National Human Exposure Assessment Survey (NHEXAS)

Maryland Study

Quality Systems and Implementation Plan for Human Exposure Assessment

Emory University
Atlanta, GA 30322

Cooperative Agreement CR 822038

Standard Operating Procedure **NHX/SOP-F02**

Title: Collection, Storage, and Shipment of Indoor and Outdoor Air Samples for Metal, Pesticide, and PAH Analysis

Source: Harvard University/Johns Hopkins University

U.S. Environmental Protection Agency
Office of Research and Development
Human Exposure & Atmospheric Sciences Division
Human Exposure Research Branch

1. Title of Standard Operating Procedure

   NHEXAS Harvard/Emory/Johns Hopkins Standard Operating Procedures:
   F02 Collection, Storage, and Shipment of Indoor and Outdoor Air Samples for Metal, Pesticide, and PAH Analysis, Rev. 1.1.1

2. Overview and Purpose

   This Standard Operating Procedure describes the procedures to be used for collecting, storing, and shipping indoor air samples to be analyzed for metals, pesticides, and PAHs, and outdoor air samples to be analyzed for metals.

   A Black Box pumping unit will be used indoors; a programmable timer-controller activates two solenoid valves, which sequentially direct air flow through each sampler (one sampler for metals, one for pesticides/PAHs) for 10 minutes out of each 70 during one week. The SKC personal pump used outdoors (one sampler for metals only) is programmed to sample one minute out of every seven and uses no additional timer or solenoid valve. Both pumps will run at 4 LPM (liters per minute). Total sampling time for each sampler will be 24 hours; the sample air volume target will be 5.76 cubic meters. The elapsed time meter on the Black Box is activated by a pressure-sensitive switch downstream of the sampler, and should read a nominal 24 hours as calculated from the timer-controller initial and final readings.

   For metals (indoors and outdoors), using a Harvard Impactor (HI) with a 10-μm cutoff, air samples will be collected on PTFE filters. The samples will be stored, shipped, then extracted and analyzed for metal content in accordance with EPA Method 200.8 and/or 200.9.

   For pesticides and PAHs (indoors only), an URG PUF (polyurethane foam) sampler will be used. The samples will be shipped to Southwest Research Institute (SwRI) for extraction. SwRI will extract and analyze the filters for pesticides.

   The samplers will be set up by Field Technician 1 (FT1) on Day 1 of each Cycle, and taken down on Day 8.

3. Discussion

   Ambient air particles and aerosols can be a significant source of human exposure to toxic metals, pesticides, and PAHs. Any of these toxic substances may be generated indoors, or outdoors followed by partial or complete penetration indoors.

   Toxic metallic elements such as lead are associated with particles generated from paint sanding operations, sandblasting, or combustion of lead-containing fuels; many of these particles are in the inhalable-size range (less than 10 μm). Pesticides can be inhaled during and after application to crops, lawns, and gardens. PAHs are produced during incomplete combustion of organic materials such as fossil fuels and tobacco.

4. Personnel Responsibilities

Staff present at the Field Coordination Center (FCC) include the FCC supervisor (FCC-S), Assistant FCC Supervisor (FCC-AS), FCC Clerks (FCC-C), and Field Technician 1 (FT1). All staff are trained in each procedure and are fully qualified to implement all procedures at the FCC. The assignment of a specific staff member to a task is at the discretion of the FCC-S. Specific reference to individuals in this SOP, other than the FCC-S, should be viewed as representing any staff member drawn from the above-mentioned group. Activities assigned to the FCC-S require the intention of him or her. Such activities may be passed on to other staff only if oversight by the FCC-S is given.

4.1 Sampler Preparation

The Emory Exposure Assessment Group is responsible for all maintenance, primary calibration, and shipment of pumps, and for calibration of rotameters.

SwRI is responsible for preparing and assembling the unexposed URG PUF cartridges that will be used to collect pesticides and PAHs, and shipping packaged samplers (complete with protective housings) to the Field Coordination Center (FCC).

The Field Supervisor is responsible for assigning to Field Technicians the weekly tasks: which locations are to be sampled, when sampling begins, what samples are to be collected, and the sample IDs.

The FCC Supervisor (FCC-S) is responsible for assembling Harvard Impactors, labeling PUF samplers, programming indoor timers, and checking pump flow rates at the FCC before approving pumps for sampling. Both the FCC-S and the assistant FCC-S will inspect filters before the HIs are assembled. The assistant FCC-S is also responsible for other tasks as assigned by the FCC-S.

The FCC Clerk is responsible for receiving unexposed sample media from Emory, SwRI, or the manufacturer, and for preparing Field Packets (including printing ID labels and affixing them to forms) before sampling.

The Field Technician 1 (FT1) is responsible for checking equipment and paperwork at the FCC before going to the field; for checking the air flow calibration of the pump in the field; for keeping custody of samplers in the field; and for returning samplers, other equipment, and paperwork to the FCC.

4.2 Sample Collection

At the field site, the Field Technician 1 will select optimal sampler locations so that the samples collected will be representative of air breathed by adults, and the apparatus will be minimally obtrusive. S/he will set up the sampling devices, program the outdoor timer, and ensure that all equipment is operating within specifications. S/he will complete all paperwork in a timely way.

4.3 Storage

The FCC Supervisor, the Field Technician 1, and the Field Coordinator or his/her designate

are responsible for ensuring that sample integrity is maintained by using the best sample storage system possible. Related paperwork, particularly chain-of-custody and shipping forms, must be kept up-to-date as well as complete.

The FCC-S will receive unexposed filters (for metals) from the assistant FCC-S, and prepared URG PUF samplers (for pesticides and PAHs) from the Field Coordinator. Both parties will sign a chain-of-custody form recording each transfer.

The Field Technician 1 is responsible for setting up and taking down the sampling equipment in the field, making measurements, and delivering the exposed samplers to the FCC.

At the FCC after sample collection, the FCC-S will disassemble the Harvard Impactors, transfer the exposed filters to sealed containers, then transfer the sealed containers to the Field Coordinator for shipment to Emory for analysis. The FCC-S is also responsible for preparing PUF samplers for shipping.

The FCC Clerk will complete and transfer all associated paperwork to the FC.

4.4 Shipment

The FCC Clerk will:

- maintain an inventory of appropriate shipping containers, paperwork forms for all modes of transport, and the necessary tools and apparatus of a shipping/receiving area.
- assemble samples to be shipped, scan the bar codes, and prepare shipping lists for the Field Coordinator to inspect.
- copy, file, and ship forms.
- ship collected samples and field blanks, along with copies of necessary paperwork, from the FCC to the analytical laboratories.

The FCC Supervisor will:

- ensure that all paperwork, particularly chain-of-custody, is kept up-to-date.
- inspect samples and shipping lists before shipping.

4.5 Analysis

The Emory Trace Metals Laboratory will be responsible for analyzing all air samples for metals. Some sample extracts may be split for interlaboratory comparisons.

SwRI will be responsible for extracting samples and analyzing for pesticides and PAHs.

5. Required Equipment and Reagents

5.1 Field Coordination Center (for one home)

5.1.1 Before Field Sampling (day 1)

Metals	Pesticides and PAHs
3 Petri slides (Analyslides) for filters 3 "Harvard Impactor" particle samplers (HI) (2 + backup): 4 LPM (thin), 10 µm (red) 3 Teflon filters: Gelman RJP 041 (41 mm) 3 cellulose support pads, 37 mm (Millipore AP-10 or equivalent) 3 rectangular plastic filter holders w/snap rings 3 orange silicone gaskets for HI housing 4 orange silicone O-rings for HI nozzle and top 1 tube of silicone adhesive Millipore tweezers silicone lubricant spray	2 URG PUF 2000-25A samplers (1 + backup)

5.1.2 After Field Sampling (day 8)

Metals	Pesticides and PAHs
labeled Petri slides in plastic bags	shipping container cooler with dry ice

5.1.3 After Each Cycle (for cleaning HI sampler parts)

4 dishpans, labeled for washing and rinsing HI O-rings, impactor plates, and jet nozzles
deionized water
detergent, Liquinox or equivalent
wipes, Kimwipes or equivalent
brush
alcohol: ethanol, technical grade
impactor plate oil: mineral oil obtained from Marple, labeled for impactor plates
laboratory tape, 3/4" wide

5.2 Field Sampling

2 "Black box" flow-controlled pumping units (4 LPM) with solenoid valves, and preprogrammed timers (1 primary, 1 backup)
2 SKC personal pumps (1 primary, 1 backup)
3 electrical extension cords (25 ft.), outdoor rating
2 3:2 prong adapters, 1 3:1 outlet adapter
spare parts for pumps: 10 ft. rubber tubing, fuses (3/4 A 250-V Slo-Blo), mufflers, air filters
tapes: yellow CAUTION tape, laboratory tape, wide plastic tape, duct tape
plastic ties -- locking, black UV resistant, and white
clamps and connectors
tool belt with tools, including screwdrivers (slotted & Phillips), pliers, wrench (adjustable), hammer, wire cutter, scissors, knife or razor blade, markers, ruler
calibrated rotameter, 4 LPM (Matheson 604 or equivalent)

wristwatch (24-hour display optional but recommended)
2 temperature & relative humidity meters (maximum-minimum):
one to be left in outdoor pump box, one for indoor readings (can be used for every house)
gloves: unpowdered disposable gloves, cotton work gloves
plastic bags -- sealable (Ziplok or equivalent) -- 6" x 9", 9" x 12"
boxes: tote box ("each house"), tool box (with tool list)

Metals	Pesticides and PAHs
3 Harvard Impactors (HI) complete samplers, labeled (2 primary, 1 backup) HI flow adapter for measuring flow	2 URG PUF samplers, labeled rubber tubing for measuring flow cooler with cold pack to store exposed samplers (Day 8)

Outdoor	Indoor
tripod to hold sampler at height of 3-6 feet pump box (cooler with hole for cord and tubing) rain cover for HI (plastic bucket with holes and tiedown) insulating foam for window through which extension cord is run	TASS (telescoping air sampler support) to hold samplers at height of 3-6 feet protective floor mat (carpet sample)

5.3 Sample Tracking and Paperwork

11 sample ID labels with sample type 11 (outdoor air: metals): 3 on FCC logsheets, 3 on field logsheets, 1 on chain-of-custody form, 2 on sampler, 2 spare
11 sample ID labels with sample type 13 (indoor air: metals)
11 sample ID labels with sample type 14 (indoor air: pesticides & PAHs)
Field Packet for household: 3 logsheets (FCC, outdoor air, indoor air), 3-part carbonless, 3 chain-of-custody forms (outdoor metals, indoor metals, indoor pesticides/PAHs), maintenance record sheets for timers
Cycles 2-8: copies of previous Cycle house and yard plans (kept by Field Technician 2 for dust soil sampling)
Field Manual (SOPs)
clipboard and paper
pens, ballpoint (black or blue, and red or green)
marker

6. Procedure

6.1 Preparation for Collection

Only complete and sealed sampler assemblies will be transported between the FCC and the sampling location. Loading and unloading of filters will be done at the FCC only.

6.1.1 Preparing ID Labels

At the FCC, the FCC Clerk will:

< Print ID labels and inspect them to make sure that they are correct for the household, Cycle, and sample types.

< Affix a "11" ID label to each of the 3 layers of the Outdoor Air logsheet.

< Affix a "13" ID label and a "14" label to each of the 3 layers of the Indoor Air logsheet.

< For each of the three chain-of-custody forms, check the box indicating the sampling medium and affix an ID label.

6.1.2 Section deleted with 15 April 1996 modification.

6.1.3 Preparing, Assembling and Labeling Harvard Impactors Filters

At the FCC, the FCC-AS will:

< Put on unpowdered gloves.

< Visually inspect each filter for integrity. Check that the lot number is the standard lot number. If not, record it on the logsheet. Place the labeled Petri slides in a plastic bag and seal it. Keep them to use after the filter is exposed.

< If the filter is defective because of holes, it can be used as a blank. If it is defective for another reason, place it in a designated container to be returned to the manufacturer for exchange. Obtain another filter.

< If the filter is acceptable, assemble the rectangular plastic filter holder:

÷ place a rectangular holder with the circular recess facing up.

÷ Remove the filter from the Petri slide and center it in the recess.

÷ snap a retaining ring into the recess.

÷ carefully place a 37-mm cellulose backing pad on top of the filter in the recess.

< Get the HI base and HI body with ID number matching the filter ID. Assemble the HI as shown in Figure 1:

÷ check the body for correct placement and alignment of the 10 μm jet fitted with a downstream impactor plate inside the air inlet.

÷ invert the HI aluminum base and center the shoulder on the top of the backing pad inside the plastic retaining ring. Use gentle pressure and a slight circular motion to help "lock" the shoulder inside the ring while maintaining the fit of the plastic holder. Then place it upright, maintaining the lock.

÷ place the HI top on the base, aligning the pegs and their corresponding holes using alignment scribes as needed. Be careful not to misalign the plastic filter holder.

÷ lock the top to the base using the side "snap-lock" lever tabs and check integrity both visually and mechanically.

< Place the assembled HI in a sealable plastic bag and seal.

< Remove and discard the gloves.

- If the HI is being prepared in advance with no designated HIN, then store it in the appropriately labeled shelf and proceed to 6.1.4. Otherwise:

- Just before transferring the HI to FT1, affix two identical labels to each of 2 HIs (sample type 11 on one and sample type 13 on the other). A third HI with no label will serve as a spare. Obtain 3 Analyslides and affix to them labels that match those on the HI. Note that one Analyslide (corresponding to the spare) will not have a label.

- Obtain the corresponding Chain(s)-of-Custody and Logsheet and record all requested information including filter Lot No. Affix Id labels.

- Transfer custody to FT1 by completing the COC and Logsheets as needed.

- Note that for stored HIs, the FCC-S will keep custody of the samplers until they are transferred to FT1.

6.1.4 Labeling PUF Samplers

At the FCC, FCC-S will label two PUF samplers as follows:

- Get two PUF samplers from the storage area. Ensure that each end is firmly sealed with a plastic cap. (See Figure 2.)

- Record the sampler lot number on the Indoor Air logsheet.

- Attach 2 identical ID labels to one PUF sampler: sample type 14. (The other sampler is the spare.)

- Place each sampler in a plastic bag and seal the bags. The FCC-S will keep custody of the samplers until they are transferred to FT1.

6.1.5 Checking Pump Flow Rates

At the FCC, FCC staff will test Black Box pumps and SKC pumps:

- Plug each pump directly into a 110v outlet, close the lid, and let the pump warm up for at least1 hour for Black Box pumps and at least 10 minutes for SKC pumps.

- Check the flow rate with a calibrated rotameter. The black glass ball reading should be within 0.2 LPM small divisions of the rotameter's target value of 4.0 LPM.

- If the pump does not fall within specification, label it "to be reset or repaired" and place it in the area designated for equipment to be repaired; obtain and test another pump.

6.1.6 Setting the Timer for the Indoor Air Sampler

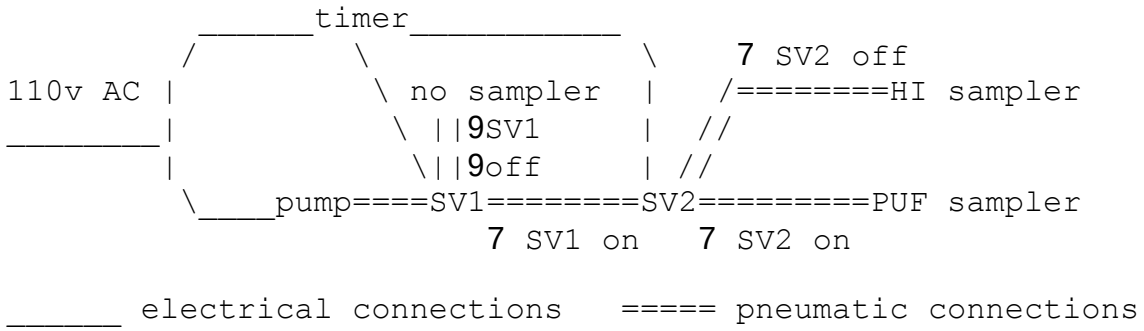
Programming is normally done at the FCC by the FCC Supervisor. However, FT1

should be able to program a timer in the field if necessary. Record all programming and maintenance on the Timer Check Record for each timer.

Batteries and plug must be disconnected for at least 5 minutes for the timer defaults to be reset. Definitely do this if the timer is not working properly, for example not reatianing the program, or has not been used for more than 14 days. A fresh lithium battery retains the program memory for at least 3 months.

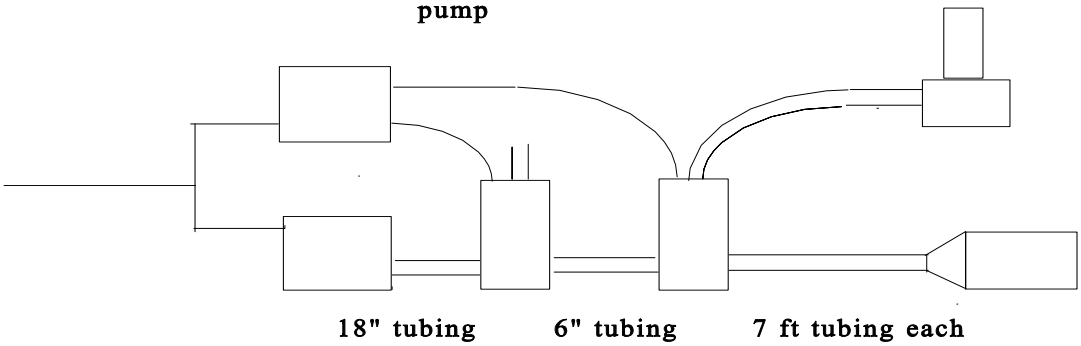
The Chrontrol Timer allows two samplers to be sequentially activated by individual solenoids for 10 minutes every 70 minutes. The pump is turned on four hours before the sampling sequence begins and remains on for the full week, avoiding warm-up errors.

The following diagram shows the arrangement of the indoor set of samplers. When solenoid valve sv1 is off, air flows through the pump without passing through either sampler. When sv1 and sv2 are both on, air flows through the PUF sampler. When sv1 is on and sv2 is off, air flows through the HI sampler.



The timer activates sv1 for 20 minutes and sv2 for 10 minutes. For the first 10 minutes, sv1 and sv2 are both activated; air flows through the PUF sampler, through sv2, then through sv1 to the pump. For the second 10 minutes, sv2 is shut off; air flows through the HI sampler, through sv2, then though sv1 to the pump. Then the timer shuts off sv1 so that flow continues from ambient through sv1 to the pump for 50 minutes. This cycle repeats once every 70 minutes.

In the black box, connect the rubber tubing from the pump to solenoid valve 1, port 2. Connect solenoid valve 1 port 3 to solenoid valve 2, port 2. In the field the FT1 will connect the samplers to solenoid valve 2: the PUF sampler to port 1 and the HI sampler to port 3.



6.1.6a Programming the Timer (Indoor Air Sampler)

For the following programs, first make sure the timer's battery and line cord have been disconnected for at least 5 minutes to reset the timer's defaults. Open the cardboard cover. Insert the plug into the outlet. The display should read 1200. Enter the current time and date as follows:

Time:

1. Press TIME. The display will go blank.
2. Set the current time in hours and minutes, e.g. for 2:45 PM press 2 4 5.
3. If the time is PM press the AM/PM key.
4. Press ENTER.

Date:

1. Press DATE. 301 is displayed.
2. Set the current month and day, e.g. for Jan. 5 press 1 0 5.
3. Press SECOND/YEAR. 00 is displayed.
4. Type the last two digits of the current year, e.g. for 1995 press 9 5.
5. Press ENTER. The time is displayed.

Key in each of the following programs sequentially, i.e., the first program completely, then the second, third, fourth, and fifth, as shown below.

PROGRAM 1	*PROGRAM 2*	*PROGRAM 3*	*PROGRAM 4*	*PROGRAM 5*
ENTER **1** **PROGRAM** **2** **PROGRAM** **5** **ON** **INTERVAL** **400** **ENTER** **TIME**	**ENTER** **2** **PROGRAM** **3** **PROGRAM** **4** **ON** **INTERVAL** **0** **ENTER** **CYCLE** **70** **ENTER** **TIME**	**ENTER** **3** **CIRCUIT** **1** **INTERVAL** **20** **ENTER** **TIME**	**ENTER** **4** **CIRCUIT** **2** **INTERVAL** **10** **ENTER** **TIME**	**ENTER** **5** **PROGRAM** **2** **OFF** **INTERVAL** **400** **DATE** **7** **ENTER** **TIME**

6.1.6b Checking the Timer (Indoor Air Sampler)

To make sure that the program is operating, make sure that the timer is attached properly to the solenoid valves. Key in

PROGRAM
2
ON

The lights for circuits 1 and 2 should go on and the solenoid valves should click.

If they do not, unplug the battery and line cord for at least 5 minutes. Plug in the line cord. (The display should read 1200.) Attach the battery. Enter the time, date, and programs 1-5 above. Key in **PROGRAM 2 ON** again. The lights for circuits 1 and 2 should go on and the solenoid valves should click.

If the lights and valves work properly, key in

PROGRAM	
2	
OFF	
CIRCUIT	
1	
OFF	(The solenoid valves click and the corresponding light goes off.)
CIRCUIT	
2	
OFF	(The solenoid valves click and the corresponding light goes off.)
TIME	(The current time should be displayed.)

To start the timer, key in

PROGRAM
1
ON

When you are sure that the program is operating properly, close the cardboard cover and tape it down securely to prevent accidental keypresses if the box is jostled.

6.1.6c Testing a New or Questionable Timer (Indoor Air Sampler)

To test a new or questionable timer, make sure that the solenoids are connected properly. Disconnect the timer's battery and line cord for at least 5 minutes to reset the timer's defaults. Key in the following set of test programs:

PROGRAM 1	*PROGRAM 2*	*PROGRAM 3*	*PROGRAM 4*	*PROGRAM 5*
ENTER	**ENTER**	**ENTER**	**ENTER**	**ENTER**
1	**2**	**3**	**4**	**5**
PROGRAM	**PROGRAM**	**CIRCUIT**	**CIRCUIT**	**PROGRAM**
2	**3**	**1**	**2**	**2**
PROGRAM	**PROGRAM**	**INTERVAL**	**INTERVAL**	**OFF**
5	**4**	**0**	**0**	**INTERVAL**
ON	**ON**	**SEC**	**SEC**	**400**
INTERVAL	**INTERVAL**	**2**	**1**	**DATE**
0	**0**	**ENTER**	**ENTER**	**7**
ENTER	**ENTER**	**TIME**	**TIME**	**ENTER**
TIME	**CYCLE**			**TIME**
	0			
	SEC			
	7			
	ENTER			
	TIME			

Activate the timer's programs by keying in **PROGRAM 1 ON**. The timer should go

through a 7-second cycle: both solenoid valves click on, then after one second sv2 clicks off, then after another second sv1 clicks off, then after five more seconds both click on. If the timer performs the test programs properly, disconnect the line cord and battery for at least five minutes and then program it as in section 6.1.6a. If it does not, give it to the FCC-S to be checked again and, if necessary, returned to the manufacturer for repair.

6.1.7 SKC Pump for Outdoor Air Sampler

6.1.7a Setting the Timer of the SKC Pump

The timer on the SKC pump used outdoors must be set in the field by the Field Technician 1. See section 6.3.1.

6.1.7b Setting the Flow Rate of the SKC Pump

FCC-S will test each SKC pump as in section 6.3.1. If the flow rate is not within the acceptable range, adjust it as follows:

1. Prepare the test pump by attaching a latex or Tygon tube from it to the FCC calibrated rotameter 001. Attach the battery charger.

2. Remove the protective plastic cover by loosening the retaining screw then gently lifting off as described in the SOP F02 section on programming the pump.

3. With the accompanying tool kit flip the pump switch ON and allow 10 minutes for warm-up.

4. While observing the reading at the center of the black glass ball in the rotameter, adjust the flow rate by turning the adjusting screw with the special tool provided, until the rotameter ball reads the target value corresponding to 4.0 LPM. Wait 5 minutes. Readjust if necessary. Repeat this step until the reading is stable for at least 15 minutes.

5. Record the rotameter reading, the pump ID, the date, your initials, and the SKC rotameter reading in the pump calibration logbook.

6. Record the SKC rotameter reading, initials, and date on a piece of lab tape and secure it to a convenient location on the pump. REMEMBER this rotameter reading is "ballpark" only, to be used as an aid when convenient.

7. Flip the pump switch to the OFF position and replace the protective plastic cover with the screw.

8. Repackage the pump and label it ready for field use. Store it appropriately.

6.1.8 Transfer and Inspection of Equipment

Before going to the field, FT1 will:

< Receive custody of the samplers from FCC-S. Both will sign the chain-of-custody form.

< Check the FCC logsheets to make sure that all preparation has been done. File the logsheets in the folder for the household and Cycle.

< Make sure that all equipment and paperwork is present and that the ID labels are correct. Pack the tote boxes to go to the field.

6.2 Selection of Sampling Location

6.2.1 Cycle 1

At the respondent's home, FT1 will:

< Select suitable locations for the samplers.

Outdoor	Indoor
The bases of selection include a reasonably expansive area representative of unrestricted air flow (e.g., not near a tree, a large rock, a wall, or a fence), an area in which inhabitants and friends do not congregate, and one that is not subject to unusual air contaminants, e.g. car exhaust.	The sampler will be placed in the activity room. The bases of selection include an area representative of unrestricted air flow, e.g., a non-traffic area, at least 6" away from a wall, and one that is not subject to unusual air contaminants (such as a cooking stove vent or a heated air register) nor near an air cleaner.

< Discuss the locations with the respondent to make sure that the equipment will not be in the family's way, either now or later in the year. Remind the respondent that the samplers will be placed in the same location in every Cycle.

< Mark the sampler locations as (A) on the yard plan (outdoors) and activity room plan (indoors) kept by FT2.

< Check the "Cycle 1" box on the logsheet.

6.2.2 Cycles 2-8

< Check the location marked on the yard or house plan from the previous Cycle. If the location is still available, use it. Check "Same" on the logsheet.

< If the location is not available (e.g., because the respondent has moved furniture or built a tool shed there), choose a new location nearby and note the change on the logsheet. Also note the change on the plan as A*, using red or green ink.

6.3 Sample Collection Procedure

6.3.1 Day 1, beginning the one-week sampling period:

< Record the pump ID, rotameter ID, FT1's name and signature, and the date on the

logsheet.

< Set up the sampler support and plug in the pump to let it warm up for at least 30 minutes:

Outdoor	Indoor
<Set up the tripod. <Set the pump storage box on the ground under the tripod. <Plug the pump into an extension cord, and the extension cord into a 110v line. Use yellow CAUTION tape on electrical cords as needed.	<Put a carpet square face down on the floor. Set the black box on the carpet square. <Attach the TASS (telescoping air sampler support) to the black box. <Plug the pump into a 3:1 outlet adapter and the adapter into an extension cord, keeping the adapter inside the black box. <Plug the extension cord into a 110v line. Use yellow CAUTION tape on electrical cords as needed. <Make sure that the fan in the black box is working. If not, check the electrical connection at the side of the fan toward the back of the black box.

< During the 30-minute warmup period for the pump, attach the sampler(s) to the support:

Outdoor	Indoor
< Attach the HI sampler to the top of the tri	< Attach the sample mounting bar to the TASS. Adjust the TASS so that the top of the HI sampler will be about 5 feet off the floor. < Attach the HI sampler to the sample mounting bar with the thumbscrew-nut combination. The HI should point upward. < Attach the PUF sampler to the sample mounting bar with cable ties. The input (slightly conical) end should point

pod with the eyebolt.	toward the pole, and the air inlet should be at the end of the bar. Allow enough space between the sampler and the pole to attach the hose. Leave the bubble wrap and bag in the black box.

< Make sure that the timer is properly programmed:

Outdoor	Indoor
< Program the	< Check the program by the procedure in section 6.1.6b. If necessary, reprogram the timer according to section 6.1.6a. < When the timer is properly programmed, set it to turn on by pressing: **PROGRAM** **1** **ON**

Outdoors -- Instructions for programming SKC timer (must be done in the field)
1. Using the larger of the two screwdrivers provided, unscrew the screw that holds the plastic cover over the touch pad; this screw is permanently attached to the cover, so do not try to remove it from the cover. Grasping the screw, gently lift then lower the cover free. 2. Use the screwdriver carefully to move the pump switch to ON. 3. Press HOLD. 4. Press SET-UP. SET-UP & DELAYED START are displayed. Rightmost digit blinks 0.
5. Press DIGIT SELECT once. Second digit from right blinks 0. 6. Press DIGIT SET four times. Second digit from right blinks 4. 7. Press DIGIT SELECT once. Third digit from right blinks 0. 8. Press DIGIT SET two times. Third digit from right blinks 2.
9 Press MODE. SET-UP & SAMPLE PERIOD are displayed. Rightmost digit blinks 0. 10. Using steps similar to 5.-8., set the display to 9999.
11. Press MODE. SET-UP & PUMP PERIOD are displayed. Rightmost digit blinks 0. 12. Using steps similar to 5.-8., set the display to 1440.
13. Check the program by pressing MODE four times to observe the displays. 14. The displays are in order: 0240 DELAYED START 9999 SAMPLE PERIOD 1440 PUMP PERIOD 0240 DELAYED START
15. Press START to activate the program.

< Check the pump flow rate:

Outdoor	Indoor
< Press HOLD to pause the timer program. Press FLOW CHECK. < Check the SKC pump 4 LPM flow rate with a calibrated rotameter; the tolerance is "0.2 LPM. (The acceptable rotameter range is written on the side of the rotameter case.) Record the reading as "test flow." The SKC pump's internal rotameter cannot be used to make this measurement. However, it has a mark to indicate the expected reading at 4 LPM. < If the pump is out of specification, use the spare pump. Check the "backup system" square on the logsheet, cross out the ID number of the pump with one line, initial it, and write the ID number of the backup pump. Cross out and initial the test flow, and measure and record a test flow for the backup pump. < Remove the sealing caps from the HI and connect the outlet to the pump. Wait a few minutes. Check the flow rate, using the sampler adapter. Record the rotameter reading. If the rotameter reading is outside the acceptable range, then connect the spare sampler and recheck. < If either sampler is damaged, replace it in its plastic bag and seal the bag. Mark the bag DAMAGED. Use the backup sampler. < When you are sure that the pump and HI are performing acceptably, press FLOW CHECK and then START to restart the program.	< Check the black box pump 4 LPM flow rate with a calibrated rotameter; the tolerance is "0.2 LPM. (The acceptable rotameter range is written on the side of the rotameter case.) Connect the rotameter to port 1 of SV1. Record the reading as "test flow." < If the primary black box pump is out of specification, the backup system must be used. Check the "backup system" square on the logsheet, cross out the ID number of the pump with one line, initial it, and write the ID number of the backup pump. Cross out and initial the test flow, and measure and record a test flow for the backup pump. < For each sampler, remove the sealing caps or plugs and connect the sampler's downstream outlet directly to the pump. Wait a few minutes. Check the flow rate, using the appropriate sampler adapters. Record the rotameter reading. If the rotameter reading is outside the acceptable range, then connect the spare sampler and recheck. Disconnect

< Replace the plastic protective cover and secure it with the attached screw.	the sampler from the pump. < If either the HI or the PUF sampler is damaged, replace it in its plastic bag and seal the bag. Mark the bag DAMAGED. Use the corresponding backup sampler.

< Connect the sampler(s) to the pump:

Outdoors	Indoors
< Connect the HI sampler to the SKC pump.	< Connect the samplers to solenoid valve 2: the PUF sampler to port 1 (side of valve, red tape on tubing) and the HI sampler to port 3 (top of valve).

< Secure the tubing so as not to present a hazard.

< Record the time (24-hour clock), elapsed timer reading, temperature, and relative humidity on the logsheet. Reset max/min temperature memory by depressing MAX until is displays, then press Reset; do the same for MI. To reset the relative humidity, depress BOTH MAX and MIN buttons simultaneously, then releases. Check every setting by depressing first the temp MAX button and confirming that it reads ambient temperature, then the remaining buttons. If it does not read ambient temperature, repeat this procedure until it does. Place the temperature-relative humidity meter in the Black Box. Note any observations that may affect the sample.

Outdoors	Indoors
< Attach the rain cover to the sampler. < Close the pump box and make sure that the lid is in the “locked” position. < Use 2 inch wide postal or wrapping tape to “seal” the lid from weather. Ensure that the tape helps to secure the rubber hose in a downward fashion at the entrance of the cooler, but does not kink it, and helps to deflect ant droplets during a windy rainstorm.	< Check that the black box lid is closed and latched.

6.3.2 Day 8, ending the one-week sampling period:

< Record your (FT1's) name and signature, the date, time, elapsed timer reading, temperature, and relative humidity on the logsheet.

< Read the max/min temperature and relative humidities by depressing first the temp MAX button until MAX is displayed then record the reading on the logsheet; repeat procedure for other three parameters (min temperature and max/min humidity).

< Perform and record a final flow measurement of each sampler:

Outdoors	Indoors
< Perform and record a final flow measurement of the HI sampler by attaching the rotameter to it.	< Perform and record final flow measurements of the HI and PUF samplers by attaching the rotameter to each sampler. You may have to attach either or both of the samplers momentarily to sv1 port 3 to obtain the final flow rate.

< Record each reading as "Day 8 rotameter reading" for the appropriate sampler. Note on the logsheet if the final flow was not within 10% of 4 LPM.

< Record any anomalies observed. Use the flags and codes in the following table. (Flags refer to readings of time and flow rate; codes deal with sampler integrity issues.)

< If the table says “VOID the sample,” write VOID on the logsheet and chain-of custody form. The sample will not be analyzed.

< If you are not sure which flag or code applies, describe the problem on the logsheet.

Field Data Flags, VOIDs, and Codes

Flags are to be assigned by computer at the time of data processing. These are to be determined based on information supplied by the field team as well as calculations based on the data as entered. The issues raised will be subsequently evaluated by the QA and Project Management teams.

Codes indicating field problems are to be assigned by the field teams. These codes are a shorthand substitute for the field technicians written notes describing the circumstances, and are based upon common field problems noted in other studies over the years. They are to be recorded in addition to the data and must be recorded and initialed by FT1. Data as collected must also be recorded. For example, id the elapsed timer failed, the FT1 would record that observed Day 8 elapsed time reading but would also include the appropriate code (995) in the logsheet.

Flag	*Meaning*
0	Both duration and rotameter "off" readings are within the acceptable ranges: 20-28 hours and Low-High respectively.
1	Duration is not within the range of 20-28 hours.
2	Rotameter "off" reading is not within the range of Low-High.
VOID	*Meaning*
9	Both duration and rotameter "off" readings are not within the acceptable

	ranges: **VOID** the sample
98	Flow rate is outside valid data limits (not between 3.2 and 4.8 LPM):
99	**VOID** the sample Duration is outside valid data limits (duration is not 18-30 hours): **VOID** the sample
Code	*Meaning*
880	Local conditions noted by operator/sample specific source
884	Field operator error affecting data e.g. switched or missing filters; sample run on wrong date
885	Inlet problems e.g. inlet plugged: **VOID** the sample
886	Filter distorted or holes observed at removal time
887	Filter mishandled or ripped in field
888	Miscellaneous operator error e.g. late
894	Filter wet
897	Data recording error in flow readings or timer readings; data value inferred
898	Miscellaneous operator error: **VOID** the sample
991	Insects in the assembly
992	Hose fell off or leaked, pump failed
995	Timer malfunction
996	Power failure
997	Preparation laboratory error, e.g. filter omitted, labeling error
998	Miscellaneous problem noted, flag the sample.
999	Miscellaneous problem noted, **VOID** the sample.

< Make sure that the ID labels are still affixed to the samplers. If not, affix correct labels.

< Disconnect the sampler(s) from the pump:

Outdoors	Indoors
< Disconnect the HI from the pump and the tripod. Leave the bolt or screw attached to the tripod. Replace the sealing caps on the HI to maintain sample integrity. Seal the HI	< Disconnect the PUF sampler from the pump and the TASS. Be careful when you cut the cable ties; they may spring away and could damage the respondent's property. Wrap the PUF sampler securely in bubble wrap and seal it into a plastic bag. Transfer custody to the Interviewer, who will transport it to the FCC with the food sample. < Disconnect the HI from the pump and the TASS. Store the HI attaching bolt in the tool box. Replace the sealing caps on the HI to maintain sample integrity. Seal the HI in a plastic bag and place it in the sampler carrier.

in a plastic bag and place it in the sampler carrier.	

< Unplug the pump to shut it off.

< Take down the tripod or TASS. Pack away all equipment.

< Return the equipment to the FCC and transfer custody of the HIs and equipment to the FCC Supervisor.

6.4 Labeling of Samples

A unique ID number will be assigned for each sample (see SOP G03 "Identification Numbers"). Printed labels will show the ID number in bar-code and human-readable format. The field technician will affix identical labels to the sampler, the logsheet, and the chain-of-custody form.

6.5 Preservation and Storage

6.5.1 HI Sampler

At the FCC, the FCC Supervisor will package the filters from the two HI samplers:

< Get the Petri slides with ID labels corresponding to the filters in the HI samplers. Check the ID numbers. Put each slide with the corresponding HI.

< Put on gloves.

< Carefully disassemble a sealed HI.

< Check that the filter was well secured into the plastic holder. Using Millipore tweezers, remove the filter by holding it by the edge. Inspect the filter for holes, uneven discoloration, spots that are or have been wet, and other anomalies. If there are any problems, note them on the logsheet.

< Place the filter in the appropriately labeled Petri slide with the edge of the filter touching the inner wall of the dish. Put the lid on so that the inner edge of the lid catches the edge of the filter and prevents it from moving.

< Place the slide with the filter in a plastic bag. Transfer the remaining ID label from the HI to the bag. Check that the bag contains the indoor and outdoor air filters from the correct household in the correct Cycle.

< Transfer custody of the bag of filters to the Field Coordinator, signing the chain-of-custody form to record the transfer.

- < Remove the impactor plate. Clean the red impactor with a damp wipe.

- < Spray the O-rings with silicone lubricant.

- < If this was not the last sampling week of a Cycle, recycle the impactor plate by doing a Level B cleaning as follows:
  - ÷ Use a wipe to rub off the material collected during sampling. Apply a small drop of impactor plate oil. Allow the oil to penetrate into the porous stainless steel disk for about half a minute. Fold a wipe in half and press it onto the oiled disk, then inspect the wipe to see if any oil came off the disk. If a significant amount of oil is transferred to the wipe, blot again with a clean section of the wipe. Repeat the blotting until very little oil is transferred.
  - ÷ Wipe the bottom edge of the impactor plate with an ethanol-moistened wipe and place it on a clean wipe to dry.
  - ÷ When the impactor plate is dry, put it back in the impactor (with the oiled surface up) and reassemble the upper part of the sampler.
  - ÷ After handling impactor plates (even after they have been cleaned), wash your hands before handling other pieces.

- < If this was the last sampling week of a Cycle, place it in a container designated for impactor plates to be given a Level A cleaning. Obtain a clean impactor plate and reassemble the upper part of the sampler.

- < Remove the gloves.

6.5.2 Cleaning and Preparing O-Rings

At the FCC, the FCC Supervisor will:

- < Clean O-rings after each Cycle, after a maximum of four uses.

- < Wash O-rings in plastic dishpan with deionized water and Liquinox. Use a dishpan designated for washing HI O-rings only. Rub each one gently by hand to remove traces of grease.

- < Rinse thoroughly with deionized water. Use a dishpan designated for rinsing HI O-rings only. After handling used O-rings (even after they have been cleaned), wash your hands before handling other pieces. Spread out the O-rings on wipes to dry. Place them in a container labeled for clean O-rings.

6.5.3 Cleaning and Preparing Impactor Plates (Level A cleaning)

At the FCC, the FCC Supervisor will:

- < Clean and prepare impactor plates after each Cycle, after a maximum of four uses.

- < With dry wipes, wipe as much excess grease from the fritted surface as possible. The fritted surface should become lightly tacky to the touch. Wash in a plastic dishpan with deionized water and Liquinox, using a dishpan and brush designated only for washing

HI impactor plates. Place the impactor plates in the custom made holder and put it in the ultrasonic bath and sonicate for 15 minutes. Rinse thoroughly with deionized water, using a dishpan designated only for rinsing HI impactor plates.

< Place each impactor plate in a shallow container filled with ethanol to approximately half the height of the impactor plate. This helps remove any grease remaining on the walls of the impactor plate. The fritted surface must never come in contact with ethanol. After dipping, place on wipes to dry. After handling impactor plates (even after they have been cleaned), wash your hands before handling other pieces.

< Prepare the impactor plates: Apply a small drop of impactor plate oil. Allow the oil to penetrate into the porous stainless steel disk for about half a minute. Fold a wipe in half and press it onto the oiled disk, then inspect the wipe to see if any oil came off the disk. If a significant amount of oil is transferred to the wipe, blot again with a clean section of the wipe. Repeat the blotting until very little oil is transferred.
< Wipe the bottom edge of the impactor plate with an ethanol-moistened wipe and place it on a clean wipe to dry. Stack each two plates with the oiled surfaces facing each other. Stack four pairs of plates and put 3/4" wide laboratory tape around the circumference to hold them together. Put them in the container labeled for clean impactor plates.

6.5.4 PUF Sampler

At the Field Coordination Center, the FCC Supervisor will:

< Place the sampler in a shipping container. Transfer the 2 remaining ID labels to the shipping container.

< Transfer custody of the sampler to the Field Coordinator, signing the chain-of-custody form to record the transfer. The FC will store the sampler on dry ice until it is shipped to SwRI.

< If the primary sampler was used, remove the ID labels from the bag containing the backup sampler and transfer custody of the backup sampler to the FC, who will store it to be used on another day.

< If the backup sampler was used because the primary sampler was damaged, transfer custody of the damaged sampler to the FC, noting on the chain-of-custody form that it is damaged. The FC will return the sampler to SwRI for cleaning, repair, and reassembly.

6.6 Handling and Shipping

6.6.1 Metal Samples

Unexposed filters are stored in their original plastic containers until HIs are assembled for field use. Exposed filters in Petri slides will be stored in a safe secure cool location at the FCC; although refrigeration is not needed, care should be taken that the samples are not exposed to high temperatures.

At the end of each two-week sampling subcycle, samples to be analyzed for metals will be shipped or transported to the Emory laboratory by 2-day delivery. They will be packaged in sealed plastic bags and packed with adequate bubble-wrap and/or other cushioning material.

6.6.2 Pesticide/PAH Samples

Unexposed PUF samplers are stored at ambient temperature until they are taken to the field. Exposed samplers are kept in a cooler with dry ice while being transported from the field to the FCC, and then stored in the freezer until they are shipped for extraction and analysis.

During every 2-week sampling period, exposed samplers will be shipped to the SwRI lab twice by overnight delivery: about half on Wednesday evening of the second week and half on Monday of the following week. They will be packed in a cooler with dry ice; the cooler will be packed with adequate bubble-wrap or other cushioning material.

6.7 Laboratory Analysis

At the Emory Trace Metals Laboratory, samples will be prepared using EPA Method 200.8 as a guideline. This will involve extended digestion in one or more concentrated or diluted mineral acids followed by dilution to an optimal volume for analysis by GF-AAS. Any analyses by another laboratory will be done according to EPA Method 200.8 or a documented equivalent. See the appropriate laboratory (L) SOPs for more information.

Samples to be analyzed for pesticides and PAHs will be extracted by SwRI within 10 days of collection. The filter and PUF plug will be analyzed together. Extracts will be analyzed for pesticides and PAHs by SwRI using GC/MS.

6.8 Data Workup

Field and laboratory data will be returned to Emory in both magnetic and hardcopy format. Data will be coded and checked, computer entry verified, and discrepancies resolved. Analytical results will then be merged with questionnaires and other data, using the ID number as the merge parameter.

6.9 Sample Tracking

The ID number will allow tracking of each sample. A data base management system will ensure knowledge of the status and location of any sample at any time including retrospectively.

The chain-of-custody form will accompany the sample wherever it goes. Anyone who receives, transfers, or ships the sample will sign and date it, and keep a photocopy. It must clearly contain all necessary information so that the custody of the sample can be determined at any time. Airbills, bills of lading, etc., are acceptable substitutes when a commercial or government carrier is used; copies of such bills will be attached to the chain-of-custody form.

7. Quality Assurance Procedures

7.1 Laboratory and Field Blanks

An additional 4% of the total number of planned exposure filters and PUF samplers will be used for field blanks. A field blank will be carried through procedures identical to the sample but will never be attached to an operating sample pump; filters will be mounted in an HI and samplers will be attached to a tripod or TASS, then removed, to mirror the physical processes that the actual sample undergoes. Like exposed samples, field blanks will be transferred to containers, stored for a time equivalent to a real sample, then shipped to the laboratory with the exposed media.

Blank filters and cartridges will be from the same lot as sample media analyzed at the same time. In the lab the blanks will be analyzed identically to the exposed filters. Results from the blanks will allow for correction due to any contamination or interference in either the sampling system or the filter itself.

7.2 Duplicate Sampling

2% of all samples will be taken in duplicate. The entire set of duplicates encompassing all Cycles will selected using a random number generator. Unlike the blank samples, each duplicate will require an additional independent pumping system. The pump will undergo the same checks as the sample pump.

To reduce respondent burden, when duplicate indoor air samples are needed, duplicate metal and pesticide/PAH samples will be taken at the same time, using one pump.

Duplicate data will be used to establish the precision associated with a given sample as well as to provide information on the precision associated with the technique in this project. The latter value can be compared to other studies.

7.3 Tolerance Limits

The tolerance limits for the black box pump flow rate are "5% before sampling and "10% after sampling as given in Section 6.3. These limits have been established over a long period of time with much experience in many studies that have used the black box pumping system. They reflect not only what can be expected from the pump and its associated mass flow controller, but also the precision of the calibrated rotameter that is used to measure the flow rate. The rotameter is calibrated over a relatively narrow range only: for use at 4.0 LPM. It can discriminate to 0.05 LPM minimally, or approximately 1% of the targeted flow.

Sampling time is checked by recording both the reading of the elapsed timer and the clock time. The elapsed timer reading, in 0.1 hour units, is the usual way of calculating the exposure time. The sampling exposure time must be within 20% of the intended time; otherwise the sample may not be comparable with others.

For both flow rate and time, an established list of flags and codes will be employed to document any observed anomalies. The logsheet will contain an area for notes by the field technician. S/he is encouraged to write down anything that may be questionable or noteworthy because it may allow for a later explanation of otherwise puzzling data.

All instruments and scientific apparatus used will be calibrated with standards and instrumentation that are traceable to the pertinent NIST standard. Both primary standards (e.g. traceable soap bubble flowmeters) and transfer standards (e.g. spirometers and rotameters) may be used. We will obtain manufacturers' or suppliers' specifications for NIST traceability for any instrumentation that we are not equipped to certify.

The table below shows expected concentrations and equivalent limits of detection given the sampling protocol.

Category	Pollutant	Expected Concentration		Expected Limit of Detection Given Sampling Protocol
		Indoor	Outdoor	
Metals	Arsenic (As)	10 ng/m^3	15 ng/m^3	4.1 ng/m^3
	Cadmium (Cd)	34 ng/m^3	34 ng/m^3	0.41 ng/m^3
	Chromium (Cr)	38 ng/m^3	22 ng/m^3	0.8 ng/m^3
	Lead (Pb)	33 ng/m^3	26 ng/m^3	6.0 ng/m^3
Pesticides	chlordane	40-300 ng/m^3		12 ng/m^3
	chlorpyrifos	5-400 ng/m^3		26 ng/m^3
	4,4'-DDD	0 ng/m^3		1.8-5.3 ng/m^3
	4,4'-DDE	1-5 ng/m^3		1.4-3.6 ng/m^3
	4,4'-DDT	0.2-1 ng/m^3		2.2-4.1 ng/m^3
	dieldrin	1-10 ng/m^3		0.5-3.3 ng/m^3
	heptachlor	0.3-200 ng/m^3		0.5-3.1 ng/m^3
	malathion	1-20 ng/m^3		18 ng/m^3
PAHs	anthracene	3.0 ng/m^3		31 ng/m^3
	benzo(a)pyrene	1.0 ng/m^3		19 ng/m^3
	chrysene	2.3 ng/m^3		14 ng/m^3
	phenanthrene	18.0 ng/m^3		12 ng/m^3

8. References

EPA Method 200.8: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry 4/91

EPA Method 200.9: Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrophotometry 4/91

EPA Method TO13: Determination of Polynuclear Aromatic Hydrocarbons (PAHs) in Ambient Air Using High Volume Sampling with Gas Chromatography/Mass Spectrometry (GC/MS) 6/88

Quality Assurance for Environmental Measurements, ASTM Special Publication 867, Taylor, J. K. and Stanley, T. W., editors, 1985.

Manual for the 4.0 and 10.0 LPM Indoor Air Sampler: July 1987
Harvard School of Public Health, Exposure Assessment and Engineering Program

Calibration Procedures For Rotameters Revision 0 March 11, 1994 Harvard School of Public Health, Exposure Assessment and Engineering Program

NHEXAS Harvard/Emory/Johns Hopkins Standard Operating Procedures:
G03 Identification Numbers
G04 Chain-of-Custody and Sample Tracking
G05 Storage and Shipping of Samples
L06 Extraction of Metals from Sampling Media
L07 Analysis of Metals by GF-AAS
L08 Analysis of Metals by ICP-MS
L09 Preparation of Exposure Media (PUFs and Quartz Fiber Filters) for Air Samplers for Pesticide and PAH Collection
L10 Extraction of Neutral Pesticides and PAHs from Sampling Media
L14 Determination of Pesticides, Acid Herbicides, and PAHs by GC/MS

Timer Check Record Timer number _____

Record all programming and other checks.

Date: Name: Signature:	Check:
Date: Name: Signature:	Check:
Date: Name: Signature:	Check:
Date: Name: Signature:	Check:
Date: Name: Signature:	Check:
Date: Name: Signature:	Check:

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Black Box Maintenance Record Black Box number: _____ Timer: 1-week 2-week

Service Date	Technician	Rotameter ID	Rotameter Reading	Flow Rate LPM	Autotimer Check	Elapsed Timer Check

Service Date	Technician	Muffler Replaced?	Pump Filter Replaced?	Final Pot. Setting	Triac Replaced?	Photocell Replaced?	Volt. Reg. Replaced?	Other Maintenance Notes

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