Designation: ~~D4532 – 15~~ D4532 – 22

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~~0.5 mg/m^3 to ~~10~~ 10 mg ~~mg/m~~/m^3 in workplace ~~atmospheres.~~ atmospheres, depending on sampling time. 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~~dependent [2]on ~~the filter.~~ sampler type and time of sampling. 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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

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[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.
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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.

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

ISO 18158 Workplace Atmospheres - Terminology

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 For definitions of terms used in this test method, refer to Terminology D1356.

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

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

3.2.1 *respirable fraction*—mass fraction of total airborne particles which penetrate to the unciliated airways. **(ISO 18158)**

3.2.2 *respirable sampler*—aerosol sampler that is used to collect the ~~respirable~~ fraction of airborne particles according to the respirable convention (see Terminology D1356) from the surrounding air. **(modified from ISO 18158)**

3.2.2.1 *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~~convention 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.

3.3 For ~~the~~ terms and definitions related to characterizing uncertainty, see ISO GUM (ISO Guide 98) 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.

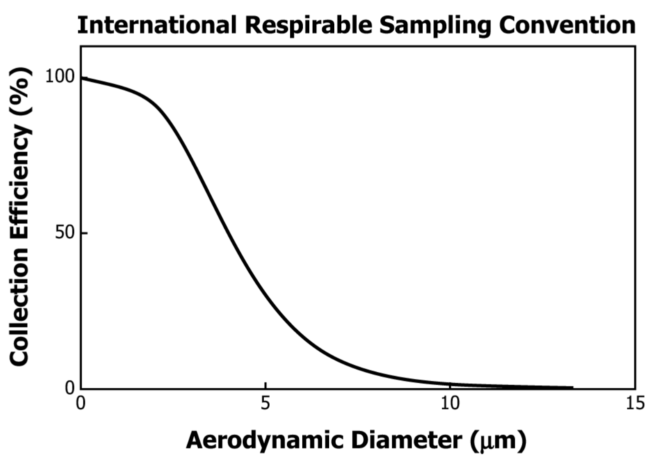

FIG. 1 Collection Efficiency of an Ideal Sampler Following the International Sampling Conventions

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

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

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.~~ are commercially available. 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.

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~~4.2 L ⁄min – ~~10~~10 L ~~L/min)~~ ⁄min) and medium flow rate cyclone samplers (flow rate range ~~1.7~~1.7 L ⁄min – ~~2.75~~2.75 L ~~L/min).~~ ⁄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)~~h) (**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.~~

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6.1.2 *Filter Cassette Assembly*—Filter, filter-support pad, and filter cassette holder with suitable caps. The filter shall be non-hygroscopic and provide 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 preweighed by the user.~~

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~~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.~~

6.1.2.2 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 3—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 Pumps shall be completely charged with an appropriate charger following the manufacturer's instructions or disposable batteries may be used.or with replacement disposable batteries, following manufacturer's instructions.

6.3 *Weighing Room*—With temperature (20(20 °C ± 2°C)2 °C) and humidity (50 ± 5 % (50 % Relative Humidity (RH) ± 5 % (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.

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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 Measuring volumetric flow with precision equal to 2 % or better within the range of the flow rate used. Various flow meters are introduced in Practice D5337. Calibration Flow verification of rotameterrotameters can be performed using Practice D3195.

6.8 *Thermometer*—Capable of covering the temperature range of interest with divisions every 0.1°C0.1 °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 0.70.7 m 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.

6.13 *Tape*—Adhesive (for example, plastic electrical), for sealing samplers.

7. Preparation of Samplers Prior to Sampling

7.1 Inspect the interior of the cyclone and clean it to keep away from reentrainment ensure no re-entrainment 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.24 h.

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. holder with adhesive tape. If necessary, use the press described in 6.6.

7.5 Place caps toon 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 to determine 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.

8.3.1 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 (for example, task-based exposures) are to be investigated.
Determinations of variations of the exposure during a shift are made.

8.3.2 The sampler must be observed during sampling to the extent possible in order to ensure that it is not inverted at any time, otherwise re-deposition of particles from the cyclone body onto the filter may occur. Instances of inversion, whether observed directly or reported by the person being sampled should be included in the report.

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.~~

8.9.1 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.

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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.~~

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~~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.~~

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~~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.~~

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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.~~

10.4.1 The balance calibration shall be regularly 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~~shall 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~~ identifier (may include name and contact ~~information;~~ information where permitted); 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 4—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).

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			