

**Standard Operating Procedure for
Air Sampling for Metals
Using the Dichotomous Sampler**

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1.0 Overview

This SOP is intended to provide a step by step procedure for collecting samples of airborne particles on Teflon filters for metals analysis using the dichotomous sampler.

The data collected from analyses of 37 mm Teflon filters from the dichotomous samplers will be used primarily for the Lake Michigan Loading Study (LMLS) and the Integrated Atmospheric Deposition Network (IADN) programs. Samples at the Sleeping Bear Dunes site, which is part of the Integrated Atmospheric Deposition Network, were sampled and analyzed by Indiana University. The sampling method is identical apart from a few minor differences in QC samples. This site represents 10 % of the samples for this method. The objectives of the programs are to determine the loadings of persistent toxic contaminants from the atmosphere to the Great Lakes from both urban and regional sources. Sampling sites have been strategically located around the Great Lakes basin to provide these estimates.

The dichotomous sampler is used for the collection of airborne particles for analysis of trace elements. Specific analytes of interest that will be collected from this sampler are listed in Table 1. The sampler operates for four 24-hour periods during each four-week sampling cycle. The flow rate through the sampler is 1 cubic meter per hour. This interval is used because of the need to collect about 100 cubic meters of air in order to get a reliable measurement of the target chemicals at the remote sites in the network. Because of the low concentrations, the operator must follow this protocol carefully to insure sample integrity.

The samples will be collected by passing air through a 37 mm Teflon filter. The sampler inlet is mounted in a standard Hi-Vol shelter. The filters are pre-weighed at the Illinois State Water Survey (ISWS), shipped to the site for collection of airborne particles, and returned to ISWS, weighed, and shipped to the U.S.EPA labs in North Carolina for analysis of the trace elements listed in Table 1 by X-ray fluorescence (XRF) methods. These methods are documented in laboratory SOPs.

The following procedure is used by the field operator to maintain the dichotomous sampler, and to remove and replace Teflon filters in a manner that will maintain sample integrity. Although a single composite sample will be collected every four weeks, the collector must be checked and reset each week to ensure proper operation and to collect samples on the prescribed sampling periods. Any questions on the sampling methods or operation of equipment should be directed to the following individuals. The Principal Investigator will be the prime contact for all methodological and general questions. The EPA Project Lead is the second contact if the Principal Investigator cannot be contacted.

Sampling Protocol and General Operations

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Supplies and Packaging

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Table 1. Trace Elements Determined on Teflon Filters

Aluminum	Silicon	Phosphorus	Sulfur	Chlorine
Potassium	Calcium	Titanium	Vanadium	Chromium
Manganese	Iron	Cobalt	Nickel	Copper
Zinc	Arsenic	Selenium	Bromine	Lead
Strontium	Tin	Iodine	Cadmium	

2.0 Summary of Method

Site operators will visit the site weekly to check for proper functioning of equipment and to set the sampler timer for the next prescribed sampling day. If it is raining or snowing, or hazardous conditions prevail, samples may be collected later in the day at the discretion of the site operator. If the sample can not be collected on the prescribed sampling day, the Principal Investigator must be notified. The following sampling activities will take place in the order listed.

- 1) Initial equipment inspection and testing.
- 2) Resetting the sampler timer (weekly).
- 3) Changing the Teflon filters (every four weeks).
- 4) Filling out the Sample Data Sheet (weekly).
- 5) Packaging filters and sample report form for shipment.
- 6) Installation of a new filters and setting flow rate.
- 7) Waste disposal and clean up.
- 8) Sample shipment.

Steps 1, 2, and 4 will be conducted weekly; Steps 1 through 8 will be conducted when the filters are changed (every four weeks). Each of these steps will be detailed in the following sections.

3.0 Sample Handling and Preservation

Due to the expense of sampling and analyzing the Teflon filters, a limited number of sites have been selected in order to achieve the goals of this study. Therefore, every sample is important and represents a significant portion of that site's yearly estimate. Any contamination through mishandling or lack of preservation could cause a bias in the program estimates. The filters are very fragile and should not be removed from the polypropylene filter holders. As the new filters are being installed, if a hole is discovered, the filter should not be installed but returned to ISWS.

Once in place, the filters should not be removed until the end of the sampling cycle (four 24-hour sampling periods over a four-week period). Follow all procedures for filter removal, packaging and shipment.

4.0 Interferences

Due to the nature of the chemicals being collected, all precautions should be taken to avoid contamination of the sample and sampler during weekly visits and sample collection. The sampler functions to collect samples of airborne particles that will be analyzed for trace elements. It is very important to avoid touching the filters and to prevent any dust or dirt from contaminating the deposit on the filter. The surfaces on the inlet should be inspected each week and any dust or dirt wiped away with a damp cloth.

5.0 Safety

In any field operation, emphasis must be placed on safety. Site operators must be aware of the potential safety hazards to which they are subjected. Follow all safety protocols and equipment guidelines, and be prepared for emergency situations. The site operator is responsible for his/her safety from potential hazards including but not limited to:

- Travel: When traveling to the site be sure to check on road conditions and weather advisories. Carry emergency supplies (warm clothing, food, water) when traveling in the winter. Always let someone know where you're going and when you expect to be back. Always carry a first aid kit.
- Electrical: For obvious problems (fire, scorching, blown fuses), turn off the power for the circuit involved and notify ISWS. Never attempt electrical repairs other than replacing fuses and circuit boards. Unplug the sampler before any replacements are made. Be especially cautious if conditions are wet.
- Insect pests: If you are allergic to insect stings, you should carry a kit prescribed by a physician. Be especially cautious if nests or large numbers of stinging insects are present. Notify ISWS of all problems.

6.0 Equipment and Supplies

Careful use, proper maintenance and cleaning extends the life of serviceable field equipment. Permission should be obtained from the Principal Investigator to use anything other than the equipment and supplies mentioned in this list (supplied by ISWS).

Serviceable Equipment

These items will stay at the site at all times.

- Dichotomous sampler (pump and timer unit, inlet shelter).
- Calibration filters in polypropylene holders.
- Pre-weighed Teflon filters in polypropylene holders in snap-lock Petri dishes.
- Kimwipes.
- Spare fuses.

7.0 Calibration and Standardization

The dichotomous sampler will be recalibrated quarterly against a mass flow meter by ISWS personnel. New rotameter settings will be marked on the instrument and entered in the log book along with the date of recalibration.

7.1 Sampler Inlet

Each week check the condition of the inlet surfaces. Wipe up any dust and dirt using a damp (DI water) Kimwipe.

7.2 Timer and Pump Unit

Figure 1 shows the timer. Each week check the operation of the timer and pump. The following checks should be made:

- 1) The time of day should be correct to the present local time.
- 2) The "Total Sampling Time" should have advanced 24 hours (1440 minutes) if a sample period was programmed during the preceding week.

Turn on the pump manually and let it run for one or two minutes. When the filters are changed every four weeks, reset the rotameter using the calibration filters (Section 8.4) before installing the new clean filters.

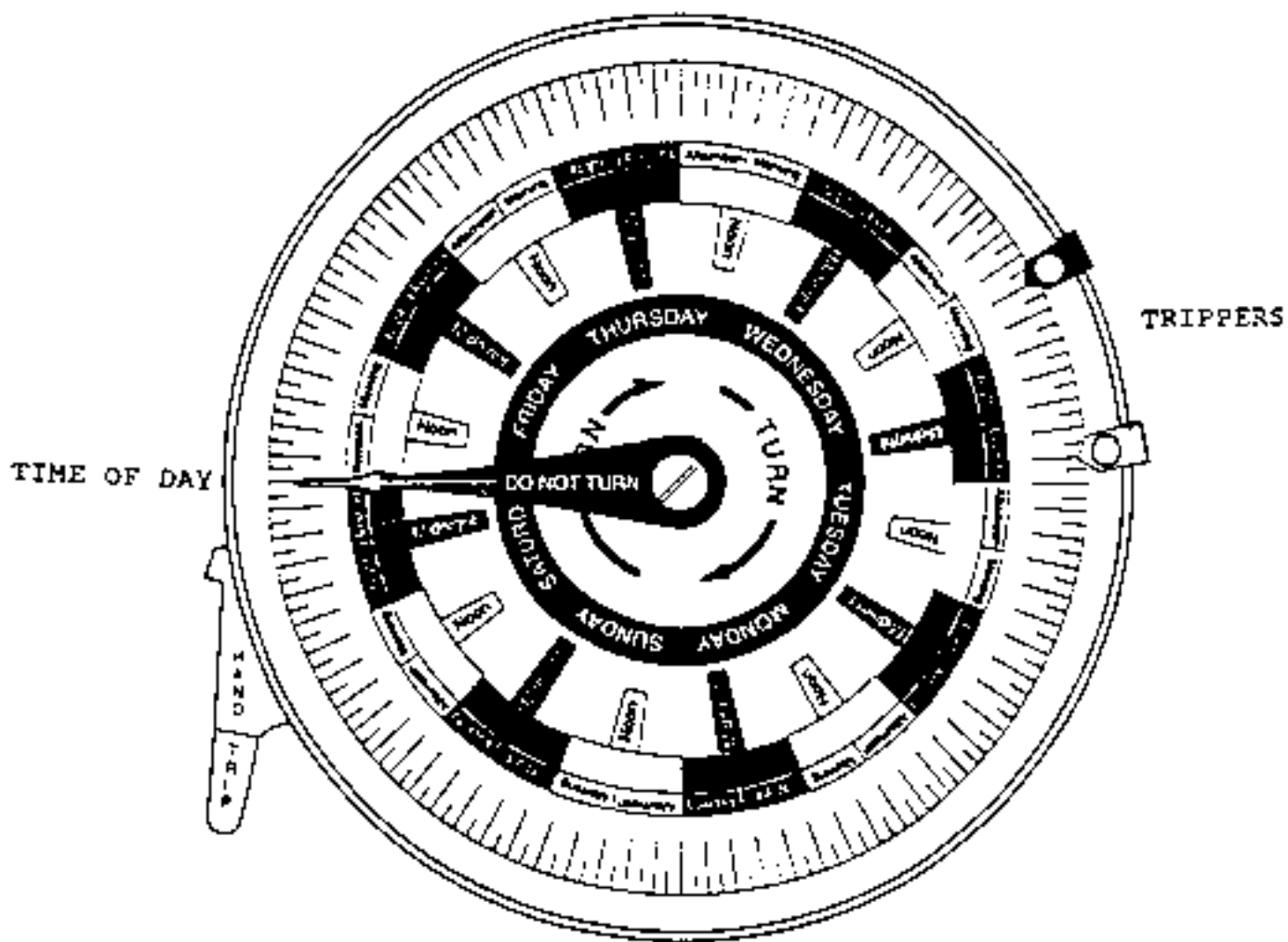


Figure 1. Mechanical Timer

8.0 Procedures

The following procedures will be discussed:

- 1) Initial Inspection.
- 2) Setting the clock and timer.
- 3) Filter removal and labeling.
- 4) Filter packaging for shipment.
- 5) Adjusting sampler flow rates.
- 6) Installation of new filters.
- 7) Setting the clock and timer.
- 8) Waste disposal/clean-up.
- 9) Sample shipment.

Steps 1 and 2 will be conducted weekly; Steps 1 through 8 will all be conducted every four weeks when the filters are changed.

8.1 Initial Inspection

Upon arrival at the site, make an initial inspection of the equipment to determine proper operation for the week. This procedure is accomplished every week. When a sample is set up, this procedure should be used to check final settings before leaving the site. Refer to Figure 1 for timer details. Check the elapsed time counter reading on the lower left corner of the timer. Record this number on the Sample Data Sheet. The counter reads in hundredths of an hour or minutes. The large red arrow should point to the correct day and time. Note any discrepancies on the Sample Data Sheet. The switch trippers should be firmly attached to the timer rim with the silver tripper at the last scheduled start time and the black tripper at the last scheduled stop time.

Turn on the sampler by moving the "Hand Trip" switch to the "On" position and note whether the pump is running normally. After two minutes, record the value on the rotameters on the Sample Data Sheet. Turn the sampler off after two minutes.

This inspection which should be entered onto the Weekly Site Visit Sheet and the Sample Data Sheet will include:

- 1) General comments. Comments that might affect the sample collection that week, i.e., fire in the area, wind storms, abnormal precipitation, vandalism, etc.
- 2) Equipment evaluation. Note any damage to equipment. If the sampler is not operating properly, notify ISWS as soon as possible.
- 3) Rotameter reading.
- 4) Total Sampling Time reading.

8.2 Setting the Clock and Timer.

If a sampling period is scheduled for the next week but no filter change is required, set the clock and timer at this point. Follow the instructions in Section 8.7.

8.3 Filter Removal and Labeling

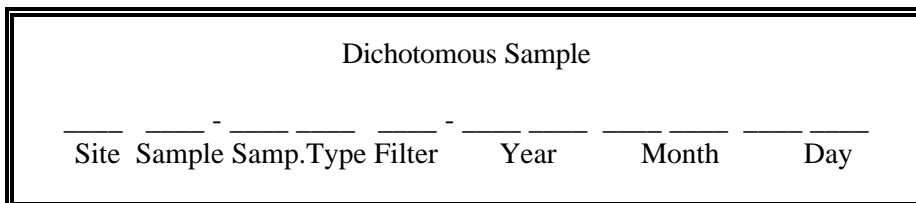
At the end of a sampling cycle, the filters are removed and replaced by the following procedure. It is extremely important that the filters not be touched, and should be placed in the snap-lock Petri dish as soon as possible. The following procedures are accomplished only during the replacement of filters and not every week.

- 1) Remove the two Teflon filters by unscrewing the locking nut (Figure 2). The filters must remain in their polypropylene holders. There will be a coarse particle filter in a yellow holder and a fine particle filter in a white holder. Place the each filter and holder in a separate snap-lock Petri dish for shipment. Be careful not to touch the filter.
- 2) Sample Labeling

All dichotomous (dichot) air samples should be lbeled on the outside of the Petri dish using the same alphanumeric system. The label includes:

- The “Site ID” letter for the site,
- the “Sample” which will always be “D” for dichotomous samples,
- the “Sample Type”, designating either a routine sample (01) or a QA sample (FB, TB),
- the “Filter” size designation, a “C” for course or an “F” for fine, and
- the “Date” of collection in a year-month-day format.

An example label and the valid codes are listed below.



Valid Codes

<u>Site ID</u>	<u>Sample</u>	<u>Sample Type</u>	<u>Filter</u>
U-Brule River	S-Sleeping Bear Dunes	D-Dichotomous	C-Coarse
C-Champaign	B-Beaver Is.	01- Routine Sample	F-Fine
N-Manitowoc	E-Eagle Harbor	FB- Field Blank	
W-Chiwaukee	T-Sturgeon Point	TB- Trip Blank	
V-South Haven	I-Indiana Dunes		
M-Muskegon	J-IIT Chicago		
L-Lake Guardian			

Example: SD-01C-930119 is the code for a routine dichot coarse particle sample collected at the Sleeping Bear Dunes site on January 19, 1993 (date filters are removed from the sampler).

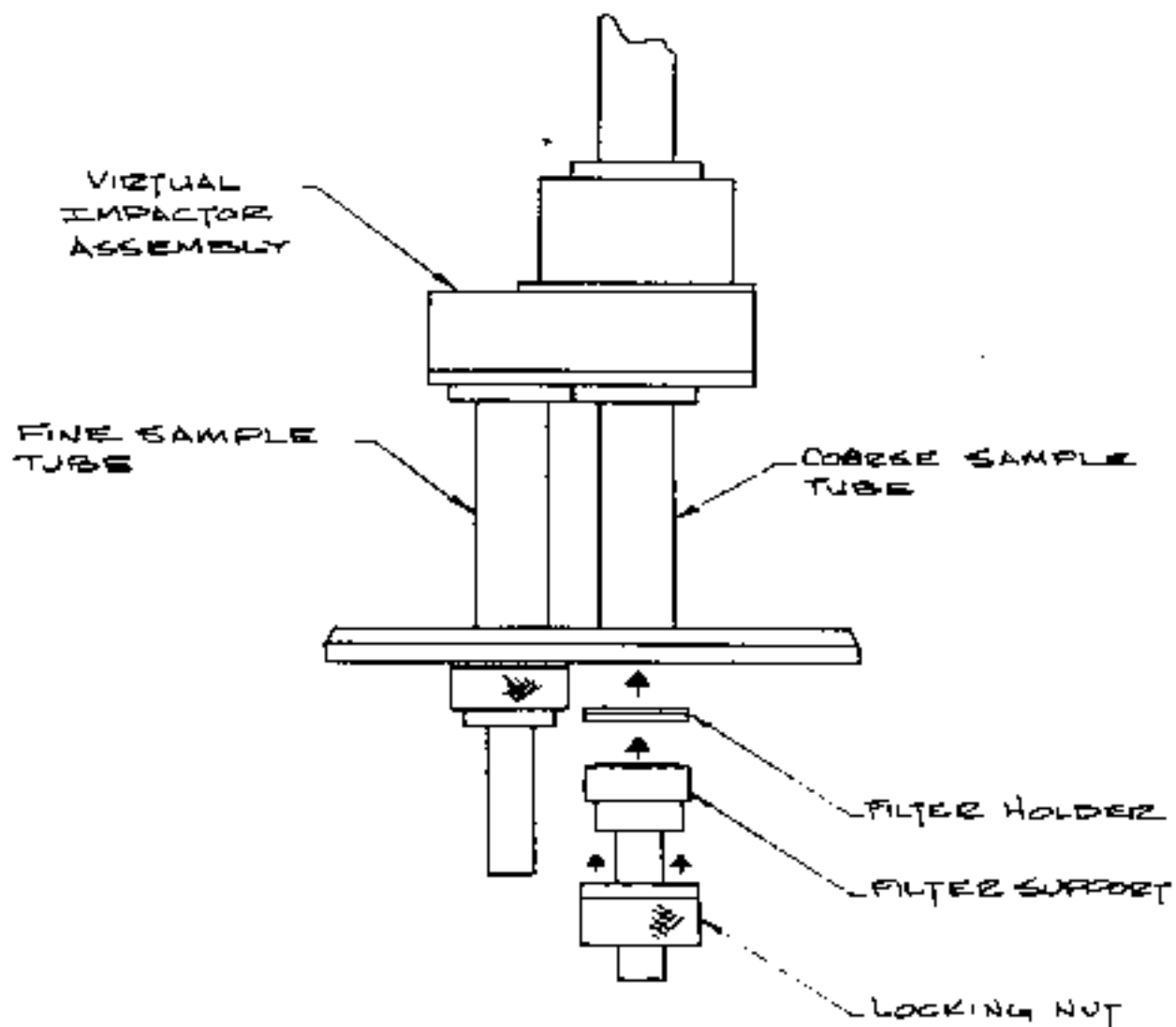


Figure 2. Schematic of the Dichotomous Sampler

8.4 Filter Packaging for Shipment

The filters in labeled Petri dishes should be shipped in a padded envelope or in a box with packing material. They may be shipped together with other samples.

8.5 Adjusting Sampler Flow Rates

8.5.1 Install the calibration filters (labeled side facing up) and tighten the locking nut (Figure 2).

8.5.2 Turn the sampler on using the hand trip switch (Figure 1) and allow it to warm up for at least 10 minutes.

8.5.3 Set the rotameters on the instrument to the most recent calibration set points. These should be marked on the instrument and entered into the site log. The set point on the rotameter scale should be lined up with the center of the metal ball using the adjustment knobs at the base of the rotameters. The rotameter on the left sets the flow to the coarse particle filter and the one on the right sets total flow. If the ball is stuck or there is some other problem with the rotameter, do not attempt to adjust it; but notify ISWS as soon as possible.

8.5.4 Turn off sampler and remove calibration filters.

8.6 Installation of New Filters

At the start of a new sampling cycle (every four weeks), fresh filters should be installed after the flow has been adjusted (Section 8.5).

8.6.1 Place new pre-weighed filters in their color-coded filter holders into the instrument. The labels should face up and the holder color should match the color patch on the instrument (yellow for the coarse position and white for the fine position). Once the filters are in place, tighten the locking nut. Be careful not to touch the filters themselves.

8.6.2 Set the timer for the next sampling period as described in next section.

8.7 Setting the Clock and Timer

8.7.1 Turn the large ring (Figure 1) clockwise so that the red pointer points to the correct day and time.

8.7.2 Attach the switch trippers to the timer ring (see Figure 1). The *silver*-colored tripper should be positioned at the start day and time and the *black* tripper on the end day and time specified in the monthly site protocol. The trippers should be attached so that the thumb screw is to the front. The screws should be hand tightened so that the trippers rest firmly against the rim of the ring.

8.7.3 Be sure to record the elapsed time reading on both the Weekly Site Visit Sheet and the Sample Data Sheet.

8.8 Waste Disposal Clean-up

Waste may include materials used to clean the inlet and packaging materials. Dispose of these properly.

8.9 Sample Shipping

Once they are properly labeled and packaged (Sections 8.3 and 8.4), send the samples, Sample Data Sheet, and Weekly Site Visit Sheet to the Principal Investigator. Keep a copy of both Sheets in the site log book. UPS 2nd day delivery is the preferred shipping method. U.S. Priority mail may also be used.

9.0 Quality Assurance Samples

Occasionally the protocol will require collection of quality assurance samples. Travel blanks are filters that are shipped with regular sample filters and stored at the site during the collection period. They should be returned to ISWS unopened after the specified period. Field blanks are filters that are installed in the sampler during the sampling period. These samples are run to assess contamination of the filters during periods when the sampler is not running. When field blanks are run the sampler should be unplugged. These samples should have a "TB" or "FB" in the sample code (Section 8.3). Specific instructions will be included in the Monthly Site Protocol with the requirements for these samples.

10.0 Equipment Maintenance and Trouble Shooting

The sampler is exposed to weather, and wind-blown dust and should be cleaned each week by wiping dirty surfaces with a clean damp cloth.

The operation of the sampler should be checked each week. If the pump does not run or there is a problem with the timer display, consult the trouble shooting guide below and contact ISWS. For more information, consult the site operator's manual or contact the manufacturer, Andersen Samplers Inc., 4215 Wendell Dr., Atlanta, GA, 800-241-6898. Table 2 includes some trouble shooting information.

Table 2. Trouble shooting

CAUSE	REMEDY
<u>Collector fails to operate</u> No power to instrument	Check switches and power source. Reset circuit breaker.
<u>Circuit breaker continues to break</u> Electrical short	Instrument needs servicing
<u>Operates for a short period then shuts off</u> Overloaded filter or plugged line	Check filters and lines. Call ISWS.

Dichot Air Sample Summary SOP

This summary does not take the place of the detailed SOP and should be used strictly to reinforce the procedure when in the field. Steps 1 and 2 will be conducted weekly; Steps 1 through 8 will be conducted when the filters are changed (every four weeks).

1.0 Initial Inspection

Upon arrival at the site make an initial inspection of the equipment to determine proper operation for the week. This inspection which will be entered into the site operators weekly activity sheet would include:

- 1.1 Comments on site and area conditions that might have affected the sample collection activity that week.
- 1.2 Determine whether the pump is operating properly by turning it on and allowing it to operate for two minutes. Record the rotameter and timer readings on the Sample Data Sheet and Weekly Site Visit Sheet.
- 1.3 Wipe clean the surfaces on the inlet.

2.0 Setting Clock and Sample Timer

This is done when a 24-hour sampling period is scheduled for the coming week and no filter change is required. Follow the procedure in Section 6.0.

3.0 Filter Removal and Labeling

- 3.1 Unscrew the locking nut (Figure 2) and remove the filters in their plastic holders being careful not to touch the filter. Place each filter directly into its own snap-lock Petri dish.
- 3.2 Label the Petri dish with the appropriate code (see Section 8.3).

4.0 Filter Packaging for Shipment

Carefully pack the filters in padded containers. Ship to ISWS as soon as possible.

5.0 Adjust Flow Rates

- 5.1 Install calibration filters.
- 5.2 Turn the pump on and let it warm up for at least 10 minutes.
- 5.3 Adjust the flows to the latest calibration set point using the adjustment knob at the bottom of the rotameters.
- 5.4 Turn off the pump and remove the calibration filters.

6.0 Installation of New Filters

- 6.1 Install new pre-weighed filters in the sampler. The labels on the filter holders should face up, and the holder color (yellow for coarse and white for fine) should match the color code patches on the sampler. Tighten the locking nut.
- 6.2 Attach the switch trippers to the timer ring (see Figure 1). The *Silver-colored* tripper should be positioned at the start day and time and the *Black* tripper on the end day and time specified in the monthly site protocol. The trippers should be attached so that the thumb screw is to the front. The screws should be hand tightened so that the trippers rest firmly against the rim of the ring.

7.0 Waste Disposal/Clean-up

Dispose of all trash properly.

8.0 Sample Shipping

Once packaged properly send the samples, the Weekly Site Visit Sheets for the month, the Sample Reporting Forms to ISWS via UPS or Priority Mail.

Appendix A
SAMPLE DATA SHEET

1. Station Name BRULE RIVER 2. Operator _____

3. Sample Start _____ End _____

Yr Mo Da Time Yr Mo Da Time

4. Sample Type _____ Sample Codes _____
Precipitation Column UP - - Total Vol: _____ L

TSP/TOC Filter UT - Filter ID _____

Sampler Timer End _____ Magnehelic End _____

- Timer Start _____ Magnehelic Start _____

- Set-up Date _____, + _____ days

Organics Filter UH - F- Filter ID _____

High Volume Sampler Cartridge UH - C-

- Timer End _____ Magnehelic End _____

- Timer Start _____ Magnehelic Start _____

Dichot Sampler Codes UD- UD-

- Filter IDs: Fine _____ Coarse _____

(T) 1st Timer end _____ Rotameters _____ (C) _____
start _____

(T) 2nd Timer end _____ Rotameters _____ (C) _____

start _____
(T) 3rd Timer end _____ Rotameters _____ (C) _____
start _____
(T) 4th Timer end _____ Rotameters _____ (C) _____
start _____

5. Comments on sample condition or site operation:

6. Date Shipped: _____
Yr Mo Da initials

Received: _____
Yr Mo Da initials

Appendix B

WEEKLY SITE VISIT SHEET

INSTRUCTIONS: Fill in all applicable space, enter general weather conditions (sunny, raining, etc.) and approximate values for weather variables. Enter “OK” after OPERATION for each sampler tested if the sampler is operating properly; if there is a problem, enter “X” and describe the problem at the bottom of the page. For the Hi-Vols and Dichots, fill in the TIMER, MAGNEHELIC, or ROTAMETER (Coarse and Total) readings in the appropriate spaces. For the MICs and metals AEROCHEM, enter the temperature inside the sampler and the approximate volume in the overflow container (MIC only). For all samplers, indicate with an “X” whether a sample was collected this week and if the sampler was set up for another run. Indicate with an “OK” whether the wind vane is pointing in the proper direction and whether the anemometer is turning.

SITE _____	DATE _____	TIME _____	
WEATHER _____	TEMP _____	WIND DIR _____	WIND SP _____
ORGANICS HIVOL #1	OPERATION ____	TIMER ____	MAGN ____
	Sample: Collected ____	Set up ____	
ORGANICS HIVOL #2	OPERATION ____	TIMER ____	MAGN ____
	Sample: Collected ____	Set up ____	
TSP HIVOL	OPERATION ____	TIMER ____	MAGN ____
	Sample: Collected ____	Set up ____	
DICHOT #1	OPERATION ____	TIMER ____	C ____ T ____
	Sample: Collected ____	Set up ____	
DICHOT #2	OPERATION ____	TIMER ____	C ____ T ____
	Sample: Collected ____	Set up ____	
MIC #1	OPERATION ____	TEMP ____	VOL ____
	Sample: Collected ____	Set up ____	
MIC #2	OPERATION ____	TEMP ____	VOL ____
	Sample: Collected ____	Set up ____	
METALS AEROCHEM	OPERATION ____	TEMP ____	
	Sample: Collected ____	Set up ____	
STANDARD AEROCHEM	OPERATION ____		
	Sample: Collected ____	Set up ____	
MET SYSTEM	WIND VANE ____	ANEMOMETER ____	

PROBLEMS AND GENERAL OBSERVATIONS:

OPERATOR _____