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it may not be technically possible to adequat

Aerosols¹

Standard Practice for Controlling and Characterizing Errors in Weighing Collected

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1. Scope

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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 accuracy of 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 D 6062M, 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 the 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
- D 1356 Terminology Relating to Sampling and Analysis of Atmospheres
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D 4096 Test Method for Determination of Total Suspended Particulate Matter in the Atmosphere (High-Volume Sampler Method) of the user of Hus Standard to establish appropriate safety and health practices a
2. **Referenced Documents**
2.1 *ASTM Standards*:²
2. **Referenced Documents**
5 D 4550 Terminology Relating to Sampling and Analysis of Atmo

D 4532 Test Method for Respirable Dust in Workplace Atmospheres

D 6062M Guide for Personal Samplers of Health-Related Aerosol Fractions (Metric)²

2.2 *International Standards:*³

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

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

Current edition approved May 10, 2000. Published July 2000.

ISO 20988 Air Quality—Guidelines for Estimating Measurement Uncertainty

¹ This test method practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Atmospheres .

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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* $\overline{\text{b}}$ 11.03. 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, 11 W 42nd Street, 13th Floor, New York, NY 10017.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

3. Terminology

3.1 *Definitions*:

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

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 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* \times 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: $3 \times$ 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: $10 \times$ 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:*

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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 D 4532 or D 4096). 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 may reduce such problems. Lawless and Rodes **(7)** present details on electrostatic effects and their minimization (see also Engelbrecht et al **(8)**).

function of all (8)).
5.1.3 *Effects of Volatile Compounds (other than water)*—Volatile compounds may be present in unused collection media **(3)** or
ay be adocrhed onto media during sampling 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 conditioning and weighing. Alternatively, losses may be compensated by the use of blanks (see Se 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.

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

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. Filter matching. Analysis of uncertainty is similar to the presentation here, out also involves estimation of the uncertainty of the internations of the matching.
6.2 *Minimum Number of Blanks*—Generally, at least one blan

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 a 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).

riations in mass (for example, due to sorption or evaporation or a contaminant during weighing).
6.4 *Conditioning Times*—Conditioning times for reaching equilibrium with the weighing environment may vary from a few
urs to 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
contagi whose environmental conditions do not differ too graphy from the belonge environment. Provisi 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*:

7.2.1Each substrate that is not mounted in a sampling cassette shall be transported in a petri dish, a tin, or a similar closed container.

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. A 1-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. 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 gravimetric analysis depends strongly on the quality of the environmental 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
 Example 18.2.2 For sensitive (that is, hygroscopic) samples, temperature and humidity show in are important. In these cases, temperature should be maintained constant within $\pm 2^{\circ}C$ of the set point, and humidity should be constant to within \pm 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, $20 \pm 2^{\circ}$ C and 50 ± 5 % RH). Very dry atmospheres (for example, $\lt 20\%$ 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 room or laboratory. Turbulent air movement generated by ventilation or humidity control in the balance room or chamber should be minimal so as not to affect the balance reading.

8.3 *Other Equipment Requirements* —The balance should be located in an area that is free from excessive vibration (for example, from elevators and rotating machinery). A massive weighing table (for example, one made of 200 kg of marble) is one means to dampen ambient vibration. The area should be away from doors, windows, air ducts, and sources of radiant energy such as direct sunlight or ovens. The electrical supply to the balance should be stable, and the balance should not be located near strong sources of electromagnetic radiation.

8.4 *Procedure*:

8.4.1 The weighing procedure shall be documented.

8.4.2 Equilibration of the sample to the temperature and humidity of the balance room or chamber shall be done for a period appropriate to the sample. The samples shall be kept in clean containers but open to the atmosphere so that equilibration can take place.

Note 11—If sampling took place in a humid environment, then, prior to equilibration, dessication may be needed to remove excess water from the samples.

8.4.3 Elimination of static electricity from the sample is extremely important and should be done immediately prior to placement of the sample on the balance pan. Alternatively, a static eliminator can be placed inside the balance chamber.

8.4.4 The balance shall not be recorded until after it has stabilized.

8.4.5 Rezero the balance between every reading.

8.4.6 Defective substrates may be identified and rejected if their initial weights differ from normal.

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9. Reporting of Measured Weight

9.1If the measured mass exceeds the LOQ (see

9.1 Details on reporting weights depends on the specific application. The following is an example of a minimal reporting policy. 9.1.1 If the measured mass exceeds the LOQ (see Annex A1-Annex A3), then it is would be reported.

9.1.2 If the result falls between the LOD (see Annex A1-Annex A3) and the LOQ, then it is would be reported that the measured mass is between LOD and LOQ.

9.1.3 If the estimate falls below the LOD, it iswould be reported that the mass is below the LOD.

NOTE 12—If the LOD is exceeded, then the false-positive error rate in asserting detection is $\lt 1\%$ if the method is evaluated with as many degrees of freedom (25) as specified in Annex A1-Annex A3.

NOTE 13—In some applications, a series of measured masses, each below the LOD, may be of value, for example, in asserting the presence of mass over the entire series, even if the individual measurements are too small to assert detection with confidence.

9.1.4 LOD and LOQ shall be determined as described in Annex A1-Annex A3 and shall be given in the report.

10. Keywords

10.1 blank; control filter; filter; foam; gravimetric; limit of detection; limit of quantitation; LOD; LOQ; sampling media; weighing; weight

ANNEXES

(Mandatory Information)

A1. ESTIMATION OF MEASUREMENT ERRORSUNCERTAINTY

A1.1 The Imprecision σ_w **The Variance** σ_w^2 ** in Weight Estimates**

A1.1.1 The standard deviation variance σ_{w}^2 in any collected mass estimate depends on the number of blanks (preferably field ALLI The standard deviation variance σ_w in any collected mass estimate depends on the number of blanks (preferably field blanks) used to correct for correlated sampling medium variation. The collected mass imprecision outlined here in Annex A1 through an extensive evaluation of blank samples. The estimate s_w is important for computing the LOD $\langle \mathbf{x}, \mathbf{s}_{u_n} \rangle$ and the LOQ (10 \times \mathbf{s}_{u_n}) of the method.
A1.1.2 Annex A2 interprets the meaning of LOD in terms of the rate of false-positive mass detection assertions at specific

$(3 \times s_{\underline{u}_\nu})$ and the LOQ $(10 \times s_{\underline{u}_\nu})$ of the method.

confidence in the method evaluation. Similarly, Annex A2 interprets the meaning of LOQ in terms of the accuracy achieved at confidence in the method evaluation. Similarly, Annex A2 interprets the meaning of LOQ in terms of the accuracy achieved given confidence in the system evaluation. Annex A3 provides a worked example of how the method evalu

A1.1.3 *No Blanks*—Because of its inaccuracy, this measurement scheme is generally not to be used. Aside from the fact that the imprecision variance σ_w^2 may be excessive, its estimation is difficult. A large number of repeat measurements of samples would necessarily be carried out on separate days. Between measurements, the samples would be exposed to environments of expected

application so as to include realistic effects of environment on substrate (14) . From such a set of measurements, σ _{. From such a set} *of measurements, the uncertainty component u ^w may be estimated. Because of the diffıculty in covering all or most environments of intended use, the confidence in the estimate may be low. In addition to imprecision, bias between pre- and post-weighing of substrates may be significant and diffıcult to characterize.*

A1.1.4 *One or More Blanks*—In the case that N_b blanks per sample are used, the imprecision variance is given by:

$$
\sigma_w^2 = \sigma^2 \times (1 + 1/N_b) \tag{A1.1}
$$

The quantity σ^2 is the uncorrectable variance associated with each mass difference measurement requiring two balance readings (before and after exposure). The factor, 1/**N** *^b*, quantifies how the blank weight-change is more accurately known using multiple blanks, which therefore can reduce the overall imprecisionvariance to a degree, at the cost of weighing extra blanks. Furthermore, at $N_b \ge 2$, a protocol could be established for voiding a sample if the blank values differ excessively. Also, at $N_b \ge 3$, one of the blanks could be eliminated if an outlier.

NOTE A1.1—If lab blanks are used instead of field blanks, it should be verified that the imprecisionvariance in weighing is representative of field conditions.

A1.2 EstimationDetermination of the Uncorrectable Weight-Change Imprecision s**Uncertainty** *u*

A1.2.1 The *imprecisionvariance* σ^2 required in Eq A1.1 is estimated through a set of method evaluation experiments. One approach to the estimation of σ^2 is presented here. Equivalent schemes can be devised. Condition and weigh a batch of at least six blank substrates. Place the substrates in clean transport containers or sampling heads, and remove them from the balance room or weighing chamber for a suitable period. If the expected handling and sampling environment is suspected of affecting the sampling medium, then all the samples should be placed in such an environment (without exposure to dust) for a normal sampling period. Repeat with at least four additional batches of blank substrates. Typically, five different days would be required at a minimum.

A1.2.2 Suppose then that a number *F* (for example, 6) of substrates is weighed twice in each of the *B* (for example, 5) batches, giving a set of measured weight differences $\{\Delta m_{fb}\}$, $f = 1, \ldots, F$; $b = 1, \ldots, B$. Then Δm_{fb} is modelled by means of: