Designation: D 6061 – 01

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. 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 D 4532, 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 D 4532.

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:*

D 1356 Terminology Relating to Atmospheric Sampling and Analysis[5]

D 4532 Test Method for Respirable Dust in Workplace Atmospheres[5]

D 6062M Performance Specifications for Samplers of Health-Related Aerosol Fractions[5]

D 6552 Practice for Controlling and Characterizing Errors in Weighing Collected Aerosols[5]

2.2 *International Standards:*

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

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[1] This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres 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] *Annual Book of ASTM Standards*, Vol 11.03.

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

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 D 1356 and ISO GUM.

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:*

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:

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

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*).

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*.

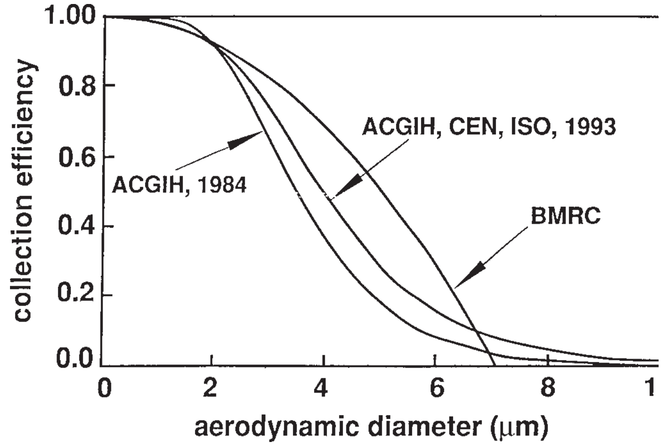

FIG. 1 Respirable Aerosol Collection Efficiencies

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:

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

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:

$$c_R = \int_0^\infty dD\,E_R\,dC/dD \tag{3}$$

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:

$$c_s = \int_0^\infty dD\,E_s\,dC/dD \tag{4}$$

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

3.2.9 *uncertainty components*:

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.

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[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.

3.2.9.3 *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.

$\epsilon_{eval\ s}$—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_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:

$$D_0 = \theta_1 \times (Q/2.0\ L/\text{min})^{-\theta_2} \quad (7)$$

$$exp[\sigma_0] = \theta_3 \times (Q/2.0\ L/\text{min})^{-\theta_4}$$

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 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 in accordance with an experimental design similar to the following:

[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.

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
n = 4 replicates, numbered $r = 1, ..., n$.

10. Measurement Uncertainty

10.1 *Systematic Deviation Relative to Convention*:

10.1.1 *Background*—As no real sampler follows the aerosol fraction conventions exactly, bias always exists between real and conventional (ideal) samplers with sampling efficiency given by Eq. 1. With minimal loading effects, this bias depends only on the particle size-distribution of the aerosol sampled, and is therefore a constant when expressed as a fraction of the conventional concentration c_R. The largest values of bias occur 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.

It has, therefore, become conventional to compare samplers as applied in sampling aerosol distributed in size. Particularly, bias is estimated in the sampling of specific lognormal size distributions (3.2.3.1). Such a comparison is then also applicable to those more realistic size distributions which can be approximated as a superposition of several lognormal distributions.

As with EN 13205, this practice requires a comparison over all lognormal particle size distributions with geometric standard deviations between 1.75 and 3.5 and mass median diameter <25 µm. Furthermore, respirable samplers would only be evaluated at aerosol size distributions with the fraction of respirable to total aerosol greater than 5 %. This omits sizes beyond the line defined by: (mass median diameter, geometric standard deviation) = (10 µm, 1.5) to (25 µm, 2.75). The performance tests are therefore not applicable to the sampling of rarely occurring narrow distributions of large-size aerosols.

Note that the variety of environments in which respirable aerosol measurements are taken precludes a simple elimination of this bias in the mean through calibration, with associated imprecision from variation of *influence parameters* (ISO GUM). For example, assuming a lognormal size-distribution, the aerosol size distribution parameters, *MMD* and *GSD* may be regarded as influence parameters. It is simplest to explicitly account for the bias in the development of confidence intervals about the measurand values (the conventional concentrations c_R).

10.1.2 *Bias Estimate*—Compute the estimated concentration $\hat{c}_s$ numerically for each sampler s at each lognormal size distribution (*MMD*, *GSD*) of interest, as indicated in (3.2.7). Estimate the constant c by the sampler average:

$$\hat{c} = \frac{1}{S} \sum_s \hat{c}_s, \tag{8}$$

then compute the bias estimate $\hat{\Delta}$ as in Eq. 5.

10.2 *Random Variations*—In the sampling of aerosol, several sources of random variation have been found (**5**) significant. These include inter-sampler variability (RSD_{inter}(3.2.9.3)), caused by physical variations in the samplers; intra-sampler variability, from inaccuracy in the setting and maintenance of required airflow (RSD_{pump}(3.2.9.2)), and analytical error ($RSD_{analytical}$(3.2.9.1)), for example, from variations in the weighing of filters, or, as another example, in the measurement of collected α-quartz mass. Like the relative bias, the relative standard deviations, RSD_{inter} and RSD_{pump} are roughly constant, whereas $RSD_{analytical}$ may depend on the conventional concentration c_R. For example, a recent assessment (**25**) by the Mine Safety and Health Administration (MSHA) indicated an uncertainty σ_{mass} in measuring filter mass changes equal to 9.1 µg. From such an estimate $RSD_{analytical}$ can be computed, given the flow rate Q (L/min), sampling time t (for example, 8 · 60 min), and conventional respirable concentration c_R of interest:

$$RSD_{analytical} = \sigma_{mass} \cdot 1000\ L/m^3/(c_R \cdot Q \cdot t), \tag{9}$$

which depends inversely on the conventional concentration c_R.

10.3 *Measurement Model*—The various aspects of concentration measurement accuracy covered in 10.1 and 10.2 lead to the following approximation for modeling the measurement:

$$\begin{aligned} \hat{c}_s &= \hat{m}_s/(\hat{Q} \cdot t) \\ &= [\ (1+\Delta) + \epsilon_s + \epsilon_{pump} + \epsilon_{analytical}] \cdot c_R, \end{aligned} \tag{10}$$

where ϵ signifies random variables approximated as normally distributed about zero:

$$\begin{aligned} \epsilon_s &\approx N[0, RSD_{sampler}] \\ \epsilon_{pump} &\approx N[0, RSD_{pump}] \\ \epsilon_{analytical} &\approx N[0, RSD_{analytical}], \end{aligned} \tag{11}$$

remembering that $RSD_{analytical}$ depends specifically on the analytical method and is not necessarily constant.

The measurement model specified in Eq. 10 indicates that the total *relative standard deviation RSD* (the *combined relative uncertainty* u_c/c_R (ISO GUM)) in the estimate $\hat{c}_s$ is given through the lowest order approximation to the law of propagation of uncertainty (ISO GUM) by:

$$RSD = \sqrt{RSD_{inter}^2 + RSD_{pump}^2 + RSD_{analytical}^2} \tag{12}$$

10.4 *Symmetric-range Accuracy A*—The definition in (3.2.11) is equivalent to the following implicit definition of the function A in terms of relative bias Δ and RSD, assuming approximately normal distributions of the concentration estimates:

$$\Phi\left[\frac{\Delta + A}{RSD}\right] - \Phi\left[\frac{\Delta - A}{RSD}\right] = 95\ \%, \tag{13}$$

where Φ is the cumulative normal function. The accuracy $A[\Delta, RSD]$ may be computed numerically and is depicted in Fig. 2. Alternatively, Eq. 13 has an approximate solution (**8**) for $A[\Delta, RSD]$ given by:

$$A[\Delta, RSD] = 1.960 \times MSE_c^{\frac{1}{2}}, \tag{14}$$

where the *combined mean square element* MSE_c is defined as:

$$MSE_c \equiv \Delta^2 + RSD^2 \tag{15}$$

The approximation of Eq. 14 is extremely accurate for small bias magnitude $|\Delta|$ (that is, for $|\Delta| < RSD/1.645$), A being overestimated fractionally by up to 1 %, only in a narrow region close to $|\Delta| = RSD/1.645$. In fact, over the region

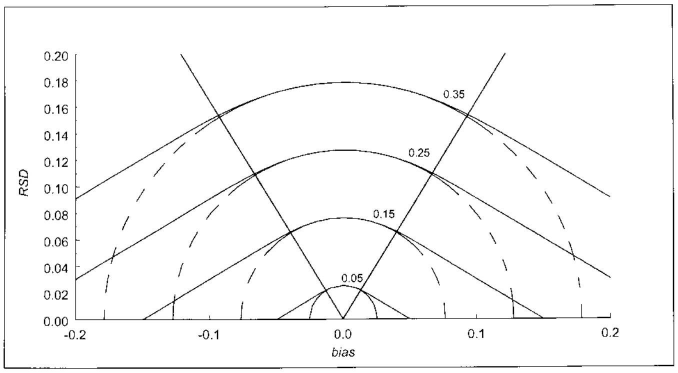

FIG. 2 Symmetric-Range Accuracy. Plotted are (solid) curves of constant accuracy = 5 %, 15 %, 25 %, and 35 %. The dashed curves identify circles in the approximation of Eqs. 14 and 15.

$|\Delta| < RSD$, Eq. 14 overestimates the accuracy fractionally by less than 5 %. Therefore, Eq. 14 may be regarded as a minimally conservative estimate of the symmetric range accuracy over ranges of bias and *RSD* of general interest. Ref (**8**) indicates how to handle yet larger bias magnitudes.

10.5 *Estimating Components of the Combined Mean Square Element* MSE_c

10.5.1 The components (Δ^2, RSD^2_{inter}, RSD^2_{pump}, and $RSD^2_{analytical}$) of the combined mean square element MSE_c (Eqs. 12 and 15) can be estimated as follows. The components, Δ^2 and RSD^2_{inter}, may be categorized as *Type A standard uncertainities* (ISO GUM), meaning that their estimates are obtained by statistical means from the data obtained during sampler evaluation. RSD^2_{pump} can be, and has, also been estimated by statistical means in specific applications. However, for illustration, RSD^2_{pump} is estimated here as a *Type B standard uncertainty*, meaning, determined on the basis of "experience with, or general knowledge of, the behavior and property of relevant materials and instruments" (ISO GUM). $RSD^2_{analytical}$ may be obtained from experiment separate from this practice as a Type A standard uncertainty, as in Practice D 6552.

10.5.2 Compute estimates of Δ^2 and RSD^2_{inter} at each size distribution (*MMD*, *GSD*) of interest. The statistical details required for these estimates are presented in Annex A.

10.5.3 Assume, as suggested in the NIOSH Manual of Analytical Methods, that $RSD^2_{pump} = 5\ \%$, with infinite degrees of freedom. As described in ISO GUM, this assumption corresponds to stating that variation from pump fluctuation follows an approximately rectangular distribution with estimates ranging within $\pm\sqrt{3} \times 5\ \%$ of the mean.

10.5.4 $RSD^2_{analytical}$ depends on the specific analysis required and therefore is not estimated within the sampler evaluation described in this practice.

10.6 *Confidence Limit on the Combined Mean Square Element* MSE_c

10.6.1 Statistical details of this calculation may be found in Annex A. However, the basic idea is as follows: The variances of each component of MSE_c are estimated. Then the part of the estimate of MSE_c which varies (that is, excluding the constant RSD^2_{pump}) is approximated as proportional to a chi-square variable with an effective number of degrees of freedom determined so that the variance is consistent (Satterthwaite approximation (ISO GUM)). The result is a 95 %-confidence level for MSE_c, and therefore, through Eq. 14, the symmetric-range accuracy confidence limit ${}_{95\ \%}A$.

10.6.2 The confidence limit ${}_{95\ \%}A$ (accounting for evaluation uncertainty) is a counterpart to what is denoted the *expanded uncertainty U* in (ISO GUM). Aside from differences in application, both quantities are used for bracketing the measurand by confidence intervals. The expanded uncertainty *U*, used for constructing symmetric intervals about measured values in the case that bias is negligible, is equal to the combined uncertainty u_c multiplied by a *coverage factor* given in terms of a Student-t quantile, indicating continual re-evaluation of a method at each application. In contrast, ${}_{95\ \%}A$ leads, with 95 % confidence in a single (extensive) initial method evaluation to intervals that enclose the conventional concentration at least 95 % of the time. For example, suppose ${}_{95\ \%}A$ is approximately independent of the measurand value c_R and that the likelihood that ${}_{95\ \%}A > 1$ is negligible. Then 3.2.11 implies the following inequality:

$$\frac{\hat{c}}{1 + {}_{95\ \%}A} < c_R < \frac{\hat{c}}{1 - {}_{95\ \%}A} \qquad (16)$$

for > 95 % of estimates ĉ, at 95 % confidence in the evaluation experiment. Note that the interval of Eq. 16 is not exactly symmetrical about the estimate ĉ, unlike intervals using the expanded uncertainty *U* (ISO GUM), with bounds $\hat{c} \pm U$.

10.6.3 An example of the difference between ${}_{95\ \%}A$ and $\hat{A}$ can be given: At $MMD = 10\mu m$ and $GSD = 3$, the Higgins-Dewell cyclone has (**5**) $\hat{\Delta} = 7\ \%$, $\widehat{RSD}_{inter} = 5\ \%$ $RSD_{pump} < 1\ \%$. Now suppose that $c_R = 2$ mg/m^3 and that (**25**) $\sigma_{mass} = 9.1$ µg; then Eq. 9 gives $RSD_{analytical} = 0.4\ \%$. Thus, the total random variation is $RSD = 5.1\ \%$, and so $\hat{A} = 15\ \%$. Following Annex A, it is found that ${}_{95\ \%}A$ is about 40 % larger than $\hat{A}$. This value is expected to be typical of the evaluation uncertainty (at 95 % confidence) over a wide range of size distributions at $c_R = 2$ mg/m^3 and analytical error $\sigma_{weight} = 9.1$ µg. For other specific applications, the corresponding figure can be calculated.

11. Non-Performance Items

Because of the complexity of aerosol sampling, several respirable aerosol sampler characteristics remain unevaluated. These may be controlled as suggested in this section through sampler specification, rather than performance criteria. Any of the suggested features not presently available are to be considered recommendations for future sampling equipment.

11.1 *Recommendation of the Use of Only Conductive Samplers*—This practice presents a recommendation that only conductive samplers be used in aerosol sampling.

11.1.1 *Justification for Recommendation*— Various authors have reported sampling problems specifically posed by the nonconductive 10-mm cyclone. The basic problem is that charges on a nonconducting sampler are immobile and therefore provide a localized source of electric field. This can strongly affect the trajectories of charged aerosol particles in the air flowing into the sampler. Quantitatively, a 10 % variability has been reported to be associated with charge effects **(26)**. Furthermore, evidence exists that a charged sampler may undersample moderately charged aerosol by as much as 40 % **(27)**. Finally, the conductivity of the filter holder itself following the 10-mm cyclone may be significant. A 25 % increase in the aerosol collected upon increasing the holder's conductivity has been reported **(28)**. Electrical charging typical on aerosol to be found in many workplaces has also been documented **(29)**.

11.1.2 *Availability of Samplers*—The presently used 10-mm cyclones are fashioned out of a poorly conductive plastic relative to metals. At one time, however, a conductive graphite-filled plastic was used in the construction of the sampler. Therefore, with a shift in the manufacturing process, a 10-mm conductive cyclone could again be available. The Higgins-Dewell cyclone,[4] now available in the United States, is made of metal and is therefore conductive. The 37-mm filter cassette[4,11] which is used with the cyclone should be made of a conductive material, for example, graphite-filled plastic.

11.2 *Recommendation of Controlled Pump Fluctuations*—Pulsation amplitude must be less than 20 % of the mean flow. This amplitude may be measured with an in-line hot-wire anemometer placed close to the sampler, analyzing the output using an oscilloscope or ac voltmeter.

11.2.1 *Justification*—Bias has been shown **(30,31)** to be caused in a cyclone by pulsation of the personal sampling pump. Cyclone samplers with pulsating flow can have negative bias as large as −22 % relative to samplers with steady flow. The magnitude of the bias depends on the amplitude of the pulsation at the cyclone aperture and the aerosol size distribution. For pumps with instantaneous flow within 20 % of the mean, the pulsation bias is estimated at less than −2 % for most size distributions encountered in the workplace.

11.3 *Recommendation of Controlled Pump Accuracy*—In accordance with 10.5.3, control the relative standard deviation of the pump flow rate RSD_{pump} through the use of a self-regulating network to $RSD_{\mathrm{pump}} < 5\ \%$ (NIOSH Manual of Analytical Methods).

12. Report

12.1 Several alternatives exist for using the results of the experimental evaluations described in this practice. For example, it is possible to classify the samplers in accordance with specific accuracy criteria. Alternatively, the NIOSH accuracy criterion (**10-13** and the NIOSH Manual of Analytical Methods) presents a pass/fail requirement that acceptable sampling methods have better than 25 % symmetric-range accuracy at the 95 % (evaluation) confidence level. What is denoted as *sampler accuracy* itself may, in fact, be defined in alternative manners. Here it is suggested simply that sufficient information is presented that most performance criteria selected for specific applications can be easily implemented. Therefore, the following should appear in the report of the sampler evaluation.

12.2 Describe the sampling efficiency model used. Present a short table giving the fitted sampling efficiency parameters θ_p, $p = 1, ..., P$ (for example, 4). Plot sampling efficiency data, averaged over sampler and replicate, together with the model curves at the four sampler flow rates of the evaluation.

12.3 Present maps giving iso-curves over $MMD = 1$ to 25 µm and $GSD = 1.5$ to 3.5 for estimates of the following: inter–sampler variation RSD_{inter}, and bias Δ.

12.4 Prepare tables of estimates of Δ^2, RSD^2_{inter}, and RSD^2_{eval}, and MSE (A1.5) in digital form. Relevant estimates of the combined mean square element MSE_{c} (Eq. 15) and confidence limit (equivalent to $_{95\ \%}A$) can then be constructed, given external knowledge of $RSD_{\mathrm{analytical}}$. The tables should be at $MMD = 1$ µm, 2 µm, ..., 25 µm and $GSD = 1.5, 1.6, ..., 3.5$.

12.5 Present maps of estimates of A and $_{95\ \%}A$ by setting $RSD_{\mathrm{analytical}}$ equal to zero. A note should be included stating that $RSD_{\mathrm{analytical}}$ of a particular analytical application would generally increase the values of the estimates of A and $_{95\ \%}A$.

12.6 It may also be useful to give a brief statement as to the purpose behind estimating $_{95\ \%}A$. An example would be:

"With 95 % confidence in the method evaluation, the symmetric-range accuracy confidence limit $_{95\ \%}A$ results in confidence intervals enclosing measurands >95 % of the time. $_{95\ \%}A$ then plays the role of the expanded uncertainty U (ISO GUM)."

13. Keywords

13.1 aerosol; air monitoring; bias; confidence; conventions; deposition; evaluation; fractions; particle; particulates; penetration; performance; random variation; respirable; sampling and analysis; sampling efficiency; size-selective; tolerance; uncertainty; workplace atmospheres

ANNEX

(Mandatory Information)

A1. STATISTICAL DETAILS

A1.1 The sampler performance assessment of this practice accounts for uncertainty in the sampler evaluation by computing a confidence limit on the combined mean square element MSE_{c}(Eq. 15) as well as an estimate of MSE_{c} itself. This is accomplished by analyzing the concentration estimates $\hat{c}_s$ from sampler s in accordance with the following model characterizing the sampler evaluation:

$$\hat{c}_s = c + \epsilon_{eval\ s} + \epsilon_s, \qquad (A1.1)$$

where random variables, $\epsilon_{\mathrm{eval\ s}} = N[0, \sigma^2_{\mathrm{eval}}]$ and $\epsilon_{\mathrm{s}} = N[0, \sigma^2_{\mathrm{inter}}]$, are represented by their respective standard deviations, σ_{eval} and σ_{inter}. The quantity σ_{eval} contains, for example, evaluation concentration fluctuations and aerosol counting errors. The quantity σ_{inter} characterizes the intersampler variability.

A1.2 The variance $\sigma^2_{inter} + \sigma^2_{eval}$ of $\hat{c}_s$ is estimated with S - 1 df by:

$$\hat{\sigma}^2_{inter} + \hat{\sigma}^2_{eval} = \frac{1}{S-1}\sum_s (\hat{c}_s - \hat{c})^2 \quad (A1.2)$$

A1.3 σ_{eval} is itself estimated from the uncertainty in the fitted parameters at fixed sampler *s* from the assumption that all the uncertainty is from experimental error and no part from lack of fit to the model. In other words, var($\hat{c}_s$) is estimated at fixed *s* from the nonlinear regression's asymptotic variance-covariance matrix ${}_s\text{cov}_{ij}$ as:

$$\hat{var}(\hat{c}_s)|_s \approx \sum_{i,j}\frac{\partial \hat{c}_s}{\partial \theta_i}\, {}_s\hat{cov}_{ij}\frac{\partial \hat{c}_s}{\partial \theta_j} \quad (\textit{fixed } s) \quad (A1.3)$$

This quantity is proportional to $(n \cdot F - P)^{-1}$, where *n* is the number of replicates, *F*, the number of flow rates in the evaluation, and *P* is the number of model parameters. The derivatives, $\partial\hat{c}_s/\partial\theta_j$, are computed numerically. Averaging over the samplers tested, an estimate of σ_{eval} is therefore given by:

$$\hat{\sigma}^2_{eval} \approx \frac{1}{S}\sum_s \sum_{i,j}\frac{\partial \hat{c}_s}{\partial \theta_i}\, {}_s\hat{cov}_{ij}\frac{\partial \hat{c}_s}{\partial \theta_j} \quad (A1.4)$$

with approximately $S{\cdot}(n{\cdot}F{-}P)$ degrees of freedom, since $P{\cdot}S$ degrees of freedom determine the fitted parameters.

A1.4 The estimate for σ_{inter} is then found from Eqs. A1.2 and A1.4.

A1.5 Estimation of the combined mean square element MSE_c is simplified through computing an estimated mean square element $\hat{MSE}$ (**32**) defined by:

$$\hat{MSE} \equiv \frac{1}{S}\sum_s(\hat{c}_s - c_R)^2/c_R^2 \quad (A1.5)$$

$$= \hat{\Delta}^2 + R\hat{S}D^2_{inter} + R\hat{S}D^2_{eval}$$

Given knowledge of $R\hat{S}D^2_{analytical}$ and $R\hat{S}D^2_{pump}$, the estimate of MSE_c may then be directly obtained (Eqs. 12 and 15) by using $\hat{MSE}$ (Eq. A1.5), eliminating $R\hat{S}D^2_{eval}$(that is, σ^2_{eval}/c^2_R) through Eq. A1.4.

A1.6 Finally, a confidence limit on MSE_c, and therefore (from Eq. 14) the symmetric-range accuracy ${}_{95\,\%}A$, may be calculated in accordance with the sketch given in 10.6.1. To this end, the estimation of the various variance components is simplified by the following:

A1.6.1 $\hat{MSE}$ and $R\hat{S}D^2_{eval}$ are uncorrelated.

A1.6.2 $R\hat{S}D^2_{eval}$ may be approximated in terms of a chi-square variable.

A1.6.3 $S \times \hat{MSE}/(R\hat{S}D^2_{inter} + R\hat{S}D^2_{eval})$ is a noncentral chi-square random variable (**33**). In terms of the number of degrees of freedom *S* and noncentrality parameter λ, the expected value and variance of the noncentral χ^2 are $S + \lambda$ and $2S + 4\lambda$, respectively. The parameter λ is given by:

$$\lambda = S \times \Delta^2/(RSD^2_{inter} + RSD^2_{eval}) \quad (A1.6)$$

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