2021 STANDARD OPERATING PROCEDURES

for the

HAWAII IR-4 FIELD RESEARCH CENTER

in the

Department of Plant and Environmental Protection Sciences
University of Hawaii at Manoa
College of Tropical Agriculture & Human Resources
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University of Hawaii
College of Tropical Agriculture & Human Resources
Department of Plant and Environmental Protection Sciences

HAWAII IR-4 FIELD RESEARCH CENTER
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SOP Numbering Format: [SOP category]-[SOP number].[Revision number]

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SOP Numbering Format: [SOP category]-[SOP number].[Revision number]

SOPs Submitted and Reviewed by:

James Kam, FRD, University of Hawaii — 3/31/21 Date

Julie Coughlin, FRD, University of Hawaii — 3/31/21 Date

SOPs Approved by: S.R. Flanagan
Stephen Flanagan, Assistant Regional Field Coordinator — 4/5/21 Approval Date

SOP 1-1.3
STANDARD OPERATING PROCEDURES (SOPs)

1.0 PURPOSE

To provide guidelines and assure a uniform format for developing SOPs.

2.0 SCOPE

All SOPs involved in conducting studies that are required to comply with 40 CFR, Part 160, Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Format

a. *NUMBERING:* Use the following format for numbering SOPs: SOP #: U-W.X; where U = SOP category; where W = SOP number; and where X = SOP revision number (e.g., 1-1.0).

b. *TITLE:* State the title of the SOP.

c. *PURPOSE:* Briefly describe the purpose of the SOP.

d. *SCOPE:* State the extent to which the SOP is applicable.

e. *PROCEDURES:* Clearly describe the operating procedure so that a competent person with some knowledge of the situation can complete the task independently.

f. When SOPs are referenced within the text of an SOP, the revision number will be listed as ‘x’. The ‘x’ represents the current revision number.

3.2 Review and Approval

a. Program personnel will meet approximately annually to review SOPs and make necessary revisions and additions.

b. A revision is defined as a change in procedure. Formatting changes, spelling or grammatical corrections, and minor wording changes to clarify the intended meaning are not considered revisions.

c. Revisions made to the SOPs shall be submitted to the Research Director(s) and Management for approval. Approval will be documented by signatures on the index page of the SOPs.

d. The effective date, (i.e., the date that the SOP becomes effective), is the date when management approves the SOP. This effective date is located next to the management approval signature on the index page of the SOPs.

e. Retired SOPs shall be deleted from the SOP manual in the year that they are retired. Retired SOP titles will remain listed in the SOP Index with "(retired)" added at the end of the title.

3.3 Training and Distribution

a. New and revised SOPs shall be distributed in the following manner:

1. Program personnel will attend a training session to discuss changes made to the SOPs. Each person will sign in at the meeting to document attendance. The sign-up sheet will be retained in the SOP distribution file. Personnel unable to attend will receive training at a later date.

2. The training session will cover the following:

i. A new SOP manual will be distributed to program personnel.
ii. Old SOP manuals will be collected and discarded.
iii. New SOPs and changes to old SOPs will be discussed.

3. Julie Coughlin, or designee will be responsible for distribution of new SOPs and maintenance of the SOP distribution file.

SOP 1-2.2

PERSONNEL RECORDS

1.0 PURPOSE

To ensure that training records for all personnel are kept current.

2.0 SCOPE

All personnel involved in studies that are required to comply with 40 CFR, Part 160, Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Have on file a current curriculum vitae, training record, and job description of each person engaged in a study. An updated original curriculum vitae, training record, and job description shall be submitted approximately annually to IR-4 Headquarters. The Field Research Center will retain a copy of the personnel records in the Field Lab, St. John Room 006.

a. Temporary workers may occasionally be necessary for assistance with harvest, sample collection, or other aspects of a trial. 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.

3.2 Include the following information in training records:

a. Employee's name
b. Date
c. Description of experience and/or training
d. Employee's signature and/or initials
e. Documentation, if available, of training

3.3 Document the review of SOPs in training records.

3.4 At the termination of an employee's hire, retain copies of that employee's personnel and training records at the Field Research Center.

SOP 2-1.1

RECORDING RAW DATA

1.0 PURPOSE

To ensure that the raw data is collected and recorded accurately and that the data properly documents the results of the study.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160, Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Raw data is defined as any worksheets, records, correspondence, 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. Raw data can also be data and records, or exact copies thereof, received from a non-GLP source, such as weather data.

3.2 Collect and record all data required by the study protocol and various forms.

3.3 Record raw data entries in indelible ink.

3.4 Complete forms “as is” or as an electronic copy (i.e., on the computer) and print the “hard” copy as soon as possible. Complete forms with legible writing or in typewriting. Whether completing forms by hand or electronically, all entries should be dated and signed or initialed by the entering person at the time of entry to verify that the description or procedure was followed as described.

3.5 Accurately complete forms. Provide sufficient detail or an appropriate reference when describing data collection methods.

3.6 When making corrections, draw a single line through the mistake. Initial and date the correction, and provide a reason for the correction. Correction codes may be used. See IR-4 Field Data Book for correction codes.

3.7 The separation of data entered onto the same page on different dates should be as distinct as possible. Date and initial each day's entry.

3.8 Clearly identify notebooks, Field Data Books, equipment and other logbooks, data sheets, summaries, etc., with the project title or field ID number, dates data were generated, and other information that may be needed to identify the data and its source.

3.9 Date and sign or initial each filled or partially filled page. All unused pages should be lined out, dated, and initialed.

3.10 Initial and date all copies. Indicate that it is an exact copy of the original and note the location of the original.

3.11 All transcribed data shall be noted as such and the location of the original point of entry shall be specified.

SOP 2-2.0

DETERMINING SIGNIFICANT FIGURES AND ROUNDING NUMBERS

1.0 PURPOSE

To describe procedures for determining significant figures and rounding numbers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160, Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Data is defined as values obtained from measuring, monitoring, counting, or ranking. Record the entire number that was obtained to the appropriate level of accuracy.

3.2 Calculating

a. Perform all calculations with data using as many places as possible, but retaining at least one insignificant figure for intermediately calculated numbers.

b. Distinguish data from multiplication/dilution factors or other constants in equations. Do not use these numbers in determining significant figures.

c. Note datum with fewest significant figures.

3.3 Reporting Values

a. Report the final value with the same number of significant figures as the datum with the fewest significant figures.

b. The final value should be meaningful with respect to the number of significant figures used.

3.4 Rounding Numbers

a. If the last digit to be dropped off the number is <5, round down.

b. If the last digit to be dropped off the number is ≥5, round up.

SOP 2-3.1

TRANSFERRING IR-4 FIELD DATA BOOKS AND RAW DATA TO IR-4

1.0 PURPOSE

To ensure that IR-4 receives the Field Data Book, and that these records are placed in the archives.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Forward the original Field Data Book to Western Region IR-4 in a reasonable period of time after the study has been completed.

3.2 Retain a copy of the Field Data Book at the testing facility. Changed pages from Quality Control reviews are placed into the Field Data Book copy. The corresponding page is removed from the Field Data Book copy and discarded. These changed pages will be used for subsequent Quality Assurance responses. A current scanned copy of the Field Data Book can also be accessed on the Western Region IR-4 website.

3.3 At least one time per year, original raw data and other records pertaining to a study should be archived at IR-4 Headquarters. These types of data include, but are not limited to, raw data entries in equipment log books, personnel records and other original study raw data not included in the Field Data Book.

3.4 Photocopy original records being transferred to IR-4; retain photocopies at the testing facility.

3.5 Include a chain of custody form and an inventory form for raw data and other GLP records transferred to IR-4 Headquarters. Send copies of chain of custody and inventory forms to IR-4 Western Region office.

3.6 Send securely-packaged originals to the IR-4 Western Region office or IR-4 Headquarters via an air carrier that allows for tracking of packages (e.g., FedEx, UPS).

3.7 Notify the IR-4 Western Region office or IR-4 Headquarters that the package was sent.

SOP 3-1.1

SITE SELECTION

1.0 PURPOSE

To ensure that the site is appropriate for obtaining the required representative data and/or samples.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Select the site according to agriculturally acceptable conditions for the crop. Test sites may be located at University Experimental Stations or commercial production sites.

3.2 Consider the required number of replicates, buffer zones, and treatments according to the protocol when selecting the site.

3.3 If the commodity is not newly established, seek out locations where the stand is as uniform as possible.

3.4 Prepare a map showing the location of the study site in reference to the nearest city and show the north azimuth.

3.5 Try to find a site where field history (e.g., crops and pesticide use) is known. A 3-year field history is ideal, a 1-year field history is acceptable (if the protocol does not require a longer period).

SOP 3-2.0

PLOT LAYOUT REQUIREMENTS

1.0 PURPOSE

To ensure that plot size, buffer zones, and plot numbers are addressed and incorporated into the plot layout.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Determine minimum plot size from protocol requirements (e.g., number of plants) as well as the size and number of samples that are specified in the protocol.

3.2 Plots shall reflect commercial cropping conditions.

3.3 Make plots large enough to avoid sampling/harvesting borders.

3.4 Include buffer zones between plots to reduce the chance of contamination and to preserve the integrity of plots. If the dimensions of the buffer zones are not specified in the protocol, contact the Study Director.

3.5 When conducting residue studies, establish the prevailing wind direction of the study site. Lay out plots taking prevailing winds and slope of the land into consideration (e.g., control plot upwind and upslope).

3.6 Prepare a plot map showing the location of the plots in relation to a permanent reference point so that plots can be located after the study is terminated. Also, show the north azimuth.

3.7 Include in plot maps the distances and relative locations of other experimental plots treated with test chemicals.

3.8 Lay out plots using suitable measuring devices and markers to accurately measure plots.

3.9 Flag or stake both ends of each plot so that plots can easily be seen for the duration of the project.

3.10 Label each plot with the field identification number, treatment number, treatment name, test substance name and crop, and/or as required by the study protocol.

3.11 Post signs stating that the crop is for experimental purposes and not for human or animal consumption.

SOP 3-3.0

CROP ESTABLISHMENT, MAINTENANCE AND DESTRUCTION

1.0 PURPOSE

To ensure that the crop is established and maintained following good agricultural practices.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Monitor crop status as needed. Provide factors (e.g., fertilizer, irrigation, etc.) for optimum crop growth. Uniformly maintain the test area (e.g., borders, treated, and control plots).

3.2 For questions concerning crop production, current literature may be used for reference or a person familiar with the production practices may be consulted.

3.3 When field trials are conducted at commercial production sites, accepted grower practices will be considered as standard procedure unless it conflicts with the protocol. Crop establishment and maintenance will usually be the responsibility of the grower.

3.4 Document the use of maintenance pesticides and cultural practices in the Field Data Book.

3.5 When required, at the conclusion of the field study, crops treated with the test substance will be destroyed in a manner that will render the crop unusable (i.e., inedible and unmarketable). In most cases, commodities will be dropped to the ground or taken to a dump site.

SOP 3-4.1

COLLECTING PHYOTOXICITY DATA

1.0 PURPOSE

To describe procedures for recording phytotoxicity data.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Review the protocol to determine the method and timing of collecting phytotoxicity data. If no method is cited, follow the procedure below:

a. When possible, record phytotoxicity data at an appropriate time after the application

b. Observe all plots and rate the phytotoxicity on a scale of 0 to 100%. Zero percent = no phytotoxicity and 100% = completely dead. The rating between 0 and 100% indicates the degree of injury expressed as stunting, necrosis, chlorosis, leaf deformation, etc.

c. Document the use of other rating scales.

SOP 4-1.4

RECEIPT AND STORAGE OF TEST SUBSTANCE AND ADJUVANTS

1.0 PURPOSE

To ensure that test substances and adjuvants are properly received and stored.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Test Substance

a. Upon receipt of a test substance, unpack on the lab bench designated for test substances. Do not store application or sampling equipment on the lab bench designated for test substances.

b. After unpacking the test substance, record the required information in the Field Data Book test substance records form. Read the pesticide label and compare it to the requirements of the protocol to assure the correct test substance was sent.

c. Place original shipping waybill and characterization document(s) (if available) in the Field Data Book.

d. Read the test substance SDS.

e. Note any special conditions for storage and record.

f. Note any special incompatibilities (e.g., low pH incompatibility induced by CO_2 pressurization).

g. Before storing, ensure that all test substance containers are sealed securely and labeled with the following:

i. Name, CAS or code number

ii Batch or lot number
iii. Expiration date
iv. Storage requirements

h. Record the daily storage temperature of test substances (e.g., Hobo Temp) to ensure that the temperature is maintained within limits specified on the label, certificate of analysis, or SDS. Retain temperature charts in the appropriate log book.

i. If a test substance is exposed to temperatures outside the manufacturer's recommended storage range for a significant amount of time (i.e., 2 days or more), contact the Study Director as soon as practical for guidance.

3.2 Adjuvants

a. Upon receipt of an adjuvant, record the required information in the Field Data Book on the Identification and Receipt of Spray Additives form.

b. Before storing, ensure that the adjuvant container is sealed securely and labeled with the following:

i. Name
ii. Identity and concentration
iii. Storage conditions
iv. Date received or purchase date
v. Expiration date

If an expiration date is not available, the Field Research Director can assign one that does not exceed 5 years from purchase or receipt date.

c. Adjuvants should be in good condition prior to use. Adjuvants that appear to be compromised (i.e., change in color, consistency or smell) should not be used for GLP residue trials.

d. If a subsample is dispensed from the original container, the excess material poured into a temporary container should be discarded (i.e., not returned to the original container). Adjuvants must be handled in a manner to prevent cross contamination with test substances or other spray additives.

e. If secondary containers are used, they must be properly labeled per the original container. These requirements do not apply to temporary

containers used for measuring, but they should be adequately labeled to uniquely identify the product.

f. Document adjuvant handling procedures used to prevent cross contamination in the Field Data Book.

3.3 If test substance or adjuvant is shipped to another island for a field trial, in the original container or in secondary containers, a chain of custody form should accompany the shipment.

SOP 4-2.0

DETERMINING SPRAY VOLUME

1.0 PURPOSE

To determine the volume of spray required for the test plots.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Method 1 (volume area^{-1} basis), follow the procedure below:

a. Determine the gallons of spray to be applied per acre (gpa) according to the approved protocol.

b. Determine the plot area (ft^2) for each treatment replicate.

c. Calculate the gallons of spray needed for plots:

$$\text{gal of spray for plots} = \frac{\text{gpa} \times \text{plot area } (ft^2) \times \text{no. of plots}}{43560\ ft^2\ A^{-1}}$$

total volume = gal of spray for plots + overage*

*Overage = extra spray volume required to prime the spray system, and to avoid running out of spray in the test plots.

(20% overage is a reasonable starting point)

3.2 Method 2 (volume tree^{-1} basis), follow the procedure below:

a. Determine the gallons of spray to be applied per acre (gpa) according to the approved protocol.

b. Obtain commercial planting density (i.e., number of plants A^{-1}) for the tree crop.

c. Calculate:

$$\frac{\text{spray volume } A^{-1}}{\text{no. of plants } A^{-1}} = \text{spray volume plant}^{-1}$$

total volume = spray volume plant^{-1} × number of plants + overage*

*Overage = extra spray volume required to prime the spray system, and to avoid running out of spray in the test plots.

Use overage if needed.

3.3 For a liquid test substance and adjuvant, if the estimated amount of carrier displaced by the test substance and adjuvant combined is >1% of the spray volume, then subtract that amount from the calculated spray volume to obtain the amount of carrier. If the estimated amount of water displaced by the test substance and adjuvant is ≤1%, then subtracting that amount from the carrier is optional.

SOP 4-3.1

DETERMINING TEST SUBSTANCE AND ADJUVANT AMOUNTS FOR SPRAY MIX

1.0 PURPOSE

To determine the amount of test substance and adjuvant required to spray the test plots.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Method 1 (amount of product area^{-1}), follow the procedure below:

a. Determine the test substance rate in pounds active ingredient per acre (lb ai A^{-1}) according to the approved protocol.

b. Determine the plot spray volume in gallons per plot (gal plot^{-1}) following SOP 4-2.x.

c. Determine the gallons of spray to be applied per acre (gal A^{-1}) from the approved protocol.

d. Calculate the test substance amounts for the spray tank including overage following the procedure below:

Dry formulations

$$\text{Amount (lb) of test substance for plots} = \frac{\text{lb ai A}^{-1} \times \text{gal plot}^{-1} \times \text{no. of plots}}{\text{gal A}^{-1} \times \% \text{ ai in formulation}}$$

For % ai, use decimal form (e.g., 30% = 0.30)

Liquid formulations

$$\text{Amount (gal) of test substance for plots} = \frac{\text{lb ai } A^{-1} \times \text{gal plot}^{-1} \times \text{no. of plots}}{\text{gal } A^{-1} \times \text{lb ai gal}^{-1} \text{ in formulation}}$$

or

$$\text{Amount (lb) of test substance for plots} = \frac{\text{lb ai } A^{-1} \times \text{gal plot}^{-1} \times \text{no. of plots}}{\text{gal } A^{-1} \times \% \text{ ai in formulation}}$$

For % ai, use decimal form (e.g., 30% = 0.30)
(Note: This equation is suitable for liquid formulations because active ingredient percentages are also expressed on a w/w basis.)

Total amount of test substance = amount of test substance for plots + overage* for total spray volume.

*Overage = extra spray volume required to prime the spray system, and to avoid running out of spray in the test plots.

(20% overage is a reasonable starting point)

3.2 Method 2 (amount of product tree^{-1}), follow the procedure below:

a. Determine the amount of product to be applied per acre according to the approved protocol.

b. Obtain commercial planting density (i.e., number of plants A^{-1}) for the tree crop.

c. Calculate:

$$\frac{\text{amount of product } A^{-1}}{\text{no. of plants } A^{-1}} = \text{amount of product plant}^{-1}$$

d. Use overage if needed.

3.3 When using an adjuvant, follow the procedure below:

$$\text{Amount of adjuvant for spray mix} = \text{Total carrier volume (including overage)} \times \text{dilution rate (v/v) recommended on adjuvant label}$$

a. Choose an adjuvant rate that is within the label range, not the exact minimum or maximum amount.

SOP 4-4.0

MEASURING TEST SUBSTANCES

1.0 PURPOSE

To ensure that test substances are measured accurately.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Use appropriate PPE while handling test substances, per SDS and/or label.

3.2 Use one of the following methods to measure a liquid test substance:

a. Method 1, use a calibrated positive displacement pipettor if appropriate, otherwise follow Methods 2 or 3.

b. Method 2, use a graduated cylinder; follow the procedure below:

1. Use a clean, graduated cylinder large enough to hold the required volume of test substance. The graduations should be accurate within 1% of the total volume required (e.g., if 100 mL are required, the smallest division should be 1 mL or less).

2. If the opening of the graduated cylinder is too small to accommodate the pouring of the test substance from the original container, follow the procedure below:

i. Pour the test substance from the original container into a clean, glass container (e.g., beaker), then fill the graduated cylinder.

ii. Or, pour the test substance from the original container into the graduated cylinder using a clean glass or stainless steel funnel.

3. Fill the graduated cylinder with the test substance and read the volume at the bottom of the meniscus.

c. Method 3, weigh a liquid test substance; follow the procedure below:

1. Use a clean secondary container large enough to hold the required volume of test substance.

2. Use SOP 5-1.x to weigh a liquid test substance.

3.3 Follow SOP 5-1.x to weigh a solid test substance.

3.4 Maintain a log of the amount of test substance removed from the original container.

3.5 When practical, have another person double-check measurements.

SOP 4-5.1

TRANFERRING A TEST SUBSTANCE OR ADJUVANT FROM THE ORIGINAL CONTAINER TO SECONDARY CONTAINERS

1.0 PURPOSE

To ensure that when a test substance or adjuvant is transferred to secondary containers, that the secondary containers are suitable, properly labeled and stored.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 When necessary, transfer the test substance or adjuvant to secondary containers for field research to have pre-measured aliquots of test substance in the field.

3.2 Preferably use secondary containers that are made of dark or amber colored glass. Use containers large enough to accommodate expanding liquids and that are tightly fitted with teflon-lined caps.

3.3 Prepare and label test substance secondary containers with at least the following information:

a. Test substance trade name
b. Test substance common name
c. EPA registration number
d. Test substance batch or lot number
e. Caution, warning, or danger, if applicable
f. Field ID number
g. Application number
h. Treatment
i. Weight or volume of test substance
j. Storage requirements
k. Expiration date

3.4 Prepare and label adjuvant secondary containers with at least the following:

a. Name
b. Identity and concentration
c. Batch or lot number (if available)
d. Field ID number
e. Application number
f. Treatment
g. Volume of adjuvant
h. Storage conditions
i. Date received or purchase date
j. Expiration date

3.5 Calibrate the balance following SOP 5-1.x. If measuring liquids, calibrate the pipettor following SOP 5-3.x.

3.6 Measure the test substance or adjuvant into the secondary container.

3.7 Record the use of the test substance in the Field Data Book.

3.8 Store secondary containers filled with test substance or adjuvant in a pesticide storage area until they are to be used. Storage temperatures of all secondary containers should be monitored and recorded until use.

SOP 4-6.1

GENERAL PROCEDURES FOR MIXING A TEST SUBSTANCE

1.0 PURPOSE

To ensure that the test substance is properly mixed.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Restrict mixing of test substances to properly trained personnel (i.e., a certified applicator or personnel under the direct supervision of a certified applicator).

3.2 Method 1 (when using a pre-measured aliquot of test substance in a secondary container), follow the procedure below:

a. Add some carrier volume to container. Reserve sufficient carrier to rinse secondary test substance and adjuvant containers.

b. Add test substance to container (triple rinse the secondary container with reserved carrier and add to container).

c. If needed, make a slurry, then follow 3.2, b.

d. Mix test substance and carrier.

e. If using a spray adjuvant, add to the container. If using a pre-measured aliquot of adjuvant, triple rinse the secondary container with reserved carrier.

1. If the adjuvant label includes mixing instructions, follow the recommended order of mixing on the label, and document that in the Field Data Book.

f. Add the remaining volume of carrier to the container and mix.

g. Agitate the spray mixture before and during (when practical) the application to ensure that the test substance is thoroughly mixed.

3.3 Method 2 (when test substance is measured from the original container), follow the procedure below:

a. Add some carrier volume to container. Reserve sufficient carrier to rinse graduated cylinder, if using.

b. Measure test substance directly into container. If using a graduated cylinder to measure test substance, triple rinse the graduated cylinder with reserved carrier.

c. Mix test substance and carrier.

d. If using spray adjuvant, add directly to container. If using a graduated cylinder to measure adjuvant, triple rinse the graduated cylinder with reserved carrier.

e. Add remaining volume of carrier to container and mix.

f. Agitate the spray mixture before and during (when practical) the application to ensure that the test substance is thoroughly mixed.

3.4 Record the time of mixing and application of the test substance in the Field Data Book.

SOP 4-7.1

GENERAL PROCEDURES FOR APPLICATION OF TEST SUBSTANCES

1.0 PURPOSE

To ensure that test substances are applied uniformly to field plots.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Restrict application of test substances to trained personnel (i.e., a certified applicator or personnel under the direct supervision of a certified applicator).

3.2 Inspect and calibrate the equipment used in the application of test substances following the appropriate SOPs. Use clean application equipment.

3.3 Ensure that all settings of pressure, speed, granular flow, etc., are according to specifications from the calibration SOPs.

3.4 Apply the test substance spray to all reps within a treatment by ascending concentration (i.e., first, lowest concentration to all reps; then middle concentration to all reps; then, highest concentration to all reps).

3.5 Release the spray or granules just before the beginning of each plot. Maintain the correct speed through the plot.

3.6 Stop the spraying or spreading just after the end of the plot. Pass times should be taken and recorded in the Field Data Book.

3.7 Test substance application to plots shall be within -5% to +10% of the protocol specified rate. Notify the Study Director when the range is exceeded.

3.8 Do not discharge excess spray or granules immediately adjacent to experimental plots.

3.9 Do not use surfactants, adjuvants, stickers, etc., unless specified in the protocol or pre-approved by the Study Director.

3.10 In the event of sprayer malfunction, troubleshoot the problem and document what was done to correct it. If study integrity is jeopardized, contact the Study Director immediately for further instructions.

SOP 4-8.0

CLEANING TEST SUBSTANCE APPLICATION EQUIPMENT

1.0 PURPOSE

To ensure that test substance application equipment are clean.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Restrict cleanup of test substance application equipment to trained personnel (i.e., a certified applicator or personnel under the direct supervision of a certified applicator). Test substance application equipment includes all items that come into contact with the test substance.

3.2 Rinse equipment in areas that shall not contaminate water or aquatic environments. Rinse spray tanks thoroughly with water. For the granular applicator, at the end of each trial, wash with soapy water and rinse to remove test substance dust from the inside and outside.

3.3 When appropriate, use soap and water to clean equipment, and rinse.

SOP 5-1.2

CALIBRATION, USE, AND MAINTENANCE OF BALANCES

1.0 PURPOSE

To ensure that chemicals and other substances are weighed accurately.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with operating instructions.

3.2 Calibration

a. Calibrate balances using only standard weights that have been properly calibrated according to SOP 5-2.x.

b. Calibrate the balance prior to weighing substances. Follow one of the methods cited below.

1. Method A

i. Tare the weighing vessel.
ii. Weigh standard weight(s) to within ± 10% of the weight of substance(s) needed.
iii. Record results in balance log.

2. Method B

i. Tare the weighing vessel.
ii. Weigh standard weights that bracket the expected weight of the substance needed.
iii. Record results in balance log.

c. If the measured weight of the standard weight is > ±1% of the actual standard weight, the scale should not be used.

d. Tare the weighing vessel for Methods A and B. Weigh the chemical in/on the tared vessel.

e. A balance does not need to be calibrated prior to weighing samples if the balance is listed as non-GLP compliant on the GLP compliance statement.

3.3 Use

a. Use the appropriate safety equipment while handling substances.

b. Check to make sure the balance is level. To set the balance level, adjust the feet until the bubble is in the center of the level indicator.

c. Clean pan and housing as needed.

d. Follow operating instructions in the balance manual.

3.4 Maintenance

a. Approximately annually, have balance calibrated and serviced by an authorized service person.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer or an authorized service representative regarding repair or replacement.

3.5 Documentation

a. Designate the person responsible for each operation in the balance log.

b. Record in the balance log the maintenance and calibrating operations. Include dates of maintenance and calibrating operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the balance as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-2.0

CALIBRATION OF STANDARD WEIGHTS

1.0 PURPOSE

To ensure proper calibration and accurate measurements with standard weights.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Calibration

a. Standard weights used for checking the performance of balances must be checked for accuracy approximately annually.

b. Standard weights shall be checked for accuracy by checking weights immediately following annual calibration of the balances.

c. If the measured weight is within ±1%, the weight is considered calibrated. If the measured weight falls outside the 1% tolerance, send weight to a qualified weight calibration service company for calibration. A company is considered qualified if they test with reference standards traceable to the National Institute of Standards and Technology (NIST).

3.2 Documentation

a. Designate the person responsible for each operation in the standard weights log.

b. Record in the standard weights log the calibration operations. Include the dates of calibration and record the balance used for calibration. Retain calibration certificates in the standard weights log.

SOP 5-3.0

CALIBRATION, USE, AND MAINTENANCE OF AUTOMATIC PIPETTORS

1.0 PURPOSE

To ensure proper calibration and accurate measurements with automatic pipettors.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with pipettor operating instructions inside the automatic pipettor log book before using.

3.2 Calibration

a. Calibrate automatic pipettors prior to each day's use. Calibrate the balance following SOP 5-1.x using the milliliter amount (1 mL H_2O = 1 g) as the weight to be calibrated. Record balance calibration information in the balance log.

b. Use water for calibrating the pipettors.

c. Use the appropriate plastic tips for pipettors.

d. Ensure that the tip is seated correctly in the pipettor.

e. Dial in the exact milliliters of liquid to be calibrated.

f. Fill the pipette tip with the desired amount of water and dispense the water into the weighing vessel. Record the resulting weight in the pipettor log. If the measured weight is > ±5% of the expected weight, the pipettor should not be used.

g. Use clean, dry tips, correctly seated in the pipettor before measuring out the desired substance.

h. Pipettors used for non-critical measurements do not need to be calibrated prior to use.

3.3 Use

a. Choose the automatic pipettor with the correct range.

b. Follow operating instructions for filling and dispensing pipettor.

3.4 Maintenance

a. Clean automatic pipettors as needed.

b. In the event of failure or malfunction, consult the manual and/or return the pipettor to the manufacturer for repair or replacement.

3.5 Documentation

a. Designate the person responsible for each operation in the automatic pipettor log.

b. Record in the automatic pipettor log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the pipettor as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-4.1

CALIBRATION, USE, AND MAINTENANCE OF BOOM SPRAYERS

1.0 PURPOSE

To ensure accurate liquid spray application.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, and tanks for obvious wear and potential leaks. Replace or repair the deficiencies as needed.

3.2 Select appropriate nozzles according to the volume and spray pressure guidelines on the approved protocol; if not specified, select nozzles that will provide desired coverage.

3.3 When calibrating for a field trial, log all calibration data in the Field Data Book. Calibrate the boom sprayer prior to use.

3.4 The criteria for selection and use of nozzles are described below:

a. Determine whether all nozzles are discharging uniformly by spraying water through them at a given pressure. Prime the boom. Catch the water from each nozzle in separate containers for a set length of time. Variation of output among nozzles shall be less than ±5% of the mean of all nozzles. Nozzle calibration should be performed at least 3 consecutive times.

b. If a nozzle output is greater than ±5% of the mean of all nozzles, replace that nozzle, and recalibrate.

3.5 Calculation Method 1 (volume area^{-1} basis), follow the procedure below:

a. Calculate the spray swath (spacing × no. of nozzles, in ft) at the recommended nozzle height. Measure a sample plot length (e.g., 50 ft). Calculate the area to be sprayed (spray swath x plot length, in ft^2).

b. Record spray pressure (psi). Record and determine average total discharge of nozzles (mL sec^{-1}). Record comfortable walking speed or tractor speed (ft sec^{-1}) while operating the spray equipment.

c. Calculate the total volume applied per area:

($\mathbf{B}$ mL sec^{-1})($\mathbf{C}$ ft sec^{-1})$^{-1}$($\mathbf{D}$ ft [spray swath])$^{-1}$ = $\mathbf{B}(\mathbf{C}\ \mathbf{D})^{-1}$ mL ft^{-2}

Convert to gal A^{-1}:

(3785 mL gal^{-1})$^{-1}$(43560 ft^2 A^{-1})[$\mathbf{B}(\mathbf{C}\ \mathbf{D})^{-1}$ mL ft^{-2}]

d. Adjust the sprayer's speed and/or pressure for the desired gal A^{-1}.

e. If the desired mean gal A^{-1} calculated from three consecutive calibration runs is within ±5% of the calculated gal A^{-1}, consider the sprayer calibrated.

3.6 Method 2 (volume $tree^{-1}$ basis), follow the procedure below:

a. Obtain commercial planting density for the tree crop, (i.e., number of plants A^{-1}).

b. Calculate:

$$\frac{\text{spray volume } A^{-1}}{\text{no. of plants } A^{-1}} = \frac{\text{spray volume}}{\text{plant}}$$

c. Determine the appropriate time required to spray the calculated volume per plant with the nozzle, at the specified pressure.

d. Or, mix a batch for the entire treatment and remove aliquots for individual plants.

3.7 Speed Calibration

a. Prior to each spray application, perform a speed calibration.

b. Conduct the speed calibration in an area similar to the test plot or on similar terrain.

c. Perform the speed calibration at least 3 consecutive times.

d. The calculated mean speed from 3 runs should be within ±5% of the target speed.

3.8 Use

a. Be sure boom is clear of spray solution from previous use or calibration.

b. Assemble appropriate sprayer components (e.g., attach regulator to CO_2 tank, attach boom to spray handle, etc.).

c. Fill spray tank/bottle with spray solution.

d. If necessary, prime boom. Collect spray in beakers, or other suitable devices.

e. Spray plots.

f. If necessary, collect overage in the same beakers. Measure and record overage.

g. When finished, clean with soap and water, rinse with water.

3.9 Maintenance

a. Before each application, visually inspect sprayer components for obvious wear; replace or repair as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.10 Documentation

a. Record in the Sprayer Use, Calibration, Maintenance, and Repair Log, use, maintenance, and calibration operations. Include the dates of operation, whether maintenance operations were routine, and whether written standard operating procedures were followed. Also, record assembly, dismantling, and other pertinent information.

b. Record any non-routine maintenance (e.g., repairs) performed on a sprayer as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial actions taken.

c. Designate the person responsible for the specific sprayer in the Sprayer Use, Calibration, Maintenance, and Repair Log.

d. Start a new log for each sprayer assembled.

e. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-5.2

CALIBRATION, USE, AND MAINTENANCE OF BACKPACK MISTBLOWERS

1.0 PURPOSE

To ensure accurate test substance application using a backpack mistblower.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the manual and mistblower operation and safety precautions before operating the mistblower.

3.2 Calibration

a. Calibrate the backpack mistblower prior to use.

b. Because spray output varies with respect to nozzle position and blower speed, the applicator should determine the appropriate calibration method for each study. For example, the applicator may choose to calibrate by applying a known amount of water to a specified area of crop (ft^2) or crop unit(s) [e.g., trees]; or, to just spray out a known amount of water with the nozzle in a fixed position. The specific calibration method used should be documented in the Field Data Book.

c. Fill tank with a known amount of water.

d. Set nozzle to desired opening. (Attach special nozzle attachments if needed).

e. Start engine.

f. Set throttle to desired position.

g. Spray out water according to the calibration method determined in step b. above. Start timing when water first leaves nozzle.

h. Stop spraying after a determined time period; or, spray out entire volume and stop timing when spray is finished.

i. Calibration should be performed at least 3 consecutive times.

j. Determine mean discharge rate and perform appropriate calculations to uniformly apply spray solution to the plot(s). Walking speed, time (e.g., seconds) per tree, time per area (ft^2), time per linear feet, are some variables that may be calculated from the specified calibration method.

k. In some instances, discharge rates are checked only to determine whether the mistblower is working properly, and has no bearing on the actual application.

3.3 Speed Calibration

a. Prior to each spray application, perform a speed calibration.

b. Conduct the speed calibration in an area similar to the test plot or on similar terrain.

c. Perform the speed calibration at least 3 consecutive times.

d. The calculated mean speed from 3 runs should be within ±5% of the target speed.

3.4 Use

a. Wear appropriate personal protection equipment (PPE) while operating the backpack mistblower.

b. The spray solution should be mixed before placing in tank; make sure shut-off valve is closed at handle.

c. Start engine. Check throttle speed and dosage sleeve for correct setting. Rock sprayer to agitate spray solution in the tank.

d. Put mistblower on back, use appropriate safety gear, and move to plot area while agitating mixture.

e. Open shut-off valve and perform spray passes determined in calibration. If overage is used, measure and record after spray passes have been completed.

f. Rinse tank, lid, and lines with soapy water. Rinse tank completely with water until all soap is removed.

3.5 Maintenance

a. Approximately annually, maintain mistblower following manual instructions.

b. Before each use, visually inspect hoses, pipes, fittings, valves, and tank for obvious wear and potential leaks. Replace and repair if needed.

c. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.6 Documentation

a. Designate in the Mistblower Log the person responsible for the performance of each operation.

b. Record in the Mistblower Log the use, maintenance, and calibration operations. Include dates of the operation, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the mistblower as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-6.1

CALIBRATION, USE, AND MAINTENANCE OF GRANULAR APPLICATORS

1.0 PURPOSE

To ensure accurate granular test substance application.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the manual and operating instructions before operating applicator.

3.2 Calibration

a. Calibrate granular applicators prior to use. Follow the procedure stated below for the calibration method. Refer to the applicator manual for additional information.

1. Fill the applicator at least half full with a formulation blank or with the material to be applied. Attach a collector under the applicator to catch the material as it is released.

2. Determine the approximate setting of the openings and walking or traveling speed for the desired amount of product per acre.

3. Determine the swath of application in feet. The application swath should be approximately equal to the plot width.

4. Measure a sample plot length (e.g., 50 ft). Calculate the area to be treated (application swath × plot length, in ft^2).

5. Operate the spreader over the measured distance and collect the output in the collector. Weigh the material in the collector in lbs.

6. Calculate:

   Applicator swath = **B** ft
   Plot length = **C** ft
   Amount collected = **E** lbs

   Treated area = (**B** ft)(**C** ft) = **B C** ft^2

   Convert to lbs A^{-1}:

   (**E** lbs)(**B C** ft^2)$^{-1}$(43560 ft^2 A^{-1})

7. If the calculated lbs A^{-1} is within ±5% of the desired lbs A^{-1}, consider the applicator calibrated.

8. Calibration should be performed at least 3 consecutive times.

3.3 Speed Calibration

a. Prior to each spray application, perform a speed calibration.

b. Conduct the speed calibration in an area similar to the test plot or on similar terrain.

c. Perform the speed calibration at least 3 consecutive times.

d. The calculated mean speed from 3 runs should be within ±5% of the target speed.

3.4 Use

a. Wear appropriate safety gear while operating the granular applicator.

b. Follow operating instructions for the granular applicator.

c. When applying granular materials at relatively low application rates, the equipment may not be capable of delivering the material uniformly to small plots. An alternative in these cases is to apply the material by hand. Follow the procedure below.

1. Calculate the amount of material required for a plot (e.g., 10 g per 50 ft).

2. Divide the amount of material per plot into similar sub portions (e.g., 2 g per 10 ft).

3. Weigh out sub portions and distribute each lot over subplots (e.g., 2 g lots over 10 ft lengths of plot).

4. To facilitate spreading, the material may be diluted with an inert carrier, such as sand or formulation blank. Mix thoroughly before applying.

5. Use a hand shaker with holes modified for the size of the granules to distribute the granules.

3.5 Maintenance

a. Prior to each use, inspect the applicator for obvious wear and mechanical problems. Replace or repair deficiencies as needed. Make sure that the openings are not clogged.

b. Wipe the applicator following each application. At the end of each trial, wash the applicator with soap and water. Rinse thoroughly with water.

c. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.6 Documentation

a. Designate the person responsible for each operation in the granular applicator log.

b. Record in the granular applicator log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the granular applicator as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-7.0

CALIBRATION, USE, AND MAINTENANCE OF FLOW METERS

1.0 PURPOSE

To ensure accurate liquid measurements using a flow meter.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the manual and meter operation before operating flow meter.

3.2 Calibration

a. Calibrate flow meters prior to use when the equipment is being used for a GLP application.

b. Attach meter to water source.

c. Turn on water at a moderate rate. Allow flow meter to “zero”.

d. Measure out at least 2 gallons of water. Measure with a graduated cylinder and record in Flow Meter Log. Flow meter calibration should be performed at least 3 consecutive times. If result is within ±5%, flow meter is calibrated.

3.3 Use

a. Flow meter can be attached to hose and used to measure out larger volumes of water for tank mixtures.

b. Flow meter can be attached to orchard sprayer handgun and used to measure quantity of spray mixture applied to larger plots or trees.

c. Flow meter can be attached to irrigation lines to measure quantity of water applied to plots for irrigation.

d. If flow meter is used to spray pesticides, clean out meter by flowing water through it for 3-5 minutes. Allow meter to drain and dry. Wipe down and store.

3.4 Maintenance

a. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.5 Documentation

a. Designate in the Flow Meter Log the person responsible for the performance of each operation.

b. Record in the Flow Meter Log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the flow meter as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-8.3

CALIBRATION, USE AND MAINTENANCE OF ANEMOMETERS

1.0 PURPOSE

To ensure accurate measurements with anemometers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the anemometer's instructions in the anemometer log.

3.2 Anemometer Calibration:

a. Calibrate anemometers approximately annually.

b. To calibrate for wind speed, line up all of the anemometers next to each other on a level surface, aimed in the same direction. Conduct the calibration indoors with a fan directed toward the anemometers. Take a picture of all the anemometers concurrently and document each reading. Calibrate at 3 different wind speed ranges (approximately 1-4 mph, 5-8 mph, and 9-12 mph) by altering the distance from the fan that the anemometers are positioned.

c. Calibration tolerances:
   1) 1-4 mph, tolerance range = ±1 mph
   2) 5-8 mph, tolerance range = ±2 mph
   3) 9-12 mph, tolerance range = ±2 mph

   If the wind speed reading of an anemometer varies more than the tolerance range of the mean (within a specific range) of all the anemometers tested for any of the ranges tested, replace the anemometer.

3.3 Use:

a. Read and follow the instructions for the specific anemometer being used.

b. Select a position near the treatment area to best approximate wind speed while treating plots.

c. Determine the approximate wind direction from the way flags, clouds, vegetation, flagging tape, etc. are streaming.

d. Hold the unit with the vanes positioned perpendicular to the direction the wind is blowing.

e. Read the wind speed at this position.

3.4 Maintenance:

a. Follow the manufacturers' recommendations, if any, for routine maintenance of the anemometers.

b. Replace batteries as needed.

c. In the event of failure or malfunction, consult the manual regarding troubleshooting, or replace the anemometer, if it cannot be fixed.

3.5 Documentation:

a. Designate the person responsible for each operation in the anemometer log book.

b. Record in the anemometer log the maintenance operations. Include dates of maintenance operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on anemometers as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-9.2

CALIBRATION, USE, AND MAINTENANCE OF SLING PSYCHROMETERS

1.0 PURPOSE

To ensure proper calibration and accurate measurements with sling psychrometers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with sling psychrometer instructions in the sling psychrometer log.

3.2 Calibration for the sling psychrometer:

a. Calibrate the sling psychrometer approximately annually.

b. Calibrate the psychrometers at ambient temperature in the field lab.

c. Open the sling psychrometer and place it next to a NIST thermometer in a spot where the temperature is not likely to fluctuate.

d. Allow the temperature of the sling psychrometer and NIST thermometer to stabilize - usually about 15 minutes, or longer, depending on ambient temperature.

e. Compare the temperatures of the wet and dry bulb thermometers (sling psychrometer) to the NIST thermometer. Take a minimum of three readings at different time points.

f. If the wet bulb and/or the dry bulb thermometer vary significantly, (>±5°F) from the NIST thermometer, replace.

g. Record the temperature of the NIST thermometer and the sling psychrometer thermometer in the sling psychrometer log book.

3.3 Use:

a. Saturate the wick thoroughly with water by removing end cap and immersing the psychrometer body up to the mercury reservoir on the thermometers until the wick is thoroughly wetted.

b. Fill the end cap with water and replace.

c. Tighten the cap just enough to prevent leakage.

d. Ensure that the wick is wet and that it covers the mercury reservoir on the wet bulb thermometer.

e. Ensure that the reservoir on the other thermometer is dry.

f. Pull the tube clear of the body so the body can swivel.

g. Hold the tube, whirl the body two or three revolutions per second (120 to 180 rpm).

h. Continue to whirl until the temperatures stabilize (1.5 minutes is usually ample)

i. Immediately read the wet bulb thermometer and then the dry bulb thermometer.

j. Set the wet and the dry bulb temperatures opposite each other on the slide rule type calculator scales, sliding the body into the tube as required.

k. Read the percent relative humidity indicated by the arrowhead on the lower scale.

3.4 Maintenance:

a. Keep the wick clean. When dirty cut it off below the wet bulb thermometer and pull a clean section out of the end cap and slide it over the bulb on the wet bulb thermometer.

b. Order wick replacement kits from the manufacturer when necessary.

c. Refer to the instructions for other replacement parts.

d. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.5 Documentation:

a. Designate the person responsible for each operation in the sling psychrometer log.

b. Record in the sling psychrometer log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the sling psychrometer as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-11.2

CALIBRATION, USE, AND MAINTENANCE OF DIGITAL PROBE THERMOMETERS

1.0 PURPOSE

To ensure proper calibration and accurate measurements with digital probe thermometers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Calibration:

a. Approximately annually check the accuracy of digital probe thermometers.

b. Calibrate the digital probe thermometers under temperature conditions similar to that in which they will be used.

c. Place digital probe thermometer next to a NIST thermometer in a spot where the desired temperature will be reached. Allow temperature to stabilize—usually about 15 minutes.

d. Compare temperature of digital probe thermometer to NIST thermometer. Take a minimum of three readings at different time points.

e. If temperature varies significantly (>±5°F) from NIST thermometer, replace.

f. Record the calibration data in the temperature and humidity meters log book.

3.2 Use:

a. To take soil temperature, insert the probe in the soil near the treated area.

b. Ensure that the probe is immersed at least 2 inches into the soil.

c. Keep the probe clean.

3.3 Maintenance:

a. Replace batteries as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer for repair or replacement.

3.4 Documentation:

a. Designate the person responsible for each operation in the temperature and humidity meters log.

b. Record in the temperature and humidity meters log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the digital probe thermometer as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-12.2

CALIBRATION AND MAINTENANCE OF COMBINATION TEMPERATURE AND HUMIDITY METERS

1.0 PURPOSE

To ensure accurate measurements with combination temperature and humidity meters.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Calibration:

a. Approximately annually check the accuracy of combination temperature/humidity meters.

b. Temperature:

1. Calibrate the temperature meter under temperature conditions similar to that in which it will be used (i.e., ambient).

2. Place meter next to a NIST thermometer in a spot where the desired temperature will be reached.

3. Allow temperature to stabilize – usually about 15 minutes.

4. Compare temperature of meter to the NIST thermometer. Take a minimum of three readings at different time points.

5. If temperature varies significantly (>±5°F) from the NIST thermometer, replace.

c. Relative humidity:

1. Place meter in a spot where relative humidity is not likely to fluctuate.

2. Operate a calibrated sling psychrometer in the same area as the temperature/humidity meter, and record the relative humidity.

3. Compare the relative humidity of the meter to the sling psychrometer. Take a minimum of three readings at different time points.

4. If the relative humidity varies significantly (>±5%) from the sling psychrometer, replace the temperature/humidity meter.

d. Record the calibration data in the temperature and humidity meters log book.

3.2 Maintenance:

a. Replace batteries as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.3 Documentation:

a. Designate the person responsible for each operation in the temperature and humidity meters log.

b. Record in the temperature and humidity meters log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the temperature and humidity meter as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-13.2

CALIBRATION, USE, AND MAINTENANCE OF HOBO RECORDERS

1.0 PURPOSE

To ensure proper calibration and accurate measurements with Hobo temperature recorders.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the Hobo recorder manual in the Hobo temperature recorder log.

3.2 Calibration:

a. At minimum, calibrate recorders approximately annually.

b. Launch the Hobo recorder following the instructions in the manual. Select appropriate interval setting. Select the measurement unit that matches the units of the NIST thermometer (i.e., °C or °F).

c. Place the launched Hobo recorder next to the NIST thermometer under temperature conditions similar to that in which they will be used (e.g., room temperature, freezer, etc.).

d. Allow temperature of the Hobo and the NIST thermometer to stabilize. Compare temperature of the Hobo and the NIST thermometer. Take a minimum of three readings at different time points.

e. Record the temperature of the NIST thermometer at appropriate interval(s), in the temperature recorder log book.

f. Readout and print Hobo data following the instructions in the manual. Document in the temperature recorder log book.

g. If the temperature of the Hobo recorder varies significantly, (>±5°C) from the NIST thermometer, replace it.

3.3 Use:

a. Follow the operating instructions in the manual to launch, readout, and print output from the Hobo recorder.

b. The first printout is the original raw data. The person printing data from a Hobo recorder shall sign/initial and date the sheet(s).

3.4 Maintenance:

a. Replace the battery as needed.

b. In the event of failure or malfunction, contact the manufacturer regarding repair or replacement.

3.5 Documentation:

a. Designate the person responsible for each operation in the temperature recorder log.

b. Record in the Hobo temperature recorder log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above. Also, retain Hobo printed output in the Hobo temperature recorder log.

c. Record any non-routine repairs performed on the Hobo temperature recorder as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and /or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-14.1

CALIBRATION, USE, AND MAINTENANCE OF GPS RECEIVERS

1.0 PURPOSE

To ensure accurate measurements with GPS receivers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Calibration:

a. Approximately annually check the accuracy of the GPS receiver.

b. 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.

c. Record the waypoint of the known location and the waypoint displayed on the GPS unit in the GPS Log book.

d. Compare the waypoint recorded on the GPS receiver and the known waypoint for accuracy.

e. If the measured waypoint varies significantly from the known waypoint (>±50 ft, latitude or longitude), the GPS receiver should not be used.

3.2 Use

a. Keep the GPS unit in clear view of the sky while operating.

b. Follow the GPS receiver owner's manual and reference guide to mark waypoints and use other mapping features.

3.3 Maintenance:

a. Replace batteries as needed.

b. If the LCD display window needs cleaning, use a soft cloth dampened with tap water. Do not use chemicals.

c. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.4 Documentation:

a. Designate the person responsible for each operation in the GPS log.

b. Record in the GPS log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the GPS receiver as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-15.0

CALIBRATION, USE, AND MAINTENANCE OF NEW AND BORROWED EQUIPMENT

1.0 PURPOSE

To ensure proper calibration and accurate measurements with the use of borrowed equipment, or newly acquired equipment for which SOPs have not yet been written and approved.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with any available manuals pertaining to the use, calibration, and maintenance of borrowed equipment (e.g., sprayers, ovens, etc.)

3.2 Calibration:

a. Calibrate all borrowed equipment before use if appropriate for its intended use.

b. Follow the calibration instructions in the specific equipment manual, if available.

c. If no calibration instructions are available, follow the industry standards for calibrating a specific type of equipment.

d. Record the calibration method followed in the equipment log.

3.3 Use:

a. Prior to using any borrowed equipment, visually inspect the equipment to determine that it is working properly and its condition is appropriate for its intended use.

b. Make any necessary repairs or adjustments prior to using borrowed equipment in the study.

c. Be sure equipment is clean prior to use.

d. Be thoroughly familiar with the operation of the equipment.

e. Only use borrowed equipment when personal safety can be assured.

3.4 Maintenance:

a. Check with the owner of the equipment if any major maintenance or repairs are required.

b. Check any borrowed equipment at the time of use for any necessary maintenance (e.g., oil, gas, tire pressure, etc.) or repair.

c. If assistance is required because of failure or malfunction, consult the manual and/or the manufacturer or service personnel regarding repair or maintenance to borrowed equipment.

d. Document any routine maintenance or repairs performed by the Pesticide Registration Program (PRP) personnel (or any major maintenance or repairs occurring during the period of equipment use by PRP personnel) on any borrowed equipment in the equipment log book.

3.5 Documentation:

a. Designate the person responsible for each operation in the equipment log.

b. Record in the equipment log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on borrowed equipment as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-16.3

USE AND MAINTENANCE OF FREEZERS

1.0 PURPOSE

To ensure the integrity of materials stored in freezers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160, Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Use:

a. Use sample freezer RF-17 to store residue samples. Set freezer RF-17 temperature to -10°F (-23.3°C).

b. Use ultra-low temperature freezer FF-01 to freeze coffee extracts during coffee processing and to store blue ice used in sample collection. Set freezer FF-01 temperature to -50°C.

c. Record sample storage in RF-17 log book.

3.2 Maintenance:

a. Use a continuous temperature recording device (*e.g.*, Hobo) to log freezer temperatures. Use a backup temperature recording device in freezers. Temperature recorders will be downloaded and temperature charts printed approximately once per month. Backup temperature recorders will only be downloaded and printed if the primary temperature recorder fails.

b. Follow manufacturers' instructions regarding general maintenance of freezers (e.g., defrosting, vacuuming grills, etc.).

c. In the event of a mechanical failure of a freezer:

1. Check the temperature recorder to determine if damaging temperatures have occurred. Notify the Study Director and determine if the contents were damaged.
2. If contents are undamaged, transfer contents to another freezer immediately. The transfer must be made in such a way that none of the contents reach damaging temperatures during the transfer (i.e., use coolers if necessary to maintain cold temperature).

d. In the event of a power outage, take measures to ensure that a non-deviant temperature range is maintained (e.g., use dry ice or obtain a power generator). If a suitable temperature range cannot be maintained, follow the procedures in section 3.2.c. of this SOP.

3.3 Documentation:

a. Designate the person responsible for each operation in the freezer log.

b. Record maintenance in the freezer log. Include dates of maintenance operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above. Also, retain temperature charts in the freezer log.

c. Record any non-routine repairs performed on the freezers as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 5-17.1

USE AND MAINTENANCE OF SENSAPHONE MONITORING SYSTEM

1.0 PURPOSE

To ensure proper operation of the Sensaphone for freezer monitoring. Proper Sensaphone operation ensures that program personnel are notified quickly when alarm conditions exist in the field freezers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160, Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Use

a. Install the Sensaphone Model 1400 in the Field Freezers, RF-17 and FF-01.

b. Program the Sensaphone following manual instructions.

3.2 Alarm Response

a. In the event of an alarm, the Sensaphone will follow a dial-out sequence to alert program personnel. The dial-out sequence is listed in the Sensaphone log book.

b. When an alarm alert call is received, listen to the message. Enter the acknowledgement code to stop the dial-out process.

c. Monitor the alarm condition and if there is a risk of freezer contents reaching damaging temperatures, follow SOP 5-16.x, section 3.2 c-d.

d. Record the alarm event in the Sensaphone log book. Include a description of the alarm and any actions taken.

3.3 Maintenance

a. Approximately annually, test the Sensaphone system in each freezer by removing the probe from the freezer and allowing it to reach room temperature. Wait for the alarm alert phone call. If a call is not received, consult the manual to troubleshoot the problem. Record testing in the Sensaphone log book.

b. Test the Sensaphone back-up battery at approximately 6 month intervals. Follow testing instructions listed on page 103 of the Sensaphone manual. Replace the back-up battery if power is insufficient. Record battery testing in the Sensaphone log book.

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

3.4 Documentation

a. Designate the person responsible for each operation in the Sensaphone log.

b. Record maintenance in the Sensaphone log. Include dates of maintenance operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the Sensaphone as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

SOP 5-18.1

CALIBRATION AND USE OF NIST THERMOMETERS

1.0 PURPOSE

To ensure proper calibration and accurate measurement with NIST traceable thermometers.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160, Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Calibration

a. NIST thermometers used to calibrate temperature monitoring devices should be factory calibrated at set calibrations points that will cover the temperature ranges utilized by the monitoring devices.

b. Ensure that the calibrated NIST thermometers are traceable and include calibration documentation from the factory.

c. NIST thermometers shall be re-calibrated or replaced approximately every 2 years.

3.2 Use

a. NIST thermometers should be used to calibrate temperature monitoring devices at appropriate temperatures. Compare the NIST thermometer with the device to determine whether the device is sufficiently accurate. Specific ranges will be specified in the device SOPs.

3.3 Documentation

a. Designate the person responsible for each operation in the NIST Thermometer log.

b. Record NIST thermometer re-calibration and/or replacement in the NIST Thermometer Log.

c. Retain the NIST traceable calibration certificates for each NIST thermometer in the NIST thermometer log book.

SOP 6-1.3

RESIDUE SAMPLE COLLECTION AND IDENTIFICATION

1.0 PURPOSE

To describe how samples shall be collected and identified.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Review the study protocol to determine the specific dates for sample collection. If the dates are based on uncontrollable events (e.g., plant size, fruit maturity, etc.) then tentative dates shall be established and refined as necessary. The Quality Assurance Unit shall be informed if dates are changed. If the dates change by more than a month or so, also notify the IR-4 Western Region Office of the changes.

3.2 Review the study protocol to determine sample size and special sampling instructions for the crop.

3.3 Before sample collection, obtain sample bags (i.e., plastic-lined cloth bags). Use sample bags that are strong and relatively puncture resistant. Have an adequate amount of bags for all samples (having extra bags available would be desirable).

3.4 Store sample bags in a separate room from test substance storage cabinets and spray equipment.

3.5 Before sample collection, label the tag attached to the sample bag with information as required by the study protocol.

3.6 Write the Field ID number and Sample ID number directly on the sample bag using a permanent marker.

3.7 Collect samples by ascending test substance rates, from the zero (untreated) rate through the highest rate, unless treatments are sampled by different people.

3.8 Take special care to follow the procedures listed below:

a. Do not contaminate the samples or sample bags with the test substance under investigation during sampling, labeling, storage, and shipping.

b. Avoid collecting diseased, undersized samples, loose heads, unfilled pods, or abnormal samples.

c. Avoid removing surface residues during sampling, packing, and preparation.

d. Clean harvesting tools between samples with soap and water, rinse with clean water and wipe dry with clean absorbent material.

e. Change gloves or wash hands between samples.

f. Do not remove soil, plant parts, or trim the commodity unless specified in the protocol.

3.9 Carefully place the sample, as it is collected, in the sample bag marked for that sample. Samples may be collected directly into zip-top bags or equivalent if permitted by the protocol or Study Director.

3.10 Close the sample bag to prevent loss of the sample during handling, transportation, and storage. Evacuate as much excess air from the bag as possible.

3.11 Sample Weighing Procedure

a. Tare an empty sample bag on the balance.

b. Weigh the sample and record the weight in the field data book.

c. Samples may be weighed at the field site or in the field lab prior to placing into the freezer for storage.

3.12 Document the procedure used to sample the crop.

3.13 If the harvested crop will require postharvest processing to obtain samples, the crop may be collected in intermediate containers. Intermediate containers will be labeled at minimum with the Field ID and Sample ID. For example, coffee cherry can be harvested into labeled trash bags and then transferred to the processing facility, or pineapple fruit may be harvested and placed into cardboard boxes and then transferred to the processing facility.

SOP 6-2.3

HANDLING AND STORING RESIDUE SAMPLES

1.0 PURPOSE

To ensure sample integrity after collection in the field.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Place the sample bag in a container with ice substitute (e.g., "blue ice") to preserve the samples during transit from field to freezer. Do not place samples from different treatments in the same container. All ice substitutes and containers used to hold and transport samples shall be thoroughly cleaned prior to use.

3.2 Monitor transit temperatures using Hobo temperature recorders.

3.3 When sample collection is complete, remove samples from the field and place in storage as soon as practical.

3.4 Review the study protocol for the method, temperature, and maximum length of time for storage. Store samples following protocol specifications.

Untreated and treated samples may be stored in the same freezer if there is sufficient separation to prevent contamination (e.g. double-bagging samples, storing on separate shelves, etc.). Document the method used to separate samples.

3.5 Ship samples requiring postharvest processing (e.g., juicing, roasting, freeze drying, etc.) to the processor (as per prior arrangements) as soon as possible after collection. Make sure arrangements are made and assistance provided to have processed samples properly delivered to the analytical laboratory.

3.6 Record the daily storage temperature of the samples to ensure that the temperature is maintained within the limits specified in the study protocol.

3.7 Ensure that the freezer or room where the samples are stored is under lock and key.

3.8 Maintain a freezer log which includes at least the following information:

a. IR-4 project number and/or study ID number
b. Number of bags or boxes for the project
c. Sample list
d. Date and time “in” facility

3.9 Record the removal of samples in the freezer log with the following information:

a. IR-4 project number and/or study ID number
b. Number of bags or boxes removed
c. List of samples removed
d. Date and time “out” of facility

SOP 6-3.3

SAMPLE SHIPPING PROCEDURE

1.0 PURPOSE

To ensure that residue samples are shipped to the residue laboratory with a minimum loss of integrity.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 At least 1 week before sample shipment, contact the analytical laboratory to notify the chemist of the shipment date. Preferably, send air freight shipments on Monday or Tuesday to avoid weekend layovers.

3.2 Obtain clean insulated containers of sufficient size and quantity to hold the sample and dry ice (when required). The amount of dry ice should be sufficient to ensure that samples remain frozen during shipping. Pack containers immediately prior to shipment. Use containers that are strong enough to withstand normal handling in shipping and storage.

3.3 Complete the "Sample Chain of Custody Form" (see IR-4 Field Data Book). Place a copy of the "Sample Chain of Custody Form" in every container. Retain the original "Sample Chain of Custody Form" in the Field Data Book. Distribute copies of the "Sample Chain of Custody Form" as indicated on the form.

3.4 Label each shipping container with the following information:

a. Return name and address of the Study/Research Director or Field Investigator
b. Name and address of the analytical laboratory receiving the samples
c. Sample ID
d. Affix "Experimental Samples - - KEEP FROZEN" to the containers

3.5 Untreated and treated samples may be shipped in the same box if sufficient measures are taken to prevent contamination (e.g. at minimum double bagging samples or using another appropriate physical barrier).

3.6 Tape the lid of each container firmly in place.

3.7 On the day of the sample shipment, notify the lab with all pertinent shipping information.

SOP 7-1.1

PROCESSING FRESH COFFEE CHERRIES INTO COFFEE GREEN BEANS (RAC)

1.0 PURPOSE

To describe a method for processing fresh coffee cherries into coffee green beans for RAC and processing samples.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 On the day of harvest, starting with the untreated control samples, pulp and demucilage fresh coffee cherries following SOP 7-3.x to produce wet parchment coffee beans.

3.2 Place wet parchment coffee beans into clean, plastic, ventilated baskets and into a designated oven (Precision oven or equivalent).

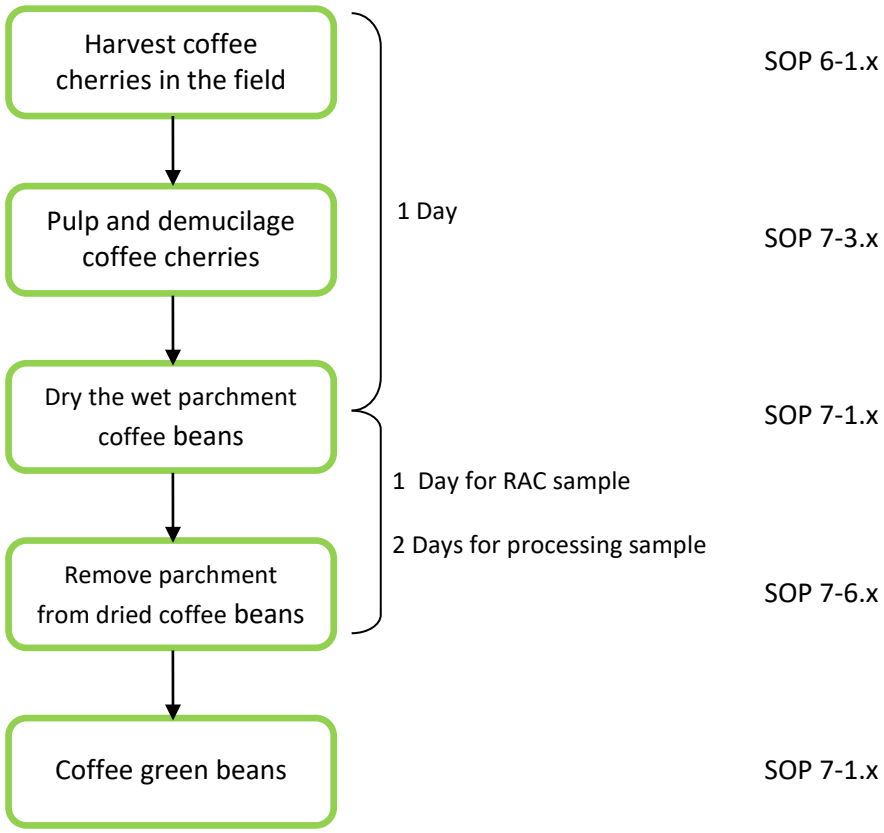
3.3 Dry the untreated control in a separate oven from the treated. Monitor oven temperatures during drying with a temperature recorder (*e.g*. Hobo). Oven drying conditions are approximately 50°C with air flow knob set at least at 7. Wet parchment coffee beans are dried until the moisture content, determined with a moisture meter, is approximately 12% or less. Follow SOP 7-5.x to determine percent moisture. (Note: Duration of drying will vary considerably with the amount of wet parchment coffee beans being dried in an oven at any given time. Drying may take from approximately 10 to 48 hours.) To measure the moisture content in parchment coffee, remove all of the parchment coffee in the oven, mix well, and take a sub-sample to fill the sample cup of the moisture meter. RAC samples are typically checked for moisture after approximately 10 hours of drying time, and processing samples after approximately 48 hours of drying time.

3.4 After drying, remove parchment from parchment coffee beans following SOP 7-6.x to produce coffee green beans.

3.5 All equipment (e.g., drying baskets, container used to mix coffee beans, etc.) should be cleaned with soap and water and rinsed between each sample.

3.6 Repeat 3.1 through 3.5 with the lowest treatment rate, and subsequently with higher rates in ascending order.

3.7 Flow chart for processing coffee cherries to coffee green beans for RAC and processing samples.



Field sample to coffee green beans sample (RAC) approximately 2 days per sample. Coffee green beans RAC sample size approximately 2.0 lbs.

Field sample to coffee green beans sample for processing approximately 3 days per sample. Coffee green beans sample size for processing approximately 15.0 lbs.

SOP 7-2.4

PROCESSING COFFEE GREEN BEANS INTO ROASTED COFFEE BEANS AND FREEZE DRIED COFFEE FRACTIONS

1.0 PURPOSE

To describe a method for processing coffee green beans into the roasted coffee beans and freeze-dried coffee fractions.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Subsampling coffee green beans

a. Start with the untreated control samples.

b. Remove the entire coffee green beans sample for processing (typically 15 lbs) from the lab freezer. Place entire sample into an appropriately-sized container and thoroughly mix (e.g., by gently shaking or stirring, bringing coffee green beans from the bottom of the container to the top several times).

c. From the container of thoroughly mixed coffee green beans, subsample the protocol specified amount (typically 1 or 2 lbs) for the coffee green beans "grab" sample which will be sent to the analytical laboratory for residue analysis.

3.2 Roasting coffee green beans

a. After the coffee green beans "grab" sample has been taken, roast the entire sample of coffee green beans received from the field phase.

b. Follow SOP 7-7.x to use the coffee roaster.

c. Follow these specific roasting conditions:

1. Roast coffee to light to medium roast.

2. Listen for the “1st pop”, i.e., when moisture quickly vaporizes from within the coffee green beans, and crackling sounds can be heard. The roasted coffee beans will subsequently puff up or expand. The coffee is roasted to the appropriate level after the 1st pop is completed. (Note: Typically, the roasting coffee beans will have two “popping” phases, the “2nd pop” signifying a medium to dark roast.)

d. Subsampling

1. After the entire sample of coffee green beans has been roasted, place the entire sample into an appropriately-sized container and thoroughly mix (e.g., by gently shaking or stirring, bringing the roasted coffee beans from the bottom of the container to the top several times).

2. From the container of thoroughly mixed roasted coffee beans, subsample the protocol-specified amount (typically 1 or 2 lbs) for the roasted coffee bean sample which will be sent to the analytical laboratory for residue analysis.

3.3 Grinding roasted coffee beans

a. Follow SOP 7-8.x to use the coffee grinder.

b. Follow these specific procedures for grinding the roasted coffee beans:

1. Subsampling

i. Obtain all the roasted coffee beans for a given sample, place all of the roasted coffee beans into an appropriately-sized container and thoroughly mix the sample by gently shaking and/or bringing roasted coffee beans from the bottom of the container to the top several times.

ii. Remove from the container and weigh only as much roasted coffee beans that are needed to produce the freeze-dried coffee sample.

iii. Typically, ~3200 g (2 batches of ~1600 g) of roasted coffee beans are needed for each sample.

2. Grind the subsampled roasted coffee beans in the coffee grinder at the FINE setting. Collect ground coffee in new paper bags.

c. Label the paper bags of ground coffee with the Field ID number, treatment number, and batch number. Place the paper bags into a new Ziploc bag, and then into the freezer. Hold the ground coffee in the freezer until extraction.

d. Place the remaining roasted coffee beans in an appropriately labeled sample bag, to include:

1. Extra roasted coffee beans
2. Field ID and/or study ID number
3. Common chemical name and formulation
4. Treatment number
5. Application rate (lb ai A^{-1})
6. Name of Processing Research Director
7. Processing (roasting) date

Place extra roasted coffee beans into a freezer for storage. Contact Study Director for approval to dispose of extra roasted coffee beans. Document disposal procedure in freezer log.

3.4 Clean all containers and utensils used in subsampling with soapy water and rinse between samples.

3.5 Extracting ground coffee

a. Place 10 L of tap water into a large pot, place pot on hot plate and bring water to a rolling boil.

b. Turn OFF hot plate, add ~1600 g ground coffee to the boiling water.

c. Stir mixture thoroughly and frequently (especially when adding the ground coffee), making sure that all the ground coffee is mixed into the water, and the mixture does not boil over.

d. Steep the mixture for at least 10 minutes, and until the ground coffee no longer floats at the surface.

e. Remove pot from hot plate.

3.6 Filtering coffee extract

a. Filter the brewed coffee mixture through a vacuum funnel using no. 4 filter paper (be sure to moisten the filter paper before adding the brewed coffee mixture to the vacuum funnel).

b. Before removing coffee grounds from the vacuum funnel, rinse grounds with ~500 mL tap water, allowing the filtrate to mix with the coffee extract already in the vacuum flask.

c. After all the brewed coffee mixture has been filtered, take a reading of the total dissolved solids (TDS) using the TDS meter (SOP 7-9.x). (Note: The value obtained is for reference only.)

d. Place the filtered coffee extract into a container large enough to hold the entire amount (<10 L), and cool the container in an ice water bath to bring the extract's temperature to ≤10°C.

e. Clean the large pot, utensils, vacuum funnel, and vacuum flasks used in the extraction process with soapy water and rinse between batches.

3.7 Freezing coffee extract

a. Obtain a freeze-dryer tray and place it on a shelf in an ultralow temperature freezer (VWR Ultralow Temperature 5702 or equivalent) set at approximately -50°C.

b. Add desired amount of coffee extract to the tray (maximum of ~2500 mL per tray).

c. Repeat for a total of 4 trays.

d. Freeze the extract overnight. (Note: Trays may be left in the freezer for longer periods, until ready to be freeze-dried.)

e. Clean the container and utensils used for the coffee extract with soapy water and rinse between batches.

3.8 Freeze-drying frozen coffee extract

a. Follow SOP 7-10.x to use the freeze-dryer.

b. Follow these specific procedures for freeze-drying the frozen coffee extract:

1. During set up, set shelf set point temperature to 0°C, and fully close the gas ballast on the vacuum pump (for maximum vacuum).

2. Load trays of frozen coffee extract onto freeze dryer shelves.

3. On the third day of freeze-drying, set the shelf set point temperature to 35°C.

4. Freeze-drying is complete when:

    i. V1 is approximately 150 mTorr or less.

5. Remove trays from freeze-dryer and feel the bottom of each tray for cool or cold spots, and if cool or cold spots are felt, immediately return trays to the freeze-dryer (using the existing shelf set point temperature) and continue to dry for up to an additional 24 h.

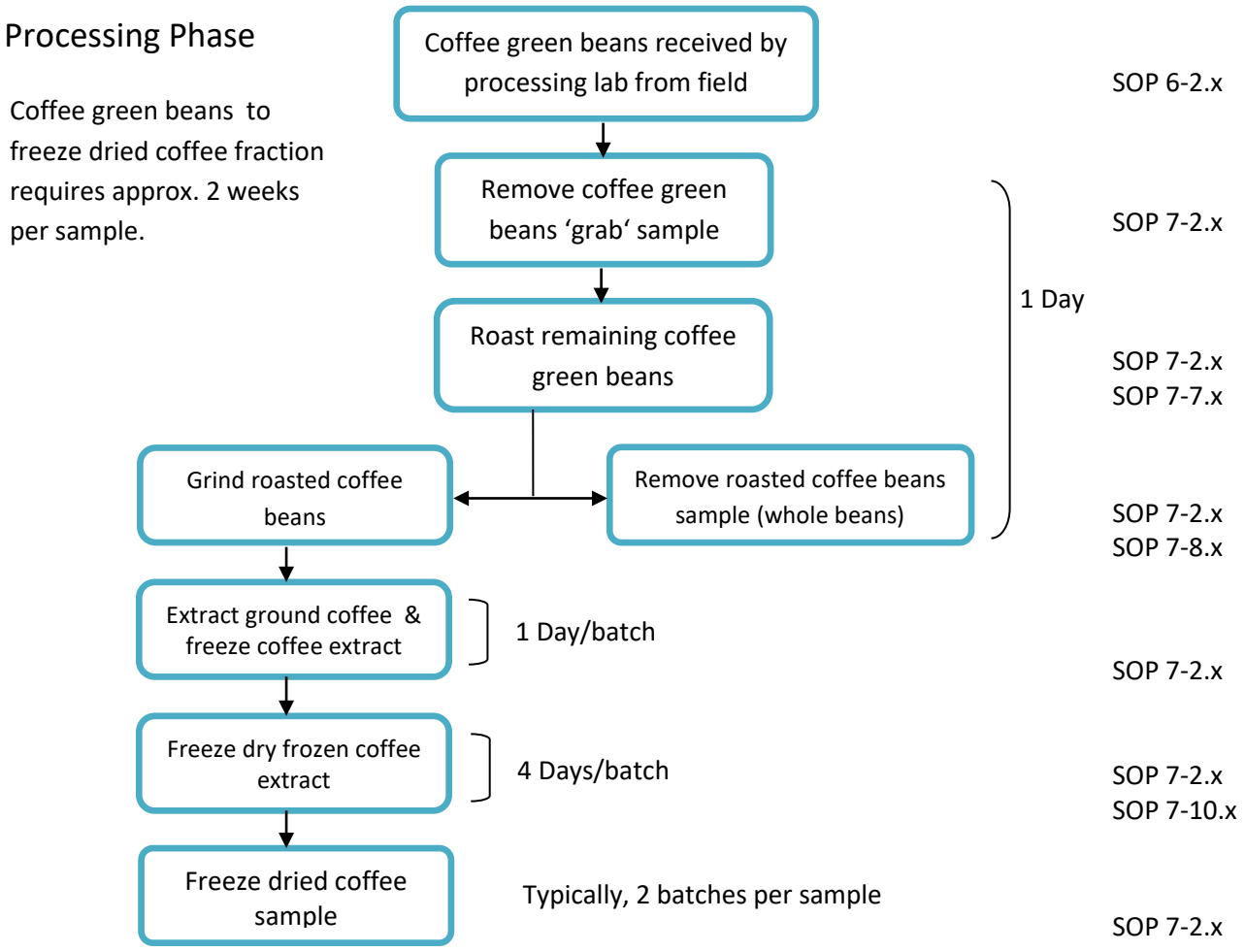
c. Carefully scoop freeze-dried coffee into a plastic bag . (Note: Freeze-dried coffee is hygroscopic, so it should be bagged immediately after removing trays from the freeze-dryer.)

d. Clean the freeze dryer trays and utensils with soapy water and rinse between batches.

e. Repeat steps 3.5 a. to 3.8 d. for the second batch of a given treatment.

3.9 Repeat steps 3.1 to 3.8 with the lowest treatment rate and subsequently with higher rates in ascending order.

3.10 Flowchart for processing coffee green beans to roasted coffee beans and freeze dried coffee fractions:



SOP 7-3.1

USE AND MAINTENANCE OF THE COFFEE PULPER-DEMUCILAGER

1.0 PURPOSE

To ensure the coffee pulper-demucilager is operating properly so coffee cherries may be processed into parchment coffee. Parchment coffee is the green seed of the coffee cherry after the soft tissue is removed and still retains a "paper-like" endocarp covering the seed.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the coffee pulper-demucilager (Penagos Ecoline 400 or equivalent) instructions in log book.

3.2 Calibration of the coffee pulper-demucilager is not necessary.

3.3 Visually inspect the coffee pulper-demucilager for obvious wear; and perform a brief test run (without coffee cherries). Replace or repair the deficiencies as needed.

3.4 Use:

a. Consult and follow the instructions for use.

b. Clean (wash down) the coffee pulper-demucilager between samples with dilute soapy water. Finish with a clean water rinse.

c. Hook up water and electricity. Turn on water and adjust water flow to the post-pulper chute such that pulped cherries flow freely into the demucilager.

d. Place coffee cherries into hopper of the Penagos Ecoline 400 pulper-demucilager, turn on pulper and demucilager motors. For RAC samples, place the entire sample into the hopper. For processing samples, place coffee cherries into the hopper in increments of approx. 30-35 lbs in a continuous loading process until the sample is all gone.

e. As pulped cherries fall into the demucilager, adjust water flow rate such that mucilage-water freely flows out of the demucilager.

f. The pulper motor can be turned off after all cherries of a sample have been pulped. But, continue to run the demucilager until the mucilage-water flowing out of the demucilager feels relatively free of mucilage (i.e., not too slimy). Turn off demucilager. Note: Do not run demucilager too long or it may result in a higher percentage of broken coffee green beans.

g. Remove wet parchment coffee beans from the demucilager.

3.5 Maintenance:

a. Visually inspect the coffee pulper-demucilager for obvious wear; and perform a brief test run (without coffee cherries). Replace or repair the deficiencies as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.6 Documentation:

a. Designate the person responsible for each operation in the coffee pulper-demucilager log.

b. Record in the coffee pulper-demucilager log, the use and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the coffee pulper-demucilager as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 7-4.1

CALIBRATION, USE, AND MAINTENANCE OF THE PRECISION OVEN

1.0 PURPOSE

To ensure proper calibration and accurate measurements with the Precision oven.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the Precision oven manual in the Precision oven log.

3.2 Calibration:

a. Any instrumental calibration to the Precision oven must be performed by trained service personnel.

b. Calibrate with a NIST thermometer approximately annually. To calibrate with a NIST thermometer, turn on the Precision oven and wait for it to reach the set temperature.

c. When the control panel indicates the set temperature has been reached, open the oven and place a NIST thermometer on the bottom shelf.

d. Wait 15 minutes, then open the oven and read the temperature of the NIST thermometer.

e. Record the set temperature and the NIST thermometer temperature in the Precision oven log book.

f. If the set temperature of the Precision oven differs significantly (>±5°C) from the NIST thermometer, call the manufacturer for service.

g. If oven is used for non-critical temperature, then calibration is not necessary.

3.3 Use:

a. Follow the instructions in the Precision Instruction Manual.

b. Keep the side diffuser panels clear when loading the chamber.

c. Keep the bottom of the chamber free and clear of objects.

d. Always use ventilated shelves.

e. Avoid spillage of liquids.

f. DO NOT evaporate noxious fumes.

g. DO NOT place sealed containers in the chamber.

3.4 Maintenance:

a. Record any maintenance in the Precision oven log book.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.5 Documentation:

a. Designate the person responsible for each operation in the Precision oven log.

b. Record in the Precision oven log the maintenance and calibration operations. Include dates of maintenance and calibration and describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the Precision oven as a result of failure and malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 7-5.1

CALIBRATION, USE, AND MAINTENANCE OF MOISTURE METERS

1.0 PURPOSE

To ensure accurate measurements with the moisture meter.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the moisture meter (Dickey John Multi-Grain Moisture Tester or equivalent) instructions in log book.

3.2 Calibration:

a. Calibrate the moisture meter approximately annually.

b. Follow the moisture meter's instruction manual (pp. 7-10) for calibration.

c. Verify calibration using the following method:

1. Weigh approximately a 300 g sample of parchment coffee dried to a commercially acceptable percent moisture (typically 9% to 12% moisture).

2. Measure percent moisture of the parchment coffee sample using the moisture meter. Three successive readings should be performed on the same sample and averaged.

3. Place parchment coffee into the oven set at 105°C for 24 hours.

4. Weigh the dried parchment coffee.

5. Calculate percent moisture in the parchment coffee:

$$\% \text{ Moisture} = (\text{Starting Weight} - \text{Dry Weight}) \times \text{Starting Weight}^{-1} \times 100$$

6. Compare calculated percent moisture with average reading from moisture meter (from Step 2).

7. If the average reading from the moisture meter varies by >±5percentage units from calculated value, repair or replace moisture meter.

d. Record calibration and verification in the moisture meter log book.

3.3 Use

a. Press the on key.

b. Select the appropriate crop for moisture testing.

c. Fill the sample cup of the moisture tester by pouring the grain/seed at a slow steady rate over the cone of the sample cup. (Display will indicate if the pouring rate is too fast.)

d. Continue pouring the grain/seed until a number begins to flash on the display. When the display stops blinking (less than 15 seconds), the temperature-compensated moisture reading is displayed.

3.4 Maintenance:

a. Visually inspect the moisture meter for obvious wear or damage. Replace or repair the deficiencies as needed.

b. Clean the sample cup (between samples) with a damp paper towel.

c. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.5 Documentation:

a. Designate the person responsible for each operation in the moisture meter log book.

b. Record in the moisture meter log the calibration and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the moisture meter as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

SOP 7-6.2

USE AND MAINTENANCE OF THE COFFEE HULLER

1.0 PURPOSE

To ensure the coffee huller is operating properly so parchment coffee may be processed into coffee green beans.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the coffee huller (Penagos K60 parchment coffee huller or equivalent) instructions in log book.

3.2 Calibration of the coffee huller is not necessary.

3.3 Visually inspect the coffee huller for obvious wear; and perform a brief test run (without parchment coffee beans). Replace or repair the deficiencies as needed.

3.4 Use:

a. Consult and follow the instructions for use.

b. If necessary, clean the coffee huller (appropriate internal and external parts) with dilute soapy water. Finish with a clean water rinse; then dry.

c. Hook up electricity. Check the placement of the two weights on the exit door of the huller; start with them all the way in (nearest to the door). Place a clean bucket at the exit door of the coffee huller to collect coffee green beans.

d. Make sure the hopper door is closed. Place parchment coffee beans into hopper, turn on the huller, slowly open hopper door.

e. As parchment coffee beans make their way into and out of the huller, adjust the parchment coffee bean inflow such that the ammeter needle operates at approximately the targeted (marked) line.

f. Also, if there are a lot of unhulled coffee beans as the coffee green beans exit the hulling chamber, adjust the weights further away from the door, such that the parchment coffee remains in the chamber longer to assure complete removal of the parchment.

g. Unhulled coffee beans may be sorted out from the green beans and returned to the hopper.

h. Clean the coffee huller between treatments. Clean appropriate internal and external parts with dilute soapy water. Finish with a clean water rinse; then dry.

3.5 Maintenance:

a. Visually inspect the coffee huller for obvious wear; and perform a brief test run (without parchment coffee beans). Replace or repair the deficiencies as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.6 Documentation:

a. Designate the person responsible for each operation in the coffee huller log.

b. Record in the coffee huller log, the use and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the coffee huller as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records

may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 7-7.4

USE AND MAINTENANCE OF THE COFFEE ROASTER

1.0 PURPOSE

To ensure the coffee roaster is operating properly so coffee green beans may be processed into the roasted fraction.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the coffee roaster (1 kg custom-made roaster or equivalent) instructions located in the log book.

3.2 Calibration of the coffee roaster is not necessary.

3.3 Visually inspect the coffee roaster for obvious wear; and perform a brief test run (without coffee green beans). Replace or repair the deficiencies as needed.

3.4 Use:

a. Before use, make sure roaster is clean. If necessary, clean following step 3.4 h

b. Set up the coffee roaster in a fume hood and hook up electricity.

c. Load coffee green beans (up to 1 kg per run) into the roasting basket. Attach the roasting basket to the motor.

d. Turn on roaster motor and heat elements. Coffee bean temperature can be monitored during roasting using an infrared thermometer. Temperature readings are for reference only and do not need to be recorded.

e. When the desired level of roast (e.g., light, medium, dark) is achieved, turn off heat elements and open roasting box top. Leave motor on to continue rotating the roasting basket to cool coffee beans. Alternatively, transfer roasting basket to second motor and rotate basket until beans are cool.

f. When roasted coffee beans are cool, turn off motor, remove roasting basket, and dump roasted beans into a clean container.

g. Continue to roast coffee green beans until all coffee green beans in the sample are roasted.

h. When done, cool roaster completely. Clean roaster between samples as follows:

1. Vacuum the chaff tray at the bottom of the roaster box, and wipe with a damp paper towel.

2. Wash the coffee loading container with soapy water, rinse and dry.

3. Wipe roasting basket with a paper towel soaked in solvent (e.g., ethanol) to remove oil residue.

4. Clean roasting basket with soapy water, rinse, and dry. Basket can be placed into a drying oven briefly to speed the drying process.

i. Repeat steps 3.4 b to 3.4 h with the lowest treatment rate and subsequently with higher rates in ascending order.

3.5 Maintenance:

a. Visually inspect the coffee roaster for obvious wear; and perform a brief test run (without green coffee beans). Replace or repair the deficiencies as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.6 Documentation:

a. Designate the person responsible for each operation in the coffee roaster log.

b. Record in the coffee roaster log, the use and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the coffee roaster as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 7-8.2

USE AND MAINTENANCE OF THE COFFEE GRINDER

1.0 PURPOSE

To ensure the coffee grinder is operating properly so coffee samples may be ground appropriately.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the coffee grinder (Bunn-O-Matic G3 or equivalent) instructions in log book.

3.2 Calibration of the coffee grinder is not necessary.

3.3 Before use, visually inspect the coffee grinder for obvious wear; and perform a brief test run (without roasted coffee beans). Replace or repair the deficiencies as needed.

3.4 Use:

a. Consult and follow the instructions for use.

b. If necessary, clean the coffee grinder following steps 3.4 g.- 3.4 h.

c. Hook up electricity. Place roasted coffee beans into hopper.

d. Set the desired fineness of grind.

e. Place collection bag or equivalent under exit chute.

f. Turn on grinder.

g. When done grinding, clean the coffee grinder with paper towels, a sponge, brushes or equivalent, dampened with dilute soapy water. Finish wiping with paper towels, a sponge or equivalent, dampened with clean water. Removable parts may be washed with dilute soapy water and rinsed with clean water.

h. Reassemble coffee grinder and adjust according to instruction manual.

3.5 Maintenance:

a. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.6 Documentation:

a. Designate the person responsible for each operation in the coffee grinder log.

b. Record in the coffee grinder log, the use and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the coffee grinder as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 7-9.0

CALIBRATION, USE, AND MAINTENANCE OF TDS (TOTAL DISSOLVED SOLIDS) METERS

1.0 PURPOSE

To ensure accurate measurements with the TDS meter.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the TDS meter (TDSTestr11 or equivalent) instructions in log book.

3.2 Calibration:

a. Calibrate the TDS meter approximately annually.

b. Follow the TDS meter's instruction for manual calibration.

c. The temperature reading on the TDS meter can be standardized to an accurate (standardized) thermometer per instruction manual.

d. Record calibration data in the TDS meter log book.

3.3 Use:

a. Turn TDS meter on by depressing on/off button.

b. Leave TDS meter in auto-range mode, unless it is necessary to select a specific range to measure. If a specific range is needed, follow instruction manual in log book.

c. Remove protective cap from electrode end of TDS meter.

d. Immerse electrode end of TDS meter into liquid to be measured, stir to release any trapped air bubbles from the electrode and allow reading to stabilize.

e. The upper reading on the display is the TDS, the lower reading is the temperature of the solution. (The TDS reading is automatically compensated to the normalized temperature of 25°C.)

3.4 Maintenance:

a. Visually inspect the TDS meter for obvious wear or damage. Replace or repair the deficiencies as needed.

b. Clean an excessively oily or dirty electrode by soaking the electrode in alcohol for 10 to 15 minutes, then rinse with water (preferably deionized). Otherwise, rinse with dilute soapy water, then rinse with water (preferably deionized). Wipe dry before replacing the protective cap.

c. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.5 Documentation:

a. Designate the person responsible for each operation in the TDS meter log book.

b. Record in the TDS meter log the calibration and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the TDS meter as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 7-10.3

USE AND MAINTENANCE OF THE FREEZE DRYER

1.0 PURPOSE

To ensure the freeze dryer is operating properly so samples may be processed into the freeze dried fraction.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the freeze dryer (Virtis Genesis 25LL or equivalent) instructions located in log book.

3.2 Calibration of the freeze dryer is not necessary.

3.3 Visually inspect the freeze dryer for obvious wear. Replace or repair the deficiencies as needed.

3.4 Use:

a. Consult and follow the instructions for use.

b. Before use, make sure that freeze dryer is clean. If necessary, clean following step 3.4 r.

c. Set up freeze dryer:

1. Hook up electricity
2. Close doors to freeze drying chamber and condenser
3. Check vacuum pump, make sure it has oil filled to the appropriate level, vacuum hoses are hooked up, and pump is plugged in.
4. Make sure all switches on the front panel are off.

d. Turn on power.

e. Turn on condenser.

f. Turn on vacuum pump for approximately 10 seconds, then turn off vacuum pump.

g. Set the shelf set point (i.e., the targeted) temperature to the desired shelf temperature; then turn shelf on and heat on. Observe shelf temperature readout to determine if the set point temperature of the shelf is obtained and maintained. (Note: The set point temperature may be adjusted over a wide range (e.g., -30 to 30°C or wider), depending on the matrix being freeze-dried and how much volume is being freeze-dried.

h. When the shelf temperature is relatively stable at the set point, the freeze dryer is ready to be loaded. Turn on release to release vacuum; when vacuum has dissipated, open door to sample chamber (do not force open) and load samples. Close sample chamber door.

i. Turn off release.

j. Turn on vacuum pump.

k. Depending on the sample size and type, it may be necessary to adjust the following at the beginning of or during the run:

    1. Gas ballast on the vacuum pump, and/or
    2. Shelf set point temperature

l. To remove freeze-dried samples, turn off shelf, vacuum, and heat; then, turn on release.

m. When vacuum has dissipated, open door to sample chamber (do not force open) and unload samples. Turn off release.

n. If there is another set of samples to freeze-dry, repeat from step h. Clean freeze drying and condenser chambers between each sample following step r.

o. To shut down freeze-dryer after having removed samples:

    1. Turn off all remaining switches, except power; and if necessary,
    2. turn on defrost; or,
    3. turn off all remaining switches, including power, and defrost at ambient temperature.

p. When condenser is defrosted, drain water and/or remove ice.

q. Turn off all switches if not freeze-drying more samples.

r. Clean the freeze drying and condenser chambers with paper towels, a sponge or equivalent, dampened with dilute soapy water. Finish wiping with paper towels, a sponge or equivalent, dampened with clean water.

s. Change vacuum pump oil when necessary.

3.5 Maintenance:

a. Visually inspect the freeze-dryer for obvious wear. Replace or repair the deficiencies as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.6 Documentation:

a. Designate the person responsible for each operation in the freeze dryer log.

b. Record in the freeze-dryer log, the use and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the freeze-dryer as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.

SOP 8-1.4

PROCESSING FRESH PINEAPPLE FRUIT INTO PINEAPPLE JUICE AND PROCESS RESIDUE

1.0 PURPOSE

To describe a method for processing fresh pineapple fruit into the pineapple juice and process residue sample fractions.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Clean all processing equipment (*e.g.*, cutting board, knife, fruit scraper, containers, juicer, food chopper, *etc.*) with soap and water and rinse thoroughly before use and between each sample.

3.2 Start with the untreated control samples. Fresh fruit samples consist of whole fruit without crowns.

3.3 Cut both ends off whole fruit, and place into a container. These will be referred to as end cuts.

3.4 Peel fruit using a knife. Scrape flesh from peels. Retain scraped flesh and peels in separate containers.

3.5 JUICE PROCESS

a. Juice peeled fruit, scraped flesh and end cuts in a commercial juicer (Waring Juicer or equivalent). If needed, fruit and end cuts may be cut into smaller pieces to fit in the juicer. Follow SOP 8-2.1 for use of the Waring Juicer

b. Collect juice and pulp in separate containers. Set pulp aside.

c. Add antifoam agent to juice (KFO™ 305 Silicon Defoamer or equivalent). Stir to mix. Use an antifoam rate that is comparable to commercial practice. For KFO 305 Silicone defoamer, use a rate of 30 ppm.

d. Heat juice in a water bath to 88°C. Hold at 88°C for 30 seconds.

e. Cool juice to 8°C in ice water bath.

f. Stir juice with a large paddle to mix thoroughly. From the container of thoroughly mixed juice, subsample the protocol specified amount for the juice sample (typically 1000 to 2000 mL). Pour juice sample into multiple, labeled containers as specified in the protocol. Place containers into a labeled IR-4 sample bag.

g. Place juice sample into field sample freezer.

3.6 PROCESS RESIDUE

a. Chop retained peels to a coarse consistency in a food cutter. (Robot Coupe or equivalent). Place chopped peels into a container.

b. Add pulp left over from juicing to the chopped peels.

c. Stir together using a large spoon to mix thoroughly.

d. From the container of thoroughly mixed process residue, subsample the protocol specified amount (typically 2 to 4 lbs) for the process residue sample. Place the process residue sample into labeled, plastic Ziploc bag(s) (or equivalent). Place Ziploc bag(s) into a labeled IR-4 sample bag.

e. Place process residue sample into field sample freezer.

3.7 Repeat 3.3 through 3.6 with the lowest treatment rate, and subsequently with higher rates in ascending order.

3.8 Store processed samples in the field sample freezer until shipment to the residue lab.

SOP 8-2.1

USE AND MAINTENANCE OF THE WARING JUICER

1.0 PURPOSE

To ensure the pineapple juicer is operating properly so fresh pineapple fruit may be processed into pineapple juice.

2.0 SCOPE

All studies that are required to comply with 40 CFR, Part 160 Good Laboratory Practice Standards.

3.0 PROCEDURES

3.1 Be familiar with the juicer instructions (Waring Commercial Juice Extractor, model WJX50 or equivalent).

3.2 Calibration of the juicer is not necessary.

3.3 Use:

a. Follow instruction manual for use.

b. Before use, make sure that juicer is clean. If necessary, clean following step 3.3 i.

c. Cut fruit into pieces sized to fit in the juicer feed chute.

d. Plug in juicer. Select desired power setting (LO=6,500 rpm, HI=13,000 rpm). Turn power on.

d. Feed fruit into juicer feed chute. Using the food pusher, slowly push the fruit down the chute.

e. The juicer is equipped with a pulp separator. After feeding fruit down the chute, the juice will flow into a juice cup and the separated pulp will accumulate in a pulp container.

g. Collect juice in the juice collector cup. When collector cup is full, empty juice into a large clean pot.

h. Collect pulp in the pulp container. When pulp container is full, empty pulp into a large clean container. Reserve pulp for "processed residue" fraction.

i. When finished juicing, turn power off and unplug juicer. Clean all removable parts with soap and water and rinse thoroughly with clean water. If necessary, use a nylon cleaning brush to remove fiber from the filter basket. Use clean paper towels to dry, or allow to air dry. Wipe motor base with a damp paper towel. Juicer should be cleaned between samples.

j. Reassemble juicer according to instruction manual.

3.4 Maintenance:

a. Visually inspect the juicer for obvious wear. Replace or repair the deficiencies as needed.

b. In the event of failure or malfunction, consult the manual and/or contact the manufacturer regarding repair or replacement.

3.5 Documentation:

a. Designate the person responsible for each operation in the juicer log.

b. Record in the juicer log, the use and maintenance operations. Include the dates of the operations, describe whether the maintenance operations were routine and followed the written standard operating procedures above.

c. Record any non-routine repairs performed on the juicer as a result of failure or malfunction. Document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

d. Because this equipment may be shared with personnel outside this program, some entries in the equipment log book and/or other records may not be GLP compliant. All equipment documentation relevant to IR-4 studies will be GLP compliant.