Appendix C:

Air Sampling Field Standard Operating Procedures

Appendix C Documents:

C1. SOP1: Sample Apparatus

SOP 2: Withdrawn

C2. SOP 3: Perimeter Sample Collection

C3. SOP 3A: Perimeter Sample Collection Addendum

C4. SOP 4: Receptor & Ambient Sample Collection

C5. SOP 4A: Receptor & Ambient Sample Collection Addendum

C6. SOP 5: Labeling

C7. SOP 6: DryCal Calibration in Laboratory

C8. SOP 7: Rotameter Calibration in Laboratory

C9. SOP 8: Chain of Custody

Pilot OP Pesticide Air Monitoring Project
Standard Operating Procedures
SOP 1: Sampling Apparatus Set-up

1.1 Objective

The objective of SOP1: Sampling Apparatus Set-up is to develop an appropriate and consistent manner to set up the equipment to collect perimeter, receptor, and ambient air samples. Modified from: *Ambient Concentrations of Organophosphorus Pesticides Caused by Volatization During Seasonal Pesticide Application, appendix A, attachment 2: sampling protocol* Master's Thesis. Lisa A. Tolbert 28 May 2007

1.2 Materials

Sampling mast materials, for each mast

- ☐ Concrete blocks with center hole (8 in x 8 inx 8 in)
- ☐ 4 ft metal stake (not used with locations that are on cement or asphalt)
- ☐ 12 in x 1 in galvanized steel pipe
- ☐ 1.5 in PVC pipes (12 inches long, 2 pieces)
- ☐ 1.5 in PVC pipe (60 inches long)
- ☐ Galvanized steel coupling piece
- ☐ 3 ft x 1 in galvanized steel pipe
- ☐ Hose clamp (small)
- ☐ Hammer
- ☐ Plastic rain gutters (~ 24 inches long)

Sample Train Materials

Perimeter	Receptor and Ambient
☐ OVS XAD-2, quartz filter sorbent tubes (SKC Cat #226-58)	☐ OVS XAD-2, quartz filter sorbent tubes (SKC Cat #226-58)
☐ Tygon tubing (3/16 in ID, R3603, VWR #63010-042)	☐ Tygon tubing (3/16 in ID, R3603, VWR #63010-042)
☐ Tygon tubing (3/8 in ID, R3603, VWR # 63010-133)	☐ Tygon tubing (1/4 in ID, R3603, VWR #63010-064)
☐ SKC Hi-Lite® air sampler (SKC Cat # 223-350, 25 L/min capacity)	☐ SKC AirChek® samplers (SKC Cat #224-PCXR8, 5 L/min capacity)
☐ Pipe adaptor 1/8 in NPT to 3/8 inID (Cole-Parmer # 30610-37)	☐ Battery eliminators (SKC Cat # 223-325)
☐ Pipe adaptor 1/8 in NPT to 3/16 inID (Cole-Parmer #0 6444-16)	☐ Pipe adaptor 1/8 in NPT to 1/4 inID (Cole-Parmer #30610-36)
☐ Reducing connectors ¼ in to 3/16 in ID (Cole-Parmer # 30622-32)	☐ Pipe adaptor 1/8 in NPT to 3/16 inID (Cole-Parmer #06444-16)
☐ Y-shape connector (3/8 in ID, VWR # 62850-106)	☐ Reducing connectors ¼ in to 3/16 in ID (Cole-Parmer #30622-32)
☐ Sampling masts (9)	☐ Sampling masts (5, four in receptor and one in ambient)
☐ Rotameters (18)	☐ Rotameters (10, eight in receptor and 2 in ambient)
☐ Plastic tubs (9)	☐ Plastic tubs (5)
☐ Zip-ties	☐ Zip-ties

☐ Bricks (2/tub)	☐ Bricks (2/tub)

Power Source Materials

Perimeter	Receptor and Ambient
☐ Generators ☐ Plastic tubs, extra large size ☐ Heavy-duty chains ☐ Gas cans ☐ Plywood platforms ☐ Metal stakes ☐ Aluminum wiring ☐ Padlocks ☐ Extension cords ☐ Bricks	☐ Marine deep-cycle batteries ☐ Plastic battery case ☐ Velcro ☐ Bricks ☐ Scissors ☐ Extension cords

Rotameters

☐ PTFE thread seal tape (1/2 in wide)
☐ Flowmeters range 1-10 Lpm (Dywer, VRB #66-SSV)
☐ Flowmeters range 1-20 Lpm (Dywer, VRB #67-SSV)
☐ Flowmeters range 1-10 Lpm (Key Instruments, Item # FR4A41SVVT)

1.3 Preparation

1.3.1 Sampling mast

- Construct a T-shape using the PVC pipes. The 60 in long pipe serves as the support stand (upright) while the two 12 in long pipes will be the cross-arm.
- Attach the rain gutter over the cross arm.
- Drill two small holes on the bottom of the cross arm at each end.

1.3.2 Plastic tubs

- Drill four holes (2 on each side) toward the top of the plastic tubs; these will serve as vent holes.
- For perimeter sampling
    - Place 2 bricks in the bottom of each tub
- For receptor/ambient sampling
    - Cut strips of Velcro, attach two sticky strips one side against the inside wall of the tubs and attach the fuzzy strips on the back of the SKC AirChek® sampler.
    - Stick the SKC AirChek® samplers to the inside wall of the tubs.
    - Set two bricks inside the tub, the SKC AirChek® sit on top of the bricks.

1.3.3 Rotameters

- Use the PTFE thread seal tape to wrap around the pipe adaptors' thread. Do not cover the ends.
- For all pipe adaptors, twist the Pipe adaptor 1/8 in NPT to 3/16 inID to the rotameter outlet (bottom of rotameter).
- For perimeter sampling

- Twist the Pipe adaptor 1/8 in NPT to 3/8 inID to the rotameter inlet (top of rotameter).

- For receptor/ambient sampling
  - Twist the Pipe adaptor 1/8 in NPT to 1/4 inID to the rotameter inlet (top of rotameter).

1.4 Sampling mast set-up (at site)

- Place the concrete block on level ground.
- Insert 4 ft metal stake into block and hammer about 1 foot into the ground.
- Insert the 12 in x 1 in galvanized steel pipe into the concrete block (goes over the stake).
- Attach the galvanized steel coupling piece to the 12 in x 1 in galvanized steel pipe.
- Place the 3 ft galvanized steel pipe over the 12 in steel pipe. This sits on top of the coupling piece.
- Make a 6 in cut parallel cut at the bottom of the PVC pipe with saw.
- Place the T-shape PVC pipe mast over the 12 in steel pipe.
- Secure the bottom of 60 inch PVC pipe to the 12 in steel pipe with a small hose clamp.

Figure 1: Sampling mast set-up with sampling train

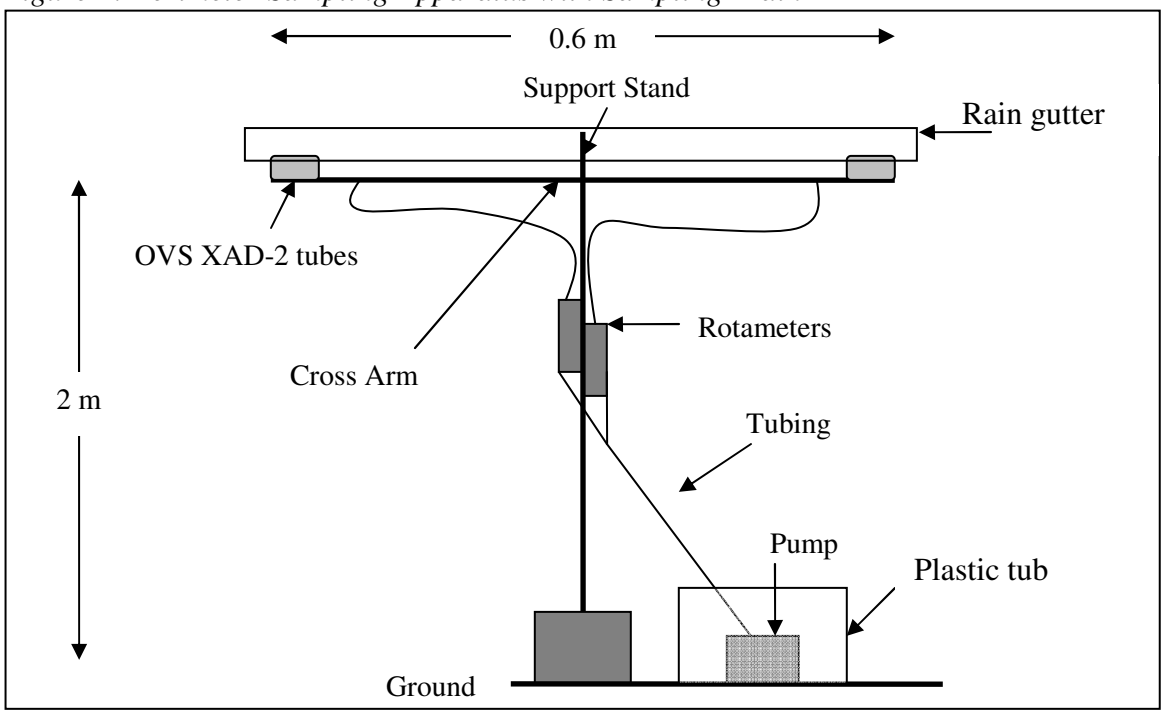
1.5 Sampling train set-up

1.5.1 Perimeter

- Place the SKC Hi-Lite® air sampler on the ground inside the plastic tub.
- Pass the extension cord through the vent holes.
- Connect the sampler's power plug to the extension cord.
- Attach the 3/8 in ID Tygon tubing to Hi-Lite® sampling pump inlet.
- Attach the Y-shape connector to the 3/8 in tubing.

- Connect each end of the Y-shape connector to the 3/8 in tubing (split sampling).
- Attach 3/8 in tubing to the inlet of the rotameter.
- Attach 3/16 in tubing to the outlet of the rotameter.
- Connect the 3/16 in tubing to the ¼ in tubing using the reducing connector.
- Pass the ¼ in tubing through the vent holes on the bottom of the cross-arm.
- Connect the sampling tube to the 1/4 in tubing once sampling starts.
- Position the sampling tube at a 90 degree angle inside the cross arm, one tube at each end.

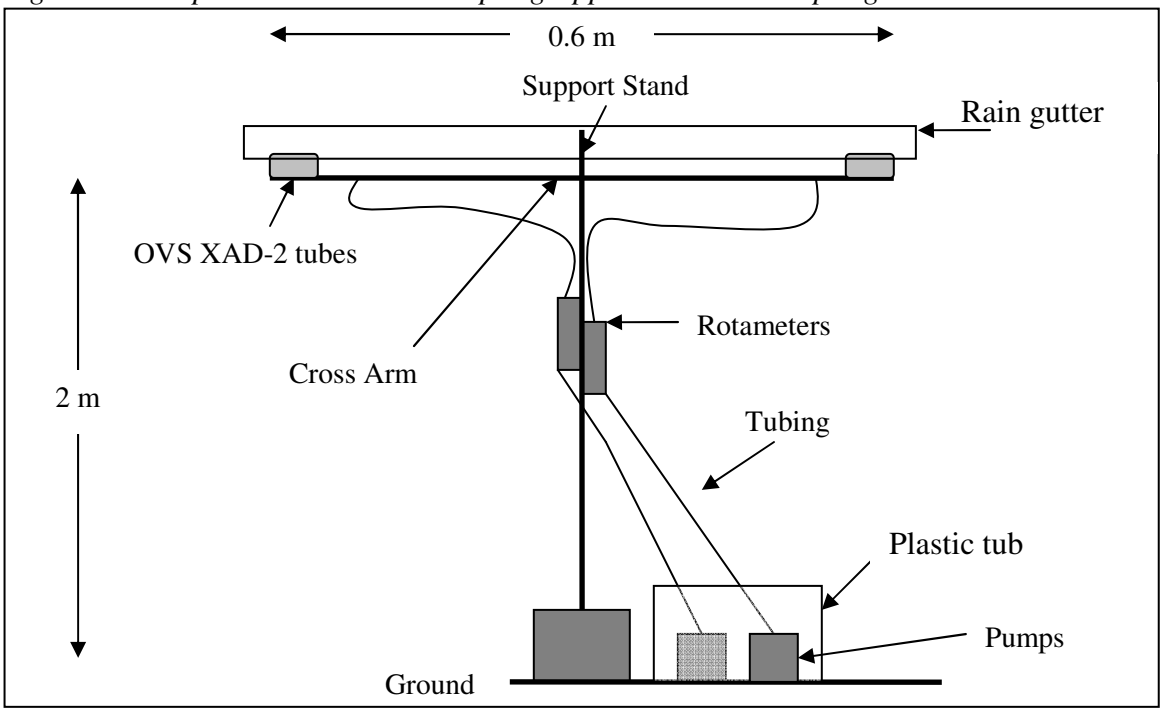
Figure 2: Perimeter Sampling Apparatus with Sampling Train



1.5.2 Receptor/ambient

- Place two SKC AirChek® samplers inside the tub and on top of the bricks.
- Secure the samplers by attaching them to the Velcro strips along one tub wall.
- Pass the extension cord through the vent holes.
- Connect the sampler's power plug to the extension cord.
- Attach the 1/4 in ID Tygon tubing to the SKC sampling pump inlets.
- Pass the 1/4 in tubing through the vent holes.
- Attach 1/4 in tubing to the inlet of the rotameter.
- Attach 3/16 in tubing to the outlet of the rotameter.
- Connect the 3/16 in tubing to the ¼ in tubing by using the reducing connector.
- Pass the ¼ in tubing through the vent holes on the bottom of the cross-arms.
- Connect the sampling tube to the 1/4 in tubing once sampling starts.
- Position the sampling tube at a 90 degree angle inside the cross arm, one tube at each end.

Figure 3: Receptor and Ambient Sampling Apparatus with Sampling Train



1.6 Power source

1.6.1 Perimeter

Two generators will supply the AC power source to the 9 SKC Hi-Lite® air samplers. The site layout will determine the locations of the generators. Use different lengths of extension cords to connect the generators to the air samplers.

- Cut vent flaps (rectangle shape) on the shorter sides of the plastic tubs.
- Select an area where the ground is leveled.
- Lay the plywood on the ground.
- Place the generator on top of the plywood.
- Place the plastic tub over the generator. The generator's exhaust must be near one opened flap.
- Use stakes, brick, and aluminum wiring to hold the tub.
- Use the chains and locks to secure the generators to a tree or wooden pole.

Figure 4: Perimeter Generator cover

1.6.2 Receptor/ambient

Use either AC power or marine deep-cycle batteries for power. This depends on the availability and security of an AC power source.

- AC power sources
  - Connect SKC Airchek® samplers with extension cords.
  - Protect cords from foot or vehicle traffic with barriers, covers, or other methods as appropriate to the site.
- Marine deep-cycle batteries
  - Store the marine deep-cycle batteries in secure locked battery cases.
  - Set fully-charged batteries before the start of the sampling period.
  - Keep detailed records to know when the battery is running low and must be replaced with another fully-charged battery.

Pilot OP Pesticide Air Monitoring Project
Standard Operating Procedures
SOP 3: Perimeter Sample Air Collection

3.1 Objective

To develop an appropriate and consistent manner to collect perimeter air samples.
Modified from: *Ambient Concentrations of Organophosphorus Pesticides Caused by Volatilization During Seasonal Pesticide Application, appendix A, attachment 2: sampling protocol* Master's Thesis. Lisa A. Tolbert 28 May 2007.

Referenced SOPS for the OP Pesticide Air Monitoring Project
SOP 1 Sampling Apparatus Set-up
SOP 5 Labeling

3.2 Materials

Sampling collection- daily
- ☐ OVS XAD-2, quartz filter sorbent tubes (SKC Cat # 226-58) (2-tubes/location within the site)
- ☐ Nitrile gloves
- ☐ Perimeter Field Data Sheet
- ☐ Pens - permanent
- ☐ Clipboard
- ☐ Small Ziplock bag sandwichor snack size (1/sample)
- ☐ large Ziplock bag quartz size (1/sample period)
- ☐ Clear packing tape
- ☐ Aluminum foil shields (pre-cut into squares)
- ☐ Screwdriver for sampling pump
- ☐ Field spikes (See Section 3.5 Sampling Plan)
- ☐ Field blanks (See Section 3.5 Sampling Plan)
- ☐ Lab diapers
- ☐ Handiwipes
- ☐ Cooler
- ☐ Blue ice
- ☐ Watch

3.3 Preparation

3.3.1 Sampling train
- Set up sampling apparatus according to *SOP 1 Sampling Apparatus Set-up*

3.3.2 Pre-labeling Ziplock bags.
- Label a quart Ziplock bag with sample start date and sample period.
- Label eighteen small Ziplock bags with the location numbers. (This is for organizing the sampling tubes.)
- Label small Ziplock bag per blank with "Field Blank", sample day (day1, day2, day3, day4, and sample period (morning, lunch, or evening).
- Label one small bag per spike with Ziplock bag with "Field Spike" sample day (day1, day2, day3, day4, and sample period (morning, lunch, or evening).

3.3.3 Sample tubes preparation before going to the field.

- Write the sample start date on the sample label.
- Write the sample start date on the field blank label
- Write the sample start date on the field spike label.
- Write the sample start date and location numbers (2 sample tubes for each location on) labels. *(Note: Pull up the ends of the sample labels you are preparing to make it easier to peel and stick on the sample tubes.)*
- Pre-cut packaging tape to fit over the sample labels.
- Layout lap diaper.
- Open the sample tubes envelopes and gently place the number of tubes required for the sample period on the lab diaper.
- Put on nitrile gloves.
- For each sample tube
  - Place the sample label on sample tube.
  - Put one packaging tape strip over the sample tube label and place the tube in its corresponding bag, by site number, small Ziplock bag.
  - Follow the same steps for the remaining tubes.
- For the field spike,
  - Wear nitrile gloves.
  - Remove the spike from the freezer.
  - Record the lab label already on the field spike onto the data sheet.
  - Remove the spike from its Ziplock bag.
  - Place the field spike label on the tube.
  - Place packaging tape over the label and replace tube back in its Ziplock bag.
  - Place the sample tube Ziplock bag in the quart size Ziplock bag that will contain all tubes collected for that sampling day and period.
- Put duplicate sample labels on the *Perimeter Field Data Sheet* filling in the start date and the location numbers.
- Record sample tube lot number (found on the tube envelop) on the *Perimeter Field Data Sheet.*

3.4 Sample collection

3.4.1 Set up sample collection area

- Lay out lab diaper.
- Designate a clean (new sample) and dirty (old sample) area.
- Lay out supplies and nitrile gloves.

3.4.1 Sample take down - Removing previous day's sample tubes

- Record rotameter reading on *Perimeter Field Data Sheet.*
- Turn off pump.
- Record stop date and time on *Perimeter Field Data Sheet.*
- Don nitrile gloves.
- Remove aluminum foil shield from sampling tube.
- Remove sample tube from tubing (only handle the tube with clean nitrile gloves).
- Put caps on the tube.
- Place each tube in a small Ziplockk bag and seal bag.

- Place all tubes in the quart Ziplockk bag label for the region, sample day, and sampling period.
- Place the labeled bags in a cooler filled with blue ice for transport back to field office freezer.

3.4.2 Sample set-up: starting sample tubes for the next sampling period

- Check all tubing and rotameter connections to ensure they are tight.
- Put on nitrile gloves.
- Remove prelabeled sample tubes from small Ziplockk bags.
- For each sampling tube
  - Remove the thinner end cap and store cap in Ziplock bag.
  - Connect the thinner end of the tube to the 1/4" tubing attaching it to the rotameter.
  - Wrap tubes with aluminum foil so that no sampling media is visible. Leave the tube inlets unobstructed.
  - Angle the sampling tube downward.
  - Hold sampling tube in place with duct tape.
- Check that tubing is securely fastened to the sampling stand.
- Record on *Perimeter Field Data Sheet*
  - Pump number
  - Rotameter number
- Turn pump on.
- Record start time.
- Adjust flow rate to setting indicated on rotameters (6 liters per minute)
  - Verify rotameter valves are open (turn counter clockwise).
  - Use pump screwdriver to turn flow screw on pump.
  - Adjust flow so that one rotameter is at the designated flow rate and the other is higher.
  - Turn (clockwise) the rotameter valve with the higher flow rate until flow rate decreases and both are within +/- 0.2 lpm.
    NOTE: It may be necessary to go back and forth to adjust both rotameters so that they read the designated flow rate.
- Record final flow rates setting on the Perimeter Sampling Field Data Sheet.

3.4.2 Flow rate check

- Midway through sampling period check flow rates and
  - Record time and flow rate.
  - Adjust flowrates to rotameter setting.
- Record new flowrate.

3.5 Sampling Plan

Table 1: Sampling Plan

	Sampling Day			
	Pre-spray	*Spray*	*Post-spray 1*	*Post-spray 2*
Sampling locations (including QA)*	4 locations + co-located QC mast	8 locations + co-located QC mast	8 locations + co-located QC mast	8 locations + co-located QC mast
Number of sample periods per day	1 Morning	3 Morning Afternoon Night	3 Morning Afternoon Night	1 Morning
Duration of Sampling periods	24-hr	8-hr	8-hr	24-hr
Number of field samples/day	10	54	54	18
Number of field blanks/day	1	6 2/sampling period	6 2/sampling period	2 2/sampling period
Number of field spikes/day	1	6 2/sampling period	6 2/sampling period	6 2/sampling period

*Each site had 8 locations are situated approximately equidistant from each other around the perimeter of the sampling block. At each location a single sampling mast holds 2 side by side samples. The QC sample mast was co-located at one of the eight sampling locations, which held 2 side by side samples as well.

3.6 Field blanks and spikes. *One field blank and one field spike per 10 samples per day.*

3.6.1 Field blanks

Field blanks will be treated the same way as the sample tubes. For each sampling period, the field blank(s) will be started just prior to setting up the sampling tubes at the first location. To start the field blank.

- Don nitrile gloves.
- Remove sample tube from its pre-labeled Ziplock bag.
- Remove both end caps.
- Replace both end caps.
- Place back in small Ziplock bag.
- Place small Ziplock bag in the large Ziplock bag for the day/period.
- Record on data sheet
  - Time that the field blank(s) were started.
- Store field blank in the cooler with blue ice.

3.6.2 Spikes EH Lab will prepare the field spikes in advance. Store the supply of field spikes in the field office freezer (-10C).

- Use nitrile gloves when handling field spikes.
- Obtain the field spike for the day from -10 C freezer and transport on blue ice.
- Remove field spike from Ziplockk bag.
- Label field spike tube with sample label.
- Replace Place field spike in small Ziplockk bag.
- Place small Ziplockk bag in the large Ziplockk bag for the day/sampling period.
- Transport and store field spike in the cooler with blue ice.

3.7 Sample tube transport and storage

- Store all sample tubes, field spikes, and field blanks in ice chest with blue ice in the field
- Transport all sample tubes, field spikes, and field blanks in ice chest with blue ice to field office freezer as soon as possible.
- Store all samples tubes, field spikes, and field blanks in Field freezer (-10C) until transport to UW-Fenske lab.
- Transport all samples tubes; field spikes, and field blanks to UW-Fenske lab in cooler with dry ice.

3.8 Sample labeling

- See SOP 5: Labeling

3.9 Data Storage and Backup

- Store all Perimeter Field Data Sheets in the offices and in password protected computer resources of R. Fenske at the University of Washington.
- Scan all data sheets and make PDFs of all data sheets for back up.
- Store all electronic files on the department server, with password access limited to research team staff. The electronic files are backed up daily.

Pilot OP Pesticide Air Monitoring
Standard Operating Procedures
SOP 3A: Perimeter Air Sample Collection
Addendum

3A.0 Purpose

The purpose of this addendum is to provide changes in the SOP 3 for phase 2 of the project.

3A.1 Sampling Plan – Phase 2

Table 1: Sampling Plan – Phase 2. ***Changes in Bold.***

	Sampling Day			
	Pre-spray	*Spray*	*Post-spray 1*	*Post-spray 2*
Sampling locations (including QA)	4 locations + co-located QC mast	8 locations + co-located QC mast	8 locations + co-located QC mast	8 locations + co-located QC mast
Number of sample periods per day	1 Morning	3 Morning Afternoon Night	**2** **Morning** **Night**	1 Morning
Sampling duration/time	24-hr	**6-hr spray** **6-hr after spray** **12-hr overnight**	**12-hr**	24-hr
*Number of field samples/day******	10	54	**36**	18
Number of field blanks	1	6 2/sampling period	**4** 2/sampling period	2 2/sampling period
Number of field spikes	1	6 2/sampling period	**4** 2/sampling period	6 2/sampling period

*****Each site had 8 locations are situated approximately equidistant from each other around the perimeter of the sampling block. At each location a single sampling mast accommodates 2 side by side samples. The QC sample mast was co-located at one of the eight sampling locations.

Pilot OP Pesticide Air Monitoring
Standard Operating Procedures
SOP 4: Receptor & Ambient Sampling Collection

4.0 Objective

To develop an appropriate and consistent manner to collect receptor and ambient air samples. Modified from: *Ambient Concentrations of Organophosphorus Pesticides Caused by Volatilization During Seasonal Pesticide Application*, *appendix A, attachment 2: sampling protocol* Master's Thesis. Lisa A. Tolbert 28 May 2007.

Referenced SOPs for the Pilot OP Pesticide Air Monitoring Project are

SOP 1: Sampling Apparatus set-up
SOP 5: Labeling
SOP 7: Rotameter Calibration in Laboratory

4.1 Materials

Sample collection - daily
- ☐ SKC Airchek sampler (SKC Cat #224-PCXR8, 5 L/min capacity) with battery eliminator
- ☐ OVS XAD-2, quartz filter sorbent tubes (SKC Cat # 226-58) (2-tubes/day/location)
- ☐ Nitrile gloves
- ☐ Receptor and Ambient Field Data Sheet
- ☐ Permanent pens
- ☐ Clipboard
- ☐ Small Ziploc® or similar bag sandwich or snack size (1 /sample)
- ☐ Large Ziploc® or similar bag quart size (1/day)
- ☐ Sample labels
- ☐ Clear packaging tape
- ☐ Aluminum foil (pre-cut into squares)
- ☐ Duct tape
- ☐ Screw driver for pump
- ☐ Lab diapers
- ☐ Handiwipes
- ☐ Field spike (1/day)
- ☐ Field blank (1/day)
- ☐ Plastic trash bag
- ☐ Cooler
- ☐ Blue ice
- ☐ Watch

4.2 Preparation

4.2.1 Sampling train (prior to first sample start day)

Set up sampling apparatus according to SOP 1, including pumps and rotameters.

4.2.2 Pre-labeling Ziploc® bags. In field office

- Label a quart size Ziploc® bag with today's date, initials of staff member collecting the samples, and region.
- Label three small size Ziploc® with the site numbers (this will store the newly labeled sample tubes).
- Label one small Ziploc® as "Field Blank" with date.

4.2.3 Sample label preparation. (See SOP 5 for complete description of labels.) In field office

- Note: For each tube, labels were preprinted in quadruplicate to be used as follows
  - 1 for sample tube
  - 1 for Receptor and Ambient Field Data Sheet
  - 1 for initial chain of custody form
  - 1 for additional chain of custody form

- Complete the receptor, ambient, and QC sample labels by writing on the quadruplicate labels
  - sample period start date
  - site number

- Complete the field spike and field labels by writing on the quadruplicate labels
  - sample period start date

4.2.4 Sample tubes preparation. In field office

- Pre-cut packaging tape to fit over the sample labels.
- Place the sample tubes envelopes and gently place the sample tubes on the lab diaper.
- For each sample tube
  - Place the sample label on tube. Do not cover any part of the sample id number the unique identifier.
  - Put packaging tape strip over the sample tube label and place the sample in its corresponding, small Ziploc® bag labeled by site number.
  - Put matching labels on the data sheet.
  - Record tube lot number (printed on the tube envelope) on data sheet.
  - Follow the same steps for the remaining tubes.
- For the field blank tube
  - Put field blank label on tube. Do not cover the unique identifier.
  - Wrap packaging tape strip over the label and small Ziploc®.
  - Put matching label on the Receptor and Ambient Field Data Sheet.
  - Record tube lot number (on the tube envelop) on Receptor and Ambient Field Data Sheet.
- For the field spike tube
  - Wear clean nitrile gloves.
  - Remove the spike tube from the freezer.
  - Record the lab label already on the field spike onto the data sheet.
  - Remove the spike from its Ziploc®.
  - Place the field spike label on the tube.

- Put matching sample label on the Receptor and Ambient Field Data Sheet.
- Wrap packaging tape over the label and put tube back in its Ziploc® bag.
- Write the corresponding sample start date on the Ziploc® Bag.
- Place the Ziploc® in the quart size Ziploc® labeled with the corresponding sample start date.

4.3 Sample collection - In the field

4.3.1 Set up sample collection area (applies to both sample start up and sample take down)

- Lay out lab diaper.
- Designate a clean (new sample) and dirty (old sample) area.
- Lay out supplies and nitrile gloves.

4.3.2 Sample start-up

- Put on clean nitrile gloves.
- Place the two prelabeled sample tubes in clean area.
- Record on field data sheet.
  - Pump number
  - Rotameter number
- For each sampling tube, follow these steps
  - Remove the caps and store in Ziploc® bag.
  - Attach narrow end of sample tube to end of 1/4" tubing, letting the tube inlet (wider opening) facing downward.
  - Check that the tubing is securely fastened to the sampling stand.
  - Wrap tubes with aluminum foil so that no sampling media is visible. Leave the tube inlet and outlet unobstructed.
  - Hold sample tube slanting downward with a piece of duct tape. Do not cover tube inlet
  - Turn pump on.
  - Record sample start time.
- Adjust pump flow rate to setting indicated on rotameter
  - Verify rotameter valve is open.
  - Use pump screwdriver to turn flow screw on in-line pump.
  - Adjust flow 2.0 liters per minute on the rotameter. (Actual flow rate will be calculated from the rotameter calibration curves in *SOP 7: Rotameter Calibration in Laboratory.)*
- Wait 5 minutes.
- Record rotameter reading.
- Adjust flow rate to setting indicated on rotameter if needed.
- Record final rotameter setting.

4.3.3 Sample Collection- sample take down

- Record rotameter reading on Receptor and Ambient Field Data Sheet.
- Turn off pump.
- Record stop time on Receptor and Ambient Field Data Sheet.
- Don clean nitrile gloves.
- Gently tug the tube out of the mast by pushing out the tubing.
- Remove aluminum foil.
- Remove sample tube from tubing (only touch the sample tube with clean nitrile gloves – nothing else).

- Put caps on the tube. Push each capped end down on firm surface to secure the cap is secure.
- Place each tube in a small Ziploc® bag.
- Place all tubes in a large Ziploc® bag labeled with the project ID, date, region, and initials (for example, AMO8Region2_1Feb08kg).
- Place the labeled bags in a cooler filled with blue ice for transport back to freezer.

4.5 Field blanks and Field spikes

4.5.1 General

- One field blank and one spike for each sampling day.
- Field blanks and field spikes for a given sample period are set-up on the start day for that sample period.
- Field blanks and field spikes are taken into the field during the sample start-up for the same sample period.
- Store and transport the field blank and field spike in a cooler filled with blue ice during the sample period.

4.5.2 Field Blanks

- See section 4.2 for preparation of field blank tubes in the field office.
- Field blanks are uncapped and recapped the first site for start-up at the beginning of the sampling period.
- To cap and recap field blank
  - Don clean nitrile gloves.
  - Remove sample tube from its pre-labeled Ziploc® bag.
  - Remove both end caps.
  - Replace both end caps.
  - Place in small Ziploc® bag.
  - Place small Ziploc® bag in the large Ziploc® bag for the day.
  - Record time on Receptor and Ambient Field Data Sheet.

4.5.3 Field Spikes

- Field spikes were prepared by the University of Washington Environmental Health Laboratory.
- The supply of field spikes will be stored in the field office freezer.
- Start the field spike for a given sample period in the field office before going into the field to start-up the samples.
- See section 4.2 for preparation of field spikes in field office.

4.6 Sample, field blank, and field spike tube transport and storage

- Transport all sample, field blank, and field spike tubes in cooler filled with blue ice when in the field.
- Store samples, field blanks, and spikes in freezer (-10C) until lab analysis. Freezers are located at
  - Yakima field office
  - Wenatchee field office
  - Seattle UW-Fenske lab freezers located in F-216 HSB.
- Transport all tubes from field offices to UW in cooler under dry ice.

4.7. Cleanup

- Every day at each field site pick up all trash and used gloves and put in plastic garbage bag.

4.8 Sampling Plan

Table 1: Ambient and Receptor Sampling plan.

# of sample periods	28 consecutive days
sample period duration	24 hours (1 day)

	sample type		
	ambient	**receptor**	**qc[a]**
# sites	1	3	1
# samples/site/period[b]	2	2	2
# samples/type/period	2	6	2
# field spikes/period	1	1	1
# field blanks/period	1	1	1

[a]The qc sample mast was co-located at on of the receptor sites.
[b]Each site has one sampling mast for each sample site. The sampling mast holds 2 side-by-side samples.

4.9 Data Storage and Backup

- Store all Receptor/Ambient Data Sheets in the offices and in password protected computer resources of R. Fenske at the University of Washington.
- Scan all data sheets and make PDFs of all data sheets for back up.
- Store all electronic files on the department server, with password access limited to research team staff. The electronic files are backed up daily.

Pilot OP Pesticide Air Monitoring
Standard Operating Procedures
SOP 4A: Receptor & Ambient Air Sample Collection
Addendum

4A.0 Purpose

The purpose of this addendum is to provide changes in the SOP 4 for phase 2 of the project.

4A.1 Sampling Plan

Table 1: Ambient and Receptor Sampling plan – Phase 2. (changes in italics)

# of sample periods	*24, sampling every third day*
sample period duration	*24 hours (1 day)*

	sample type		
	ambient	**receptor**	**QC[a]**
# sites	1	3	1
# samples/site/period[b]	2	2	2
# samples/type/period	2	6	2
# field spikes/period	1	1	1
# field blanks/period	1	1	1

[a]The qc sample mast was co-located at on of the receptor sites.
[b]Each site has one sampling mast for each sample site. The sampling mast holds 2 side-by-side samples.

Pilot OP Pesticide Air Monitoring Project
Standard Operating Procedures
SOP 5: Labeling

5.1 Objective

The object of SOP5: Labeling is to develop an appropriate and consistent system for labeling sample collection tubes using unique sample identification (ID) codes.

5.2 Materials

- ☐ Labels (Avery number 5267, ½" x 1 ¾")
- ☐ Thin Sharpie
- ☐ Packaging tape
- ☐ Lab diaper
- ☐ Latex or nitrile gloves

5.3 Project ID

Project ID: All ID numbers for project samples collected in 2008 start with the project code:

- AM8

5.4 Near-field Receptor/Ambient Sampling code descriptions

5.4.1 Region

- T= Toppenish (Region 2)
- W= Wenatchee (Region 1)

5.4.2 Site

- Assigned 1-4, (list of sites maintained separately to protect confidentiality).
- Assign new sites starting number 5 and above (Phase 2).

5.4.3 Sample type

- A= ambient
- R= receptor
- B= field blank
- S= field spikes
- Q= quality assurance

5.4.4 Sample pairs

For each location and sample period, two side-by-side samples are collected. The samples are designated by a "1" or "2" after the sample type.

- 1 = first of pair
- 2 = second of pair

5.4.5 Unique identifier

- Assigned a unique identifier number each sample.
- Start with 1001

5.4.6 Ambient & receptor sampling label format

AM8_ [_____] _ [_____] _ [_____] _ [_____]
day, mo _ region, site _ sample type, sample pair number _ unique ID

For example, the following sample label represents a sample with this description: the DOH air monitoring project in 2008 on 30 March in Toppenish (Region 2), at site 15 the 1st tube of a receptor sample. The samples unique identifier was 1020.

AM8_3003_T15_R1_1020

5.5 Near-field Perimeter Sampling code descriptions

5.5.1 Sampling day

- 1: day one or pre-spray day
- 2: application/spray day
- 3: post-spray 1
- 4: post-spray 2

5.5.2 Sampling period

On spray day (sampling day 2) and post-spray 1 (sampling day 3), the sampling period was divided into the following periods. For sampling day 1 and sampling day 4, an "X" was used since there was only one sampling period

- M= morning
- L= lunch
- E= evening
- X= only one sampling period (Day 1, Day 4)

5.5.3 Location

Refers to specific location of the sampling apparatus at the site

- Sample locations:1-8
- Quality assurance location: 9 (co-located at one of the sampling locations)

5.5.4 Sample pairs

For each location and sample period, two side-by-side samples are collected. The samples are designated by an "A" or "B" after the sampling day.

- A = first of pair
- B = second of pair

5.5.5 Sample type

No distinction between the air samples. During Phase 1, there were two different spike loads.

- SH= field spike, high (Phase 1)

- SL= field spike, low (Phase 1)
- FS= field spikes (Phase 2)
- FB= field blank (Phase 1, Phase 2)

5.5.6 Perimeter sampling label format

AM8_ [_____] _ [_____] _ [_____]
day, mo _ sample day, sample period, location, sample pair _ unique ID

For example, on 4 April 2008 a post-spray 1 day, 2nd tube of perimeter evening sample was collected in location site 7. The unique identifier was 9999.

AM8_0404_3E7B_9999

Another example, on 28 March 2008 the spray day, 1st tube of perimeter lunch sample was collected in location site 2. The unique identifier was 4501.

AM8_2803_2M2A_4501

Another example, on 21 April a pre-spray day, 1st tube sample was collected on location site 4. The unique identifier was 5234.

AM8_2104_1X4A_5234

An example for field spike *Phase 1*, field spike high was collected on 22 April a spray day morning sample period. The unique identifier was 7001.

AM8_2204_2MSH_7001

An example for field spike *Phase 2*, field spike was collected on 18 April a background day. The unique identifier was 6524.

AM8_1804_1FS_6524

An example for field blank, field blank was collected on 21 April a background day. The unique identifier was 6587.

AM8_2104_1FB_6587

5.6 Sample label preparation

Print four copies of the sample labels. One sample label copy goes on the tube, the second sample copy on the data sheet, the third on the chain of custody data sheet, and the fourth (last) copy goes on the final transport chain of custody data sheet.

- Pre-print the following codes on the labels.
- For receptor/ambient
    - Project ID

  - Blank line for day and month
  - Region
  - Blank line for site
  - R1 and R2 (each region has 3 receptor sites)
  - A1 and A2 (each region has 1 ambient site)
  - Assigned unique identifier
- For perimeter
  - Project ID
  - Blank line for day and month
  - 1X, 2M,2L, 2E, 3M, 3L, 3E, 4X
  - Sample type *(field blanks and field spikes only)*
  - Pre-assigned location sites for day 1
  - All location sites for days 2-4
  - Sample pairs at each location site
  - Assigned unique identifier

5.7 Pre-labeling sample tubes

- Write the dates on the labels using a fine point Sharpie.
  - Write the dates for all four copies (across the sheet) of the labels.
- For receptor/ambient labels, also write in the site number.
  - Write the site numbers for all four copies of the labels.
- Pre-cut thin strips of packaging tape.
- Lay out a lab diaper.
- Don gloves.
- Lay the sample tubes on the lab diaper.
- Stick the label on the sample tube. Do not cover any part of the sample ID label.
- Wrap the packaging tape over the sample label.

Pilot OP Pesticide Air Monitoring Project
Standard Operating Procedures
SOP 6: DryCal Calibration in Laboratory

6.1 Objective

The objective of SOP6: DryCal Calibration in Laboratory is to develop an appropriate and consistent manner to calibrate the DryCal in the laboratory to a primary bubble flow meter. Modified from: *Ambient Concentrations of Organophosphorus Pesticides Caused by Volatization During Seasonal Pesticide Application, appendix A, attachment 2: sampling protocol* Master's Thesis. Lisa A. Tolbert 28 May07

6.2 Materials

- ☐ DryCal calibrator (BIOS International, model # DCLT-12K)
- ☐ 1,000 ml burette
- ☐ SKC Hi-Lite® air sampler (SKC Cat # 223-350, 25 liter per min (lpm) capacity)
- ☐ SKC AirChek® sampler (SKC Cat #224-PCXR8, 5 lpm capacity)
- ☐ Battery eliminators (SKC Cat # 223-325)
- ☐ Bowl wider than 1,000 ml burette
- ☐ Bubble solution
- ☐ Rubber tubing
- ☐ Stopwatch
- ☐ DryCal calibration data sheet
- ☐ Lab stand
- ☐ Glassware clamps
- ☐ Pen

6.3 Calibration Equipment Set-up

- Invert the burette and attach to the lab stand using the glassware clamps.
- Burette (inverted)→ DryCal→ pump (connect equipment with rubber tubing).
- Partially fill the bowl with bubble solution and place bowl below the inlet of the burette.
    - Do not let top layer of bubble solution touch the burette inlet.

6.4 DryCal Calibration

"True" flowrates come directly from the bubble flow meter's (primary flowrate) travel volume and the time it took for the bubble to move through the volume. The calibration flowrates are 2, 3, 4, 8, and 11 lpm. Use the SKC AirChek® sampler for the 2-4 lpm flowrates and the SKC Hi-Lite® air sampler for the 8-11 lpm.

1. Pour a small amount bubble solution inside the burette.
2. Swirl the bubble solution to coat inside of the burette.
3. Set-up the calibration set up *(Section 6.3).*
4. Turn on pump and DryCal.
5. As measured by DryCal, set pump speed to the lowest calibration flowrate.
6. Briefly lift bowl to the burette rim so that top layer of bubble solution in bowl touches rim of burette and thin bubble is form that travels up length of burette.

7. Repeat several times to re-coat inside of burette with bubble solution.
8. Do not start timed trials until bubbles travel entire burette length without popping.
9. For timed trials, reset DryCal by re-starting it and briefly touch top layer of bubble solution in bowl to the burette rim so that thin bubble is formed.
10. When bubble reaches **1,000 ml** mark on inverted burette, start stopwatch.
11. When bubble reaches the **0 ml mark** on inverted burette, stop stopwatch and Drycal.
12. Record travel volume (1000ml), travel time, and the flowrate displayed on DryCal.
13. Repeat timed trials three times per pump speed setting.
14. Record the Drycal average per pump speed setting.
15. Increase flowrate to the next flowrate.
16. Allow pump time to equilibrate (approximately 2-3 min) before repeating burette coating and timed trials at higher flowrate.
17. Continue calibration process until pump speed reaches above highest anticipated sampling flowrate of 11 lpm.
18. Calculate the "true" flowrate (lpm) dividing the bubble travel volume (1000 ml) by the bubble travel time per trial.

    $$\text{flowrate (lpm)} = \frac{\text{travel volume (l)}}{\text{travel time (min)}}$$

19. Plot DryCal flowrate versus "true" flowrate (as measured by travel volume and travel time) and fit linear model to data.
20. Calculate the slope equation from the data (calibration curve)
    - $Y = mx + b$

      y= rotameter reading lpm
      m=slope
      x= DryCal reading lpm
      b=y-intercept

21. Use equation of best-fitting line to adjust the DryCal flowrates to be equivalent to the desired "true" flowrate.

6.5 Data Storage and Backup

- Stored all of the Drycal calibration data in the offices and in password protected computer resources of R. Fenske at the University of Washington.
- Scan all data sheets and make PDFs of all data sheets for back up.
- Store all electronic files in the department server, with password access limited to research team staff. The electronic files are backed-up daily.

Pilot OP Pesticide Air Monitoring Project
Standard Operating Procedures
SOP 7: Rotameter Calibration in Laboratory

7.1 Objective

The objective of SOP7: Rotameter Calibration in Laboratory is to develop an appropriate and consistent manner to calibrate the rotameters in the laboratory. Modified from: *Ambient Concentrations of Organophosphorus Pesticides Caused by Volatization During Seasonal Pesticide Application, appendix A, attachment 2: sampling protocol* Master's Thesis. Lisa A. Tolbert 28 May 2007

Referenced SOPs
SOP6: DryCal Calibration in Laboratory

7.2 Materials

- ☐ DryCal calibrator (BIOS International, model # DCLT-12K)
- ☐ Rotameters
- ☐ SKC Hi-Lite® air sampler (SKC Cat # 223-350, 25 L/min capacity)
- ☐ SKC AirChek® sampler (SKC Cat #224-PCXR8, 5 L/min capacity)
- ☐ Battery eliminators (SKC Cat # 223-325)
- ☐ Tygon tubing (1/4" ID, R3603, VWR #63010-064)
- ☐ Tygon tubing (3/8" ID, R3603, VWR # 63010-133)
- ☐ Rotameter Calibration Data Sheet
- ☐ Pen

7.3 Calibration Equipment Set-up

- DryCal→ rotameter→ pump (connect equipment with Tygon tubing)

7.4 Rotameter Calibration

The calibration range is 2, 4, 6, and 8 L/min. Use the SKC AirChek® sampler for the 2-4 L/min flowrates and the SKC Hi-Lite® air sampler for the 6-8 L/min.

1. Turn the rotameter valve counter-clockwise so that the valve is fully "open."
2. Turn on SKC AirChek® sampler.
3. Turn on DryCal.
4. As measured by the Drycal, set pump speed to the lowest calibration flow rate.
5. Allow pump time to equilibrate (2-3 min).
6. Take 10 flowrate readings by reading the rotameter flowrate and the flowrate measured on the DryCal.
7. Record rotameters and Drycal flowrates on the Rotameter Calibration Data Sheet.
8. Record the average of 10 DryCal readings on the data sheet.
9. Increase flowrate to the next calibration flowrate by adjusting the pump's speed.
10. Repeat steps 5-10.
11. Switch to the Hi-Lite® pump for the 6-8 L/min calibration range.
12. Repeat steps 5-10 for all four calibration range flowrates.
13. Repeat the calibration process (steps 1-11) with the remainder of rotameters.

14. Plot rotameters flowrates versus DryCal flowrates for each rotameter.
15. Calculate the slope equation from the data (calibration curve). This equation of the slope is unique to the rotameter.
    - y= mx + b

      y= rotameter reading lpm
      m=slope
      x= DryCal reading lpm
      b=y-intercept

7.5 Rotameter flowrate correction

- Correct each rotameter flowrate to the primary standard value by using the equation of best-fitting line "true" flow rate *(see Sop 6: DryCal Calibration in Laboratory).*

7.6 Data Storage and Backup

- Store all rotameter calibration data in the offices and in password protected computer resources of R. Fenske at the University of Washington.
- Scan all data sheets and make PDFs of all data sheets for back up.
- Store all electronic files in the department server, with password access limited to research team staff. The electronic files are backed-up daily.

Pilot OP Pesticide Air Monitoring Project
Standard Operating Procedures
SOP 8: Chain of Custody

8.1 Objective

The objectives of SOP:8 Chain of Custody is to ensure the appropriate and consistent system for tracking samples.

8.2 Materials

- ☐ Chain of custody data sheets
- ☐ Sample labels
- ☐ Lab diaper
- ☐ Thermometers
- ☐ Watch
- ☐ Masking tape
- ☐ Permanent pen
- ☐ Nitrile gloves

NOTE: At the end of each sample period, all samples for that period are put in a quart Ziploc® bag. The bag is labeled with sample period, sampling date, region, staff initials (receptor/ambient sampling only). In the field, this Ziploc® bag is stored inside an ice cooler filled with blue ice.

8.2 Filling out chain of custody (if **same** person collecting the sample is transporting the samples to the freezer)

At site or at Field lab

1. Record the date of the sample collected.
2. Don nitrile gloves.
3. Pull out all of the sample tubes by their Ziploc® bag and set inside the ice chest.
4. Select a sample tube.
5. Locate its matching sample label and place on the chain of custody data sheets.
6. Store this sample tube back inside the sampling day's Ziploc® bag.
7. Record time when the sample tube is place inside the quart size Ziploc bag.
8. Measure the temperature of the cooler.
9. Write down your initials under the "Prepared/Given By" column.
10. Repeat the steps above for all remaining sample tubes.
11. Seal the Ziploc® bag with masking tape.

At Field lab

12. Once arriving to the lab, write down your initials under the "Received By" column.
13. Record the arrival temperature of inside of cooler.
14. Write how the samples arrived (ie. cooler).
15. Note any disturbances of the sample tubes.
16. Store the samples inside the freezer.
17. Record the field freezer temperature.

8.3 Filling out chain of custody (if person collecting the sample **differs** than the person transporting the samples to the freezer)

8.3.1 Person collecting the samples: Repeat steps 1-10

8.3.2 Person transporting/ receiving the samples

- Record the arrival temperature of the inside of the cooler.
- Don nitrile gloves.
- Lay out lab diaper.
- Lay out the sample tubes by Ziploc® bag.
- Select a sample tube.
- Locate this sample tube on the chain of custody data sheet.
- Once found, place this sample tube back in its sampling day's Ziploc® bag.
- Write down your initials in the "Received By" column.
- Repeat the above 8 steps for all remaining sample tubes.
- Note any disturbances of the sample tubes. For example, missing or fallen tube cap.
- Seal the Ziploc® bag with masking tape.
- Store the samples inside the freezer.
- Record the field freezer temperature.

8.4 Transport from field freezer to UW freezer

8.4.1 Final Transport Materials

☐ On location freezer transport to UW freezer chain of custody data sheets
☐ Thermometers: freezer and coolers
☐ Coolers (2-3)
☐ Dry ice
☐ Watch

8.4.2 Preparation

- Purchase dry ice.
- Break dry ice into small pieces and place inside the coolers.
- Place sample labels on the chain of custody sorted by date.
    - If a premade sample label is not available record the complete sample ID on the data sheet

  NOTE: if person transporting samples **differs** than the last person responsible for the samples, then another chain of custody sheet must be filled out.

- Place a thermometer inside the cooler.

8.4.3 Removing sample tubes from freezer

- For each individual quart size Ziploc® bag containing samples
  1) Remove one quart size Ziploc® containing samples.
  2) Locate the date of the Ziploc® on the data sheets.
  3) Record the removal date and time under the "Date/Removal Time from Storage."

4) Write down freezer as the storage type and the temperature under the "Storage Type/Temp."
5) Keep track of the dates of the Ziploc® bags and in which coolers they are being placed.

- Ensure all samples designated for transport from the field freezer have been accounted for and placed in the coolers for transport.
- Record the departure cooler temperature once all Ziploc® bags have been transferred from the freezer to coolers.

8.4.4 Transport

- Do not close the cooler lid too tightly, the dry ice needs to vent.
- Place the cooler in the trunk or in the trunk area.
- Drive with the windows slightly open.

8.5 Arrival at UW freezer

- Repeat Section 8.3.2
- Store the samples in the UW freezer.

8.6 Data Storage and Backup

- Stored all of the chain of custody data in the offices and in password protected computer resources of R. Fenske at the University of Washington.
- Scan all data sheets and make PDFs of all data sheets for back up.
- Store all electronic files in the department server, with password access limited to research team staff. The electronic files are backed-up daily.