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Standard Practice for Evaluating the Performance of Respirable Aerosol Samplers[1]

This standard is issued under the fixed designation D6061; 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.

ε[1] NOTE—Editorially removed the Metric designation from Specification D 6062 in April 2007.

1. Scope

1.1 This practice covers the evaluation of the performance of personal samplers of non-fibrous respirable aerosol. The samplers are assessed relative to a specific respirable sampling convention. The convention is one of several that identify specific particle size fractions for assessing health effects of airborne particles. When a health effects assessment has been based on a specific convention it is appropriate to use that same convention for setting permissible exposure limits in the workplace and ambient environment and for monitoring compliance. The conventions, which define inhalable, thoracic, and respirable aerosol sampler ideals, have now been adopted by the International Standards Organization (Technical Report ISO TR 7708), the Comité Européen de Normalisation (CEN Standard EN 481), and the American Conference of Governmental Industrial Hygienists (ACGIH, Ref (**1**)),[2] developed (**2**) in part from health-effects studies reviewed in Ref (**3**) and in part as a compromise between definitions proposed in Refs (**3**,**4**).

1.2 This practice is complimentary to Test Method D4532, which specifies a particular instrument, the 10-mm cyclone.[3] The sampler evaluation procedures presented in this practice have been applied in the testing of the 10-mm cyclone as well as the Higgins-Dewell cyclone.[3,4] Details on the evaluation have been recently published (**5-7**) and can be incorporated into revisions of Test Method D4532.

1.3 A central aim of this practice is to provide information required for characterizing the uncertainty of concentration estimates from samples taken by candidate samplers. For this purpose, sampling accuracy data from the performance tests given here can be combined with information as to analytical and sampling pump uncertainty obtained externally. The practice applies principles of ISO GUM, expanded to cover situations common in occupational hygiene measurement, where the measurand varies markedly in both time and space. A general approach (**8**) for dealing with this situation relates to the theory of tolerance intervals and may be summarized as follows: Sampling/analytical methods undergo extensive evaluations and are subsequently applied without re-evaluation at each measurement, while taking precautions (for example, through a quality assurance program) that the method remains stable. Measurement uncertainty is then characterized by specifying the evaluation confidence (for example, 95 %) that confidence intervals determined by measurements bracket measurand values at better than a given rate (for example, 95 %). Moreover, the systematic difference between candidate and idealized aerosol samplers can be expressed as a relative bias, which has proven to be a useful concept and is included in the specification of accuracy (3.2.9-3.2.10).

1.4 Units of the International System of Units (SI) are used throughout this practice and should be regarded as standard.

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 establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[5]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D4532 Test Method for Respirable Dust in Workplace Atmospheres

D6062 Guide for Personal Samplers of Health-Related Aerosol Fractions

D6552 Practice for Controlling and Characterizing Errors in

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

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

[3] 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.

[4] The sole source of supply of the Higgins-Dewell cyclone known to the committee at this time is BGI Inc., 58 Guinan Street, Waltham, MA 02154.

[5] 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.

Weighing Collected Aerosols

2.2 *International Standards:*

ISO TR 7708 Technical Report on Air Quality—Particle Size Fraction Definitions for Health-Related Sampling, Brussels, 1993[6]

ISO GUM Guide to the Expression of Uncertainty in Measurement, Brussels, 1993[6]

CEN EN 481 Standard on Workplace Atmospheres. Size Fraction Definitions for the Measurement of Airborne Particles in the Workplace, Brussels, 1993[7]

CEN EN 1232 Standard on Workplace Atmospheres. Requirements and Test Methods for Pumps used for Personal Sampling of Chemical Agents in the Workplace, Brussels, 1993[7]

CEN EN 13205 Workplace Atmospheres- Assessment of Performance of Instruments for Measurement of Airborne Particle Concentrations, 2001[7]

2.3 *NIOSH Standards:*

NIOSH Manual of Analytical Methods, 4th ed., Eller, P. M., ed.: Dept. of Health and Human Services, 1994[8]

Criteria for a Recommended Standard, Occupational Exposure to Respirable Coal Mine Dust, NIOSH, 1995 [9]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice, refer to Terminology D1356 and ISO GUM.

3.1.2 Aerosol fraction sampling conventions have been presented in Performance Specifications D6062. The relevant definitions are repeated here for convenience.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aerodynamic diameter, D* (μm)—the diameter of a sphere of density, 10^3 kg/m, with the same stopping time as a particle of interest.

3.2.2 *respirable sampling convention, E_R*—defined explicitly at aerodynamic diameter *D* (μm) as a fraction of total airborne aerosol in terms of the cumulative normal function **(9)** Φ as follows:

$$E_R = 0.50\,(1 + \exp[-0.06\,D])\;\Phi\;[\ln[D_R/D]/\sigma_R] \qquad (1)$$

where the indicated constants are $D_R = 4.25$ μm and $\sigma_R = \ln[1.5]$.

3.2.2.1 *Discussion*—The respirable sampling convention, together with earlier definitions, is shown in Fig. 1. This convention has been adopted by the International Standards Organization (Technical Report ISO TR 7708), the Comité Européen de Normalisation (CEN Standard EN 481), and the American Conference of Governmental and Industrial Hygienists (ACGIH, Ref (**1**)). The definition of respirable aerosol is the basis for the recommended exposure level (REL) of respirable coal mine dust as promulgated by NIOSH (*Criteria for a Recommended Standard, Occupational Exposure to Respirable Coal Mine Dust*) and also forms the basis of the NIOSH sampling method for particulates not otherwise regulated, respirable (*NIOSH Manual of Analytical Methods*).

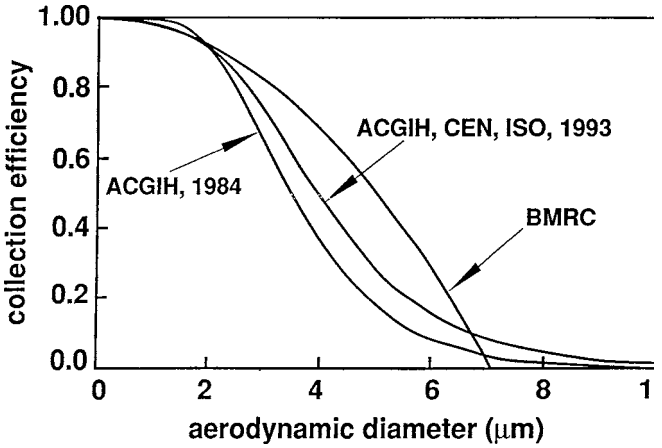

FIG. 1 Respirable Aerosol Collection Efficiencies

3.2.3 *size-distribution C^{-1} dC/dD (μm^{-1})*—of a given airborne aerosol, the mass concentration of aerosol per unit aerodynamic diameter range per total concentration *C*.

3.2.3.1 *lognormal size distribution*—an idealized distribution characterized by two parameters: the *geometric standard deviation (GSD)* and *mass median diameter (MMD).* The distribution is given explicitly as follows:

$$C^{-1}\,dC/dD = \frac{1}{\sqrt{2\pi}\,D\,\ln[GSD]}\exp\left[-\frac{1}{2}\ln[D/MMD]^2/\ln[GSD]^2\right] \qquad (2)$$

where *C* is the total mass concentration.

3.2.4 *conventional respirable concentration c_R* (mg/m^3)—the concentration measured by a conventional (that is, ideal) respirable sampler and given in terms of the size distribution *dC/dD* as follows:

$$c_R = \int_0^\infty dD\,E_R\;dC/dD \qquad (3)$$

3.2.4.1 *Discussion*—Note that samples are often taken over an extended time period (for example, 8 h), so that *dC/dD* of Eq. 3 represents a time-averaged, rather than instantaneous, size-distribution.

3.2.5 *sampler number s = 1, ..., S*— a number identifying a particular sampler under evaluation.

3.2.6 *sampling efficiency $E_s(D, Q)$*—the modeled sampling efficiency of sampler *s* as a function of aerodynamic diameter *D* and flow rate *Q* (9.1).

3.2.6.1 *model parameters θ_p, where p = 1, ..., P (for example, 4)*—parameters that specify the function $E_s(D, Q)$.

3.2.7 *mean sampled concentration c_s*—the concentration that sampler *s* would give, averaged over sampling pump and analytical fluctuations, in sampling aerosol of size-distribution C^{-1} *dC/dD* is given as follows:

$$c_s = \int_0^\infty dD\,E_s\,dC/dD \qquad (4)$$

3.2.8 *mean concentration c*—the population mean of c_s.

3.2.9 *uncertainty components*:

[6] Available from International Organization for Standardization, Caisse Postale 56, CH-1211, Geneva 20, Switzerland.

[7] Available from CEN Central Secretariat: rue de Stassart 36, B-1050 Brussels, Belgium.

[8] Available from Superintendent of Documents, U.S. Government Printing Office, Stock No. 917-011-00000-1, Washington DC 20402.

[9] Available from NIOSH Publications, 4676 Columbia Parkway, Cincinnati, OH 45226.

3.2.9.1 *analytical relative standard deviation* $RSD_{analytical}$—the standard deviation relative to the true respirable concentration c_R associated with mass analysis, for example, the weighing of filters, analysis of α-quartz, and so forth.

3.2.9.2 *pump-induced relative standard deviation* RSD_{pump}—the intra-sampler standard deviation relative to the respirable concentration c_R associated with both drift and variability in the setting of the sampling pump.

3.2.9.3 *inter-sampler relative standard deviation* RSD_{inter}—the inter-sampler standard deviation (varying sampler *s*) relative to the respirable concentration c_R and taken as primarily associated with physical variations in sampler dimensions.

3.2.10 *mean relative bias* Δ—of measurement *c* relative to the conventional respirable concentration c_R, defined as follows:

$$\Delta \equiv (c - c_R)/c_R \quad (5)$$

3.2.11 *symmetric-range accuracy A*—the fractional range, symmetric about the conventional concentration c_R, within which 95 % of sampler measurements are to be found (**8,10-13** and the NIOSH Manual of Analytical Methods).

3.2.12 *flow rate Q (L/min)*—the average flow rate of air sampled by a given sampler over the duration of the sampling period.

3.2.13 *flow number F*—the number (for example, 4) of sampler flow rates *Q* tested.

3.2.14 *replication number n (for example, 4)*— the number of replicate measurements for evaluating a given sampler at specific flow rate and aerodynamic diameter.

3.3 *Symbols and Abbreviations*:

A—symmetric-range accuracy as defined in terms of bias and precision (see 3.2.11).

$\hat{A}$—estimated accuracy *A*.

NOTE 1—*Hats* as in *A* refer to estimates, both in sampler application and sampler evaluation.

$_{95\,\%}A$—95 % confidence limit on the symmetric-range accuracy *A*.

c(mg/m^3)—expected value of the sampler-averaged concentration estimates c_s.

c_s(mg/m^3)—expected value (averaged over sampling pump and analytical variations) of the concentration estimate from sampler *s*.

$_s cov_{ij}$—covariance matrix for sampler *s* and efficiency parameters θ_i and θ_j.

c_R(mg/m^3)—concentration measured by a conventional (that is, ideal) respirable sampler.

D (µm)—aerosol aerodynamic diameter.

D_0—sampling efficiency model parameter.

D_R(µm)—respirable sampling convention parameter equal to 4.25 µm in the case of healthy adults, or 2.5 µm for the sick or infirm or children.

E—sampling convention in general.

E_R—respirable sampling convention.

E_s—sampling efficiency of sampler *s*.

F—number of flow rates evaluated.

GSD—geometric standard deviation of a lognormal aerosol size distribution.

MMD—mass median diameter of a lognormal aerosol size distribution.

MSE_c—mean square element for sampler in application (see 10.4).

MSE—mean square element for evaluation data (see A1.5).

n—number of replicate measurements.

P—number of sampling efficiency parameters.

RSD—relative standard deviation (relative to concentration c_R as estimated by an ideal sampler following the respirable sampling convention).

$RSD_{analytical}$—relative standard deviation component characterizing analytical random variation.

RSD_{eval}—relative standard deviation component characterizing uncertainty from the evaluation experiment itself (Annex Annex A1).

RSD_{inter}—relative standard deviation component characterizing random inter-sampler variation.

RSD_{pump}—relative standard deviation component characterizing the effect of random sampling pump variation.

s—sampler number.

S—number of samplers evaluated.

t—sampling time (for example, 8h).

U—expanded uncertainty.

u_c—combined uncertainty.

v (m/s)—wind speed.

Δ—bias relative to an ideal sampler following the respirable sampling convention.

$\varepsilon_{eval\,s}$—random variable contribution to evaluation experimental error in a concentration estimate.

ε_s—random variable contribution to inter-sampler error in a concentration estimate.

θ—sampling efficiency model parameter.

σ_0—sampling efficiency model parameter.

σ_{eval}—evaluation experimental standard deviation in a concentration estimate.

σ_{inter}—inter-sampler standard deviation in a concentration estimate.

σ_R—respirable sampling convention parameter equal to ln[1.5].

σ_{mass}—weighing imprecision in mass collected on a filter.

Φ[*x*]—cumulative normal function given for argument *x*.

4. Summary of Practice

4.1 The sampling efficiency from $D = 0$ to 10 µm and its variability are measured in calm air (<0.5 m/s) for several candidate samplers operated at a variety of flow rates. This information is then used to compute concentration estimates expected in sampling representative lognormal aerosol size distributions. Random variations (10.2) as well as systematic deviation (10.1) are specified relative to a conventional sampler. Overall performance in calm air can then be assessed by computing a confidence limit $_{95\,\%}A$ on the symmetric-range accuracy (3.2.11), accounting for uncertainty in the evaluation experiment, given estimated bias and imprecision at each lognormal aerosol size distribution of interest. The symmetric-range accuracy confidence limit $_{95\,\%}A$ provides conservative confidence intervals bracketing the conventional concentration at given confidence in the method evaluation, analogous to the

use of the expanded uncertainty U in ISO GUM (See Eq. 16). This performance evaluation has evolved from work described in Refs (**8**, **14-21**).

5. Significance and Use

5.1 This practice is significant for determining performance relative to ideal sampling conventions. The purposes are multifold:

5.1.1 The conventions have a recognized tie to health effects and can easily be adjusted to accommodate new findings.

5.1.2 Performance criteria permit instrument designers to seek practical sampler improvements.

5.1.3 Performance criteria promote continued experimental testing of the samplers in use with the result that the significant variables (such as wind speed, particle charge, etc.) affecting sampler operation become understood.

5.2 One specific use of the performance tests is in determining the efficacy of a given candidate sampler for application in regulatory sampling. The accuracy of the candidate sampler is measured in accordance with the evaluation tests given here. A sampler may then be adopted for a specific application if the accuracy is better than a specific value.

5.2.1 *Discussion*—In some instances, a sampler so selected for use in compliance determinations is specified within an exposure standard. This is done so as to eliminate differences among similar samplers. Sampler specification then replaces the respirable sampling convention, eliminating bias (3.2.10), which then does not appear in the uncertainty budget.

5.3 Although the criteria are presented in terms of accepted sampling conventions geared mainly to compliance sampling, other applications exist as well. For example, suppose that a specific aerosol diameter-dependent health effect is under investigation. Then for the purpose of an epidemiological study an aerosol sampler that reflects the diameter dependence of interest is required. Sampler accuracy may then be determined relative to a modified sampling convention.

6. Apparatus

6.1 *Small Single-pass Wind Tunnel* (or, equivalently, a static exposure chamber). The following dimensions are nominal:

6.1.1 Cross section: 500 by 500 mm; Length: 6 m.

6.1.2 Air speed: <0.5 m/s.

6.1.3 Air speed uniformity: ±3 % over 250 by 250-mm central cross-sectional area.

6.1.4 Turbulence <3 %.

6.1.5 *Test Aerosol Generation System*:

6.1.5.1 Generation system: ultrasonic nebulizer.

6.1.5.2 Static discharging nozzle.

6.1.5.3 Mixing with tunnel air by turbulence created by 100 by 100-mm rectangular plate 10 cm downstream of the nebulizer and perpendicular to the tunnel's airflow.

6.1.5.4 Concentration: 5000 aerosol particles/L.

6.1.5.5 Size distribution: count median diameter = 4 µm and geometric standard deviation = 2.2.

6.2 *Aerodynamic Particle Sizer (APS).*[3,10]

6.3 *Tube-Mounted Hot-Wire Anemometer Probe*, or equivalent, ac voltmeter or oscilloscope.

7. Reagents and Materials

7.1 *Reagents*:

7.1.1 *Potassium Sodium Tartrate*, A.C.S.-certified reagent grade, for generating solid spherical aerosol particles.

7.1.2 *Standard Polystyrene Latex Spheres* for calibrating APS (6.2).

7.2 *Materials*:

7.2.1 *Five-micrometre PVC Membrane Filters and Conductive Filter Cassettes.*[3,11]

8. Data Representation through Sampling Efficiency Model

8.1 Determine a sampling efficiency curve for each of the S (for example, eight) samplers by least squares fit to the data taken in four replicates at the four flow rates. Thus eight functions of aerodynamic diameter D and flow rate Q are determined. Use the following model (**5**) or equivalent for characterizing the candidate cyclones:

$$E_s(D;Q) = \Phi\left[\frac{1}{\sigma_0}\ln\left(\frac{D_0}{D}\right)\right] \quad (6)$$

where Φ is the cumulative normal function (**9**), easily computed within most statistical software packages. The indicated constants are defined in terms of model parameters θ_p, determined by the least squares fit to the data using a standard nonlinear regression routine:

$$D_0 = \theta_1 \times (Q/2.0\ L/\mathrm{min})^{-\theta_2} \quad (7)$$
$$exp[\sigma_0] = \theta_3 \times (Q/2.0\ L/\mathrm{min})^{-\theta_4}$$

In this case the curve fitting would determine eight sets (one for each sampler) of four parameters each.

9. Procedure

9.1 General procedures for evaluating respirable aerosol samplers are presented in this practice. For other details on the experimental procedures, see Refs (**5,6,22-24**).

9.2 Set up the APS (6.2) for operation in the small wind tunnel (6.1). Check the APS calibration using (nominally) 3 and 7-µm standard polystyrene latex spheres (7.1.2) by comparing measured and known particle sizes. Set up the potassium sodium tartrate (7.1.1) aerosol generator (6.1.5.1) with charge neutralizer (6.1.5.2) and adjust to achieve about 5000 aerosol particles/L in the test region of the wind tunnel. Adjust the nebulizer aperture and aerosol solution concentration to achieve a test size distribution with count median diameter ≈4 µm and geometric standard deviation ≈2.2, covering the aerodynamic diameter region of interest. Test the aerosol

[10] The TSI Aerodynamic Particle Sizer 3300 from TSI, Inc., P.O. Box 64394, St. Paul, MN 55164 is the sole aerodynamic particle sizer presently available suitable for this purpose.

[11] The sole source of supply of conductive cassettes known to the committee at this time is Omega Specialty Instrument Co., 4 Kidder Road, Chelmsford, MA 01824.