PARTICULATES NOT OTHERWISE REGULATED, RESPIRABLE 0600

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/m^3
NIOSH: no REL
ACGIH: 3 mg/m^3

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/m^3
-MAX: 400 L

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.5 to 10 mg/m^3 (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/m^3 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 flow rate shifts by a known amount between calibration and use, the nominal flow rates 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):

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

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 flow rate (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.

[3] Parobeck P, Tomb TF, Ku H, Cameron J [1981]. Measurement assurance program for the weighings of respirable coal mine dust samples. J Qual Tech *13*:157.

[4] ACGIH [1996]. 1996 Threshold limit values (TLVs™) for chemical substances and physical agents and biological exposure indices (BEIs™). Cincinnati, OH: American Conference of Governmental Industrial Hygienists.

[5] American Conference of Governmental Industrial Hygienists [1991]. Notice of intended change—appendix D—particle size-selective sampling criteria for airborne particulate matter. Appl Occup Env Hyg *6*(9): 817–818.

[6] NIOSH [1977]. NIOSH Manual of sampling data sheets. Cincinnati, OH: National Institute for Occupational Safety and Health, DHEW (NIOSH) Publication No. 77-159.

[7] Higgins RI, Dewell P [1967]. A gravimetric size selecting personal dust sampler. In: Davies CN, Ed. Inhaled particles and vapors II. Oxford: Pergammon Press, pp. 575–586.

[8] Bowman JD, Bartley DL, Breuer GM, Doemeny LJ, Murdock DJ [1984]. Accuracy criteria recommended for the certification of gravimetric coal mine dust personal samplers. NTIS Pub. No. PB 85-222446 (1984).

[9] Breslin, JA, Page SJ, Jankowski RA [1983]. Precision of personal sampling of respirable dust in coal mines. U.S. Bureau of Mines Report of Investigations #8740.

[10] Bartley DL, Breuer GM [1982]. Analysis and optimization of the performance of the 10-mm cyclone. Am Ind Hyg Assoc J *43*: 520–528.

[11] Caplan KJ, Doemeny LJ, Sorenson S [1973]. Evaluation of coal mine dust personal sampler performance, Final Report. NIOSH Contract No. PH CPE-r-70-0036.

[12] Bartley DL, Breuer GM, Baron PA, Bowman JD [1984]. Pump fluctuations and their effect on cyclone performance. Am Ind Hyg Assoc J *45*(1): 10–18.

[13] Briant JK, Moss OR [1983]. The influence of electrostatic charge on the performance of 10-mm nylon cyclones. Unpublished paper presented at the American Industrial Hygiene Conference, Philadelphia, PA, May 1983.

[14] Koqut J [1994]. Private Communication from MSHA, May 12, 1994.

[15] Vaughn NP, Chalmers CP, Botham [1990]. Field comparison of personal samplers for inhalable dust. Ann Occup Hyg *34*: 553–573.

METHOD REVISED BY:

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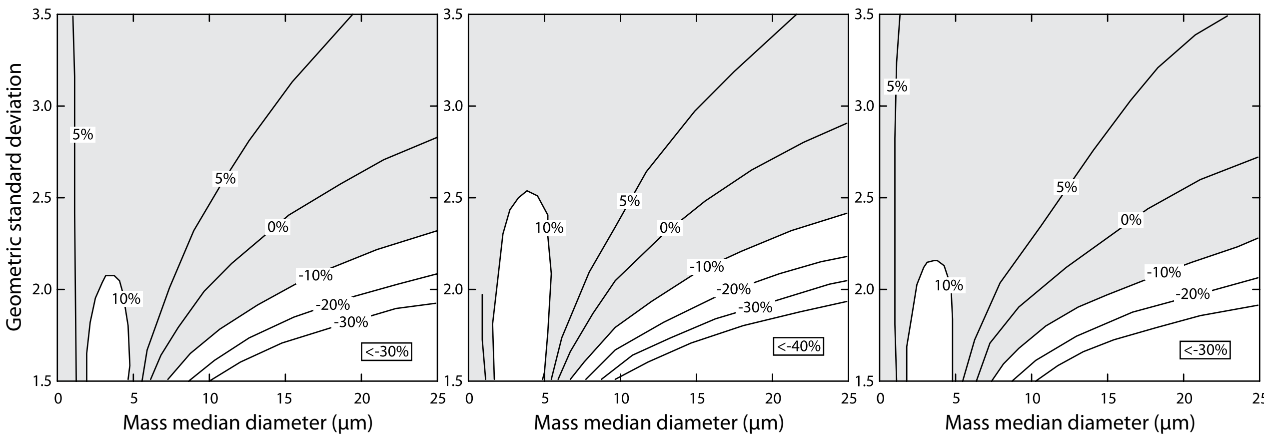

10-mm Nylon cyclone at 1.7 L/min — Higgins-Dewell cyclone at 2.2 L/min — Aluminum cyclone at 2.5 L/min

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.

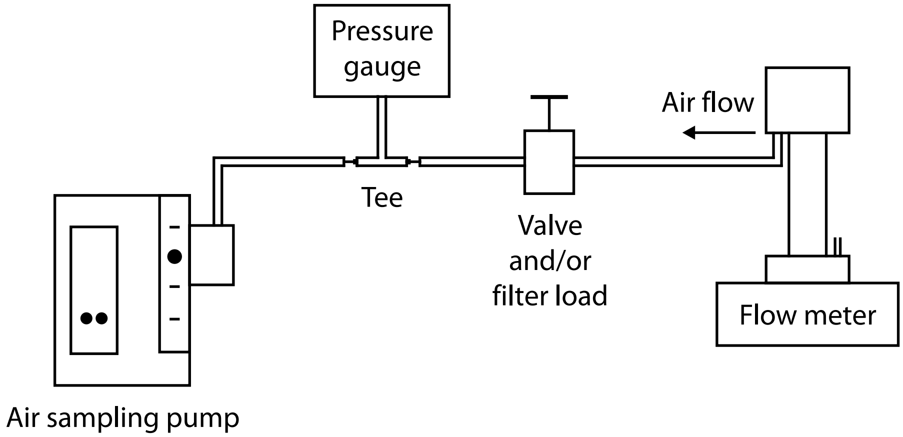

Figure 2.1. Block diagram of pump/load/flow meter set-up.

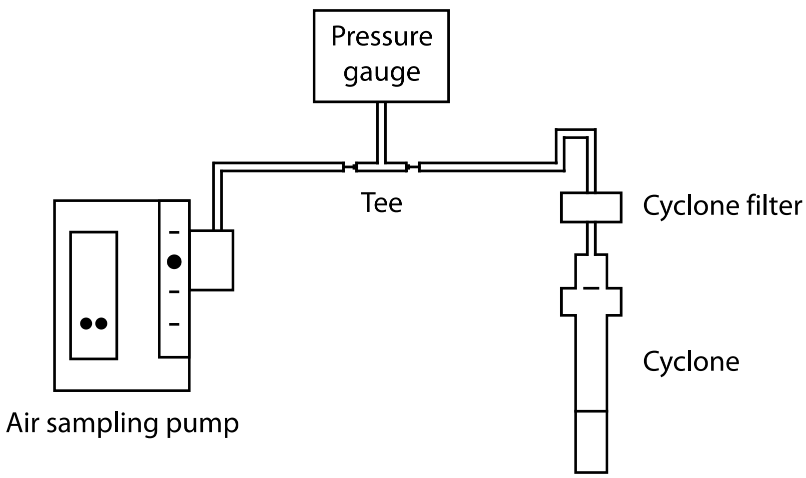

Figure 2.2. Block diagram with cyclone as the test load.