

**U.S. EPA BASE STUDY  
STANDARD OPERATING PROCEDURE  
FOR SAMPLING OF  
PARTICULATES (PM2.5 AND PM10)**

Previously submitted date: April 1996

Prepared By:

**Environmental Health & Engineering, Inc.  
60 Wells Avenue  
Newton, MA 02459-3210**

EH&E Report #11663  
September 2000

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## 1.0 OBJECTIVE

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The objective of the procedure described is to collect a sample of particles of respirable and inhalable size (approx. 0.1 to 10 microns in diameter) from indoor air and from the outdoor air supplied to the indoor space tested. The collected material is weighed and quantitated in terms of weight per unit volume of air sampled<sup>1</sup>. The particle samples are collected by inertial impaction onto a preweighed filters<sup>2</sup>. The sampling is conducted on two size cuts: particles less than 10 microns in diameter (PM10 "inhalable" fraction), and particles less than 2.5 microns in diameter (PM2.5 "fine respirable" fraction).

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<sup>1</sup> The amount of collected in each impactor is determined gravimetrically with a high sensitivity ( $\pm 0.004$ mg) analytical balance (e.g., Cahn C-31) located in a Weighing Room with regular certification and internal QA checks).

<sup>2</sup> Following procedures outlined in Method IP-10A "Size Specific Impaction", in "Compendium of Methods for the Determination of Air Pollutants in Indoor Air", USEPA, Research Triangle Park, North Carolina (1989).

## **2.0 GENERAL PROCEDURES**

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### **2.1 SAMPLING CRITERIA AND REQUIREMENTS**

The essential criterion for collecting a representative sample of airborne particulates (*i.e.*, sampling all size fractions within the size range of interest with equal efficiency) is that the sampling be done under isokinetic conditions. The isokinetic condition is met if the gas velocity at the inlet to the sampling probe is identical to that of the free stream velocity approaching the inlet. The specific collection devices employed are multi-hole microenvironmental exposure monitors (MEMs), with impactor classifiers for the two cutoff particle sizes of interest. The sampling port of the MEM impactor is considered to meet the isokinetic criterion.

The samples are collected on 37 mm Teflon air sampling (PTFE, Gelman) membrane filters. These filters are preweighed after equilibrating in a temperature and humidity controlled weighing room. After sample collection the filters plus collected sample are again equilibrated in the weighing room before weighing.

### **2.2 EQUIPMENT AND SUPPLIES REQUIRED**

The following equipment and apparatuses is used for PM<sub>2.5</sub> and PM<sub>10</sub> sampling:

- Seven MEMs<sup>3</sup> for collecting PM<sub>10</sub> (sample of particles with  $d < 10 \mu\text{m}$ ) (six to be samples; one to be a field blank)
- Six MEMs for collecting PM<sub>2.5</sub> (sample of particles with  $d < 2.5 \mu\text{m}$ )
- Fourteen vacuum pumps (12 pumps with 2 back-ups) with a capacity for drawing no less than 20 liters/minute through the MEMs and equipped with flow controllers<sup>4</sup>

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<sup>3</sup> Air Diagnostics and Engineering Inc., R.R. 1, Box 445, Naples, Maine

<sup>4</sup> The required flow rate may be 10 lpm or 20 lpm depending on the rating of the MEM nozzle installed.

- 1/4" ID flexible (e.g., latex) tubing
- 1/4" ID polyethylene tubing (for outdoor set-ups only)
- Calibrated rotameter for 20 lpm flow rates<sup>5</sup>
- Stopwatch or timer
- Eleven 37 mm Teflon Air Sampling membrane filters<sup>6</sup> each in a IADCS pre-labeled Petri dish (the analytical laboratory is supplied IADCS labels prior to the field study)
- Eleven filter supports - cellulose backing material<sup>7</sup>
- Flow cap for measuring flow rates through impactors

All pumps will be equipped with appropriate sound insulated to avoid unnecessary disturbance in the indoor environment being sampled. The pump and sound insulation modules will be mechanically ventilated to prevent overheating of the pumps.

### 2.3 SAMPLING APPARATUS

The sampling apparatus consists of MEMs with impactor classifiers (*i.e.*, orifices) for the two cut-off sizes of interest (2.5 microns and 10 microns). The impactor classifier selected defines the size (aerodynamic diameter) of the particles collected. The cut-off size is defined as the aerodynamic diameter of particles that are collected with 50% efficiency. Sampling is conducted by drawing air through the MEM at a rate of  $20.0 \pm 1$  lpm.

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<sup>5</sup> Dwyer RMC 102, or equivalent.

<sup>6</sup> e.g., PTFE, Gelman.

<sup>7</sup> e.g., Millipore AP-10037, or equivalent.

## 2.4 SET-UP AND SAMPLING

### 2.4.1 Preparation (Cleaning) Samplers

For cleaning and labeling of samplers:

- Drying oven for 200°F
- Tub for washing and rinsing
- Filter forceps
- Isopropyl alcohol
- Lint-free wipes (e.g., Kimwipes)
- Mineral oil, dropper
- Detergent or liquid soap
- Parafilm
- Cookie sheet
- Silicone spray
- IADCS sample ID labels

The preparation of an impactor takes place in three stages:

1. All impactor plates must be cleaned and dried before use
2. Plates must be oiled (a drop of oil placed on each impactor plate)
3. Impactor plates must be placed underneath impactor nozzles and filter backings and filters placed inside the impactor base

Impactors must be cleaned and assembled at the beginning of the sampling week. The first step in preparing impactors is to create a “clean” work area by swabbing a table top with alcohol and then laying down Kimwipes. Once such a “clean” work space has been prepared, the impactors are disassembled and the impactor plates set aside for proper cleaning.

The three step process for cleaning the impactor plates is the following:

1. Wash the plates in water with added detergent or liquid soap to remove visible debris and excess oil
2. Place impactor plates on a cookie sheet and oven dry at 200° F until the moisture has evaporated (approx. 20 to 30 min.)
3. Place a droplet of mineral oil in the center of each plate before re-assembling in the impactor

The other necessary tasks in preparing the impactors are:

1. Clean the inside of the impactor “candlestick” by wiping with an alcohol-impregnated, lint-free wipe
2. Lubricate the rubber gaskets with a silicone spray dampened wipe, taking care not to spray any part of the inside surface of the impactor

Once the impactor plates and the separate pieces of the impactor candlestick are properly cleaned, they are ready to be re-assembled, and filters inserted into each impactor.

NOTE: The filters employed are enclosed in plastic Dichot rings for ease of manipulation. The filters should not be removed from this mounting except by the laboratory. Special care must be taken to record the impactor ID and IADCS sample ID (from the filter’s Petri dish) as each filter is placed into an impactor. This allows to keep track of the sampling location corresponding to each filter upon disassembly of the impactors.

The final step after assembly of the impactor is to wrap Parafilm around the air inlet and outlet of the top and bottom of the impactor so it will not collect dust particles while in transit.

## 2.4.2 Set-up and Sampling

The flow rates are checked (after the pumps have been warmed up for approximately 10 minutes) on the morning of sampling, and adjusted, if necessary, to 20 lpm.<sup>8</sup> The pumps are then turned off and the impactors are installed to the corresponding sampling trains at the various sampling sites. The Parafilm is then removed from the air inlets and outlets of the samplers and the samplers.

After the sampler is attached, the pump is turned on and the time of the beginning of sampling is indicated as "Time On" on the log sheet. Immediately after the start of sampling the flow is re-checked and adjusted to  $20 \pm 1$  lpm, and this flow rate is recorded as the "Flow On" on the log sheet.

NOTE: Flow rates with the sampler (MEM) in place are checked by placing a cap connected to a rotameter over the inlet to the sampler.

The sample is collected for approximately nine hours, after which time the rotameter is reconnected to the sampling train, and the flow rate checked and recorded as "Flow Off" on the log sheet. Then the pump is turned off and this time is recorded as "Time Off" on the log sheet. The impactor is then disconnected from the sampling train and its air inlets and outlets re-wrapped with Parafilm to prevent contamination.

The final step in the sampling process is the disassembly of the impactors to retrieve the collection filters. A clean work area is prepared by wiping a table top with alcohol and then covering the surface with Kimwipes. The impactors are placed on this cleaned and covered surface and the filters are removed from the impactor with tweezers and placed into their original Petri dishes.

NOTE: The area where impactors are disassembled and filters removed should be situated away from supply air diffusers or other air currents that could perturb the sampled material.

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<sup>8</sup> 10 lpm if nozzles of that rating are installed on the MEMs.

Once the filters have been removed from their impactors, they are prepared for shipping by stacking them and securing the stack with tape.

## **2.5 SAMPLING LOCATIONS AND QC SAMPLES**

The fixed site sampling convention is as follows provided the test space can accommodate such a configuration.

Outdoor Site: One PM 10 and one PM 2.5 sample, one PM 10 and one PM 2.5 duplicate

Fixed Site 1: One PM 10 sample, one PM 2.5 sample and one field blank<sup>9</sup>

Fixed Site 3: One PM 10 sample and one PM 2.5 sample

Fixed Site 5: One PM 10 and one PM 2.5 sample, one PM 10 and one PM 2.5 duplicate<sup>10</sup>

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<sup>9</sup> Field blanks may be collected at either site F1, F3, or F5.

<sup>10</sup> Indoor duplicate samples may be collected at either site F1, F3, or F5 and may be placed based on site physical restrictions. Indoor duplicate samples shall not be collected across multiple fixed indoor sites (e.g., VOC duplicates at F1, particles at F3, and other duplicate samplers at F5).

## **3.0 QUALITY CONTROL**

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### **3.1 FLOW RATES**

The air flow rate through the impactor is measured with a secondary or transfer standard flowmeter. This standard is a calibrated rotameter reading in the flow rate range of interest (20 lpm). The performance requirement for the pumps is  $\pm 5\%$  of the nominal flow rate.

### **3.2 FILTERS**

The samples are collected on 37 mm Teflon air sampling (PTFE, Gelman) membrane filters. The filters must be preweighed after equilibrating in a temperature and humidity controlled weighing room, inserted in Dichot rings, and labeled. (These operations are conducted in the laboratory that supplies and analyzes the filters). After sample collection the filters plus collected sample are again equilibrated in the weighing room before weighing. A batch blank retained in the laboratory must be weighed before and after the samples. Should the initial and final weights of the batch blank differ by more than 0.007mg, all filters in the batch must be weighed again.

The amount of sample collected and the accuracy of the gravimetrically determined sample size depend on the size of the sample. The required precision of the balance is  $\pm 0.004$  mg.

### **3.3 QUALITY CONTROL (QC) SAMPLE**

A field blank will be included in each sample batch. The field blank will be prepared by inserting it into a MEM sampler and then removing it without running air through the instrument.

## **4.0 SAMPLE PROCESSING AND SHIPPING**

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Each filter + Dichot ring assembly (filter cassette) is placed inside a Petri dish. As the filters (previously equilibrated to the temperature and humidity conditions of the weighing chamber) are placed inside the Petri dishes they are first inspected (by holding them against light) for holes or tears. Filters that show evidence of such defects are discarded.

After collecting samples the filters are removed from the MEM samplers, returned to their corresponding Petri dishes and sealed with masking tape. The cassettes, so packaged, are shipped to the laboratory.

The filters in their Petri dish enclosure must be stored at all times in dust-free, low static containers and protected from shock.

Filter samples must be mailed for overnight delivery inside a well protected shipping container.

A chain of custody is required for each sample batch.

## 5.0 ANALYTICAL METHODS

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All weighings must be conducted in a certified laboratory which must be able to conduct the following criteria:

1. All filters must be conditioned in the temperature and humidity controlled atmosphere of a certified weighing room for at least 24 hours before initial (or final) weighing to equilibrate with its humidity conditions.
2. The balance employed must meet or exceed the performance of a Cahn Model 37 microbalance.
3. The balance zero must be checked and adjusted, if necessary, to  $0.000 \pm 0.005$  mg.
4. The balance calibration will be checked with a calibration weight of 50 to 200 mg (to  $\pm 0.002$  mg), and adjusted, if necessary.
5. The calibration will be rechecked (and readjusted, if necessary) after every ten filter weighings. The balance will be turned back to zero between filters. If either the zero or calibration are found to fall outside the specified range of variation, the last ten filters will be weighed again, after readjusting the balance.
6. After zeroing and calibrating each filter must be passed over a static eliminator unit.