Designation: D 6061 – 96

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

This standard is issued under the fixed designation D 6061; 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 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 and in the setting of and testing for compliance with permissible exposure limits in the workplace and ambient environment. 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)**. This practice is specific to respirable aerosol sampler evaluation because of simplifying characteristics particular to this fraction.

1.2 This practice is complimentary to Test Method D 4532, which specifies a particular instrument, the 10-mm cyclone.[3,4] 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.[4,5] Details on the evaluation have been recently published **(5-7)** and can be incorporated into revisions of Test Method D 4532.

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

1.4 *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:*

D 1356 Terminology Relating to Atmospheric Sampling and Analysis[6]

D 4532 Test Method for Respirable Dust in Workplace Atmospheres[6]

D 6062M Performance Specifications for Samplers of Health-Related Aerosol Fractions[6]

2.2 *International Standards:*

ISO TR 7708 Technical Report on Air Quality—Particle Size Fraction Definitions for Health-Related Sampling, Brussels, 1993[7]

CEN EN 481 Standard on Workplace Atmospheres. Size Fraction Definitions for the Measurement of Airborne Particles in the Workplace, Brussels, 1993[8]

CEN prEN 1232 Pre-Standard on Workplace Atmospheres. Requirements and Test Methods for Pumps used for Personal Sampling of Chemical Agents in the Workplace, Brussels, 1993[8]

2.3 *NIOSH Standards:*

NIOSH Manual of Analytical Methods, 4th ed., Eller, P. M., ed.: Dept. of Health and Human Services, 1994[9]

Criteria for a Recommended Standard, Occupational Exposure to Respirable Coal Mine Dust, NIOSH, 1995[10]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice, refer to Terminology D 1356.

3.1.2 Aerosol fraction sampling conventions have been presented in Performance Specifications D 6062M. The relevant definitions are repeated here for convenience.

3.2 *Definitions of Terms Specific to This Standard:*

[1] This practice 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 Analysis of Workplace Atmospheres.

Current edition approved Dec. 10, 1996. Published February 1997.

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

[3] The sole source of supply of the 10-mm cyclone known to the committee at this time is Mine Safety Appliances Co., Instrument Div., P.O. Box 427, Pittsburgh, PA 15230.

[4] 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.

[5] 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.

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

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

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

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

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

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—at aerodynamic diameter D,* defined explicitly as a fraction of total airborne aerosol in terms of the cumulative normal function **(8)** Φ as follows:

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

where the indicated constants are $D_R = 4.25$ µm and $\sigma_R = \ln[1.5]$. The function Φ may be approximated using the algorithm presented in Appendix X1.

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*).

3.2.3 *size-distribution dC/dD* (mg/m ^{3}/µm)—*of a given airborne aerosol*, the mass concentration of aerosol per unit aerodynamic diameter range.

3.2.3.1 *log-normal size distribution $dC_{\ln}/dD$*—an idealized distribution characterized by two parameters: the *geometric standard deviation (GSD)* and *mass median diameter (MMD)*. $dC_{\ln}/dD$ is given explicitly as follows:

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

where *C* is the total mass concentration.

3.2.4 *true 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:

$$c_R = \int_0^\infty dD\ E_R\ dC/dD \quad (3)$$

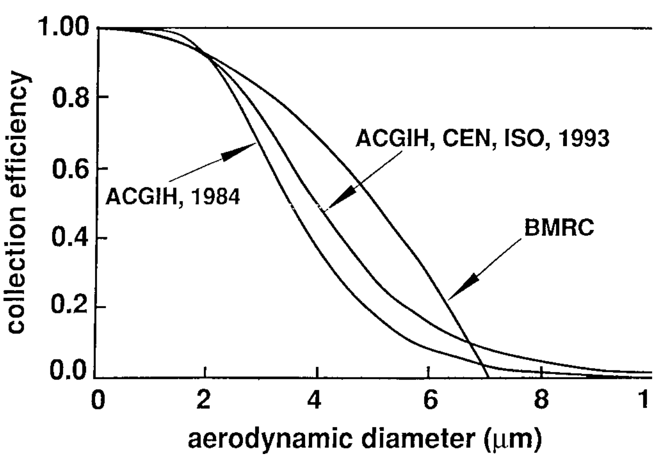

FIG. 1 Respirable Aerosol Collection Efficiencies

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_j, where j = 1, ..., J*—parameters that specify the function $E_S(D, Q)$.

3.2.7 *sampled concentration c_s*—the concentration that sampler *s* would give in sampling aerosol of size-distribution *dC/dD* and is given as follows:

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

3.2.8 *mean concentration c*—the average of c_s over the samplers tested.

3.2.9 *mean bias Δ—relative to a conventional sampler*, defined as follows:

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

3.2.10 The imprecision or relative standard deviation *RSD* is defined here in terms of independent analytical, intra-sampler, and inter-sampler components:

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

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

3.2.10.3 *inter-sampler relative standard deviation RSD_{inter}*—the inter-sampler imprecision relative to the true respirable concentration c_R and taken as primarily associated with physical variations in sampler dimensions.

3.2.10.4 The *total imprecision RSD* is then given as follows:

$$RSD = \sqrt{RSD^2_{analytical} + RSD^2_{pump} + RSD^2_{inter}} \quad (6)$$

3.2.11 *flow rate Q (L/min)*—the flow rate sampled by a given sampler.

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

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

3.2.14 *parameter number p = 1, ..., P (for example, 4)*—a number identifying parameters θ_p in modeled sampling efficiency data as in Section 9.

3.2.15 *Accuracy A, Busch probabilistic*—the fractional range, symmetric about the true concentration c_R, within which 95 % of sampler measurements are to be found (**9-12** and the NIOSH Manual of Analytical Methods).

3.2.15.1 *Discussion*—The function *A* depends only on the bias Δ and total imprecision *RSD* in the event that these quantities are independent of c_R. *A* is defined implicitly as follows:

$$\Phi[\Delta + A)/RSD] - \Phi[(\Delta - A)/RSD] = 95\ \% \quad (7)$$

where Φ is the cumulative normal function. The function *A* (Δ, *RSD*) may be computed numerically and is depicted in Fig. 2. Alternatively, Eq 7 has an approximate solution **(13)** for *A*[Δ,

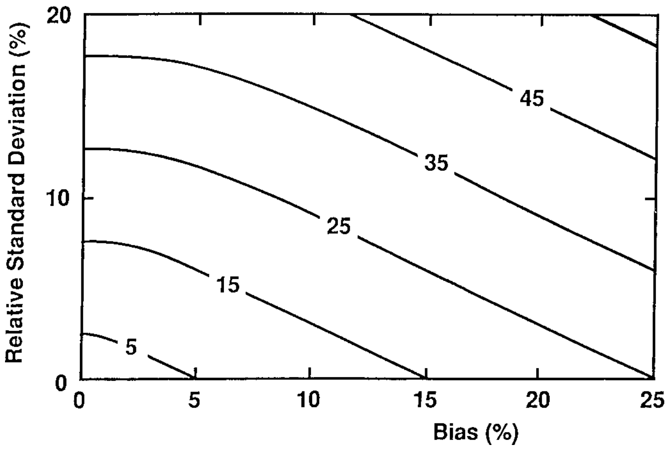

FIG. 2 Contours Showing Accuracy *A* at 95 % Confidence Level in Terms of Bias and Relative Standard Deviation (*RSD*)

RSD] given as follows:

$$A[\Delta, RSD] = \Phi^{-1}(0.95) \times RSD + Sqrt[(\Phi^{-1}(1.95/2) - \Phi^{-1}(0.95))^2 \; RSD^2 + \Delta^2] \quad (8)$$

This expression is easily evaluated using a calculator, noting that $\Phi^{-1}(0.95) = 1.645$, and $\Phi^{-1}(1.95/2) = 1.960$.

3.3 *Symbols and Abbreviations*:

A—(Busch probabilistic) accuracy as defined in terms of bias and precision (see 3.2.15).

$\hat{A}$—estimated accuracy *A*.

$_{95\,\%}A$—95 % confidence level on the (Busch probabilistic) accuracy *A*.

C (mg/m^3)—total mass concentration.

$_s cov_{ij}$—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.

$\hat{c}$—sampler-averaged concentration estimate.

$\hat{c}_s$—concentration estimate from sampler *s*.

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 representative log-normal aerosol size distribution.

MMD—mass median diameter of a representative log-normal aerosol size distribution.

n—number of replicate measurements.

P—number of sampling efficiency parameters.

Q (L/min)—sampler flow rate.

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

$RSD_{analytical}$—analytical imprecision component.

RSD_{flow}—uncertainty in the pump flow rate.

RSD_{inter}—inter-sampler imprecision.

RSD_{pump}—imprecision induced by imprecision in the sampling pump.

$R\hat{S}D_{inter}$—estimated inter-sampler imprecision RSD_{inter}.

$R\hat{S}D_{pump}$—estimated pump-induced imprecision RSD_{pump}.

s—sampler number.

S—number of samplers evaluated.

t—sampling time.

v (m/s)—wind speed.

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

Δ—estimated bias Δ.

$\epsilon_{eval\,s}$—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_{eval}—estimate for σ_{eval}.

σ_{inter}—estimate for σ_{inter}.

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

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

$\Phi[x]$—cumulative normal function defined, given argument *x*.

4. Summary of Practice

4.1 *Calm-air Evaluation*—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 log-normal aerosol size distributions. Precision and bias (4.1.1 and 4.1.2) are therefrom determined relative to a conventional sampler. Overall performance in calm air can then be assessed by computing a confidence limit on the Busch probabilistic accuracy, accounting for uncertainty in the evaluation experiment, given measured bias and imprecision at each log-normal aerosol size distribution of interest. This test has evolved from work described in Refs **(14-21)**.

4.1.1 *Precision*—In the sampling of aerosol, several components of precision have been found **(5)** significant. These include inter-sampler variability, caused by physical variations in the samplers; intra-sampler variability, from inaccuracy in the setting and maintenance of required airflow; and analytical error, for example, in the weighing of filters, or, as another example, in the measurement of α-quartz.

4.1.2 *Bias*—As no real sampler follows the aerosol fraction conventions exactly, bias always exists between true and conventional (ideal) samplers. This bias depends on the particle size-distribution of the aerosol sampled. The worst-case situation is in the sampling of monodisperse aerosol. However, in most workplaces, aerosol is present in a broad distribution of sizes. The cancellation of positive and negative components of bias at different particle sizes reduces the overall bias in this case.

5. Significance and Use

5.1 This practice is significant in providing the experimental means for replacing instrument or sampler specification by performance criteria based on accuracy relative to ideal sampling conventions. The advantages 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 according to the evaluation tests given here. The sampler is then certified as acceptable if the accuracy is better than a specific value.

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 is then 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).[4,11]

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.*[4,12]

8. Data Representation through Sampling Efficiency Model

8.1 Determine a sampling efficiency curve for each of the 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:

$$E_s(D;Q) = \Phi\left[\frac{1}{\sigma_0}\ln\left(\frac{D_0}{D}\right)\right] \tag{9}$$

where Φ is the cumulative normal function **(8)**, which may be approximated using the algorithm presented in Appendix X1. The indicated constants are defined in terms of model parameters θ_j, determined by the least squares fit to the data using a standard nonlinear regression routine:

$$D_0 = \theta_1(Q/2.0\ L/\text{min})^{-\theta_2} \tag{10}$$

$$e^{\sigma_0} = \theta_3(Q/2.0\ L/\text{min})^{-\theta_4} \tag{10}$$

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 concentration for stability in time by taking a series of size distribution measurements. Variation should be <1 % over 2-min periods.

9.3 Determine the sampler sampling efficiency from $D = 0$ to 10 µm by measuring the aerosol size distribution before and after the samplers with 1-min exposures according to the following experimental design:

F = 4 sampler flow rates: distributed between 50 and 200 % of the presumed optimal sampler flow rate,

S = 8 samplers, numbered $s = 1, ..., S$, and

[11] 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.

[12] 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.