Designation: D4532 – 15

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

This standard is issued under the fixed designation D4532; 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 provides details for the determination of respirable dust concentration defined in terms of international convention in a range from 0.5 to 10 mg/m^3 in workplace atmospheres. Specifics are given for sampling and analysis using any one of a number of commercially available cyclone samplers.

1.2 The limitations on the test method are a minimum weight of 0.1 mg of dust on the filter, and a maximum loading of 0.3 mg/m^2 on the filter. The test method may be used at higher loadings if the flow rate can be maintained constant.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 This test method contains notes that are explanatory and are not part of the mandatory requirements of the method.

1.5 *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:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres
D3195 Practice for Rotameter Calibration
D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps
D6062 Guide for Personal Samplers of Health-Related Aerosol Fractions
D6552 Practice for Controlling and Characterizing Errors in Weighing Collected Aerosols
D7440 Practice for Characterizing Uncertainty in Air Quality Measurements
E1 Specification for ASTM Liquid-in-Glass Thermometers
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *Other International Standards:*[3]

ISO GUM Guide to the Expression of Uncertainty in Measurement, ISO Guide 98
ISO 7708 Air Quality—Particle Size Fraction Definitions for Health-Related Sampling
ISO 13137 Workplace Atmospheres – Pumps for Personal Sampling of Chemical and Biological Agents – Requirements and Test Methods
ISO 15767 Workplace Atmospheres—Controlling and Characterizing Errors in Weighing Collected Aerosol
EN 481 Workplace Atmospheres—Size Fraction Definitions for the Measurement of Airborne Particles in the Workplace
EN 13205 Workplace Atmospheres—Assessment of Performance of Instruments for Measurement of Airborne Particle Concentrations

3. Terminology

3.1 *Definitions of Terms Specific to This Standard*—(otherwise, consult Terminology D1356):

3.1.1 *respirable convention*—target specification for sampling instruments when the respirable fraction is the fraction of interest.

3.1.2 *respirable fraction*—mass fraction of total airborne particles which penetrate to the unciliated airways.

3.1.3 *respirable sampler*—aerosol sampler that is used to collect the respirable fraction of airborne particles from the surrounding air.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.1.3.1 *Discussion*—Fig. 1 shows the collection efficiency of an idealized sampler following the internationally-harmonized sampling conventions of ISO 7708, EN 481, Guide D6062, and Ref. (**1**).[4]

NOTE 1—The definition of the respirable fraction is a compromise between previous definitions, available samplers, and the fraction of dust that penetrates to (rather than deposits in) the alveolar region of the lung. Local legal definitions may differ from the definition adopted in this test method.

3.2 For the terms and definitions related to characterizing uncertainty, see ISO GUM and Practice D7440.

4. Summary of Test Method

4.1 Air is drawn through a cyclone or equivalent sampler followed by a tared filter, which is then re-weighed to determine the mass of respirable dust. The air flow rate and time of sampling provide the volume from which the dust mass was sampled. A time-weighted average respirable dust concentration is calculated by dividing the mass by the total air volume.

NOTE 2—Samplers alternative to a cyclone (for example, foam-based or personal cascade impactors) may be used if they have desirable properties (for example, ease of use or uncertainty control) for intended application. Nevertheless, this test method is limited to cyclone samplers.

5. Significance and Use

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

5.2 Variations of the test method are in world-wide use for determining compliance relative to occupational exposure levels.

5.3 The test method may be used to verify dust control measures.

5.4 The test method may also be applied in research into health effects of dust in an occupational setting.

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

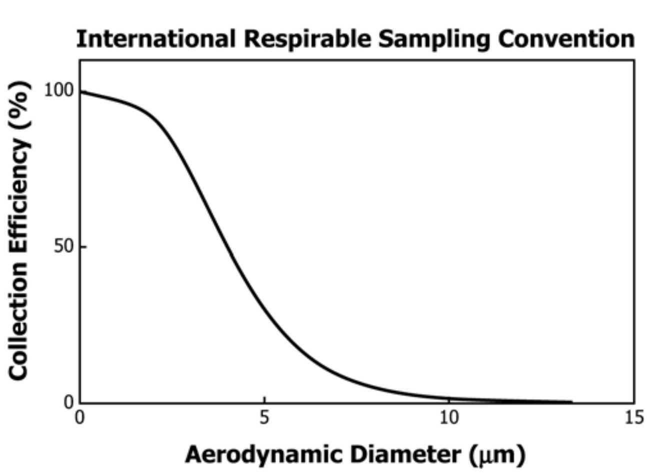

FIG. 1 Collection Efficiency of an Ideal Sampler Following the International Sampling Conventions

6. Apparatus

6.1 *Sampling Unit*—The sampling unit consists of a pump, a sampling head, and tubing connecting the pump and outlet of the sampling head. The sampling head consists of a cyclone and a filter assembly.

6.1.1 *Respirable Dust Cyclone*—Various types of respirable dust cyclones are commercially available. In general, these samplers can be categorized into two groups, high flow rate cyclone samplers (flow rate range 4.2 – 10 L/min) and medium flow rate cyclone samplers (flow rate range 1.7 – 2.75 L/min). High flow rate samplers should be considered for workplaces where airborne particle concentrations are low (for example, <0.05 mg/m^3) or where it is desirable to take short-term exposure measurements (for example, <4 hours) (**2-4**).

NOTE 3—Bias relative to the international respirable dust criterion and the dust size distribution being sampled (**2-13**) must be controlled sufficiently (see 13.2.4) for the application of intended use.

6.1.2 *Filter Cassette Assembly*—Filter, filter-support pad, and filter cassette holder with suitable caps. The filter shall be non-hygroscopic and a collection efficiency greater than 95 % for the dust of interest.

NOTE 4—As an example, most glass fiber and membrane filters with nominal pore size of 5 µm will fulfill this requirement (**14**). PVC is recommended for gravimetric analysis. The equilibrated filter is pre-weighed by the user.

NOTE 5—It is preferable to use a conductive cassette because electrostatic charge on the dust and a non-conductive cassette can result in a significant bias (**15-20**). For controlling dust which may become attracted to the interior cassette walls, several filter holders equipped with a shielded respirable dust filter and cassette are commercially available, which may be weighed together with the filter.

6.1.3 *Personal Sampling Pump*—With a flow rate uncertainty (see 13.2.1) less than 5 %. The pump pulsation amplitude may not exceed ±10 % of the mean flow according to ISO 13137 method (**21**) or ±25 % of the mean flow if pump pulsation is measured at the inlet of the cyclones (**22**). The nominal sampling flow rate for each cyclone type is adjusted using Practice D5337.

NOTE 6—Cyclone samples collected with pulsating flow have been shown to yield a negative bias as large as 70 % compared to samples collected under steady flow (**22**).

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

6.3 *Weighing Room*—With temperature (20 ± 2°C) and humidity (50 ± 5 % Relative Humidity (RH)) control to allow weighing with an analytical balance to accuracy required. See ISO 15767 and Practice D6552 for controlling and characterizing errors in weighing collected aerosols.

NOTE 7—If a weighing room is not available, a filter equilibration chamber can be used to equilibrate the filters in a temperature (20 ± 2°C) and humidity (50 ± 5 % RH) controlled chamber.

6.4 *Analytical Balance*—Capable of weighing to 0.01 mg or better, depending on application. Particular care must be given to the proper zeroing of the balance.

6.5 *Charge Neutralizer*—To eliminate static charge in the balance case and on the filters during weighing. Po-210 neutralizers if used must be replaced within nine months of their production date.

6.6 *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).

6.7 *Flow Meter*—With precision equal to 2 % or better within the range of the flow rate used. Various flow meters are introduced in Practice D5337. Calibration of rotameter can be performed using Practice D3195.

6.8 *Thermometer*—Capable of covering the temperature range of interest with divisions every 0.1°C (see Specifications E1 and E2251).

6.9 *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.

6.10 *Forceps*—Preferably nylon.

6.11 *Rod or filter lifter.*

6.12 *Petri Dishes or Filter Keepers*—With diameter slightly greater than the filter.

7. Preparation of Samplers Prior to Sampling

7.1 Inspect the interior of the cyclone and clean it to keep away from reentrainment of large particles. If the inside surfaces are visibly scored, replace the cyclone since the dust separation characteristics of the sampler might be altered.

7.2 Equilibrate all the filters in an environmentally controlled weighing room or equilibration chamber for at least 24 hours.

7.3 Weigh the filters in the weighing room.

7.3.1 Internally calibrate the balance (zero balance) before use.

7.3.2 Grasp the filter with forceps and pass the filter several times through a charge neutralizer to eliminate static charge, if necessary.

7.3.3 Record the weight of filters.

7.4 Place the tared filter and filter support in the filter cassette holder, close firmly, and tape the circumference of the filter holder. If necessary, use the press described in 6.6.

7.5 Place caps to the filter holder and suitably cover the assembly to avoid contamination if it is held for any time prior to use.

8. Sampling

8.1 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 calibrated pump's inlet with a piece of flexible tubing. Make sure all connections are free of leaks by closing off the filter inlet.

8.2 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 30-cm 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.

8.3 Initiate sampling by turning the pump on and record the flow rate and the time. For long-term sampling, periodically check the pump whether the pump functions properly. If a noticeable change of the flow rate is visually observed due to bending or blockage of tubing, turn off the pump and reset the flow rate. If unable to reset the flow rate to the original setting, terminate sampling and note the reason for termination.

NOTE 8—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 eight hours. Sampling times shorter than a full shift are permitted if the following occurs:

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

Specific working operations of shorter duration are to be investigated.

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

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

8.5 Remove the sampling unit from the worker and carefully take the sampling equipment to a clean, dust-free area.

8.6 Measure the pump flow rate using the calibrated flow meter (see Practice D5337 for the measurement and adjustment of flow rate). If the flow rates before and after sampling differ by more than 5 %, consider the sample to be invalid.

8.7 Remove the filter holder from the sampling head and replace the filter holder caps.

8.8 For each set of ten or fewer samples, submit a field 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.

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

NOTE 9—The sampler must not be inverted at any time or else re-deposition of particles from the cyclone body onto the filter may occur.

NOTE 10—The preferred procedure is to personally transport samples back to the laboratory such as by car or carry-on aircraft baggage. If collected samples need to be shipped by a shipping service, place sample packages inside larger boxes and cushion with packing materials.

9. Flow Rate Adjustment and Standardization

9.1 Air flow rate adjustment of the sampling unit should be completed before each sampling session. Maintenance and repairs, according to the manufacturer's instructions, should be performed on a regular schedule and records kept for documentation. See Practice D5337 for guidance on flow rate adjustment.

NOTE 11—Make sure that the pump is connected to an appropriate sampling train, in the order of pump, tubing, inlet of sample media holder, and the cyclone connected to the sample media holder.

NOTE 12—Some respirable dust samplers are accompanied with their own chamber to adjust nominal flow rate and thus, do not need a jar; refer to the manufacturer's manual. Alternatively, a calibrated flow meter (see 6.7) may be used to check the field flow rate at the beginning and end of sampling.

NOTE 13—It is critical that the flow rate required for the sampler be set at the time and location of sampling. If the temperature and pressure in the sampling environment differ from where the pump flow rate was set, the volumetric flow rate needs to be readjusted prior to sampling.

10. Procedure

10.1 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.

10.2 Remove the caps of the filter holder and equilibrate in an environmentally controlled room or equilibration chamber for at least two hours.

10.3 Open the filter holder and carefully remove the filter with forceps from the holder with the aid of a rod or filter lifter inserted into the outlet hole of the filter holder. Handle the filters very gently by the edge to avoid loss of dust. Transfer the filter to a petri dish. Place the filter in the weighing room.

10.4 Weigh the filter preferably on the same analytical balance that was used to determine the tare weight. If the original balance is not available or is inoperative, then an alternative analytical balance can be used for the second reading, capable of weighing to the nearest 0.01 mg or better as needed. Record anything notable about the filter such as overloading, leakage, torn and so on.

NOTE 14—The balance shall be regularly calibrated using ASTM Class I weights or equivalent NIST-traceable certified weights. An alternative balance that would be used in the procedure shall be calibrated as the same level as the original balance.

10.5 After weighing the filter, make sure to re-zero the balance prior to weighing next filter.

11. Calculation

11.1 The mass M_s (mg) of dust found on the sample filter is calculated as:

$$M_s = (m_2 - m_1) - \delta m_b \quad (1)$$

where:

m_1 = tare weight (mg) of the filter before sampling,
m_2 = mass (mg) of the filter plus aerosol sample, and
δm_b = average mass increase or decrease (mg) of the blank filter.

11.2 The sampled volume V_s (m^3) is:

$$V_s = (Q\,t)/(1000\ L/m^3) \quad (2)$$

where:

Q = mean flow rate (L/min) of air sampled, and
t = sampling time (min).

11.3 The concentration C (mg/m^3) of the respirable dust in the sampled air is:

$$C = M_s/V_s \quad (3)$$

where:

M_s = mass (mg) found on the sample filter (see 11.1), and
V_s = volume (m^3) of air sampled (see 11.2).

12. Report

12.1 A report should include the following information: date of sampling (optionally with weather conditions including temperature, pressure, humidity, velocity, and wind direction); date of shipping; person performing sampling; employee information including name and contact information; personal protective equipment used; job description; types of dust; pump number; pre- and post-sampling pump flow rate; sample duration in minutes (pump on/off time); volume of air collected; type of sample media; initial and final filter weight; dust weight gained; number of field blank filters; mean value of mass found on the field blank filters; concentration of the respirable dust in the sampled air (generally to 0.01 mg/m^3 to avoid uncertainty associated with rounding); and an uncertainty budget. Table 1 shows an example of the uncertainty budget if C_{resp} is found to be equal to 1 mg/m^3 in sampling at Q = 2 L/min for t = 8 h.

NOTE 15—The relative uncertainty associated with weighing depends on the mass sampled and will generally not equal 1 % as in Table 1 (see 13.2.3).

13. Uncertainty of Respirable Mass Concentration Estimates[5]

13.1 *General:*

13.1.1 In any sampling method, factors that can influence the overall uncertainty include:

13.1.1.1 *Components Associated with the Sampling Procedures*—Flow rate measurement (calibration), pump flow stability, sampling efficiency, inter-sampler variability, temporal variability, losses to sampler walls, environmental influence parameters (such as temperature, pressure, humidity and etc.), and sample storage and transportation.

13.1.1.2 *Components Associated with the Analysis*—Sample preparation, analytical precision of balance, operator effects, and rounding error in reporting.

13.1.2 For respirable dust sampling in workplace atmospheres, the overall uncertainty is shown to be most sensitive to four factors: pump flow rate uncertainty, inter-sampler variability, weighing, and sampler bias. The sampler bias depends on the dust size distributions and on the respirable aerosol convention used. (See Refs. (**14, 23**), and laboratory references therein.) Losses to walls may be controlled by using a sampler designed so that the part of the aerosol sample

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D22-1033. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Relative Uncertainty Budget with Specific Sampling Conditions if the Estimated Respirable Dust Concentration = 1 mg/m^3

Uncertainty Source	Uncertainty Component	Degrees of Freedom	Type
Bias Estimation	5 %	∞	B
Weighing	1 %	25	B
Flow Rate Variation	5 %	∞	B
Sampler Variation	6 %	15	B
Combined Uncertainty = 9.3 %			
Expanded Uncertainty = 18.7 % at coverage factor *k* = 2			

acquired by the walls is analyzed. Problems with sample storage and transportation are assumed under control for the purposes of this standard.

13.2 *Specific Uncertainty Components:*

13.2.1 *Sampling Pump Flow Rate Uncertainty—u_{pump}* (Type B Evaluation):

13.2.1.1 Various types of flow devices such as rotameters, mass flow meters, bubble flow meters, or dry piston flow meters are used to measure flow rate, and pump stability may be controlled electronically or by manual measurement both before and after sampling. (See Practice D5337 for the measurement and adjustment of flow rate.) Accounting for uncertainty in flow rate adjustment, reading, and pump stability during operation, the combined pump flow rate uncertainty is taken (**24**) as:

$$u_{pump} = 5\ \% \quad (4)$$

13.2.2 *Uncertainty from Inter-Sampler Variation—$u_{sampler}$* (Type B Evaluation):

13.2.2.1 Slight variations in the dimensions of the samplers can affect the collection efficiency of respirable aerosol samplers. For example, a value of 6 % was found for the relative standard deviation in sampling dust sizes of interest when laboratory tests were performed by taking four replicate efficiency measurements with eight samplers of each of two specific types of cyclone samplers (**9**). This figure is adopted here, for respirable samplers in general, assuming about 15 degrees of freedom in evaluation:

$$u_{sampler} = 6\ \% \quad (5)$$

NOTE 16—This value for the intra-sampler variation may be conservative in the case of samplers manufactured at tighter tolerance than in the original experiment.

13.2.3 *Uncertainty of Weighing—$u_{weighing}$* (Type B Evaluation):

13.2.3.1 The uncertainty of weighing depends primarily on the analytical precision of balance, climate control, and calibration of electronic circuits. Filter sensitivity to temperature and humidity variation can be minimized through the use of at least a single field blank. The combined (relative) uncertainty for weighing is estimated from the results of an evaluation (**25**) of balance readings to 0.01 mg. The weighing uncertainty is taken as:

$$u_{weighing} = 0.068\ mg/(C_{resp} Q\ t) \quad (6)$$

where:

C_{resp} = estimated respirable concentration (mg/m^3)
Q = pump flow rate (m^3/min), and
t = sampling time (min).

NOTE 17—The value taken here as the weighing uncertainty will generally be conservative if a balance read to 0.01 mg is used. A more realistic value for the weighing uncertainty can then be adopted if the laboratory conducting the weighing documents the uncertainty as with ISO 15767.

13.2.4 *Uncertainty of Sampler Bias—u_{bias}* (Type B Evaluation):

13.2.4.1 The uncertainty of sampler bias is estimated as follows. The bias at a given size distribution can be estimated from the sampling efficiency curve known for a cyclone against the respirable convention and is defined as:

$$bias = (C_{sampler} - C_{convention})/C_{convention} \quad (7)$$

where $C_{sampler}$ and $C_{convention}$ are sampler and convention estimates, respectively.

13.2.4.2 In accordance with EN 13205, the uncertainty of sampler bias is estimated from the variance in the bias computed in sampling lognormal size distributions with mass median diameters (MMD) equal to 1, 2, …, 50 µm and geometric standard deviation (GSD) equal to 1.75, 2.00, 2.25, …, 4.00 (and if the respirable fraction > 0.05, MMD*GSD < 100 µm, and MMD/GSD > 0.5 µm):

$$u_{bias} = \sqrt{\frac{1}{n}\sum_{j=1}^{n}\left(\overline{bias} - bias_j\right)^2} \quad (8)$$

where j labels one of the n (MMD, GSD) pairs.

13.2.4.3 Details are presented in Research Report RR:D22-1033 for this standard and indicate that a respirable sampler type can be selected so that conservatively:

$$u_{bias} = 5\ \% \quad (9)$$

13.2.4.4 This value is then taken as the bias uncertainty for the purposes of this standard.

NOTE 18—If a sampler has desirable characteristics, but has bias uncertainty larger than 5 %, then its documented uncertainty replaces the above value.

NOTE 19—Compliance agencies generally avoid the bias uncertainty entirely by adopting a specific single sampler type, rather than the international sampling convention.

13.3 *Combined Standard Uncertainty and Expanded Uncertainty:*

13.3.1 The combined (relative) standard uncertainty (u_c) is found from pooling the uncertainties from each source:

$$u_c = \sqrt{(u_{pump}^2 + u_{sampler}^2 + u_{weighing}^2 + u_{bias}^2)} \quad (10)$$

13.3.2 Then finally, the expanded uncertainty (U) is calculated by multiplying the combined standard uncertainty by a coverage factor k:

$$U = k\, u_c \quad (11)$$

where typically, k ranges from 2 to 3, but for simplicity this practice adopts the traditional coverage factor 2.

14. Keywords

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

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