



# 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-F03

Title: Collection, Storage, and Shipment of Personal Air Samples

for Metal 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

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), partially funded and collaborated in the research described here. This protocol is part of the Quality Systems Implementation Plan (QSIP) that was reviewed by the EPA and approved for use in this demonstration/scoping study. Mention of trade names or commercial products does not constitute endorsement or recommendation by EPA for use.

# 1. Title of Standard Operating Procedure

Harvard University/Johns Hopkins University Standard Operating Procedures: F03 Collection, Storage, and Shipment of Personal Air Samples for Metal Analysis, Rev. 1.0

# 2. Overview and Purpose

This Standard Operating Procedure describes the procedures to be used for collecting, storing, and shipping personal air samples. Personal air samples will be collected on cellulose ester membrane filters using a Personal Exposure Monitor (PEM<sup>TM</sup>) with a 10 µm cutoff at 4 LPM (liters per minute). The target individual in each household will use the sampler for 24 hours, starting on Day 1 and finishing on Day 2. The sampler will be given a Level B cleaning and the filter changed; a sampler may be used as many as three times during each two-week subcycle, after which it is given a Level A cleaning.

The samples will be stored, shipped, then extracted (the filter is consumed) and analyzed for metal content in accordance with EPA Method 200.8 and/or 200.9; and other analytes may be determined.

#### 3. Discussion

Deposition of ambient air particles in the lungs can be a significant source of human exposure to toxic metals. Toxic metallic elements such as lead are associated with particles generated from sanding operations, sandblasting, or combustion of lead-containing fuels, to name a few. Many of these particles are in the inhalable-size range (less than 10 micrometers).

A personal air sampler may provide a more representative sample of human exposure to particulate matter than an indoor or outdoor air sampler. However, it requires dedicated involvement of the subject and may have more subtle potential interferences. The PEM<sup>TM</sup> is attached to a shoulder strap of a small waist pack; the pack houses the pump, battery, electronic flow control, and noise insulating padding. Modest shielding of the PEM<sup>TM</sup> will reduce the sampling of clothing fibers immediately adjacent to the PEM<sup>TM</sup>.

# 4. Personnel Responsibilities

#### 4.1 Sampler Preparation

The Field Coordination Center Supervisor (FCC-S) is responsible for preparing and assembling the PEM<sup>TM</sup>s, checking for leaks, and measuring the flow rate. Loading and unloading of filters will be done only at the Field Coordination Center (FCC); only complete PEM<sup>TM</sup> assemblies will be transported between the FCC and the sampling location.

The FCC Clerk is responsible for preparing Field Packets (including printing ID labels and affixing them to forms) before sampling.

The Field Technician 1 (FT1) is responsible for checking the PEM<sup>TM</sup>s and having all necessary tools, equipment, and paperwork ready before going into the field.

# 4.2 Sample Collection

FT1 is responsible for setting up and testing the PEM<sup>TM</sup> at the respondent's residence, and assisting the Interviewer if needed.

The Interviewer is responsible for instructing the target individual in the use of the PEM<sup>TM</sup>. The Interviewer must ensure that the target individual understands the proper operation and positioning of the sampling device and also how to keep a record of the exposure time and anomalies.

The Phlebotomist is responsible for measuring and recording the flow at the end of sampling, and for returning the PEM<sup>TM</sup> to the FCC for disassembly.

# 4.3 Storage

FCC-S is responsible for disassembling the PEM<sup>TM</sup> at the FCC after it is collected by the Phlebotomist, and for transferring custody of the exposed filter to the Field Coordinator (FC).

The Field Technician and the Interviewer are responsible for ensuring that sample integrity is maintained by using the best sample storage system possible. Paperwork completion, particularly chain-of-custody and shipping forms, must be kept up-to-date as well as complete. This is important especially as collected samples are transferred from one individual to another.

# 4.4 Shipment

The Field Coordinator (FC) is responsible for inspecting collected samples, field blanks, and paperwork being sent from the FCC to the analytical laboratory. S/he must also receive unexposed sample filters from the laboratory. The FC must also ensure that all paperwork, particularly chain-of-custody, is kept up-to-date.

The FCC Clerk is responsible for copying and filing forms, and shipping collected samples and field blanks along with necessary paperwork from the FCC to the analytical laboratory.

# 4.5 Analysis

The Emory analytical laboratory will be responsible for analyzing all air samples. Some sample extracts will be split for interlaboratory comparisons.

# 5. Required Equipment and Reagents

#### 5.1 Field Coordination Center

# 5.1.1 Before Field Sampling

Petri slides (Analyslides) for filters 37 mm Teflon filter, 0.80 µm pore size (Millipore AA or equivalent) gloves, unpowdered Millipore tweezers 2 Personal Exposure Monitor (PEM<sup>TM</sup>): 10 μm and 4 LPM (primary and backup) wipes, Kimwipe or equivalent screwdrivers: one Phillips, one with 2 prongs for PEM<sup>TM</sup>, 2 slotted (large & small) calibrated rotameter, 4 LPM (Matheson 604 or equivalent) black box pump, 4 LPM PEM<sup>TM</sup> flow adapter cap with attached Balston particle filter silicone lubricant spray 3 resealable plastic bags, Ziplok or equivalent, about 3" x 6" 2 pump bags and straps 2 battery packs and batteries ethanol, technical grade silicone vacuum grease

#### 5.1.2 After Field Sampling

gloves, unpowdered
Millipore tweezers
washing solution (Liquinox)
sonicator
dishpans and brushes
impactor plate oil: mineral oil obtained from Marple, labeled for impactor plates

# 5.2 Field Sampling

# 5.2.1 Day 1 (Field Technician 1 -- setup)

2 samplers (primary and backup), each consisting of:

Casella pump (modified for noise reduction) with bag, straps, tubing battery pack with 8 lithium batteries

PEM<sup>TM</sup> with filter, sealed in bag (section 6.1)
tools, e.g. screwdrivers, pliers, wrench calibrated rotameter, 4 LPM (Matheson 604 or equivalent)
PEM<sup>TM</sup> flow adapter cap, sealed in bag (section 6.1)
polyurethane foam for padding/sound reduction wipes, Kimwipe or equivalent spare parts for pump and PEM<sup>TM</sup>
wristwatch or equivalent

#### 5.2.2 Day 2 (Phlebotomist -- pickup)

PEM<sup>TM</sup> flow adapter cap calibrated rotameter resealable plastic bag

# 5.3 Sample Tracking and Paperwork

12 sample ID labels with sample type 15 (personal air): 3 for FCC logsheet, 3 for field logsheet, 1 on chain-of-custody form, 1 for Petri slide, 1 for PEM<sup>TM</sup>, 1 for PEM<sup>TM</sup> bag, 2 spare

Field Packet for household: FCC and field logsheets (3-part carbonless), chain-of-custody form Field Manual (SOPs)

clipboard

pens, ballpoint

#### 6. Procedure

# 6.1 Preparation for Collection

# 6.1.1 Preparing ID Labels

At the Field Coordination Center, 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 3 ID labels to the 3 layers of the FCC logsheet, 3 to the 3 layers of the field logsheet, and one to the chain-of-custody form. Place the other labels in a plastic bag, seal the bag, and clip it to the FCC logsheet.

#### 6.1.2 Preparing Filters

At the FCC, the Field Coordinator or his designate will:

- ➤ Obtain a Petri slide and an ID label with sample type 15, and the other digits appropriate for the household and Cycle. Affix the ID label to the slide. Open the slide.
- Record the lot number of the filters on the FCC logsheet.
- ➤ Put on gloves.
- ➤ Open the box of filters. Using Millipore tweezers, remove a filter from between the paper separators by holding the filter by its edge. Hold the filter up to the light and check it for integrity.
- ➤ 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 place to be returned to the manufacturer for exchange.

- ➤ If the filter is acceptable, place it in the Petri slide and close the lid. Store labeled filters in a designated area to be distributed when needed.
- 6.1.3 Assembling and Labeling PEM<sup>TM</sup>

At the Field Coordination Center, the FCC Supervisor (FCC-S) will:

- ➤ Write your name and signature and the date on the FCC logsheet.
- ➤ Receive a filter (in a Petri slide, with ID label) from the Field Coordinator. Both FC and FCC-S will sign the chain-of-custody form recording the transfer. Check that the ID label is correct.
- ➤ Obtain a PEM<sup>TM</sup> (clean, in a plastic bag).
- > Put on gloves.
- ➤ Make sure that the top and bottom of the PEM<sup>TM</sup> have the same number. Record the number on the FCC logsheet. Affix an ID label to the outside of the base.
- ➤ Visually inspect the filter for integrity.
- ➤ If the filter is defective, return it to the FC to be used as a blank or returned to the manufacturer for exchange. Obtain another filter from the FC.

If the filter is acceptable, assemble the PEM<sup>TM</sup> as follows:

- ➤ Place a stainless steel screen into the body of the PEM<sup>TM</sup>.
- ➤ With Millipore tweezers, gently grip the edge of the filter and place it on top of the screen.
- ➤ Place the impactor on top of the filter. Place an O-ring on top of the impactor. Make sure that all components are aligned.
- ➤ Place the top of the PEM<sup>TM</sup> in position. Insert and start the screws. Alternating between the two screws, tighten them very firmly but not excessively.
- ➤ Place the labeled Petri dish in a plastic bag and seal it. Keep it to use after the filter is exposed.
- ➤ If the PEM<sup>TM</sup> is not to be leak-checked soon, seal it into a plastic bag.

# 6.1.4 Leak-Checking PEM<sup>TM</sup>

At the Field Coordination Center, FCC-S will:

➤ Write your name and signature and the date on the FCC logsheet.

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➤ Get a 4-LPM rotameter, a 4-LPM black box pump, and a clean PEM<sup>TM</sup> adaptor with attached Balston particle filter.

- ➤ Turn on the pump and let it run for 60 minutes. With the rotameter, ascertain that the flow rate is 4 LPM. Adjust if necessary. For each PEM<sup>TM</sup>:
  - (a) Check the pump flow rate (at the tubing).
  - (b) Slip the adapter onto the PEM<sup>TM</sup>.
  - © Attach the pump tubing to the PEM<sup>TM</sup> using an adapter. If it is tight, use a little silicone spray on the adapter.
  - (d) Connect the rotameter to the Balston filter via tubing.
  - (e) Check the flow rate (with PEM<sup>TM</sup> attached). Record the flow rate on the FCC logsheet.
    - ➤ If the rotameter reading with PEM<sup>TM</sup> attached is not within 2 small divisions of the rotameter reading with the pump alone, then an air leak exists. Carefully check all connections for leaks, and reseal where needed.
    - ➤ If the PEM<sup>TM</sup> flow rate is still lower, disassemble the PEM<sup>TM</sup>. Check the order and placement of the components. Look for ring crimping. Check for dimples in the stainless steel screen that extend to the rim where the compression of the impactor plate seals together the filter sandwich. If examining the interior of the PEM<sup>TM</sup> risks contaminating the filter, then reassemble the PEM<sup>TM</sup> using new interior parts and a new filter.
    - Recheck the flow rate as above.
  - (f) Seal the PEM<sup>TM</sup> assembly into a resealable plastic bag. Affix an ID label to the bag.
  - (g) Seal the adapter cap into a resealable plastic bag.

# 6.1.5 Assembling Pump and Bag

At the Field Coordination Center, FCC-S will:

- ➤ Attach the two battery packs to the lead clips of the pump electronics module. Each battery pack contains four 9V lithium batteries.
- ➤ Enclose the pump in soundproofing foam. Place it with the battery packs and module into the padded waist bag, and close the zipper.
- Make sure the rubber tubing attached to the pump unit is not crimped by the zipper.
- > Clean the naugahyde shield of the shoulder strap with ethanol and a wipe.
- Turn the pump on. Let it warm up for 15 minutes. Check the flow rate as follows:

- $\rightarrow$  Attach a rotameter to the pump. Make sure that the flow is within  $\pm 2$  divisions of the target flow.
- → If the flow rate is unacceptable, mark the pump TO BE REPAIRED, put it in the area designated for pumps needing repair, and get another pump. Test it as before.
- → If the flow rate is acceptable, record the flow rate and the pump ID number on the logsheet. Remove the rotameter and turn the pump off. Pack the pump and PEM together.

# 6.1.6 Transfer and Inspection of Equipment

At the Field Coordination Center, FT1 will:

- Receive custody of the sampler from FCC-S. Both will sign the chain-of-custody form.
- ➤ Check the FCC logsheet to make sure that all preparation has been done. File the logsheet in the folder for the household and Cycle.
- ➤ Make sure that all 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

The PEM<sup>TM</sup> will be located just below the target individual's collarbone. The shoulder strap has a continuous strip of soft velcro sewn to it. The PEM<sup>TM</sup> attaches to this. The Interviewer can position this; be prepared to improvise if necessary.

The personal pump will be carried in a padded waist bag with an adjustable shoulder strap. This allows the target individual (TI) to position the bag where it is most comfortable with respect to the TI's build or activity.

# 6.3 Sample Collection Procedure

# 6.3.1 Day 1

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At the respondent's home, the FT1 will:

- ➤ Record the date, the time (24-hour clock), the PEM<sup>TM</sup> ID number, the rotameter ID number, and the pump's elapsed timer reading on the field logsheet.
- ➤ Use the rotameter to measure the pump flow rate. Record it on the field logsheet as "test flow."
- Remove the adaptor cap from its bag and wipe it out with a clean wipe. The adaptor cap should be kept facing up and away from fibers (i.e., not face down on a rug).
- Connect the adaptor cap via Tygon tubing to a calibrated rotameter.

- ➤ Remove the PEM<sup>TM</sup> from its bag. Save the bag with its ID label to be used when the PEM<sup>TM</sup> is collected.
- Lubricate the edge of the adaptor cap with a very light film of silicone spray or grease. Attach the adaptor cap to the inlet-nozzle section. Connect the PEM<sup>TM</sup> to the pump tubing. Check the 4 LPM flow rate with a calibrated rotameter; tolerance is  $\pm 3.2$  divisions of the rotameter's target reading for 4 LPM.
- ➤ After 5 minutes, check the flow rate again: it must remain within 3.2 divisions of 4 LPM. Replace the adaptor cap in its bag.
- ➤ If the flow rate is out of range, the backup system must be used. Check the "backup system" box on the logsheet. Measure the flow rate of the backup pump twice at 5-minute intervals to make sure that it is operating properly.
- ➤ When all equipment is functioning properly and flow rates have been taken and recorded, inform the Interviewer that it is ready.

The Interviewer, with FT1's assistance if necessary, will:

- ➤ Demonstrate to the target individual (TI), by holding the sampler and bag in front of your body, the approximate locations where the equipment will ride.
- ➤ Show how the sampler should be set up for nighttime sleeping. The TI will take the sampler off and place it on a bedside table or on a chair near the bed, so that it is within 4 feet of the TI's face and at head height. Explain that for swimming or contact sports, the sampler should be placed at the sidelines on a chair. When bathing or showering, the sampler should be placed on a chair outside the bathroom door.
- ➤ Fasten the waist strap of the carry bag around the TI's waist. Fasten the shoulder strap (where the PEM<sup>TM</sup> will be) across the shoulder opposite the TI's dominant hand: for example, across the left shoulder of a right-handed person. Tighten the waist and shoulder straps so that the carry bag weight is supported most comfortably.
- ➤ Show the TI that s/he can shift the carry bag around the waist strap so that it rides in the most convenient location based on the TI's build and activities. The shoulder "crescent" (material with velcro) should be centered across the top of the shoulder.
- ➤ When the carry bag and strapping are stable, place the PEM<sup>TM</sup> on the shoulder strap velcro at or just below the collar bone. Tie wrap the carry bag zipper closed.
- ➤ Check the flow rate. If it is out of range, repeat the tests.
- Record the date and time on the logsheet.
- ➤ Give the TI the instruction sheet, and make sure that s/he understands it.

At the FCC, the FT1 will:

➤ Leave the field logsheet, the plastic PEM<sup>TM</sup> bag (with the ID label) and the adaptor cap

(in its bag) for the phlebotomist to use the next day.

➤ On the chain-of-custody form, write that it was transferred to "target individual" (not the person's name). Copy the chain-of-custody form and leave the original for the phlebotomist to use the next day.

# 6.3.2 Day 2

At the FCC, the Phlebotomist will:

➤ Pick up the chain-of-custody form, field logsheet, PEM<sup>TM</sup> bag (with ID label), the adaptor cap (in its bag), and the rotameter.

At the respondent's home, the Phlebotomist will:

- ➤ Record the date, the time (24-hour clock), the rotameter ID number, and the elapsed timer reading on the logsheet.
- > Remove the adaptor cap from its bag and wipe it out with a clean wipe. The adaptor cap should be kept facing up and away from fibers (i.e., not face down on a rug).
- ➤ Connect the adaptor cap via Tygon tubing to the rotameter. Lubricate it with a very light film of silicone spray or grease if necessary.
- ➤ Remove the PEM<sup>TM</sup> from the strap. Attach the adaptor cap to the inlet-nozzle section. Check the 4 LPM flow rate with the rotameter. Record the reading on the logsheet.
- ➤ Collect the PEM<sup>TM</sup> equipment from the target individual. Ask whether s/he had any problems. If so, record them on the logsheet.
- ➤ Disconnect the PEM<sup>TM</sup> from the tubing, place it in the resealable plastic bag with the correct ID label, and reseal the bag tightly.
- ➤ On the chain-of-custody form, write that it is transferred from "target individual" (not the person's name) to the Phlebotomist.
- ➤ Keep the PEM<sup>TM</sup> and the remainder of the sampling apparatus away from excessive heat until they are delivered to the FCC.
- ➤ Check the field logsheet for any problems that could affect the integrity of the sample, such as obstruction by clothing or spillage of food or drink on the PEM<sup>TM</sup>. If there are any, note them on the chain-of-custody form.
- ➤ At the FCC, transfer custody of the sampling apparatus to the Field Coordinator. The Phlebotomist and FC will sign the chain-of-custody form recording this transfer.

# 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.

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The field technician will affix identical labels to the PEM<sup>TM</sup>, the logsheet, and the chain-of-custody form.

# 6.5 Preservation and Storage

# 6.5.1 Disassembly of PEM<sup>TM</sup>

At the FCC, after the Phlebotomist delivers the sampling apparatus, FCC-S will:

- ➤ Receive custody of the PEM<sup>TM</sup> and filter from the FC. Check that the ID numbers on the PEM<sup>TM</sup> bag and Petri slide are correct.
- $\triangleright$  Take the PEM<sup>TM</sup> to a clean work area. Work with only one at a time.
- ➤ Open the Petri slide with the correct ID number.
- ➤ Remove the PEM<sup>TM</sup> from the bag and set the bag aside.
- ➤ Unscrew the PEM<sup>TM</sup> screws fully and put them aside. Lift the PEM<sup>TM</sup> cover vertically and perpendicularly to the table surface. Do the same with the impactor plate.
- > If anything comes in contact with the filter during disassembly, it should be recorded.

the **PEM** TM cover and impa ctor plate have been remo ved, visual 1y inspe ct them for (a) loose partic les, dust, or lint that has

When

not been trapp ed by the impa ctor greas e,

- (b) fibers/hair overhanging from the impactor plate through its center hole, and
- (c) any unusual characteristics. Record your findings on the FCC logsheet.
- ➤ Check that the filter was well secured into the 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 side 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 Petri slide in the plastic bag with the ID label. Seal the bag.
- ➤ Check the field and FCC logsheets for any problems that could affect the integrity of the sample, such as obstruction by clothing or spillage of food or drink on the PEM<sup>TM</sup>. If there are any, note them on the chain-of-custody form.
- > Transfer custody of the bag and filter to the Field Coordinator, signing the chain-of-custody form to record the transfer.
- ➤ If the PEM<sup>TM</sup> is to go back to the field the same week, do a Level B cleaning:
  - → 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 PEM<sup>TM</sup>.
- ➤ If the PEM<sup>TM</sup> is not to go back to the field the same week, place the impactor plate in a container designated for impactor plates to be cleaned. Obtain a clean impactor plate and reassemble the PEM<sup>TM</sup>.

#### 6.5.2 Level A Cleaning of PEM<sup>TM</sup>

At the FCC, the FCC-S will:

- ➤ Clean and prepare PEM<sup>TM</sup>s and impactor plates after each two-week subcycle, after a maximum of three uses.
- ➤ Washing solution for all parts is approximately 1 tsp. Liquinox to 1 liter distilled water. Rinse all parts until no visible soap is present.
- ➤ PEM<sup>TM</sup> covers and bases: Wash in a plastic dishpan with deionized water and Liquinox. Rinse thoroughly. Shake out the water in the hose barb. Lay out the PEM<sup>TM</sup> bases and covers on wipes and air-dry overnight. If the PEM<sup>TM</sup> will be needed that day, dip in alcohol after washing, and dry with a fan, if needed.
- ➤ Stainless steel screens and spacer rings: With a wipe, remove fibers if necessary. Handle screens carefully so as not to create "dings." Sonicate in Liquinox and distilled water for one sonicator cycle. Rinse thoroughly with distilled water. Rinse the sonicator clean.
- ➤ O-rings: Wash in plastic dishpan with distilled water and Liquinox. Use a dishpan used only for O-rings. Rub each one gently by hand to remove traces of grease. Rinse thoroughly with distilled water. After handling used O-rings (even after they have been cleaned), wash your hands before handling other pieces.
- ➤ Impactor plates: 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 distilled water and Liquinox. Use a dishpan and brush used only for impactor plates. Rinse thoroughly with distilled water.
- ➤ Place each impactor plate in a shallow container filled with alcohol 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 a wipe 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. When it is dry, place it in a container labeled for clean PEM<sup>TM</sup> impactor plates.

# 6.6 Handling and Shipping

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 not be exposed to high temperatures.

Every two weeks the collected samples will be shipped or transported to the Emory laboratory by 2-day delivery. They will be packed with adequate bubble-wrap and/or other cushioning media.

# 6.7 Laboratory Analysis

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 either GF-AAS or ICP-MS or both.

Any analyses by another laboratory will be done according to EPA Method 200.8 or a documented equivalent.

# 6.8 Data Workup

Field and laboratory data will be returned to Harvard 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

For personal sampling an additional 5% of the total number of planned exposure sample filters will be retained in the laboratory for use as lab blanks.

An additional 10% of the total number of planned exposure sample filters will be used for field blanks. The field blank will be carried through procedures identical to the sample but will never be attached to an operating sample pump; it will be mounted in a PEM<sup>TM</sup>, then removed, to mirror the physical processes that the actual sample undergoes. Like exposed samples, it will be transferred to a plastic Petri slide, stored for a time equivalent to a real sample, then

shipped back to the HSPH lab with the exposed filters. Blank filters will be from the same lot as sample filters analyzed at the same time.

In the lab the blank will be analyzed identically to the exposed filters. Results from the blanks will allow for correction due to any uniform contamination or interference in either the sampling system or the filter itself.

# 7.2 Duplicate Sampling

10% of all samples will be taken in duplicate. To the degree possible, these samples will be selected randomly (using a random number generator) and spread evenly during each six-week Cycle rotation. Unlike the blank samples, each duplicate will require an additional independent pump and pack accessory. The pump will have to undergo the same checks as the sample pump did before. Obtaining duplicates may be difficult, because the target individual may be too uncomfortable with two systems, which are somewhat heavy and unattractive. A skilled Interviewer will be the best resource for accomplishing this aspect of the study.

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

# 7.3 Tolerance Limits, Detection Limits, and Sensitivity Limits

The tolerance limits for the pump flow rate are given in Section 6.3 above. These limits have been established over a long period of time with much experience in many studies. The rotameter is calibrated over a relatively narrow range only: for use at 4.0 LPM. It can discriminate to at least 0.05 LPM 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 assist in documenting any observed anomalies.

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 lab 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 logsheet will contain an area for notes by the field staff. (There will be a recording sheet for the target individual to complete.) Staff are encouraged to write down anything that may be questionable or noteworthy because it may allow for a later explanation of otherwise puzzling data.

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

Metal	Expected Concentration	Expected Limits of Detection Given Sampling Protocol
arsenic	$10 \text{ ng/m}^3$	4.1 ng/m <sup>3</sup>
cadmium	34 ng/m <sup>3</sup>	$0.41 \text{ ng/m}^3$
chromium	38 ng/m <sup>3</sup>	$0.8 \text{ ng/m}^3$
lead	35 ng/m <sup>3</sup>	$6.0 \text{ ng/m}^3$

# 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

PTEAM Field Monitoring Manual: RTI/Harvard/Acurex Aug. 16, 1990

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

Harvard University/Johns Hopkins University Standard Operating Procedure:

- G03 Identification Numbers
- G04 Chain-of-Custody and Sample Tracking
- G05 Storage and Shipping of Samples
- F01 Field Sampling -- General Information
- L06 Extraction of Metals from Sampling Media
- L07 Analysis of Metals by GF-AAS
- L08 Analysis of Metals by ICP-MS