Designation: D 4532 – 97

Standard Test Method for Respirable Dust in Workplace Atmospheres[1]

This standard is issued under the fixed designation D 4532; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is useful for the determination of respirable dust (see Terminology D 1356) in a range from 0.5 to 10 mg/m^3 in workplace atmospheres.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]
D 3195 Practice for Rotameter Calibration[2]
D 5337 Practice for Flow Rate for Calibration of Personal Sampling Pumps[2]
D 6062M Guide for Personal Samplers of Health-Related Aerosol Fractions (Metric)[2]
E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *respirable fraction of the dust*—that mass which passes through a cyclone at the stated conditions **(1, 2).**[4]

4. Summary of Test Method

4.1 Air is accurately drawn for a measured period of time through a 10-mm cyclone followed by a tared filter. The respirable dust concentration is calculated from the weight gain of the filter and the total volume of air sampled.

5. Significance and Use

5.1 This test method covers the determination of respirable dust in workplace atmospheres.

5.2 The limitations on the test method are a minimum weight of 0.2 mg of dust on the filter, and a maximum loading of 0.3 mg/cm^2 on the filter. The test method may be used at higher loadings if the flow rate can be maintained constant.

6. Apparatus

6.1 The sampling unit consists of a pump and a sampling head. The sampling head consists of a 10-mm cyclone and a filter assembly.

6.1.1 *Pump*—A personal sampling pump with a flow rate accurate to ±5 %. Pump pulsation not to exceed ± 20 % of the mean flow. The pump must be capable of maintaining the mean flow constant to within ±5 % during the sampling period. Calibrate the sampling pump using Practice D 5337.

6.1.2 *Sampling Head*—The sampling head consists of a 10-mm cyclone, a filter, a filter-support pad, and a filter holder with suitable caps (see Fig. 1).

6.1.2.1 The cyclone[5] must be shown to be unbiased relative to the appropriate respirable dust criterion and the dust size distribution being sampled **(3, 4, 5, 6).** Based on the cyclone penetration curve for non-pulsating flow measured with a monodisperse aerosol, the bias in the test method is shown in Fig. 2 for sampling rates appropriate for individual cyclones. **(7).**

6.1.2.2 Cyclone samples collected with pulsating flow have been shown to yield a negative bias as large as 22 % compared to samples collected under steady flow **(8).**

6.1.2.3 Electrostatic charge on the dust and a non-conductive sampler can cause bias as large as 50 %. **(9).**

6.1.3 The filter shall be non-hygroscopic and a collection efficiency greater than 95 % for the dust cloud of interest. The filter and its filter support shall be 37 mm in diameter.

NOTE 1—As an example, most glass fiber and membrane filters with nominal pore size of 5 µm will nearly always fulfill this requirement **(10).** PVC is recommended for gravimetric analysis. The equilibrated filter is preweighed by the user. The weight of the filter holder is not used in any determination of weight gain in this test method. The filter holder material must not contribute to any weight change of the filter.

[1] This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.04 on Workplace Atmospheres.

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[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[5] The sole source of the 10-mm cyclones known to the committee at this time is Dorr-Oliver, Inc., Milford, CT 06460. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

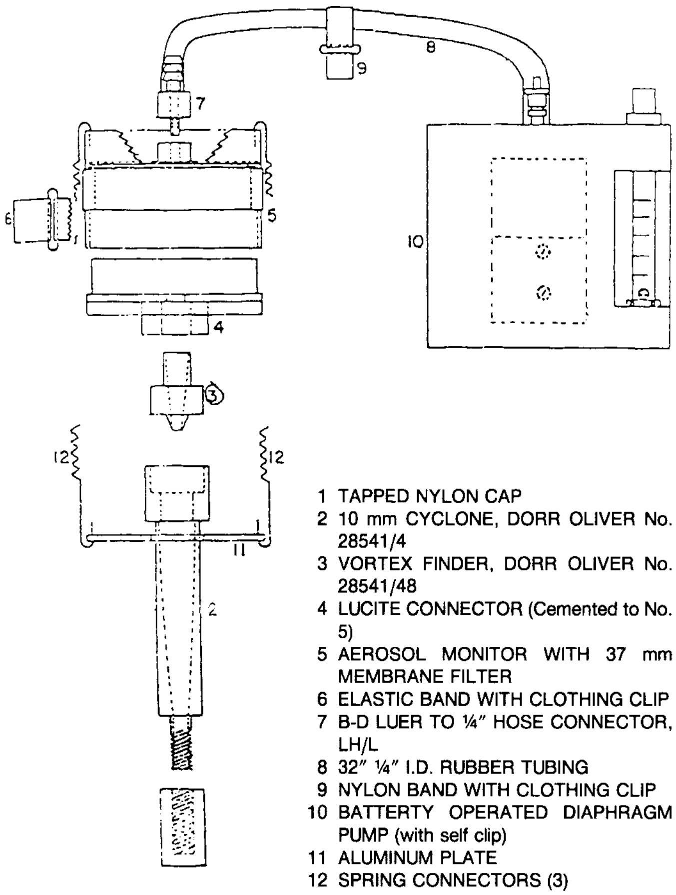

FIG. 1 Example of Personal Sampler for Respirable Dust

6.1.4 *Charger*—Pump batteries shall be completely charged with appropriate charger following the manufacturer's instructions or disposable batteries may be used.

6.1.5 Suitable means is provided for separately attaching the pump and the sampling head to the appropriate person.

6.2 *Buret*, capacity of 1 L, used as a soap bubble meter for calibration of the sampling unit.

6.3 *Barometer*, capable of measuring atmospheric pressure to ±0.1 kPa.

6.4 *Stopwatch*, capable of measuring to ±0.1 s.

6.5 *Weighing Room*, with temperature and humidity control to allow weighing with an analytical balance to ±0.01 mg.

6.6 *Analytical Balance*, capable of weighing ±0.01 mg or better. Particular care must be given to the proper zeroing of the balance. The same analytical balance and weights must be used for weighing filters before and after sample collection.

6.7 *Charge Neutralizer*, to eliminate static charge in the balance case and on the filters during weighing. Replace Po-210 neutralizers 9 months after production date.

6.8 *Plane-Parallel Press*, capable of giving a force of at least 1000 N (may be required if plastic filter holders are used that must be pressed together after insertion of the filter).

6.9 *Tapered Tube Flow Meter*, with precision ±2 % or better within the range of the flow rate used. Calibrate the meter using Practice D 3195.

6.10 *Thermometer*, dry bulb, 0 to 50°C with divisions every 0.1°C. (ASTM thermometers number 90C and 91C.) (See Specification E 1.)

6.11 *Manometer*, 0 to 250 mm of water (0 to 0.25 kPa) for measuring the pressure drop across the sampling head.

6.12 *Flexible Tube with Two Clips*, one near the sampling head, if the sampling head does not have a clip, and the other midway between the sampling head and the pump. The length of the tube is dependent on how the sampling unit is worn. A length of 0.7 to 0.9 m is suitable if the pump is attached to the worker's belt.

6.13 *Jar*, leakproof, of suitable size to contain the sampling head during calibration of sampling system. (See and Fig. 3).[6]

7. Sampling

7.1 Clean and inspect the interior of the cyclone. If the inside surfaces are visibly scored, replace the cyclone since the dust separation characteristics might be altered.

7.2 Condition all filters to a constant weight. Record the filter weight.

7.3 Place the tared filter and filter support in the filter holder, close firmly, and tape the circumference of the filter holder. If necessary, use the press described in 6.8. Suitably cover the assembly to avoid contamination if it is held for any time prior to use.

7.4 Assemble sampling apparatus as shown in Fig. 1.

7.5 Run the pump for 5 min to stabilize the flow rate.

7.6 Remove the filter holder caps and connect the filter holder to the cyclone as required by the manufacturer. Connect the outlet of the sampling head to the pump's inlet with a piece of flexible tubing. Check to be sure all connections are free of leaks by closing off the filter inlet. Flow should stop in 10 to 15 s.

7.7 Check the sampling unit for proper operation, check for leaks, and measure the flow rate.

7.8 Sample at 1.7 L/min for the Dorr-Oliver 10-mm cyclone, or as directed by manufacturer of specific cyclones. Depending on sample load, consecutive samples over the shift may be required. However, the sampling time should not exceed the operating life of the batteries or the prevailing "full shift." The nominal sampling period is 8 h. Sampling times shorter than a full shift are permitted if the following occurs:

7.8.1 The pressure drop across the filter exceeds the pump's capabilities; that is, the filter becomes clogged.

7.8.2 Specific working operations of shorter duration are to be investigated.

7.8.3 Determinations of variations of the exposure during a shift are made.

7.9 Attach the sampling head to the worker so that it is located in the breathing zone. The worker's breathing zone consists of a hemisphere 300-mm radius extending in front of the face, and measured from a line bisecting the ears. The sampling head shall be placed in such a manner to prevent dust from falling into it and to avoid restricting the inlet. The pump can be attached to the worker's belt.

7.10 Initiate sampling by turning the pump on and setting

[6] The sole source for the 10-mm nylon cyclone calibrating jar known to the committee at this time is Fischer Scientific, 711 Forbes Ave. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee[1] which you may attend.

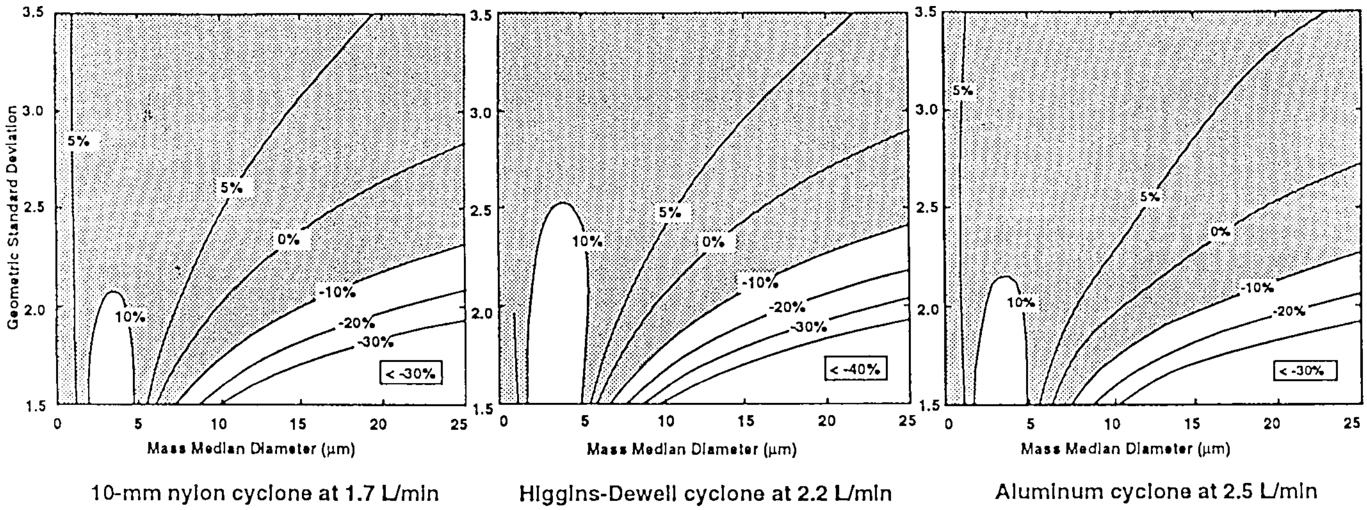

FIG. 2 Bias of Three Cyclone Types Relative to the International Respirable Dust Sampling Convention

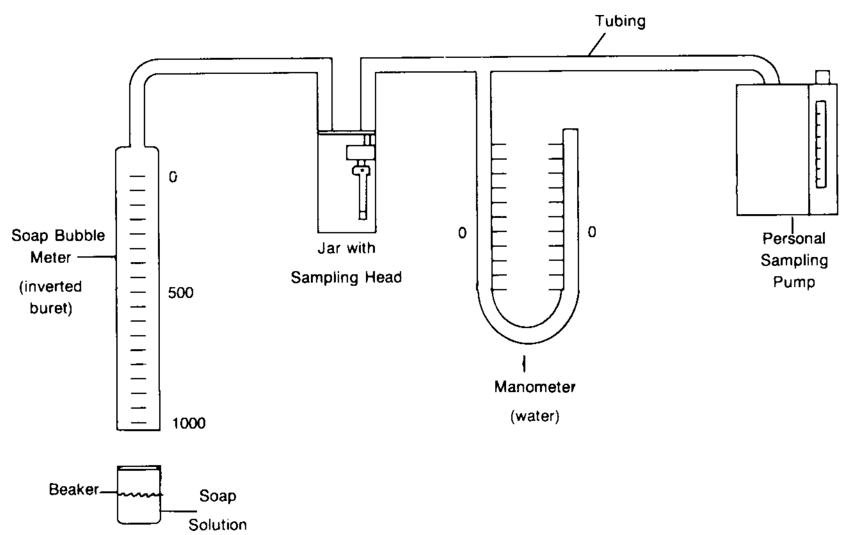

FIG. 3 Example of the Calibration Setup for Personal Sampling Pump with Respirable Dust Sampling Head

the flow rate determined in 8.5 and according to the manufacturer's instructions. Record the flow rate and the time. If the flow rate changes during sampling by more than ±5 %, record the change and the time. Reset the flow rate. If unable to reset the flow rate to the original setting, terminate sampling and note the reason for termination. Follow Guide D 6062M for personal samplers.

7.11 At the end of the sampling period, turn the pump off and record the final flow rate and time.

7.12 Replace the filter holder caps. Remove the sampling unit from the worker.

7.13 For each set of 10 or fewer samples, submit a blank sample. The filters and filter holders to be used as blanks are prepared and transported in the same manner as the samples except that no air is drawn through them. Label these as blanks.

7.14 The filter assembly should be returned to the laboratory in a suitable container designed to prevent sample damage in transit.

8. Calibration and Standardization

8.1 Air flow calibration of the sampling unit should be completed before and after each sampling session. (See Fig. 3.) Maintenance and repairs, according to the manufacturer's instructions, should be performed on a regular schedule and records kept for documentation.

8.2 The choice of calibration instrument will depend largely on where the calibration is to be performed. A soap bubble meter or spirometer is recommended. Instructions for calibration of the sampling unit with the soap bubble meter are provided in 8.3-8.9 and Fig. 3. If a spirometer is selected, an appropriate procedure shall be used. Since the flow rate given by a pump is dependent on the pressure drop across the sampling device, for instance the filter and cyclone, the pump must be calibrated while operating with a representative sampling head in line. Calibration of the sampling unit should be performed at approximately the same temperature and pressure that the sample will be collected.

8.3 Place the sampling head, with the same type of filter to be used to collect the sample, in the jar. Connect the sampling head to the outlet of the jar and connect the outlet of the jar to the pump to be calibrated.

8.4 Turn on the pump and moisten the inside of the bubble meter by drawing bubbles up the meter until the bubbles are able to travel the entire length of the buret without bursting.

8.5 Adjust the pump to provide the desired flow rate.

8.6 Start a soap bubble up the buret and measure with a stopwatch the time it takes the bubble to pass from the zero line to the 1.0 L mark.

8.7 Repeat the procedure in 8.5 at least three times. Calculate the flow rate by dividing the volume of air between the preselected marks of the buret by the time required for the soap bubble to traverse the distance and average the results. If the measured flow rate is outside the specification, readjust as in 8.5 and repeat 8.6 and 8.7.

8.8 Record the date of the calibration and the temperature and pressure at the time of the calibration.

8.9 Alternatively, the calibrated flow meter (6.9) may be used to field check flow rate at the beginning and end of sampling. Connect the outlet side of the flow meter to the inlet (vacuum side) of the pump. Connect the inlet side of the flow meter to the outlet of the jar, which contains the sampling head. Turn on the pump and determine flow rate from a calibration chart prepared for the flow meter by comparison with a soap bubble flow meter or spirometer.

9. Procedure

9.1 Carefully swab the outer surface of the filter assembly with a lintless paper towel moistened with water before opening the filter holder to minimize sample contamination.

9.2 Open the filter holder and carefully remove the filter from the holder with the aid of a rod inserted into the outlet hole of the filter holder and a filter tweezer. Handle the filters very gently by the edge to avoid loss of dust. Transfer the filter to a petri dish. Brush out the filter holder and add to the filter any dust adhering to the inside of the filter holder. Place the filter in the weighing room. Handle the equilibrated filter as stated in 7.2.

9.3 Weigh the filter on the same analytical balance that was used to determine the tare weight (6.6).

10. Calculation

10.1 Mass of dust found on the sample filter:

$$M_s = (m_2 - m_1) - m_3 \qquad (1)$$

where:
m_1 = tare weight, in mg, of the clean filter before sampling,
m_2 = the weight, in mg, of the sample-containing filter,
m_3 = the mean value of mass, in mg, found on the blank filter, and
M_s = mass found on the sample filter.

10.2 The sampled volume is:

$$Vs = Q \times t/1000 \qquad (2)$$

where:
Q = the mean indicated flow rate, in L/min of air sampled,
t = the sampling time, in min,
Vs = the volume, in m^3, of the air sampled, and
1000 = conversion from L to m^3.

NOTE 2—There are no temperature or pressure corrections for changes in sampled volume since it is critical that the flow rate required for the cyclone be set at the time and location of sampling.

10.3 The concentration of the respirable dust in the sampled air is expressed in mg/m^3.

$$C = K \times M_s/V_s \qquad (3)$$

where:
C = mass concentration of respirable dust,
K = a correction factor for the cyclone to convert to respirable mass fraction (supplied by the manufacturer),
M_s = mass, in mg found on the sample filter, see 10.1, and
V_s = the volume, in m^3, of air sampled, see 10.2.

11. Precision and Bias

11.1 *Analytical Range*, 0.2 mg to 2 mg per sample.

11.2 *Estimated Limit of Detection*, 0.2 mg per sample.

11.3 *Analytical Precision*, 68 µg when a 0.01 mg balance is used. (See Ref **(11).**)

11.4 *Range Studied*, 0.5 to 10 mg/m^3 (lab and field).

11.5 *Bias*, depends on the dust size distributions and in the respirable aerosol convention used. (See Ref **(12), (13)**, and laboratory references therein, and Fig. 2.)

11.6 *Precision*:

11.6.1 The overall same laboratory precision for 10-mm nylon cyclone collected respirable dust samples is shown to be most sensitive to two factors: the analytical precision and the sampling procedures, particularly the quality control system used in maintenance and calibration of samplers. Theoretically, the variance for the overall precision is the sum of the variances from the sampling and the analysis. The analytical variance depends on the dust loading on the filter. For an 8-h sample and dust loading above 1.5 mg/m^3, Bowman, et al. **(4)** find that the empirically determined sampling error dominates this analytical error.

11.6.2 Because of the effects of the environment, precision estimates for dust samplers are much more variable than those reported for gas and vapor sampling. In laboratory tests with 0.01-mg sensitivity balances, the overall precision for a single respirable dust sample has relative standard deviations (RS) from 0.043 to 0.145 over concentrations ranging from 0.5 to 10.0 mg/m^3. In the laboratory studies where the dust concentrations in the test chamber are more carefully controlled, the estimated RSD is less than 0.091, which is the target precision value for a bias equal to ±0.010 in the National Institute for Occupational Safety and Health validation criteria.

11.6.3 In the field tests with 0.01-mg sensitivity balances, precision estimates range from 0.144 to 0.227 over concentrations ranging from 1 to 10 mg/m^3. Whether the larger precision values in the field tests are due to sampler performance or to less homogeneous dust concentrations in the field tests cannot be determined from existing data.

12. Keywords

12.1 air monitoring; respirable dust; sampling and analysis; workplace atmospheres

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