II. AIRBORNE PARTICLE CHARACTERIZATION

Purpose

1. To become familiar with the principle of operation of the cascade impactor used for aerodynamic sizing of airborne aerosols.

2. To learn the techniques for field sampling for respirable dust using the SKC cyclone, the IOM inhalable dust sampler, and for total airborne dust using appropriate filter media.

3. To compare results from simultaneous sampling with the above devices.

4. To Investigate and compare other means of analyzing aerosol samples such as direct reading aerosol monitors and microscopic counting. *(Microscopic counting will be done during the asbestos experiment later in the semester).*

Outline

This laboratory has two parts: (i) collection of integrated particle samples using a variety of size selective and total dust samples, and (ii) measurement of airborne dust using several direct reading instruments. These measurements will be made in an enclosed chamber to which has been fitted a dynamic particle generating system. The class will be divided into 2 groups. On day one of the experiment group 1 will collect integrated particulate samples, and group 2 will use the direct reading instruments. The two groups will switch tasks for day two of the experiment.

Important reference material

- NIOSH method 0500, particles not otherwise regulated (total)
- NIOSH method 0600, particles not otherwise regulated (respirable)

Both references available online at http://www.cdc.gov/niosh/nmam/

Operating instructions for the following:

IOM sampler
Marple cascade impactor
Cyclone samplers
MIE personal dataram
Dust-trak aerosol monitor

These references are available in the cardboard box in F226, and in the teaching laboratory

Background

It has long been recognized that gravimetric analysis of airborne dust is often inadequate for determination of a health hazard because it ignores effects of particle size. Methods have been developed which provide information about the distribution of particle mass among the various particle sizes present and are commonly employed for "respirable" and inhalable dust analysis. In practice this takes the form of a cyclone pre-separator placed in front of the standard air sampling filter cassette. Used alone, this simply gives the mass concentration of dust whose particle sizes are small enough to pass through the cyclone; no information about the larger dust particles, or about the actual size distribution, is obtained.

A more sophisticated type of instrument, the cascade impactor, can provide more detail on the particle size distribution and can also be used to estimate the fraction (or concentration) of respirable dust present in the air. Its principle of operation, and the manipulation of primary data from it, are described in the references.

The inhalable dust sampler (IOM) collects a larger size distribution and corresponds to the ACGIH sampling criteria for inhalable particulate mass.

In this experiment you will take simultaneous samples with the cascade impactor, the respirable dust filter sampling train, the inhalable sampler and the total dust sampling train in order to compare and interpret their results. Further, duplicate systems will be used for the different techniques to gain insight into the extent of variability which is inherent in the filter sampling methods.

Finally, it is important to realize that different filter media are required, dictated by the different samplers that are used or the analysis method that will be applied to each sample. You should become familiar with which filter media are appropriate for specific applications.

PART 1: Integrated Sampling for Particulates

The following table summarizes the different integrated samples that will be collected in this experiment. In addition to the samples listed below, you should **prepare a lab blank for each sampler configuration**.

Sample type	Sampler	Filter media	# samples
Total dust	Cassette, closed face	PVC	1
	Cassette, open face	PVC	1
	Cassette, open face	MCE*	1
Inhalable dust	IOM	PVC	1
Respirable dust	Cyclone/cassette	PVC	2
Size distribution	Marple	Mylar/PVC	1 (6 stages)

* *The MCE samples will be used for microscopy and metals analysis. These filters do not need to be weighed.*

Procedure

Each type of sampler must be carefully prepared. For gravimetric analysis, the filters or foils must be accurately weighed before and after sampling using identical equilibration techniques that are described in the NIOSH Manual or as modified by the instructors. The techniques must take into account the effect of moisture and temperature on the sample media. You must begin this experiment by reading the manual or NIOSH method for each type of sampler to establish what type of filter is required for each application.

1. Obtain Tare Weights

a. **(Marple Impactor)** Weigh the plastic collection foil for each preceding impactor plate and the final filter on the microbalance. Filters should be weighted to constant weight. Be certain that each train is reassembled exactly as it was calibrated or as numbered and that filters or plates are not exchanged. Slots and jets must be staggered.

b. **(Cassettes)** Weigh the filters for the cassettes (including blank filters).

c. Number all samplers for later identification. Be sure to record the tare weights along with the matching sampler numbers.

2. Cascade Impactor Set Up; Model 190 Marple Personal Cascade Impactor

a. Each collecting plate should be topped with a pre-weighed mylar foil. These are extremely thin. Use only one foil per stage (not the white spacers). For sampling dry dusts the sampling substrate must be coated with adhesive, but in this case, the aerosol is moist and an adhesive is not required. The foils can pick up an electrostatic charge that must be neutralized by passing them over an anti static device just before weighing. Care must be taken to keep the films in the proper sequence, as related to each impactor stage.

b. Assemble the impactor as shown in the manual, inserting one pre-weighed foil per plate or stage. Include a 34 mm PVC absolute filter on the last stage. Connect it to the vacuum pump. Note that the corresponding media follows and is underneath the impactor stage.

c. Adjust the flow rate through the sampler to 3 LPM as specified in the description using an appropriate calibration device. Note that the absolute particle-size cut-point for each stage of the impactor is determined by the flow rate through the impactor. A flow of 3L/minute will allow collection of sufficient mass in the 2-hr sampling period for subsequent accurate gravimetic analysis. The Marple manual describes how to calculate the mass cut-points at 3 L/minute flow rate.

3. Filter and Cyclone/Filter Sampling Train Set Up

a. Using 37 mm polystyrene cassettes and pre-weighed PVC filters, assemble two respirable and two total dust sampling trains. Follow the steps outlined in the NIOSH Methods 0500 and 0600. Also, fill cassettes with **unweighed** MCE filters for microscopic tests.

b. Adjust the flow rate through each train to the specified value (that is 2.75 LPM for SKC cyclones) using an appropriate calibration device. Use a glass dessicator to calibrate pump flow for the cyclones.

c. The PVC filters will be used for gravimetric analysis and MCE will be retained for microscopic size analysis and metals analysis in later lab experiments.

4. IOM Inhalable Dust Samplers

a. Assemble the sampling cassettes (1 metal, 1 plastic) with their filters. Carefully weigh the whole cassette on the **Mettler electrobalance**. Place it in the sampler housing and attach the cover. *NB: It may help to preweigh the IOMs on the top-loading balance in the main lab, and* *use this approximate weight as a starting point for obtaining an exact weight on the* Mettler electrobalance.

b. Use the adapter and calibrate the flow to 2 LPM.

5. Aerosol Generation and Sampling

a. The zinc and iron salt aerosol will be generated using a nebulizer and aerosol dilution system. Observe and make a diagram of the generator and chamber assembly. Note the location of the sampling ports and the respective sampler locations in the chamber.

b. Place and record the location of the cascade impactor, the respirable and inhalable samplers, and total dust filters inside the chamber. Attach each to the appropriate tubing through-ports provided to the correct pumps. Be sure each sampler is properly oriented in the chamber air flow. Close and seal the chamber.

c. The nebulizer flow is adjusted to preset conditions for optimum generation of the aerosol. The build up of aerosol to uniform concentration can be followed with the MIE personal dataram and the beam of the laser pen. Other Aerosol Monitors, such as the Dust-trak will be available.

d. Once the aerosol has reached uniform distribution in the chamber, start the various samplers and sample for approximately 1-2 hours or a period designated by the instructor.

e. When sampling is complete, shut off each sampler, **recording the sampling time and flow**. The generator must be turned off and the chamber cleared before the samplers can be removed.

6. Determine gross weights

a. Remove the samplers and disassemble each sampler carefully. Allow the filters to equilibrate in the dessicator provided (~1hr). After equilibration, weigh to constant weight and record the weights as before in Section 1.

b. Mark and retain the samplers (cassettes) that were designated for later microscopic and metals analysis. Place these samplers in a safe location.

7. Calculations and Application of Data:

a. Tabulate the size selective data for the Marple cascade samplers and calculate the following according to the procedures in the Marple brochure:

I. Using computer software (e.g Microsoft excel), graph the **differential** particle size distribution size distribution as described in the marple manual. Use logarithmic scales for both axes.
II. Using computer software (e.g Microsoft excel), graph the **cumulative** particle size distribution size distribution as described in the marple manual. Use a logarithmic scale for the particle size axis.

i. From this cumulative particle size distribution graph, determine the Mass Median Aerodynamic Diameter (MMAD).
ii. From the cumulative particle size distribution graph, determine the Geometric Standard Deviation (GSD).

b. Use the cascade impactor data to calculate the fraction of the aerosol that was respirable.

c. Calculate the concentration of respirable dust from cyclone data, and the concentration of the total dust from the open faced filter samplers. Then calculate the fraction of respirable dust from the ratio of the respirable dust from the cyclone samplers to the weight of the total dust from the open faced filters.

d. Compare the respirable fraction as calculated from both b. and c. Should the result from the two different methods agree? Discuss why you feel these results are or are not comparable.

Questions:

a. Is the total concentration greater than the respirable concentration?
b. Does the sum of the weights of all the Marple stages give the same total dust concentration as the total dust samplers?
c. How does the IOM data compare to the other size fractions? Is it less than the total? Is it greater than the respirable? Is this what general principles would predict?

PART 2: Direct Aerosol Measurement Lab

In this part of the experiment, you will use the following direct reading instruments to monitor particle levels in real time:

Dust-trak
MIE personal dataram (pDR)

Procedure

1. Operation:
   Read the operating instruction for each manual, and determine how to execute the following operations. (Check with the instructors if in doubt).
   a. On/off
   b. Zero instrument
   c. Measure particle levels
   d. Store and download data to computer.

2. Measurement Locations:
   a. Sampling ports on chamber
   b. Chamber exhaust

3. Particle Measurements:
   a. The dust-trak and the personal dataram should be set up to log the PM data at 1 minute intervals. Connect the dataram to a sampling port and commence sampling co-incident with the integrated samples collected by group 1. Later, we will compare the integrated TWA measurement from the data ram with the gravimetric mass measurement from the filter samples.
   b. Using the dust-trak:
      1. Perform side-by-side comparisons of the two instruments. Do they report the same levels?
      2. Compare measurements made with and without the size selective inlet. **To what extent does the size selective inlet attenuate instrument response?**

4. Reporting Data:
   a. Download the data from the dataram and dust-trak to Microsoft excel. (Follow the instructions in the manual. Ask an instructor if you have problems). Generate a graph of particle level vs. time. Annotate the graphs as appropriate to indicate changes in sampler position, size selective inlets, etc.
   b. Prepare data tables to summarize the measurements made in part 3 (above)
   c. Share your data with the other group. **How does the data from the dust-trak compare with the mass measurements from the cyclones?**
   d. Remember to answer the questions highlighted in the text in the previous sections

Write Up (for parts I and II combined)

1. Include all data obtained in Part I, 6 & 7, and Part II 3 & 4

2. Answer all the questions that were highlighted in Part I, #7, and Part II #3 & 4

3. Compare the results from the various monitoing methods with respect to estimated respirable fraction and agreement on total mass concentration. Prepare a brief summary table.

4. Discuss the advantages and disadvantages of each of the techniques. In what circumstances would each technique be most appropriate? What situations would require the use of multiple sampling techniques vs. one particular technique?

Supplementary References:

1. Cohen, B. Air Sampling Instruments, ACGIH, 8th Edition. Chapters 13, 14, 16 (1995).

2. NIOSH. The Industrial Environment - Its Evaluation and Control. Chapter 14.

3. Mercer, T.T. Aerosol Technology in Hazard Evaluation. New York: Academic Press, 1973.

4. Raabe, O. Size-selective sampling criteria for thoracic and respirable mass fractions. Trans. A.C.G.I.H. 1984, p. 53.

5. Hinds, W. C., Aerosol Technology, Wiley Interscience, New York, 1982

6. Perkins, Jimmy. Modern Industrial Hygiene, Recognition and Evaluation of Chemical Agents, Chapter 22, Van Nostrand Reinhold, 1997.

PARTICULATES NOT OTHERWISE REGULATED, TOTAL 0500

DEFINITION: total aerosol mass CAS: NONE RTECS: NONE

METHOD: 0500, Issue 2	**EVALUATION: FULL**	**Issue 1: 15 February 1984** **Issue 2: 15 August 1994**

OSHA : 15 mg/m^3
NIOSH: no REL
ACGIH: 10 mg/m^3, total dust less than 1% quartz

PROPERTIES: contains no asbestos and quartz less than 1%

SYNONYMS: nuisance dusts; particulates not otherwise classified

SAMPLING

SAMPLER:	FILTER (tared 37-mm, 5-µm PVC filter)
FLOW RATE:	1 to 2 L/min
VOL-MIN:	7 L @ 15 mg/m^3
-MAX:	133 L @ 15 mg/m^3
SHIPMENT:	routine
SAMPLE STABILITY:	indefinitely
BLANKS:	2 to 10 field blanks per set
BULK SAMPLE:	none required

ACCURACY

RANGE STUDIED:	8 to 28 mg/m^3
BIAS:	0.01%
OVERALL PRECISION ($\hat{S}_{rT}$):	0.056 [1]
ACCURACY:	± 11.04%

MEASUREMENT

TECHNIQUE:	GRAVIMETRIC (FILTER WEIGHT)
ANALYTE:	airborne particulate material
BALANCE:	0.001 mg sensitivity; use same balance before and after sample collection
CALIBRATION:	National Institute of Standards and Technology Class S-1.1 weights or ASTM Class 1 weights
RANGE:	0.1 to 2 mg per sample
ESTIMATED LOD:	0.03 mg per sample
PRECISION ($\hat{S}_r$):	0.026 [2]

APPLICABILITY: The working range is 1 to 20 mg/m^3 for a 100-L air sample. This method is nonspecific and determines the total dust concentration to which a worker is exposed. It may be applied, e.g., to gravimetric determination of fibrous glass [3] in addition to the other ACGIH particulates not otherwise regulated [4].

INTERFERENCES: Organic and volatile particulate matter may be removed by dry ashing [3].

OTHER METHODS: This method is similar to the criteria document method for fibrous glass [3] and Method 5000 for carbon black. This method replaces Method S349 [5]. Impingers and direct-reading instruments may be used to collect total dust samples, but these have limitations for personal sampling.

EQUIPMENT:

1. Sampler: 37-mm PVC, 2- to 5-µm pore size membrane or equivalent hydrophobic filter and supporting pad in 37-mm cassette filter holder.
2. Personal sampling pump, 1 to 2 L/min, with flexible connecting tubing.
3. Microbalance, capable of weighing to 0.001 mg.
4. Static neutralizer: e.g., Po-210; replace nine months after the production date.
5. Forceps (preferably nylon).
6. Environmental chamber or room for balance (e.g., 20 °C ± 1 °C and 50% ± 5% RH).

SPECIAL PRECAUTIONS: None.

PREPARATION OF FILTERS BEFORE SAMPLING:

1. Equilibrate the filters in an environmentally controlled weighing area or chamber for at least 2 h. NOTE: An environmentally controlled chamber is desirable, but not required.
2. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
3. Weigh the filters in an environmentally controlled area or chamber. Record the filter tare weight, W_1 (mg).
   a. Zero the balance before each weighing.
   b. Handle the filter with forceps. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
4. Assemble the filter in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry and mark with the same number as the backup pad.

SAMPLING:

5. Calibrate each personal sampling pump with a representative sampler in line.
6. Sample at 1 to 2 L/min for a total sample volume of 7 to 133 L. Do not exceed a total filter loading of approximately 2 mg total dust. Take two to four replicate samples for each batch of field samples for quality assurance on the sampling procedure.

SAMPLE PREPARATION:

7. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
8. Remove the top and bottom plugs from the filter cassette. Equilibrate for at least 2 h in the balance room.
9. Remove the cassette band, pry open the cassette, and remove the filter gently to avoid loss of dust.
   NOTE: If the filter adheres to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

10. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Maintain and calibrate the balance with National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 weights.

11. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [7] or in the field [8]. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. The relative standard deviation calculated from these replicates should be recorded on control charts and action taken when the precision is out of control [7].

MEASUREMENT:

12. Weigh each filter, including field blanks. Record the post-sampling weight, W_2 (mg). Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.)

CALCULATIONS:

13. Calculate the concentration of total particulate, C (mg/m^3), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) - (B_2 - B_1) \cdot 10^3}{V}, \ mg/m^3.$$

where: W_1 = tare weight of filter before sampling (mg)
W_2 = post-sampling weight of sample-containing filter (mg)
B_1 = mean tare weight of blank filters (mg)
B_2 = mean post-sampling weight of blank filters (mg)

EVALUATION OF METHOD:

Lab testing with blank filters and generated atmospheres of carbon black was done at 8 to 28 mg/m^3 [2,6]. Precision and accuracy data are given on page 0500-1.

REFERENCES:

[1] NIOSH Manual of Analytical Methods, 3rd ed., NMAM 5000, DHHS (NIOSH) Publication No. 84-100 (1984).
[2] Unpublished data from Non-textile Cotton Study, NIOSH/DRDS/EIB.
[3] NIOSH Criteria for a Recommended Standard ... Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152, 119-142 (1977).
[4] 1993-1994 Threshold Limit Values and Biological Exposure Indices, Appendix D, ACGIH, Cincinnati, OH (1993).
[5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[6] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[7] Bowman, J.D., D.L. Bartley, G.M. Breuer, L.J. Doemeny, and D.J. Murdock. Accuracy Criteria Recommended for the Certification of Gravimetric Coal Mine Dust Personal Samplers. NTIS Pub. No. PB 85-222446 (1984).
[8] Breslin, J.A., S.J. Page, and R.A. Jankowski. Precision of Personal Sampling of Respirable Dust in Coal Mines, U.S. Bureau of Mines Report of Investigations #8740 (1983).

METHOD REVISED BY:

Jerry Clere and Frank Hearl, P.E., NIOSH/DRDS.

PARTICULATES NOT OTHERWISE REGULATED, RESPIRABLE

DEFINITION: aerosol collected by sampler with 4-μm median cut point   CAS: None   RTECS: None

METHOD: 0600, Issue 3	EVALUATION: FULL	Issue 1: 15 February 1984 Issue 3: 15 January 1998

OSHA : 5 mg/m³
NIOSH: no REL
ACGIH: 3 mg/m³

PROPERTIES: contains no asbestos and quartz less than 1%; penetrates non-ciliated portions of respiratory system

SYNONYMS: nuisance dusts; particulates not otherwise classified

SAMPLING

SAMPLER: CYCLONE + FILTER (10-mm nylon cyclone, Higgins-Dewell [HD] cyclone, or Aluminum cyclone + tared 5-μm PVC membrane)

FLOW RATE: nylon cyclone: 1.7 L/min
HD cyclone: 2.2 L/min
Al cyclone: 2.5 L/min

VOL-MIN: 20 L @ 5 mg/m³
-MAX: 400 L

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.5 to 10 mg/m³ (lab and field)

BIAS: dependent on dust size distribution [1]

OVERALL PRECISION ($\hat{S}_{rT}$): dependent on size distribution [1,2]

ACCURACY: dependent on size distribution [1]

MEASUREMENT

TECHNIQUE: GRAVIMETRIC (FILTER WEIGHT)

ANALYTE: mass of respirable dust fraction

BALANCE: 0.001 mg sensitivity; use same balance before and after sample collection

CALIBRATION: National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 weights

RANGE: 0.1 to 2 mg per sample

ESTIMATED LOD: 0.03 mg per sample

PRECISION: <10 μg with 0.001 mg sensitivity balance; <70 μg with 0.01 mg sensitivity balance [3]

APPLICABILITY: The working range is 0.5 to 10 mg/m³ for a 200-L air sample. The method measures the mass concentration of any non-volatile respirable dust. In addition to inert dusts [4], the method has been recommended for respirable coal dust. The method is biased in light of the recently adopted international definition of respirable dust, e.g., ≈ +7% bias for non-diesel, coal mine dust [5].

INTERFERENCES: Larger than respirable particles (over 10 μm) have been found in some cases by microscopic analysis of cyclone filters. Over-sized particles in samples are known to be caused by inverting the cyclone assembly. Heavy dust loadings, fibers, and water-saturated dusts also interfere with the cyclone's size-selective properties. The use of conductive samplers is recommended to minimize particle charge effects.

OTHER METHODS: This method is based on and replaces Sampling Data Sheet #29.02 [6].

EQUIPMENT:

1. Sampler:
   a. Filter: 5.0-µm pore size, polyvinyl chloride filter or equivalent hydrophobic membrane filter supported by a cassette filter holder (preferably conductive).
   b. Cyclone: 10-mm nylon(Mine Safety Appliance Co., Instrument Division, P. O. Box 427, Pittsburgh, PA 15230), Higgins-Dewell(BGI Inc., 58 Guinan St., Waltham, MA 02154)[7], aluminum cyclone (SKC Inc., 863 Valley View Road, Eighty Four, PA 15330), or equivalent.
2. Personal sampling pump, 1.7 L/min ± 5% for nylon cyclone, 2.2 L/min ± 5% for HD cyclone, or 2.5 L/min ± 5% for the Al cyclone with flexible connecting tubing.
   NOTE: Pulsation in the pump flow must be within ± 20% of the mean flow.
3. Balance, analytical, with sensitivity of 0.001 mg.
4. Weights, NIST Class S-1.1, or ASTM Class 1.
5. Static neutralizer, e.g., Po-210; replace nine months after the production date.
6. Forceps (preferably nylon).
7. Environmental chamber or room for balance, e.g., 20 °C ± 1 °C and 50% ± 5% RH.

SPECIAL PRECAUTIONS: None.

PREPARATION OF SAMPLERS BEFORE SAMPLING:

1. Equilibrate the filters in an environmentally controlled weighing area or chamber for at least 2 h.
2. Weigh the filters in an environmentally controlled area or chamber. Record the filter tare weight, W_1 (mg).
   a. Zero the balance before each weighing.
   b. Handle the filter with forceps (nylon forceps if further analyses will be done).
   c. Pass the filter over an anti-static radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
3. Assemble the filters in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette.
4. Remove the cyclone's grit cap before use and inspect the cyclone interior. If the inside is visibly scored, discard this cyclone since the dust separation characteristics of the cyclone may be altered. Clean the interior of the cyclone to prevent reentrainment of large particles.
5. Assemble the sampler head. Check alignment of filter holder and cyclone in the sampling head to prevent leakage.

SAMPLING:

6. Calibrate each personal sampling pump to the appropriate flow rate with a representative sampler in line.
   NOTE 1: Because of their inlet designs, nylon and aluminum cyclones are calibrated within a large vessel with inlet and outlet ports. The inlet is connected to a calibrator (e.g., a bubble meter). The cyclone outlet is connected to the outlet port within the vessel, and the vessel outlet is attached to the pump. See APPENDIX for alternate calibration procedure. (The calibrator can be connected directly to the HD cyclone.)
   NOTE 2: Even if the flowrate shifts by a known amount between calibration and use, the nominal flowrates are used for concentration calculation because of a self-correction feature of the cyclones.
7. Sample 45 min to 8 h. Do not exceed 2 mg dust loading on the filter. Take 2 to 4 replicate samples for each batch of field samples for quality assurance on the sampling procedure (see Step 10).
   NOTE : Do not allow the sampler assembly to be inverted at any time. Turning the cyclone to anything more than a horizontal orientation may deposit oversized material from the cyclone body onto the filter.

SAMPLE PREPARATION:

8. Remove the top and bottom plugs from the filter cassette. Equilibrate for at least 2 h in an environmentally controlled area or chamber.

CALIBRATION AND QUALITY CONTROL:

9. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Calibrate the balance with National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 weights.
10. The set of replicate field samples should be exposed to the same dust environment, either in a laboratory dust chamber [8] or in the field [9]. The quality control samples must be taken with the same equipment, procedures, and personnel used in the routine field samples. Calculate precision from these replicates and record relative standard deviation (S_r) on control charts. Take corrective action when the precision is out of control [8].

MEASUREMENT:

11. Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., visible particles, overloading, leakage, wet, torn, etc.).

CALCULATIONS:

12. Calculate the concentration of respirable particulate, C (mg/m^3), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) - (B_2 - B_1)}{V} . 10^3, \ mg/m^3$$

where: W_1 = tare weight of filter before sampling (mg)
W_2 = post-sampling weight of sample-containing filter (mg)
B_1 = mean tare weight of blank filters (mg).
B_2 = mean post-sampling weight of blank filters (mg)
V = volume as sampled at the nominal flowrate (i.e., 1.7 L/min or 2.2 L/min)

EVALUATION OF METHOD:

1. Bias: In respirable dust measurements, the bias in a sample is calculated relative to the appropriate respirable dust convention. The theory for calculating bias was developed by Bartley and Breuer [10]. For this method, the bias, therefore, depends on the international convention for respirable dust, the cyclones' penetration curves, and the size distribution of the ambient dust. Based on measured penetration curves for non-pulsating flow [1], the bias in this method is shown in Figure 1.

   For dust size distributions in the shaded region, the bias in this method lies within the ± 0.10 criterion established by NIOSH for method validation. Bias larger than ± 0.10 would, therefore, be expected for some workplace aerosols. However, bias within ± 0.20 would be expected for dusts with geometric standard deviations greater than 2.0, which is the case in most workplaces.

   Bias can also be caused in a cyclone by the pulsation of the personal sampling pump. Bartley, et al. [12] showed that cyclone samples with pulsating flow can have negative bias as large as −0.22 relative to samples with steady flow. The magnitude of the bias depends on the amplitude of the pulsation at the cyclone aperture and the dust size distribution. For pumps with instantaneous flow rates within 20% of the mean, the pulsation bias magnitude is less than 0.02 for most dust size distributions encountered in the workplace.

Electric charges on the dust and the cyclone will also cause bias. Briant and Moss [13] have found electrostatic biases as large as -50%, and show that cyclones made with graphite-filled nylon eliminate the problem. Use of conductive samplers and filter cassettes(Omega Specialty Instrument Co., 4 Kidder Road, Chelmsford, MA 01824) is recommended.

2. Precision: The figure 0.068 mg quoted above for the precision is based on a study [3] of weighing procedures employed in the past by the Mine Safety and Health Administration (MSHA) in which filters are pre-weighed by the filter manufacturer and post-weighed by MSHA using balances readable to 0.010 mg. MSHA [14] has recently completed a study using a 0.001 mg balance for the post-weighing, indicating imprecision equal to 0.006 mg.

   Imprecision equal to 0.010 mg was used for estimating the LOD and is based on specific suggestions [8] regarding filter weighing using a single 0.001 mg balance. This value is consistent with another study [15] of repeat filter weighings, although the actual attainable precision may depend strongly on the specific environment to which the filters are exposed between the two weighings.

REFERENCES:

[1] Bartley DL, Chen CC, Song R, Fischbach TJ [1994]. Respirable aerosol sampler performance testing. Am. Ind. Hyg. Assoc. J., *55*(11): 1036-1046.

[2] Bowman JD, Bartley DL, Breuer GM, Shulman SA [1985]. The precision of coal mine dust sampling. Cincinnati, OH: National Institute for Occupational Safety and Health, DHEW (NIOSH) Pub. No. 85-220721.

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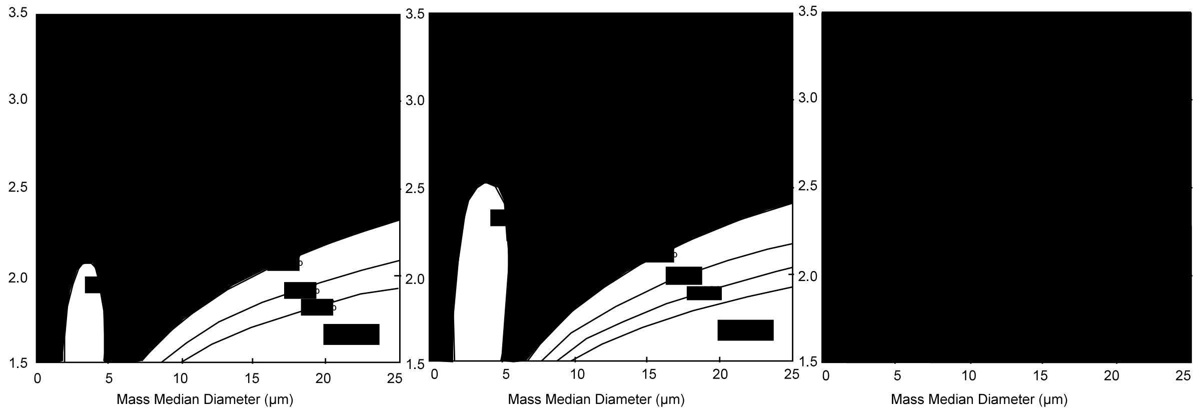

10-mm nylon cyclone at 1.7 L/min | Higgins-Dewell cyclone at 2.2 L/min | Aluminum cyclone at 2.5 L/min

Figure 1. Bias of three cyclone types relative to the international respirable dust sampling convention.

APPENDIX: Jarless Method for Calibration of Cyclone Assemblies

This procedure may be used in the field to calibrate an air sampling pump and a cyclone assembly without using the one-liter "calibration jar".

(1) Connect the pump to a pressure gauge or water manometer and a light load (adjustable valve or 5-µm filter) equal to 2" to 5" H_2O with a "TEE" connector and flexible tubing. Connect other end of valve to an electronic bubble meter or standard bubble tube with flexible tubing (See Fig. 2.1).
    NOTE: A light load can be a 5-µm filter and/or an adjustable valve. A heavy load can be several 0.8-µm filters and/or adjustable valve.
(2) Adjust the pump to 1.7 L/min, as indicated on the bubble meter/tube, under the light load conditions (2" to 5" H_2O) as indicated on the pressure gauge or manometer.
(3) Increase the load until the pressure gauge or water manometer indicates between 25" and 35" H_2O. Check the flow rate of the pump again. The flow rate should remain at 1.7 L/min ± 5%.
(4) Replace the pressure gauge or water manometer and the electronic bubble meter or standard bubble tube with the cyclone having a clean filter installed (Fig. 2.2). If the loading caused by the cyclone assembly is between 2" and 5" H_2O, the calibration is complete and the pump and cyclone are ready for sampling.

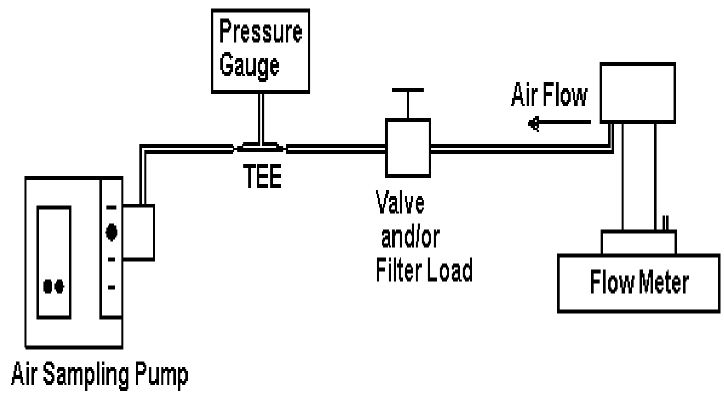

Figure 2.1 Block Diagram of Pump/Load/Flow Meter Set-up.

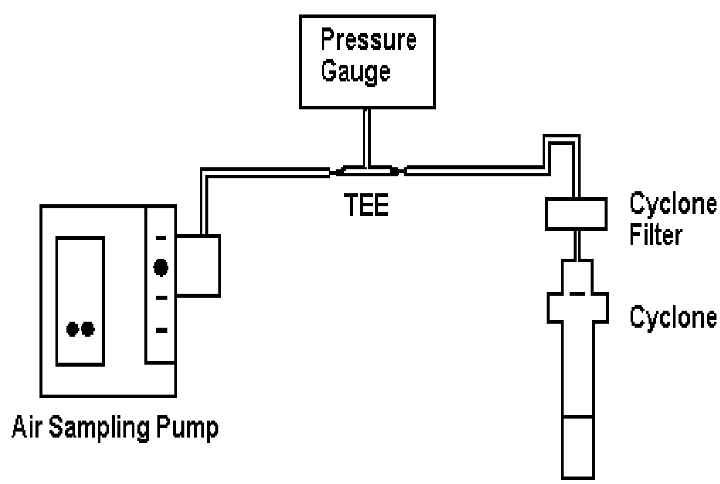

Figure 2.2. Block Diagram with Cyclone as the Test Load.