



Designation: D6552 – 06 (Reapproved 2021)

Standard Practice for Controlling and Characterizing Errors in Weighing Collected Aerosols¹

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1. Scope

1.1 Assessment of airborne aerosol hazards in the occupational setting entails sampling onto a collection medium followed by analysis of the collected material. The result is generally an estimated concentration of a possibly hazardous material in the air. The uncertainty in such estimates depends on several factors, one of which relates to the specific type of analysis employed. The most commonly applied method for analysis of aerosols is the weighing of the sampled material. Gravimetric analysis, though apparently simple, is subject to errors from instability in the mass of the sampling medium and other elements that must be weighed. An example is provided by aerosol samplers designed to collect particles so as to agree with the inhalable aerosol sampling convention (see ISO 7708, Guide D6062, and EN 481). For some sampler types, filter and cassette are weighed together to make estimates. Therefore, if the cassette, for example, absorbs or loses water between the weighings required for a concentration estimation, then errors may arise. This practice covers such potential errors and provides solutions for their minimization.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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.*

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 Air Quality.

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2. Referenced Documents

2.1 ASTM Standards:²

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D4096 Test Method for Determination of Total Suspended Particulate Matter in the Atmosphere (High-Volume Sampler Method)

D4532 Test Method for Respirable Dust in Workplace Atmospheres Using Cyclone Samplers

D6062 Guide for Personal Samplers of Health-Related Aerosol Fractions

2.2 International Standards:³

EN 481 Workplace Atmospheres — Size Fraction Definitions for Measurement of Airborne Particles in the Workplace

EN 13205 Workplace Atmospheres — Assessment of Performance of Instruments for Measurement of Airborne Particle Concentrations

2.3 ISO Standards:⁴

ISO 7708 Air quality — Particle size fraction definitions for health-related sampling

ISO 20581 Workplace atmospheres — General requirements for performance of procedures for the measurement of chemical agents

ISO 20988 Air quality — Guidelines for estimating measurement uncertainty

ISO GUM Guide to the Expression of Uncertainty in Measurement (1998)

3. Terminology

3.1 Definitions:

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

3.2 Definitions of Terms Specific to This Standard:

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

³ Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, <http://www.cen.eu>.

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

3.2.1 *blank substrate, n*—a collection medium or substrate coming from the same batch as the sampling medium, but unexposed.

3.2.2 *equilibration time, n*—For the purposes of this practice, a time constant (seconds) characterizing an approximate exponentially damped approach of the mass of an aerosol collection medium to a constant value. The constant can be defined as the mean difference of the mass from equilibrium per mean mass loss or gain rate as measured over a finite time interval.

3.2.2.1 *Discussion*—There may be important instances in which *several* time constants are required to describe the approach to equilibrium.

3.2.3 *estimated overall uncertainty (U), n*— $2 \times$ estimated standard deviation of estimated mass, in the case of negligible uncorrectable bias (see ISO 20581).

3.2.4 *field blank, n*—a blank substrate that undergoes the same handling as the sample substrate, generally including conditioning and loading into the samplers or transport containers, as well as transportation to the sampling site, but without being exposed.

3.2.4.1 *Discussion*—If blanks are not actually loaded into samplers, losses due to handling could be underestimated.

3.2.5 *lab blank, n*—a blank substrate that undergoes the same handling as the sample substrate in the laboratory, including conditioning and loading into the samplers or transport containers when this is done in the laboratory.

3.2.6 *limit of detection (LOD), n*—a value for which exceedence by measured mass indicates the presence of a substance at given false-positive rate: $3 \times$ estimated standard deviation of the measured blank substrate mass (see [Annex A2](#)).

3.2.7 *limit of quantitation (LOQ), n*—a value for which exceedence by measured mass indicates the quantitation of a substance at given accuracy: $10 \times$ estimated standard deviation of the measured blank substrate mass (see [Annex A2](#)).

3.2.8 *substrate, n*—sampling filter, foam, and so forth together with whatever mounting is weighed as a single item.

3.2.8.1 *Discussion*—The 25 or 37-mm plastic filter cassette often used for total dust sampling in either its closed-face or open-face version is NOT part of the substrate in the definition above, since it is not weighed.

3.3 Symbols:

α	= detection error rate
B	= number of substrate batches in method evaluation
b	= batch index (1, ..., B)
β	= mean substrate mass change during evaluation experiment
CV_{\max}	= maximum relative error acceptable in quantifying collected mass
$\Delta m_{fb}(\mu\text{g})$	= substrate mass change
$\varepsilon_b(\mu\text{g})$	= substrate weight-change random variable representing inter-batch variability
$\varepsilon_{fb}(\mu\text{g})$	= substrate weight change residual random variable with variance σ^2

f	= substrate index (1, ..., F)
F	= number of substrates (for example, filters) in each batch tested in method evaluation
γ	= method evaluation error rate
$LOD(\mu\text{g})$	= limit of detection: $3 \times s_w$
$LOD_{1-\gamma}(\mu\text{g})$	= LOD confidence limit
$LOQ(\mu\text{g})$	= limit of quantitation: $10 \times s_w$
$LOQ_{1-\gamma}(\mu\text{g})$	= LOQ confidence limit
N_b	= number of blanks per substrate set
ν	= number of degrees of freedom in method evaluation
Φ	= cumulative normal function
χ^2	= chi-square random variable
$\chi_{\gamma,\nu}^2$	= chi-square quantile (that is, a fixed number that exceeds the random variable χ^2 at probability γ)
RH	= relative humidity
$u(\mu\text{g})$	= uncertainty component in two balance readings, an estimate of σ
$u_w(\mu\text{g})$	= weighing uncertainty, estimate of σ_w
$\sigma(\mu\text{g})$	= uncorrectable (for example, by way of blank correction) standard deviation in (single) mass-change measurement
$\sigma_{1-\gamma}(\mu\text{g})$	= confidence limit on σ
$\sigma_w(\mu\text{g})$	= standard deviation in collected mass determination
U	= overall uncertainty

4. Significance and Use

4.1 The weighing of collected aerosol is one of the most common and purportedly simple analytical procedures in both occupational and environmental atmospheric monitoring (for example, Test Method [D4532](#) or [D4096](#)). Problems with measurement accuracy occur when the amount of material collected is small, owing both to balance inaccuracy and variation in the weight of that part of the sampling medium that is weighed along with the sample. The procedures presented here for controlling and documenting such analytical errors will help provide the accuracy required for making well-founded decisions in identifying, characterizing, and controlling hazardous conditions.

4.2 Recommendations are given as to materials to be used. Means of controlling or correcting errors arising from instability are provided. Recommendations as to the weighing procedure are given. Finally, a method evaluation procedure for estimating weighing errors is described.

4.3 Recommendations are also provided for the reporting of weights relative to LOD (see [3.2.6](#)) and LOQ (see [3.2.7](#)). The quantities, LOD and LOQ, are computed as a result of the method evaluation.

5. Weight Instability, Causes, and Minimization

5.1 Weight instability of sampling substrates may be attributed to several causes. The following subclauses address the more important of these.

5.1.1 Moisture Sorption:

5.1.1.1 Moisture sorption is the most common cause of weight instability. Water may be directly collected by the filter or foam or other substrate material that is weighed. Water

sorption by any part of the sampling system that is weighed must be suspected as well. For example, the sampling cassette itself, if weighed, may be the cause of significant error (1)⁵ (see also 8.2.2).

5.1.1.2 The effects of water sorption can be reduced by using nonsorptive materials. However, there may exist specific sampling needs for which a hydrophobic material is not feasible. Table 1 presents a list of common aerosol sampling substrates with different water adsorption features.

NOTE 1—Gonzalez-Fernandez, Kauffer et al, and Lippmann (2-4) provide further details. Also, Vaughan et al (5) report that filters of evidently the same material, but originating from different manufacturers, may have widely differing variabilities.

NOTE 2—There is generally a trade-off between hydrophobicity and conductivity in many materials (6). Therefore, one must be aware of the possibility of creating sampling problems while reducing hygroscopicity.

NOTE 3—Pretreatments of substrates, such as greasing, may also affect water sorption.

5.1.2 *Electrostatic Effects*—Electrostatic effects are a common source of weighing problems. These effects can usually be minimized by discharging the substrate through the use of a plasma ion source or a radioactive source. Using conductive materials may reduce such problems. Lawless and Rodes (7) present details on electrostatic effects and their minimization (see also Engelbrecht et al (8)).

5.1.3 *Effects of Volatile Compounds (other than water)*—Volatile compounds may be present in unused collection media (3) or may be adsorbed onto media during sampling.

5.1.3.1 Desorption of volatiles from unused media may be controlled, for example, by heating or oxygen plasma treatment prior to conditioning and weighing. Alternatively, losses may be compensated by the use of blanks (see Section 6).

5.1.3.2 When volatile materials collected during sampling form part of the intended sample, standardized written procedures are required to ensure that any losses are minimized or at least controlled, for example, by conditioning under tightly specified conditions.

NOTE 4—When volatile materials collected during sampling are not part of the intended sample, it may be difficult to eliminate them if weighing

⁵ The boldface numbers in parentheses refer to the list of references at the end of this standard.

TABLE 1 Water Sorption Characteristics of Some Aerosol Sampling Media

Substrate or Cassette Type	Very Low	Low	High	Very High
Cellulose fiber filter			*	
Glass fiber filter		*		
Quartz fiber filter		*		
Cellulose ester membrane filter			*	
Polytetrafluoroethylene filter		*		
PVC membrane filter	*			
Polycarbonate filter		*		
Silver membrane filter		*		
Polyurethane foam				*
Greased polyester film impaction substrate		*		
Greased aluminum foil impaction substrate		*		
Carbon-filled resin				*
Aluminum cassette	*			
Stainless steel cassette	*			

is the only form of analysis. Preferably nonsorptive media should be used.

5.1.4 *Handling Damage*—Lawless and Rodes (7) give recommendations on minimizing balance-operator effects. If friable substrates are used, procedures are needed to avoid mechanical damage during gravimetric analysis.

5.1.4.1 The air sampling equipment should be designed so that the substrate is not damaged during assembly and disassembly.

5.1.4.2 Flat tipped forceps are recommended for handling filters. Nonoxidizing metal tins may be used to weigh delicate substrates without direct handling.

5.1.4.3 Parts to be weighed shall not be touched with the hands, unless gloved.

5.1.4.4 Handling shall take place in a clean environment to avoid contamination.

5.1.4.5 Gloves, if used, shall leave no residue on what is weighed.

5.1.5 *Buoyancy Changes*—Corrections (9) for air buoyancy, equal to the density of air multiplied by the air volume displaced, are not necessary for small objects, such as a 37-mm diameter membrane filter. However, there may exist circumstances (for example, if an entire sampling cassette were weighed without the use of correcting blanks) in which the object to be weighed is so large that buoyancy must be corrected. For example, if the volume weighed exceeds 0.1 cm³, then correction would be required to weigh down to 0.1 mg if pressure changes of the order of 10 % between weighings are expected. If such a correction is necessary, the atmospheric pressure and temperature at the time of weighing should be recorded.

6. Correcting for Weight Instability

6.1 *Recommended Method for Correction by Use of Blanks*—The use of blanks is the most important practical tool for reducing errors due to weight instability. Correction for weight instability depends on the specific application and should follow a written procedure. The general principles are as follows. Blank sampling media are exposed, as closely as possible, to the same conditions as the active sampling media, without actually drawing air through. Correction is effected by subtracting the average blank weight gain from the weight gain of the active samples. Of course, if the atmosphere to be sampled contains water (or other volatile) droplets, then the use of blanks alone cannot correct. Kauffer et al (3) note that blanks may also offer correction for filter material losses. Blanks shall be matched to samples, that is, if the sample consists of a filter within a cassette that is weighed, the blank shall be the same type of filter within the same type of cassette.

6.1.1 An alternative procedure employs matched weight filters consisting of two nearly equal-weight filters, one placed in front of the other, with the sampler following employed as blank. The collected mass is estimated simply by subtracting the filter masses following sampling. Analysis of uncertainty is similar to the presentation here, but also involves estimation of the uncertainty of the filter matching.

6.2 *Minimum Number of Blanks*—Generally, at least one blank is recommended for each ten samples. Measurement