

PARTICULATES NOT OTHERWISE REGULATED, RESPIRABLE 0600

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

METHOD: 0600, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA : 5 mg/m³

NIOSH: no REL

ACGIH: 3 mg/m³

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

SYNOMYS: nuisance dusts; particulates not otherwise classified

SAMPLING		MEASUREMENT
SAMPLER:	CYCLONE + FILTER (10-mm cyclone or Higgins-Dewell (HD) cyclone + tared 5- μm PVC membrane)	TECHNIQUE: GRAVIMETRIC (FILTER WEIGHT)
FLOW RATE:	nylon cyclone: 1.7 L/min HD cyclone: 2.2 L/min	ANALYTE: mass of respirable dust fraction
VOL-MIN: -MAX:	20 L @ 5 mg/m ³ 400 L	BALANCE: 0.001 mg sensitivity; use same balance before and after sample collection
SHIPMENT:	routine	CALIBRATION: National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 weights
SAMPLE STABILITY:	indefinitely	RANGE: 0.1 to 2 mg per sample
BLANKS:	2 to 10 field blanks per set	ESTIMATED LOD: 0.03 mg per sample
ACCURACY		PRECISION: <10 μg with 0.001 mg sensitivity balance <68 μg with 0.01-mg sensitivity balance [3]
RANGE STUDIED:	0.5 to 10 mg/m ³ (lab and field)	
BIAS:	depends on dust size distributions [1]	
OVERALL PRECISION (S_{rt}):	depends on size distributions [1,2]	
ACCURACY:	depends on dust size distributions [1]	

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. Besides 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., $\approx\pm 7\%$ 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 the sample 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 condutive 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 or Higgins-Dewell (HD)[7], or equivalent.
2. Personal sampling pump, $1.7 \text{ L/min} \pm 5\%$ for nylon cyclone or $2.2 \pm 5\% \text{ L/min}$ for HD cyclone, with flexible connecting tubing.
NOTE: Pulsation in the pump flow must be within $\pm 20\%$ of the mean flow.
3. Balance, analytical, with sensitivity of 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., $20^\circ\text{C} \pm 1^\circ\text{C}$ and $50\% \pm 5\%$ 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 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.
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: Because of its square inlets, the nylon cyclone must be calibrated within a large vessel with inlet and outlet ports. The vessel inlet is connected to a calibrator (e.g., a bubble meter); whereas the outlet is connected to the sampling pump. The HD cyclone, on the other hand, can have the calibrator connected directly to the cyclone inlet. Note that 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 approximately 2 mg dust loading on the filter.
NOTE 1: 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.
NOTE 2: Take two to four replicate samples for each batch of field samples for quality assurance on the sampling procedure (see Step 10).

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 ($S_{\text{r}}\%$) 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):

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

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 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 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 0.029 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, D.L., C.C. Chen, R. Song, and T.J. Fischbach. Respirable Aerosol Sampler Performance Testing. *Am. Ind. Hyg. Assoc. J.*, **55**(11), 1036-1046 (1994).
- [2] Bowman, J.D., D.L. Bartley, G.M. Breuer, and S.A. Shulman. The Precision of Coal Mine Dust Sampling, DHEW (NIOSH) Pub. No. 85-220721 (1985).
- [3] Parobek, P., T.F. Tomb, H. Ku, and J. Cameron. Measurement Assurance Program for the Weighings of Respirable Coal Mine Dust Samples, *J. Qual. Tech.*, **13**, 157 (1981).
- [4] 1993-1994 Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices, ACGIH, Cincinnati, OH (1993).
- [5] ACGIH: Notice of Intended Change - Appendix D - Particle Size-Selective Sampling Criteria for Airborne Particulate Matter. *Appl. Occup. Env. Hyg.* **6**(9) (1991).
- [6] NIOSH Manual of Sampling Data Sheets, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-159 (1977).
- [7] Higgins, R.I. and P. Dewell, "A Gravimetric Size Selecting Personal Dust Sampler," in C.N. Davies, Ed., *Inhaled Particles and Vapors II*, pp. 575-586, Pergamon Press, Oxford (1967).
- [8] 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).
- [9] 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).
- [10] Bartley, D.L. and G.M. Breuer. Analysis and Optimization of the Performance of the 10-mm Cyclone, *Am. Ind. Hyg. Assoc. J.*, **43**, 520-528 (1982).
- [11] Caplan, K.J., L.J. Doemeny, and S. Sorenson. Evaluation of Coal Mine Dust Personal Sampler Performance, Final Report, NIOSH Contract No. PH CPE-r-70-0036 (1973).
- [12] Bartley, D.L., G.M. Breuer, P.A. Baron, and J.D. Bowman. Pump Fluctuations and Their Effect on Cyclone Performance, submitted to the *Am. Ind. Hyg. Assoc. J.* (1983).
- [13] Briant, J.K. and O.R. Moss. The Influence of Electrostatic Charge on the Performance of 10-mm Nylon Cyclones, American Industrial Hygiene Conference (1983).
- [14] Koqut, J. Private Communication from MSHA, May 12 (1994).
- [15] Vaughn NP, Milligan BD, Ogden TL. Filter weighing reproducibility and the gravimetric detection limit, *Ann. Occup. Hyg.* **33**(3), 331 (1989).

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Figure 1. Bias in respirable dust determination.