Designation: D6552 – 06(Reapproved 2011)

Standard Practice for Controlling and Characterizing Errors in Weighing Collected Aerosols[1]

This standard is issued under the fixed designation D6552; 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 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 TR 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 given in SI units are to be regarded as 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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 482 Workplace Atmospheres—General Requirements for Performance of Procedures for the Measurement of Chemical Agents

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

2.3 *ISO Standards:*[4]

ISO TR 7708 Air Quality—Particle Size Fraction Definitions for Health-related Sampling

ISO GUM Guide to the Expression of Uncertainty in Measurement (1993)

ISO 20988 Air Quality—Guidelines for Estimating Measurement Uncertainty

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:*

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

3.2.2 *equilibration time*—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

[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.

Current edition approved Oct. 1, 2011. Published October 2011. Originally approved in 2000. Last previous edition approved in 2006 as D6552 - 06. DOI: 10.1520/D6552-06R11.

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

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

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)*—2 × estimated standard deviation of estimated mass, in the case of negligible uncorrectable bias (see EN 482).

3.2.4 *field blank*—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*—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)*—a value for which exceedence by measured mass indicates the presence of a substance at given false-positive rate: 3 × estimated standard deviation of the measured blank substrate mass (see Annex A2).

3.2.7 *limit of quantitation (LOQ)*—a value for which exceedence by measured mass indicates the quantitation of a substance at given accuracy: 10 × estimated standard deviation of the measured blank substrate mass (see Annex A2).

3.2.8 *substrate*—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
Δm_{fb}(µg)	=	substrate mass change
ε_b (µg)	=	substrate weight-change random variable representing inter-batch variability
ε_{fb} (µg)	=	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 (µg)	=	limit of detection: 3 × s_w
$LOD_{1-\gamma}$ (µg)	=	LOD confidence limit
LOQ (µg)	=	limit of quantitation: 10 × s_w
$LOQ_{1-\gamma}$ (µg)	=	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 (µg)	=	uncertainty component in two balance readings, an estimate of σ
u_w (µg)	=	weighing uncertainty, estimate of σ_w
σ (µg)	=	uncorrectable (for example, by way of blank correction) standard deviation in (single) mass-change measurement
$\sigma_{1-\gamma}$ (µg)	=	confidence limit on σ
σ_w (µg)	=	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] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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 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.

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	*			

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 cm^3, 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 schemes in current use require between one and four blanks per batch. See A1.1 for advantages of multiple blanks.

6.3 *Weighing Times and Sequence*—Blanks shall be interspersed with samples, before and after use, so as to detect systematic variations in mass (for example, due to sorption or evaporation of a contaminant during weighing).

6.4 *Conditioning Times*—Conditioning times for reaching equilibrium with the weighing environment may vary from a few hours to several weeks, depending on the specific sampling media. Typically, for workplace sampling applications, overnight conditioning is satisfactory. For sampling media with longer conditioning times, error correction through the use of blank substrates is particularly important. Charell and Hawley (**10**) indicate that extremely short equilibration times exist for some filters.

6.5 *Storage Stability*—Unused substrates shall, where possible, be stored prior to weighing and conditioning in a clean laboratory whose environmental conditions do not differ too greatly from the balance environment. Preweighed substrates shall be stored together with weighed blanks and used in any case within the assigned shelf life. The assigned shelf life and storage requirements shall be documented as part of a written weighing procedure.

NOTE 5—Shelf life depends on substrate material, storage conditions, cassette material, and required LOQ or LOD.

6.5.1 Archived samples shall be stored together with weighed blanks in a clean laboratory whose environmental conditions do not differ too greatly from the balance environment. Note that transfers of mass between filters and cassettes could occur when these media are stored together.

7. Transport of Samples to Laboratory

7.1 *General*—The transportation of samples shall form part of a written procedure. The transport procedure shall be validated to ensure that significant losses do not occur. Follow test method of Annex A4.

7.1.1 The main problems occurring during handling and transport of sampling media are:

7.1.1.1 With substrates designed to be separated from sampling cassettes, dust may migrate from substrate to the transport container, and hence be lost.

7.1.1.2 On the other hand, contamination of the sampling cassette and cover lid (when supplied) can be a significant source of error if the cassette (including cover lid) is part of the substrate.

7.1.1.3 If a cover lid is not supplied, dust may be lost from the cassette to the transport container.

NOTE 6—Transportation losses are discussed in Awan and Burgess and van Tongeren et al (**11**, **12**).

7.2 *Recommended Packaging:*

7.2.1 Each substrate that is not mounted in a sampling cassette shall be transported in a petri dish, a tin, or a similar closed container that prevents contact with the surface of the collection medium.

7.2.2 Sampling cassettes (that is, with mounted filters) should preferably have cover lids during transport. If the sample consists of all dust deposited inside the sampling cassette (with filter), then dust that migrates during transport from cassette to cover lid shall also be weighed. The only foolproof way to handle this is to weigh the cover lid with the sampling cassette.

7.2.3 The sealed substrates shall be transported in a suitable container or package. The floor, ceiling, and walls of the container should be lined with a spongy material (preferably electrically conducting), which may absorb some mechanical shock and thus protect the samples during transport.

7.2.4 The samples shall be protected from excessive heating or cooling during transport.

NOTE 7—Special procedures are needed for the transport of unstable particles or biological materials.

NOTE 8—When there is a possibility for dust to be lost from the substrate, the losses may be recovered by transporting the substrate within a container that can itself be weighed.

8. Weighing Equipment and Procedure

8.1 *Balance*—The balance should be matched to the task. The choice of balance will depend on the desired limits of quantitation (see 3.2.7) for the application and on the maximum tare weights of the samples to be weighed. Workplace air sampling typically requires either a five- or six-figure balance. The balance shall be regularly calibrated using reference weights traceable to International Standards.

NOTE 9—The performance of different balances was compared and reported in Vaughan et al (**5**). In one experiment, repeat weighings of 25-mm filters were made with filters stored between weighings in ventilated tins with conditions not strictly controlled. A1-µg (six-figure) balance was compared to a 10-µg (five-figure) balance. It was concluded that using a 1-µg versus a 10-µg balance approximately halves the standard deviation of repeat weighing. Intra-day standard deviation was smaller than the inter-day deviation and is expected to be of greater importance when blanks are used to correct inter-day variation in the balance room (see also Feeney et al (**13**)).

8.2 *Recommended Environmental Controls:*

8.2.1 Equilibration and weighing shall be carried out under the same conditions, that is, in the same room or chamber. Environmental control can be achieved in different ways: (*1*) by means of a balance room containing balance, samples, and the weighing personnel; or (*2*) by means of an environmentally-controlled chamber containing balance and samples, sited within a clean laboratory.

NOTE 10—It may be possible to achieve an adequate level of environmental control without the need for active air conditioning. However, the quality of gravimetric analysis depends strongly on the quality of the environmental control.

8.2.2 For sensitive (that is, hygroscopic) samples, temperature and humidity control in the weighing chamber or balance room are important. In these cases, temperature should be maintained constant within ±2°C of the set point, and humidity should be constant to within ±5 % RH at the target temperature. The target temperature and humidity should be in the range of operating conditions recommended by the manufacturer of the balance (for example, 20 ± 2°C and 50 ± 5 % RH). Very dry atmospheres (for example, < 20 % RH) are to be avoided, as electrostatic buildup on the samples is more likely in such conditions. The environmental controls shall be capable of compensating for heat and humidity sources such as people working in the room or electrically-powered instruments in the room (**3**).

8.2.3 The particulate content of the balance room or chamber air should be minimized by filtration (for example, by HEPA filtration).

8.2.4 Fresh air should be supplied consistent with the health and comfort requirements of personnel working in the balance