United States Environmental Protection Agency

Office of Research and Development

National Human Exposure Assessment Survey (NHEXAS)

Arizona Study

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 821560

Standard Operating Procedure **SOP-UA-L-6.1**

Title: Calibration of Harvard PM Samplers

Source: The University of Arizona

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

Calibration of Harvard PM Samplers

1.0 Purpose and applicability

This SOP describes the procedures to be followed during the calibration of Harvard particulate matter (PM) samplers. This procedure applies directly to the calibration of Harvard particulate matter (PM) samplers used for the EPA NHEXAS and EPA Border projects of the University of Arizona/Battelle/Illinois Institute of Technology consortia, as well as future "Health in the Environment" investigations.

2.0 Definitions

2.1 Black box = Harvard particulate matter sampler

2.2 Bubble flow meter = Primary calibration source that measures volume flow by timing a bubble as it rises through a tube of known volume.

2.3 Damping array = A group of air-tight chambers connected in series that are placed in the calibration flow train between the pump and calibration device to remove the effect of flow pulsation caused by the pump.

2.4 DDW = Distilled deionized water

2.5 DVM = Digital volt meter

2.6 Impactor = Attachments to the PM Sampler designed to collect either, less than 2.5 μm (respirable DM 2.5) or less than 10 μm (inhalable) particles.

2.7 PM = Particulate matter

2.8 SLPM = Standard liters per minute

2.9 SOP = Standard Operating Procedure

3.0 References

3.1 Turner, William. Manual for the Indoor Sampler (Draft dated 10/7/85), Section 3.5, Harvard School of Public Health, Boston, MA 1985.

3.2 Chemical Rubber Company. Handbook of Chemistry and Physics, 52nd Edition, 1971-1972, pg. E-40, Constant Humidity tables.

4.0 Discussion

This procedure is primarily derived from the operating manual that accompanied the Harvard PM samplers. The procedure was modified by the Health and Environment department to include a bubble flow meter as a primary calibration device, and the addition of a saturated Calcium Chloride solution containing excess Calcium Chloride Hexahydrate to control the humidity of the flow stream to the PM sampler near normal ambient conditions, as the Harvard PM sampler has an inherent problem maintaining stable flow during fluctuations in relative humidity.

5.0 Responsibilities

5.1 The Project Director will be responsible for:

5.1.1 Final review and approval of this procedure.

5.2 The Project Lab Coordinator will be responsible for :

5.2.1 Insuring SOP procedures are followed by the Project Lab Staff.

5.2.2 Notifying the appropriate technicians with needed repairs. In cases when the item can not be fixed in house, Project Field Coordinator will generate the appropriate paperwork, notify the appropriate vendor or company, and ship the disfunctional item.

5.3 The Project Lab Staff will be responsible for:

5.3.1 Knowing and following the procedure described in this SOP.

5.3.2 Recording the information as directed in this SOP.

5.3.3 Notifying the Project Lab Supervisor with down equipment and repair supplies needed (where applicable).

5.3.4 Providing the Project Lab Supervisor with down equipment label and isolating the down equipment into the down equipment area.

5.3.5 Insuring proper labeling techniques of down equipment.

5.3.6 Repairing the item (where applicable) in a timely manner.

6.0 Equipment and Materials

6.1 Equipment

6.1.1 Digital volt meter with leads

6.1.2 Flow damping array

6.1.3 Gilibrator bubble flow calibration device

6.1.4 Harvard PM samplers (black boxes)

6.1.5 Harvard PM Sampler Log Book

6.1.6 Hot plate magna-stir

6.1.7 Magnehelic gage with 100 inches H2O range

6.1.8 PM Sampler Calibration Data Form (Figure 2)

6.1.9 Power strips, 6-outlet (2)

6.1.10 Rotameter

6.2 Materials

6.2.1 Deionized distilled water (DDW)

6.2.2 Soap solution for bubble meter

6.3 Reagents

6.3.1 Calcium chloride ($CaCl_2$)

7.0 Procedure

7.1 Preparation

7.1.1 Calcium chloride solution Preparation

A. Prepare a supersaturated solution of Calcium Chloride by warming a 1000 mL beaker filled with DDW to between 60 and 80 °C. Add $CaCl_2$ to the warm solution slowly, allowing complete dissolution. The use of a teflon magnetic stir bar, set at a slow stirring speed, aids in dissolution.

B. Continue adding salt until the solution becomes saturated and salt will no longer easily dissolve. The solution will contain Calcium Chloride Hexahydrate ($CaCl_2*6H_2O$) to remove humidity from the air stream.

C. Remove the beaker and solution from the hot plate and cool to room temperature by placing it into a cold water bath or the refrigerator.

D. Once the solution achieves room temperature, remove it and inspect it for crystal formation. If crystals have formed, it is ready for use. If no crystals are present, a seed crystal must be added to promote crystal growth.

E. To seed crystal growth, add one or two grains of Calcium Chloride to the solution and set the beaker aside for a few minutes to allow growth to occur. If there is no crystal formation after addition of a seed crystal, the solution was not supersaturated to begin with and steps A and B must be repeated.

7.1.2 Work area preparation

A. Organize (figure 1) and wipe down the workspace set up for the flow calibrations.

B. Use power strips to plug in PM samplers and set timers to allow pumping units to operate for at least 8 hours. Pumping units should warm up for at least 30 minutes before calibration procedure begins.

C. Obtain current atmospheric pressure reading from:
   1. The Pulmonary Function Lab, located in the University Medical Center.
   2. The local Weather Bureau Station (In Tucson = 881-3333).

   *Note that the pressure is often given in inches of Hg corrected to sea level thus the value given must be corrected to mm of Hg at the current elevation. See section 7.3.1 C for the correct equation.

D. Record pertinent information on the "PM Sampler Calibration Data Form" (figure 2).

7.1.3 PM box setup

A. Set the T1 timer of the sample unit to be calibrated to the on position by pushing the set pins in at the arrow or turning on the electronic timer.
B. Unplug the T2 timer.
C. All electric or sample lines should be fed through the access hole in the left side of the box, and close the lid during all warm-up and calibration operations.

7.1.4 Calibration equipment setup

A. Refer to figure 1 for help setting up the calibration equipment. Once set up, the calibration line remains in its configuration.
B. Attach a tee to the PM sampler on-line.
C. Connect the low pressure side of the magnehelic to one side of the tee.
D. Connect the damping array to the other side of the tee.
E. Connect the bubbler containing saturated $CaCl_2$ solution to the open end of the damping array to draw air through the solution.
F. Connect a 3-way tee to the other side of the $CaCl_2$ bubbler.
G. One line from the tee contains in-line an impactor with filter, and rotameter that is open to the atmosphere as shown in Figure 1.
H. The Gilibrator is connected to the other side of the 3-way tee.
I. The "in" side of the Gilibrator is positioned so that a temperature probe may be inserted. The side of the tee containing the temperature probe is open to the atmosphere.

7.2 Calibration of the PM boxes

7.2.1 Procedure

A. Prepare form L-6.0-1.0 for the Pumping unit to be checked or calibrated by filling in the date, box number, atmospheric pressure and calibration temperature on top of the form. Leave T1 and T2 spaces blank until the calibration is completed.
B. Connect the "ON" hose of pumping unit to the calibration configuration as indicated in figure 1.
C. Set the three-way tee so the airflow goes through the bubble flow meter side of the configuration and the impactor and rotometer are closed off.
D. Allow the flow to stabilize through the configuration, and record the dial setting (of the box potentiometer), DVM number and voltage, system back-pressure (from the magnehelic), and flow on the initial calibration check section of form L-6.0-1.0.
E. Use the bubble meter to check to see that the flow is close to 4.0 LPM. If the bubble flow meter indicates that the pumping unit is out of calibration, unlock the potentiometer inside the untis green electronic control box and adjust it to obtain a mass flow rate of 4.0 LPM as indicated by the bubble meter.

F. Re-lock potentiometer.
G. When the box has the desired flow obtain the average of 10 bubble measurements 3 times and calculate the SLPM on figure 3 (L-6.0-2.0) for each.
H. Record pump unit box #, date, temperature, operator initials, atmospheric pressure, elapsed timer settings, potentiometer dial setting, voltage, and volume flow rate (rotameter) on the PM Sampler Calibration Data Form (L-6.0-1.0).
I. Close a hose clamp on the air line going to the calcium chloride solution (figure 1) until a pressure of -50 inches of water is displayed on the magnehelic gage. Observe that mass flow rate remains constant (+ 0.10 SLPM) at elevated pressure drop.
J. Repeat step G and insure that the flow rate stays within 5% (± 0.2) of 4.0 SLPM. If it does the box is calibrated otherwise repeat steps E through G.
K. Repeat steps A through J above for each pumping unit to be calibrated.

7.3 Calculations

7.3.1 Calibration with bubble meter (used in conjunction with the worksheet - Figure 3, L-6-2.0)

A. $$Q_{std} = Q_m * \frac{P_{atm} - P_v}{760} * \frac{298}{T_m\,(K^\circ)}$$

Where: Q_{std} = standardized flow; Q_m = The measured flow; P_{atm} = the atmospheric pressure in mm of mercury (mmHg); P_v = vapor pressure; and T_m the temperature in degrees Kelvin at the time of measurement.

B. $$\text{mmHg} = \text{inches water} * \frac{51.75}{27.71}$$

C. mmHg(at current elevation) =

$$P\ \text{inHg} * 25.4\ \text{mm/in} - (25.4 * (\text{elevation ft}/1000\ \text{ft}))$$

Where: P inHG = is obtained for the local area; and 25.4 = conversion factor from in to mm.

7.4 Quality Control

7.4.1 Tolerance limits

A. Stable operation of the pumping unit is defined as a fluctuation of less than ± 0.5% in mass flow rate (± 0.2 VDC in flow controller output voltage).
B. The pumping unit should read within ± 5% of its calibrated flow rate (± 0.2 SLPM if set for 4.0 SLPM).
C. Constant pressure is defined as a decrease of no more than 2 inches of water from a stabilized reading of at least -75 inches of water over a period of one minute.

7.4.2 Detection limits

A. The digital volt meter will be read to within ± 0.02 volts.
B. The magnehelic gage will be read to within ± 1 inch of water.

7.4.3 Corrective actions

A. If the pumping unit fails to maintain a constant flow within ± 5% (± 0.2 SLPM for a mass flow of 4.0 SLPM), or if it does not recover to within ± 5% (± 0.2 SLPM) when -50 inches of water resistance is added to line, the flow controller should be tagged for inspection and repair and removed from service until completed.

8.0 Records

8.1 Data Collected by this Procedure

8.1.1 PM Sampler Pumping Unit

A. Pumping unit mass flow rate calibration data will be recorded on the PM Sampler Calibration Data Form (Figure 2), and stored in the PM Sampler Log Book.
B. After each pumping unit's calibration, the calibration card (figure 4, L-6.0-3.0) will be completed and attached to the inside top of the appropriate black box.
C. A brief history of the calibration date, performance, and maintenance history of each box will be kept on the PM Sampler History Sheet (L-6.0-4.0) in the PM Sampler Log Book.
D. Notation of each calibration is kept in a file in the material technicians area to follow boxes which have consistent calibration problems.

8.2 Location/Placement of Forms

8.2.1 PM Sampler

A. The PM Sampler Log Book is kept in the weight room adjacent to the PM sampler calibration work table.
B. A history of each box is kept with the Material Technician.

Figure 1; Configuration Diagram for Box Calibration (PM)

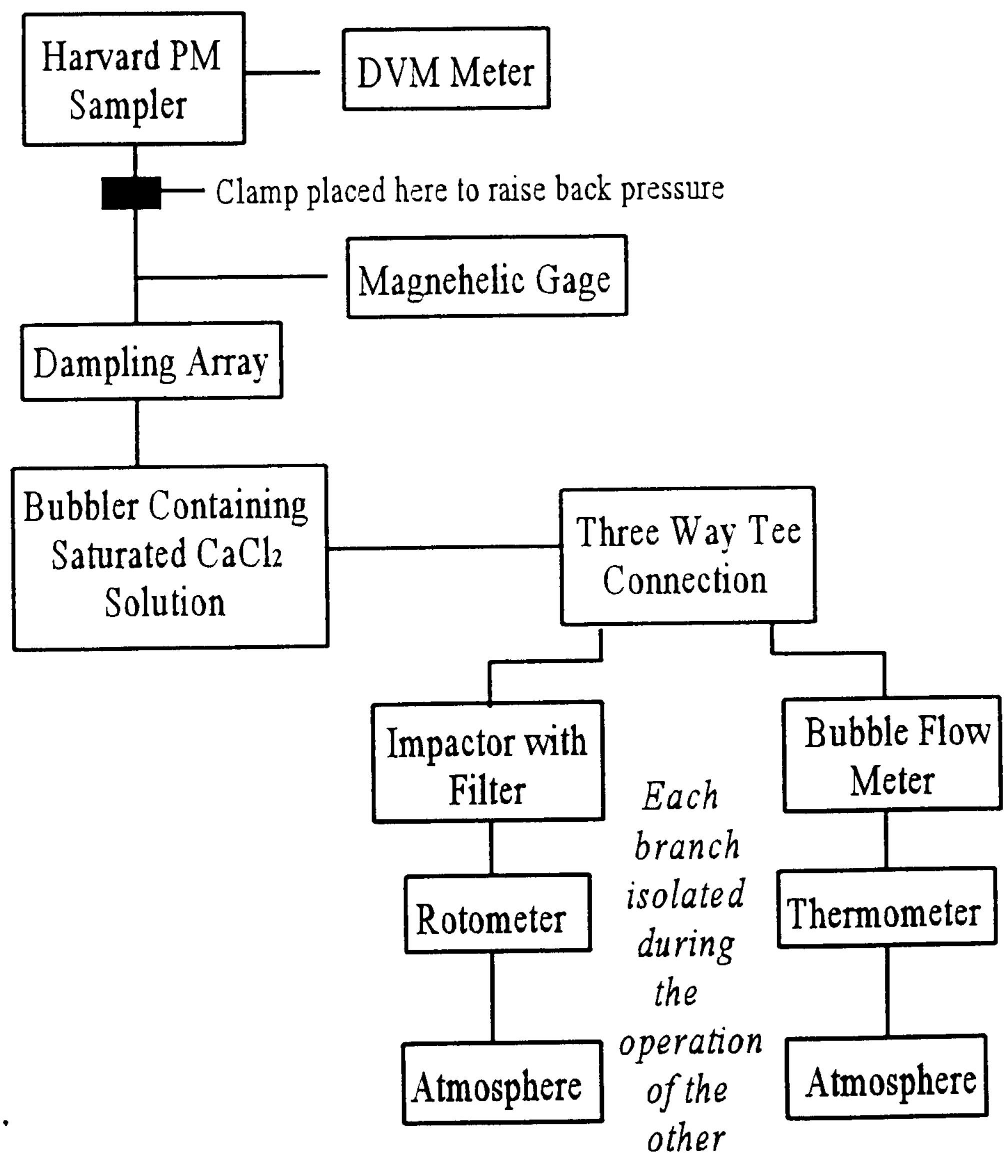

Figure 2; PM box calibration data form

PM SAMPLER CALIBRATION DATA FORM

Date: _____/_____/_____ Box#:___________ T1:_________
Atm. Pressure:______mmHg Temperature:_____ C° T2:_________
Calibration Performed by: _______ Configuration Reviewed By: _______

INITIAL CALIBRATION CHECK:

Dial Setting: __________ DVM#:_________ :_________VDC
Flow (mfm): __________LPM System back pressure:________ H2O
Rotameter#: __________

RECALIBRATION DATA:

New Dial Setting: _______ DVM#:_________ :_________VDC
Flow at Open Atmospheric Pressure
Bubble Flow SLPM: (1)__________ (2)__________ (3)__________
Average Bubble flow: ______ SLPM at _______ H_2O avg. sys. pressure
Flow at -50"
Bubble Flow SLPM: (1)__________ (2)__________ (3)__________
Average Bubble flow: ______ SLPM at _______ H_2O avg. sys. pressure
Rotameter (SN):_________(bb)

New Calibration Card Prepared By:________

ATTACH OLD CALIBRATION CARD HERE
(please staple)

Figure 3; Worksheet for PM Sampler Calibration with Bubble Meter

PM SAMPLER CALIBRATION WITH BUBBLE METER

Box#	System Back Pressure	Dial # DC	DVM Volts	Tmp C° (at Tm)	Patm mmHg	PV mmHg	Qm LPM	Qstd SLPM

Figure 4 Calibration Card

Box#: _______________ Dial setting:________ Box voltage:________ Actual airflow:______ Rotameter#:_________ bb Reading:_________ Calibration date:__/__/__ By:____________

Figure 5; PM-L2.2 PM Sampler History Sheet

PM SAMPLER HISTORY SHEET Box #:__________

Date	Tech.	Remarks/Corrective Actions