and trash will gather with RAC.

Never place hands inside of Thresher head while it is running.

Operation

1. Start Machine.
2. Place product into header of thresher.
3. If product becomes stuck in header, push through with wooden plot stake.

Never place hands inside of Thresher head while it is running.

4. When grain catcher is near full, remove and place in specimen bag.
5. Follow SOP # 5505 and protocol for choosing which samples will be threshed.

Decontamination and Cleaning

Prior to use, thresher should be cleaned of all debris with special attention to any grain of the same type as the specimen to be gathered. Clean the thresher after each period of use. To clean thresher use either air or clean by running untreated grain through machine then discarding the threshed grain.

Each use and decontamination should be documented in the use and maintenance log if there is one available on borrowed equipment, or in the appropriate field data notebook.

CONTINGENCIES

If there is a problem with the thresher during the operation, stop immediately, turn off the thresher, and fix the problem. If problem cannot be fixed, try to secure another thresher from research farm. If this is not possible, note this in data book, secure proper running thresher as soon as possible and continue to thresh. Place all portions already threshed in freezer. Generally the problem consists of a belt breaking or machine not threshing properly. If a belt is the problem, replace or fix as necessary. If machine is leaving too much trash in with grain, motor speed can be increased or fan speed can be increased to assist in separation of seed from plant debris. If machine is blowing too much grain out with trash, reduce motor speed or fan speed. If operator is harmed by machine, seek medical attention if needed and report it to farm manager and FRD.

Never place hands inside of Thresher head while it is running.

If more than one day is necessary to thresh a single study due to equipment problems, contact study director for any deviation procedures.

In all cases the data should accurately reflect what occurred and what course(s) of action was taken to preserve the integrity of the study.

Documentation

Record all necessary information in enough detail to reconstruct the procedure, including the SOPs used, into the trial notebook. Remember to make the appropriate entries into the equipment use and maintenance log.

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STANDARD OPERATING PROCEDURES

HOP COLLECTION AND SAMPLE DRYING

SOP NUMBER: 5610

REVISION NUMBER: 5

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-06-14

Purpose and scope

This standard operating procedure is for use by research personnel to ensure uniform collection and drying of hop samples.

When drying hops for RAC samples, a pre-approved method must be used to determine the moisture content of the hops samples such as the Pre Sample Dry Down Method.

Pre Sample Dry Down Method

One to three days before collecting RAC samples, collect three separate samples of hop cones to be checked for moisture content. Each sample should weigh at least two pounds. These samples are to be dried to 0% moisture. Full dry down to 0% is determined by less than 2% weight change over an hour of drying. Enter data into the Hop Data Pre Samples sheet to determine original moisture content of hops. Using Hop sheet data, make calculations to determine the target weight of the required moisture content specified in the protocol for RAC samples. While measuring weights for pre samples, weights and times should be noted as an aid in timing for dry down of RAC samples on later days.

Hop Collection

Spread a clean tarp on the ground next to the hop hills to be harvested. Cut the bines from base of the hills that are to be harvested and pull the bines down from the overhead cable. Collect hop cones into a clean and labeled burlap bag, collecting from all portions of the bines (making sure to collect cones from the inside and outside portions of the

bine, and all along the length of the bine). When enough cones have been collected for each treatment, drive the cones back to the dryer.

Hop dry down procedure

Upon completion of hops harvesting, hops must be transported to drying facility. During transport keep all hops in appropriately labeled burlap or other suitable bags and place in transport vehicle in a manner to assure separation of treatments, avoiding possibility of cross contamination. When possible, place samples in some sort of storage container such as a large cooler or Rubbermaid type bin.

Once hops have arrived at drying facility, start drying process immediately when possible. If time specifications or scheduling demands require a delay, hops may be held in facility for up to 24 hours but must remain in their labeled sacks and must be in a storage container. After the dryer has been cleaned, place the cones from each sample into clean labeled burlap bags or appropriate drying apparatus that allow adequate air flow through the samples. When sample target weights are reached, record sample weights and calculate % sample moisture based on pre-samples. Place the dried cones in sample residue bags and record weight of samples and time of sampling in the FDN. Transport sample bags to freezers to await shipment to lab.

CONTINGENCIES

If there is a problem with the oven during the operation, turn off the oven, and fix the problem. If problem cannot be fixed, try to secure another oven from research farm.

In all cases the data should accurately reflect what occurred and what course(s) of action was taken to preserve the integrity of the study.

Documentation

Record all necessary information in enough detail to reconstruct the procedure into the trial notebook. Remember to make the appropriate entries into the equipment use and maintenance log.

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STANDARD OPERATING PROCEDURES

DRYING SAMPLES FOR HAY

SOP NUMBER: 5612

REVISION NUMBER: 1

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-9-17

Purpose and scope

This standard operating procedure is for use by research personnel to ensure uniform collection and drying of hay samples.

When drying a crop for hay RAC samples, a pre-approved method must be used to verify the moisture content of the hay samples such as the Parallel Sample Method.

Crop harvesting and dry down procedure

Upon completion of harvesting, the crop may be left in the field to dry or transported to an indoor location for air drying if permitted by the protocol. If transported, keep all samples in appropriately labeled bags or containers and place in transport vehicle in a manner to assure separation of treatments, avoiding possibility of cross contamination. When possible, place samples in some sort of storage container such as a large cooler or Rubbermaid type bin.

Once the samples have arrived at the facility, spread them out on a clean surface taking care to keep treatments separated to prevent contamination, and label each one. Allow the samples to air dry, checking them periodically until they feel like hay. Record the dates and times that drying started and ended, and environmental conditions during drying. When the samples feel like hay, collect the appropriate amount into the IR-4 sample bags, and record sample weights and time of collection. Transport sample bags to freezers to await shipment to lab.

Moisture Verification - Parallel Sample Method

After collecting RAC samples, collect three separate samples of hay from the same harvested lot to be checked for moisture content. Each sample should weigh at least 100g. Weigh the samples before placing them in the drying oven, and check the weight periodically during drying. These samples are to be dried to 0% moisture. Full dry down to 0% is determined by less than 2% weight change between measurements. Record the parallel sample weights, drying start time and end time, and temperature of the oven. Enter data into the Hay Parallel Samples sheet or equivalent to determine moisture content. Add this sheet to the FDN as a verification of the moisture content of the RAC samples.

CONTINGENCIES

If there is a problem with the oven during the operation, turn off the oven, and fix the problem. If problem cannot be fixed, try to secure another oven from research farm.

In all cases the data should accurately reflect what occurred and what course(s) of action was taken to preserve the integrity of the study.

Documentation

Record all necessary information in enough detail to reconstruct the procedure into the trial notebook. Remember to make the appropriate entries into the equipment use and maintenance log.

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STANDARD OPERATING PROCEDURES

BORROWED EQUIPMENT

SOP NUMBER: 5615

REVISION NUMBER: 1

AUTHOR OR EDITOR: Will Meeks

REVISION DATE: 3-10-12

Purpose and scope:

To describe the procedures used to acquire rented/borrowed equipment for use in GLP studies. It applies to all research trials using borrowed/rented equipment.

PROCEDURE:

1.0 Procurement Procedures

- 1.1 Contact the appropriate source for the equipment to be borrowed or rented
- 1.2 Determine whether there is an operation manual available for the equipment.
Request a copy of the manual when it is available

2.0 Documentation

2.1 The information must be documented for borrowed or rented field equipment.

2.11 Owner/Source

2.1.2 Description of equipment, e.g., type, make, and model

2.1.3 Year manufactured (when available)

2.1.4 Year acquired (when available)

- 2.1.5 Purpose
- 2.1.6 Study identification
- 2.1.7 Condition upon receipt e.g., good, needed repair
- 2.1.8 Maintenance preformed (when applicable)
- 2.1.9 Modifications required (when applicable)
- 2.1.10 Cleaning/decontamination procedures preformed
- 2.1.11 Research Director statement of suitability for use
- 2.1.12 Date of use
- 2.1.13 Time procedure initiated and completed
- 2.2 The person entering the information must initial and date entries.
- 2.3 The above information is maintained in the field notebook. A copy of the operations manual, when available, will also be kept with the raw data.