

# Workplace atmospheres — Controlling and characterizing uncertainty in weighing collected aerosols

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## National foreword

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## **Workplace atmospheres — Controlling and characterizing uncertainty in weighing collected aerosols**

*Air des lieux de travail — Contrôle et caractérisation de l'incertitude de pesée des aérosols collectés*



Reference number  
ISO 15767:2009(E)

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## Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15767 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This second edition cancels and replaces the first edition (ISO 15767:2003), which has been technically revised.

## **Introduction**

Assessment of airborne aerosol hazards in occupational settings entails sampling onto a collection substrate, followed by analysis of the collected material. The weight of the collection substrate is generally many times (10 to 20, or more) larger than the aerosol sample. Weighing the aerosol sample is therefore actually the differential weighing of the substrate, where the aerosol sample is essentially a disturbance of the substrate. The result is generally an estimated concentration of a 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.

This International Standard deals with a specific type of analysis which finds the most general application in the sampling of aerosols, namely the weighing of sampled material. Gravimetric analysis, though apparently simple, is subject to uncertainty arising from instability in the mass of the sampling medium and other elements which must be weighed. An example is provided by aerosol samplers designed to collect particles so as to agree with the inhalable aerosol sampling convention. For some sampler types, the filter and cassette are weighed together to make estimates. Therefore, uncertainty may result if the cassette, for example, absorbs or loses water between the weighings required for a concentration estimation. This International Standard describes such uncertainty and provides solutions for minimization.





# Workplace atmospheres — Controlling and characterizing uncertainty in weighing collected aerosols

## 1 Scope

This International Standard provides recommendations for controlling the analytical uncertainty associated with aerosol collection medium instability, where collection medium or collection substrate includes any article used to collect particles (e.g. filter or foam material) as well as those supporting elements which must be analysed by weighing.

This International Standard is applicable to results compiled both from the literature and, if necessary and feasible, through laboratory experiment. Expected uncertainty associated with given aerosol capture methods is quantified where possible. Recommendations as to materials to be used are given. Means of minimizing uncertainty arising from instability are provided. Recommendations for the weighing procedure are given. A procedure for estimating weighing uncertainty is described. Finally, recommendations are given for the reporting of measured mass, including an uncertainty component and limits of detection and quantification.

## 2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 2.1

#### **aerosol sample**

aerosol particles collected onto the collection substrate or sampling cassette

### 2.2

#### **collection substrate**

aerosol sampling filter, foam, impaction plate or other deposition plate designed for subsequent analysis, with whatever mounting, e.g. a sampling cassette, if used, analysed (weighed) as a single item together with the collected aerosol sample, if present

**NOTE** As an example of the converse, the 25 mm or 37 mm plastic filter holder often used for “total dust” sampling in either its closed-face or open-face version is not part of the collection substrate in the definition above, since it is not weighed.

### 2.3

#### **substrate holder**

cassette primarily designed to hold a collection substrate (of any kind) and for which only the deposit on the collection substrate is analysed (weighed)

### 2.4

#### **filter holder**

substrate holder designed to hold a filter and for which only the filter deposit is analysed (weighed)

### 2.5

#### **sampling cassette**

collection substrate together with whatever mounting that is used and analysed (weighed) as a single unit

**2.6**  
**equilibration time**

time constant dependent on the type of collection substrate and characterizing an approximately exponentially damped approach of the mass of an aerosol collection medium to a constant value

NOTE 1 The constant can be defined as the mean difference of the mass from equilibrium per mean rate of mass loss or gain, averaging over any time interval.

NOTE 2 There may be important instances in which several independent time constants are required to describe the approach to equilibrium.

NOTE 3 Equilibration times range from seconds to weeks.

**2.7**  
**field blank**

blank collection substrate that undergoes the same handling as the collection substrate plus aerosol sample, including conditioning and loading into the samplers or transport containers, as well as transportation between the lab and sampling site, but without being exposed to sampling

**2.8**  
**lab blank**

blank collection substrate that never leaves the laboratory, but undergoes the same handling as the collection substrate plus aerosol sample, including conditioning and loading into the samplers or transport containers

**2.9**  
**blank collection substrate**

collection medium or substrate taken from the same batch as the sampling medium, but unexposed to sampling

**2.10**  
**limit of detection**  
**LOD**

three times the estimated standard deviation of the mass of the aerosol sample, accounting for the double weighing (exposed vs. unexposed) and for the uncertainty associated with any correction blanks used

NOTE The value of LOD, as defined here, does not take into account sources of variability beyond weighing.

**2.11**  
**false positive rate**

fraction of incorrect assertions of the presence of an aerosol sample on a substrate

NOTE Annex B describes how to estimate, on the basis of the method evaluation, the false positive rate in such assertions.

**2.12**  
**limit of quantification**  
**LOQ**

ten times the estimated standard deviation of the mass of the aerosol sample

NOTE The value of LOQ can be used as a threshold value to assure accurate measurement of a substance. For details, see Annex B.

**2.13**  
**uncertainty component**

$u_w$   
estimated standard deviation of the mass of the aerosol sample

NOTE See Annex A and ISO/IEC Guide 98-3 for details.

### 3 Weight instability — Causes and minimization

#### 3.1 General

Weight instability of collection substrates can be attributed to several causes (see References [1] to [14]). The following subclauses address the more important of these.

#### 3.2 Moisture sorption

**3.2.1** Moisture sorption is the most common cause of weight instability. Water can be directly collected by the filter or foam or other collection substrate material that is weighed. Water sorption by any part of the sampling system which is weighed must be suspected as well. For example, the sampling cassette itself, if weighed, can be the cause of significant uncertainty [1].

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

**Table 1 — Water sorption characteristics of some aerosol sampling media**

Collection substrate or cassette type	Water sorption			
	Very low	Low	High	Very high
Cellulose fibre filter			*	
Glass fibre filter		*		
Quartz fibre filter		*		
Cellulose ester membrane filter			*	
Polytetrafluoroethylene filter	*			
PVC membrane filter		*		
Polycarbonate filter	*			
Silver membrane filter	*			
Polyurethane foam				*
Greased Mylar impaction collection substrate		*		
Greased aluminium foil impaction collection substrate		*		
Carbon-filled resin				*
Aluminium cassette		*		
Stainless steel cassette	*			

NOTE 1 References [2] to [4] provide further details. Also, Reference [5] reports that filters of evidently the same material, but originating from different manufacturers, can have widely differing variabilities.

NOTE 2 There is generally a trade-off between hydrophobicity and conductivity in many materials [9]. Therefore, one must be aware of the possibility of creating sampling problems when reducing hygroscopicity.

NOTE 3 Pre-treatments of collection substrates, such as greasing, can also affect water sorption.

### 3.3 Electrostatic effects

Electrostatic effects are a common source of weighing problems. These effects can usually be minimized by discharging the collection substrate through the use of a plasma ion source or a radioactive source immediately before weighing or during weighing. Using conductive materials may reduce such problems. (See also Reference [7].)

### 3.4 Effects of volatile compounds (other than water)

**3.4.1** Volatile compounds can be present in unused collection media<sup>[3]</sup>, or can be adsorbed onto media during sampling.

**3.4.2** Desorption of volatiles from unused media can 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 Clause 4).

**3.4.3** When volatile materials collected during sampling constitute part of the intended aerosol 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.

**3.4.4** When volatile materials collected during sampling are not part of the intended aerosol sample, it may be difficult to eliminate them if weighing is the only form of analysis. Non-sorptive media should preferably be used.

### 3.5 Handling damage

**3.5.1** If friable collection substrates such as quartz filters are used, procedures are needed to control mechanical damage.

**3.5.2** The air-sampling equipment should be designed so that the collection substrate is not damaged during assembly and disassembly.

**3.5.3** Flat-tipped forceps are recommended for handling filters. Non-oxidizing metal tins may be used to weigh delicate collection substrates without direct handling.

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

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

**3.5.6** Handling shall take place in a clean environment, to avoid contamination.

### 3.6 Buoyancy changes

Corrections for air buoyancy<sup>[8]</sup>, 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 (e.g. if an entire sampling cassette was 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<sup>3</sup>, then correction would be required in order to weigh down to 0,01 mg, if pressure changes in the order of 10 % between weighings are expected (e.g. at different altitudes). If such a correction is necessary, the atmospheric pressure and temperature at the time of weighing should be recorded.

## 4 Correcting for weight instability by use of blanks

### 4.1 General

**4.1.1** Many approaches to controlling weight instability exist (see References [15] to [25]). The use of blanks is the most important practical tool for reducing uncertainty 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 mass change from the mass change of the active collection substrates plus aerosol samples. Of course, if the atmosphere to be sampled contains water (or other volatile) droplets which are weighed, then the use of blanks alone cannot correct. Similarly, water adsorbed by the aerosol sample itself may require special attention, for example if the water adsorbed is to be measured. Blanks shall be matched to the samplers used, e.g. if the sampler contains a filter within a sampling cassette which is weighed, the blank shall be the same type of filter within the same type of cassette.

**4.1.2** An alternative procedure employs matched-weight filters consisting of two pre-selected nearly equal-weight filters, one placed in front of the other, with the downstream filter employed as blank. Requiring only two rather than four mass measurements, 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 done prior to sampling.

**4.1.3** In another approach to eliminate the burden of blank handling (at the expense of high LOD), the equilibrium filter mass in terms of humidity is initially modelled. Mass estimates are subsequently corrected knowing the humidity at the application weighing [26].

### 4.2 Minimum number of blanks

Generally, at least one blank is recommended for every 10 samplers. Measurement schemes in current use require between one and four blanks per batch. See Annex A for advantages of multiple blanks.

### 4.3 Weighing times and sequence

Blanks shall be interspersed with samples, before and after use, so as to detect systematic variations in weighing or substrate mass (e.g. due to sorption or evaporation of a contaminant during weighing).

### 4.4 Conditioning times

Conditioning times for reaching equilibrium with the weighing environment may vary from a few hours to several weeks or more, depending on the specific sampling media. Typically, for workplace sampling using filters, overnight conditioning is satisfactory. For sampling media with longer conditioning times, correction through the use of blank collection substrates is particularly important.

### 4.5 Storage stability

Unused collection substrates shall be stored, prior to weighing and conditioning, in a clean laboratory, whose environmental conditions do not differ greatly from the environment of the balance. Pre-weighed collection 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 Shelf-life depends on the collection substrate material, storage conditions, cassette material and required LOQ or LOD.

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

## 5 Transport of collection substrates with collected aerosol samples to laboratory

### 5.1 General

The transportation of substrates with collected aerosol samples shall form part of a written procedure. The transport procedure shall be validated to ensure that significant losses do not occur. Follow the test method given in Annex D.

The main problems occurring during handling and transport of sampling media are described below.

- With collection substrates designed to be separated from the substrate holder, dust can migrate from the collection substrate to the transport container, and hence be lost.
- On the other hand, contamination of the sampling cassette can be a significant source of uncertainty, as this type of cassette is itself weighed.
- If a cover lid is not supplied, dust can be lost from the cassette to the transport container.
- Dust can migrate from the substrate holder to the collection substrate.

NOTE Transportation losses are discussed in References [15] and [16].

### 5.2 Recommended packaging

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

**5.2.2** Sampling cassettes (i.e. with mounted filters) should preferably have cover lids during transport. If the aerosol sample consists of all the dust deposited inside the sampling cassette (with filter), then the dust which migrates during transport from the cassette to the cover lid shall also be weighed.

**5.2.3** The sealed collection 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 aerosol samples during transport.

**5.2.4** The aerosol samples shall be protected from excessive heating or cooling during transport.

NOTE 1 Special procedures are generally used for the transport of unstable particles or biological materials.

NOTE 2 If there is a possibility for dust to be lost from the collection substrate, the losses can be recovered by transporting the collection substrate within a container that can itself be weighed.

## 6 Weighing equipment and procedure

### 6.1 The balance

The balance should be matched to the task. The choice of balance depends on the desired limits of quantification for the application (see Clause 7) and on the maximum tare masses of the collection substrates to be weighed.

Workplace-air sampling typically requires a balance capable of weighing to a resolution of 1 µg or 10 µg. The balance shall be regularly calibrated using reference masses traceable to International Standards.

NOTE The performance of different balances was compared and reported in Reference [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 balance weighing to 1 µg (six figures) was compared to a balance weighing to 10 µg (five figures). It was concluded that using a 1 µg balance approximately halves the standard deviation of repeat weighing compared with a 10 µg balance. 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 Reference [11]).

## 6.2 Recommended environmental controls

**6.2.1** Equilibration and weighing shall be carried out under the same conditions, i.e. in the same room or chamber. Environmental control can be achieved in different ways:

- by means of a balance room containing the balance and the weighing personnel;
- by means of an environmentally-controlled chamber containing the balance, sited within a clean laboratory.

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

**6.2.2** For sensitive (i.e. hygroscopic) aerosol samples or substrates, temperature and humidity control in the weighing chamber or balance room are important. In these cases, temperature should be maintained constant within  $\pm 2$  °C of the set point, and humidity should be constant to within  $\pm 5$  % relative humidity (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 [e.g.  $(20 \pm 2)$  °C and  $(50 \pm 5)$  % RH]. Very dry atmospheres (e.g.  $< 20$  % RH) are to be avoided, as electrostatic build-up on whatever is to be weighed 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].

NOTE Air conditioning in a weighing chamber is not necessary, e.g. filtered laboratory air can be fed into a positive-pressure chamber.

**6.2.3** The particulate content of the balance room or chamber air should be minimized by filtration [e.g. by high efficiency particulate air (HEPA) filtration].

**6.2.4** Fresh air should be supplied, in accordance 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.

## 6.3 Other equipment requirements

The balance should be located in an area which is free from excessive vibration [e.g. due to lifts (elevators) or rotating machinery]. A massive weighing table (e.g. 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 sources of strong electromagnetic radiation.

## 6.4 Procedure

**6.4.1** The weighing procedure shall be documented.

**6.4.2** Equilibration to the temperature and humidity of the balance room or chamber shall take place for a period appropriate to the substrates and aerosol samples. The substrates and collected aerosol samples shall be kept in clean containers but open to the atmosphere so that equilibration can occur.

NOTE Desiccation prior to equilibration is sometimes used to remove excess water from substrates or aerosol samples taken in a humid environment. (See also References [26] to [29].)

**6.4.3** Elimination (if possible) of static electricity from the collection substrates and aerosol samples is extremely important, and should be done immediately prior to placement of collection substrates on the balance pan. Alternatively, a static eliminator can be placed inside the balance chamber.

**6.4.4** The balance reading shall not be recorded until after it has stabilized.

**6.4.5** Re-zero the balance as determined necessary.

NOTE A defective collection substrate can sometimes be identified by an abnormal initial mass.

## 7 Recommendations for the reporting of measured mass relative to LOD and LOQ

Details on reporting weights depend on the specific application. The following is an example of a minimal reporting policy.

**7.1** If the measured mass is above the limit of quantification (LOQ, Annexes A to C), then it should be reported.

**7.2** If the result falls between the limit of detection (LOD, Annexes A to C) and LOQ, then it should be reported that the measured mass is between the values of LOD and LOQ, and the measured mass is reported as well.

**7.3** If the measured mass falls below the value of the LOD, it should be reported that the estimate is below the value of the LOD.

**NOTE 1** If the value of the LOD is exceeded, then the false-positive rate in asserting detection is  $< 1\%$ , if the method is evaluated with as many degrees of freedom (25) as in the examples in Annexes A to C.

**NOTE 2** In some applications, a series of measured masses, each below the value of the LOD, may be of help, e.g. in asserting the presence of mass over the entire series, even if the individual measurements are too small to assert detection with confidence. Applications such as these employ actual measured values (even if negative).

**7.4** LOD and LOQ values shall be determined and shall be given in the report. Annexes A to C contain suitable procedures for these determinations.

**7.5** The uncertainty component  $u_w$  shall be given in the report (see Clause 8).

## 8 Estimation of the uncertainty of the analytical procedure of weighing aerosol collection substrates

### 8.1 Introduction

**8.1.1** The uncertainty  $u_w$  of weighing shall be taken as the within-laboratory estimated standard deviation  $s_w$  in a mass of the aerosol sample, including any blank correction.

**8.1.2** As indicated in Annex F, the uncertainty associated with the calibration of the balance itself is generally negligible.

**8.1.3** The quantity  $u_w$  is an uncertainty component to be combined with other uncertainty components to give combined ( $u_c$ ) and expanded uncertainties ( $U$ , traditionally taken as  $2 \times u_c$ ) of the whole measurement (see ISO/IEC Guide 98-3 and EN 482). Variability, aside from the weighing, can arise from several sources:

- sampling pump-flowrate bias;
- flow fluctuation (in the case of sampling with internal aerodynamic separation);
- variation in sampler physical dimensions;
- electrostatic effects on sampling; or
- bias relative to the appropriate sampling convention.

**8.1.4** At low loadings, if the gravimetric variability dominates the uncertainty from other sources, then  $u_c \rightarrow u_w$ .



## **8.2 Within-laboratory estimated standard deviation $s_w$ obtained over an extended period**

**8.2.1** This shall be determined according to the method described in Annex A, and especially Equations (A.3) to (A.5). The test batches shall be spread out evenly throughout the year during which the laboratory usually weighs collection substrates.

NOTE If almost no weighing occurs during the summer months, this period does not need to be investigated.

**8.2.2** All collection substrates shall be treated exactly as collection substrates intended for sampling are treated.

**8.2.3** The duration between the pre-weighing and post-weighing for each batch of the test shall be identical to nominal (maximum) shelf-life that the laboratory has determined for this collection substrate.

**8.2.4** Between the pre-weighing and the post-weighing, the collection substrates shall be placed in the ordinary transport package used by the laboratory, as if they were to be transported away from the laboratory. The transport package, with the test collection substrates, shall be placed outside the weighing room/box for the time period between the weighings.

**8.2.5** If the collection substrates are not mounted into samplers by the weighing laboratory after pre-weighing, each collection substrate shall be mounted once in a sampler for a period identical to a recommended sampling time, i.e. usually 4 h to 8 h, during this part of the test.

## **9 Measures to assure the validity of previously determined measurement uncertainty**

### **9.1 Continued determination of within-laboratory reproducibility**

Each year, at least one of the batches of the experiment described in A.3 should be performed anew. The batch in the original data, whose pre-weighing and post-weighing months correspond best to those of the new batch, shall be deleted from the data and replaced with the data from the new batch.

### **9.2 Participation in laboratory performance proficiency testing**

If, there exists a group of laboratories considered acceptable for weighing specific aerosol collection substrates, then generally the group will establish a laboratory performance proficiency testing scheme with mandatory participation required of each laboratory.

### **9.3 Laboratory self-check on weighing uncertainty**

A laboratory wishing to check weighing uncertainty can, for example, follow the method described in Annex E.

## Annex A (normative)

### Uncertainty component in weighing collected aerosol

#### A.1 Symbols and abbreviated terms

The following symbols and abbreviated terms are used in both this annex and Annex B.

$A$	coverage defining an interval about true values containing measured values at a specified rate
$b$	batch index (1, ..., $B$ )
$B$	number of collection substrate batches in method evaluation
$f$	collection substrate index (1, ..., $F$ )
$F$	number of collection substrates (e.g. filters) in each batch tested in method evaluation
LOD	value of the limit of detection: $3 \times s_w$ , expressed in micrograms
LOQ	value of the limit of quantification: $10 \times s_w$ , expressed in micrograms
$N_b$	number of blanks per collection substrate set
$s$	estimate of the standard deviation $\sigma$
$s_w$	estimate of the standard deviation $\sigma_w$
$u_c$	combined uncertainty (including uncertainty sources beyond weighing) (see A.2.1)
$u_w$	uncertainty component in weighing collected aerosols: taken as the standard deviation estimate $s_w$ , expressed in micrograms
$U$	expanded uncertainty (including uncertainty sources beyond weighing) (see A.2.1)
$\alpha$	false-positive detection rate (see Annex B)
$\beta$	mean collection substrate mass change during evaluation experiment
$\Delta m_{fb}$	collection substrate mass change, expressed in micrograms
$\varepsilon_{fb}$	collection substrate mass change residual random variable with variance $\sigma^2$ , expressed in micrograms
$\varepsilon_b$	collection substrate mass change random variable representing inter-batch variability, expressed in micrograms
$\gamma$	complement of the method evaluation confidence
$\nu$	number of degrees of freedom in method evaluation
$\sigma$	uncorrectable (e.g. via blank correction) standard deviation in (single) mass-change measurement, expressed in micrograms
$\sigma_w$	standard deviation in collected mass determination, expressed in micrograms
$\Phi$	cumulative normal function
$\chi^2$	chi-squared random variable
$\chi^2_{\gamma, \nu}$	chi-squared quantile

## A.2 The uncertainty component $u_w$ in mass estimates

### A.2.1 General

The variance  $\sigma_w^2$  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 uncertainty  $u_w$  is taken as equal to the estimate  $s_w$  of the standard deviation  $\sigma_w$ . The estimate  $s_w$  is determined, as outlined in this annex, through an extensive evaluation of blanks. In addition to the uncertainty component  $u_w$  (equal to  $s_w$ ), the estimate  $s_w$  is important for computing the limit of detection LOD ( $3 \times s_w$ ) and the limit of quantification LOQ ( $10 \times s_w$ ).

Annex B interprets the meaning of LOD in terms of the rate of false-positive mass detection assertions at a specific confidence in the method evaluation. Similarly, Annex B interprets the meaning of LOQ in terms of the coverage, as known at a given confidence in the system evaluation. Annex C provides a worked example of how the method evaluation is analysed.

### A.2.2 No blanks

Because of excessive uncertainty, a measurement scheme with no blanks is generally not to be used (however, see 4.1.3 and Reference [26]). Aside from the fact that the variance  $\sigma_w^2$  may be excessive, its estimation is difficult. A large number of replicate measurements would need to be carried out on separate days. Between measurements, the blanks would be exposed to environments of expected application, so as to include realistic effects of environment on collection substrate. From such a set of measurements, the uncertainty component  $u_w$  can be estimated. Because of the difficulty in covering all or most environments of intended use, the confidence in the estimate can be low. In addition to uncertainty in individual weighings, bias between pre- and post-weighing of substrates can be significant and difficult to characterize.

### A.2.3 One or more blanks

In the case where  $N_b$  blanks per active sampling are used, the variance is given by

$$\sigma_w^2 = \sigma^2 [1 + (1/N_b)] \quad (\text{A.1})$$

The quantity  $\sigma^2$  is the uncorrectable variance associated with each mass difference measurement requiring two balance readings (before and after exposure). The first term of Equation (A.1) reflects the fact that the aerosol sample is present for a single balance reading only. The factor  $1/N_b$  quantifies how the blank mass-change is more accurately known using multiple blanks, which therefore can reduce the overall variance to a degree, at the cost of weighing extra blanks. Furthermore, at  $N_b = 2$ , a protocol could be established for voiding an aerosol sample if the blank values differ excessively. Also, at  $N_b = 3$ , one of the blanks could be eliminated, if it is an outlier.

NOTE 1 A "balance reading" may actually consist of the mean of several readings for minimizing uncertainty in the operation of the balance.

NOTE 2 Laboratory blanks are sometimes used instead of field blanks, if it can be verified that the weight of the lab blanks is constant over time and that that the weighing variance is representative of field conditions.

## A.3 Determination of the uncorrectable mass-change standard deviation $s$

The variance  $\sigma^2$  required in Equation (A.1) is estimated through a set of method evaluation experiments. One approach to the estimation of  $\sigma$  is presented here. Equivalent schemes can be devised. Condition and weigh a batch of at least six, but preferably of the order of ten, blank collection substrates. Place the collection substrates in clean transport containers or sampling heads and remove them from the balance room or weighing chamber for a suitable period as prescribed in 8.2. If the expected handling and sampling environment is suspected of affecting the sampling medium, then all the collection substrates 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 collection substrates. Typically, five different test batches spread over the year (see 8.2) would be required at a minimum.

Suppose then that a number  $F$  (e.g. 6) of collection substrates are weighed twice in each of the  $B$  (e.g. 5) batches, giving a set of measured mass differences  $\{\Delta m_{fb}\}$ ,  $f = 1, \dots, F$ ;  $b = 1, \dots, B$ . Then,  $\Delta m_{fb}$  is modelled via:

$$\Delta m_{fb} = \beta + \varepsilon_b + \varepsilon_{fb} \quad (\text{A.2})$$

The various quantities in Equation (A.2) are interpreted as follows. The constant  $\beta$  is a mean mass gain over all the blanks.  $\varepsilon_b$  is a random variable with zero mean and assumed normal distribution, expressing the inter-batch variability. Finally,  $\varepsilon_{fb}$ , the term of real interest, is the only remaining quantity upon forming the difference of one collection substrate's mass measurement, relative to a difference in blank collection substrate masses within the same batch.  $\varepsilon_{fb}$  is approximated as normally distributed about zero, with standard deviation  $\sigma$ . Note that  $\sigma$  involves uncorrelated medium instability as well as balance uncertainty and therefore will generally exceed the value appropriate to a standard mass difference.

NOTE 1 Interaction between filter, foam or cassette dimensional variations and environmental change is assumed to be negligible.

NOTE 2 The uncertainty of weighing can vary from season to season.

Each batch  $b$  provides its own estimate  $s_b$  of  $\sigma$  via:

$$s_b^2 = (F - 1)^{-1} \sum (\Delta m_{fb} - \Delta m_{.b})^2 \quad (\text{A.3})$$

where the summation is over  $f$  denoting the blank in batch  $b$ , and where  $\Delta m_{.b}$  is the mean over the collection substrates in the batch  $b$ . Pooling the batch estimates then gives the value  $s^2$  as a summation over the batches:

$$s^2 = B^{-1} \sum s_b^2 \quad \text{with } \nu = (F - 1) \times B \text{ degrees of freedom} \quad (\text{A.4})$$

Annex C provides an example of estimating  $\sigma$ .

NOTE A nearly identical evaluation experiment was reported in Reference [28].

#### **A.4 The blank-corrected standard deviation $s_w$**

The estimate of the standard deviation  $s_w$  is therefore [Equation (A.1)] determined from:

$$s_w^2 = s^2 \cdot [1 + (1 / N_b)] \quad (\text{A.5})$$

#### **A.5 The limit of detection**

Following References [30] to [38], the limit of detection (LOD) is reported as the value

$$\text{LOD} = 3 \times s_w \quad (\text{A.6})$$

The presence of a substance can be asserted if its measured value exceeds the value of LOD. The uncertainty associated with such assertions is described in Annex B.

#### **A.6 The limit of quantification**

Similarly, if the variance  $\sigma_w^2$  is constant at small loadings, the limit of quantification (LOQ) is generally defined by the value

$$\text{LOQ} \equiv 10 \times s_w \quad (\text{A.7})$$

NOTE The only known situation with aerosol weighing for which the uncertainty is not approximately constant at small loadings involves instability of the collected material itself. Characterization of this uncertainty requires statistical modelling of this instability and is not covered in this International Standard. See, however, the recommendation in 3.4.3.

## A.7 The uncertainty component

The uncertainty component  $u_w$  is taken as equal to  $s_w$

$$u_w = s_w \quad (\text{A.8})$$

## Annex B (informative)

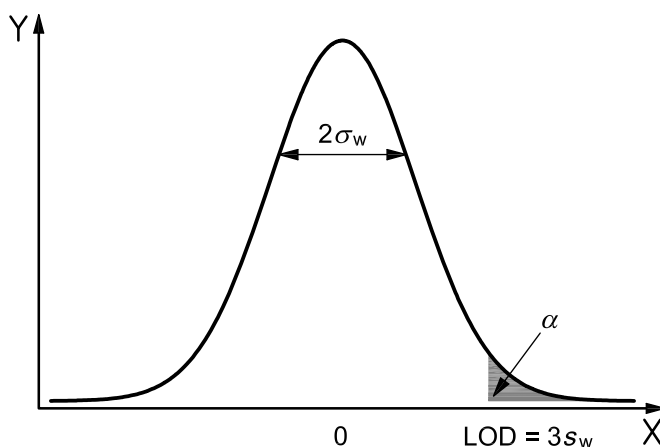
### Interpretation of LOD and LOQ

#### B.1 False-positive rate upon using the LOD for detection assertion

Suppose the variance  $\sigma_w^2$ , estimated by (known)  $s_w^2$ , is independent of the sampled mass at small loadings. The limit of detection (LOD) is defined following a weighing evaluation by

$$\text{LOD} \equiv 3 \cdot s_w \tag{B.1}$$

Denote the false-positive rate in asserting the presence of a substance by  $\alpha$  (e.g. 2 %), as depicted in Figure B.1.



**Key**

- X mass estimate
- Y probability density

NOTE The shaded area represents  $\alpha$ , the false-positive detection rate.

**Figure B.1 — (Known) LOD plotted with (unknown) distribution of mass estimates**

If  $\Phi$  is the cumulative normal function (e.g.  $\Phi[1,960] = 97,5 \%$ ), then  $\alpha$  is given by

$$\alpha = 1 - \Phi(\text{LOD}/\sigma_w) \tag{B.2}$$

The standard deviation  $\sigma_w$ , though not known exactly, is limited by

$$\sigma_w < s_w / \sqrt{\nu} / \chi_{\gamma, \nu}^2 \tag{B.3}$$

at evaluation confidence level  $(1 - \gamma)$  (e.g. 95 %). The quantile  $\chi_{\gamma, \nu}^2$  may be found in standard statistics tabulations and is defined implicitly as the value for which

$$\text{prob}(\chi^2 > \chi_{\gamma, \nu}^2) = 1 - \gamma \tag{B.4}$$

Therefore, the false-positive rate  $\alpha$  is limited by

$$\alpha < 1 - \Phi(3 / \sqrt{\nu / \chi_{\gamma, \nu}^2}) \text{ at evaluation confidence } (1 - \gamma) \quad (\text{B.5})$$

Let  $\gamma = 5\%$  (meaning 95 % confidence in the system evaluation), then at  $\nu = 25$  degrees of freedom, as in the suggested weighing evaluation experiment, Inequality (B.5) implies that the false-positive rate is less than 1 %. Similarly, if the concentration is large enough to sample LOD on average, then the false-negative (Type 2) rate is equal to 50 % due to the symmetry of the distribution of the residuals.

## B.2 Interpretation of LOQ

LOQ may be interpreted similarly. For 95 % of the estimates  $m_{\text{est}}$  of true mass  $m$ ,

$$m - 1,960 \sigma_w < m_{\text{est}} < m + 1,960 \sigma_w \quad (\text{B.6})$$

Thus, the coverage  $A$  at the 95 % rate may be defined relative to the true value  $m$  as:

$$A \equiv 1,960 \sigma_w / m \quad (\text{B.7})$$

At  $m$  equal to LOQ,

$$m = 10 s_w \quad (\text{B.8})$$

Thus, Inequality (B.3) implies that at evaluation confidence  $(1 - \gamma)$ , the 95 %-rate coverage  $A$  is limited by

$$A < \frac{1}{10} 1,960 \sqrt{\nu / \chi_{\gamma, \nu}^2} \quad (\text{B.9})$$

As an example, with 95 % confidence in the evaluation at  $\nu$  equal to 25 degrees of freedom, then over 95 % of the samples with true masses equal to LOQ will fall in an interval defined as  $\pm 25,6\%$  about true values.

NOTE 1 The NIOSH accuracy criterion [35] to [38] requires  $A < 25\%$  at the 95 %-confidence level for methods without uncorrectable bias or systematic error.

NOTE 2 Similarly,  $A < 25,6\%$  implies that the expanded uncertainty (see EN 482)  $U < 30\%$ , in the absence of bias and of significant uncertainty other than gravimetric at masses exceeding the value LOQ. Here the expanded uncertainty is taken generally as  $2 \times u_c$ , where  $u_c$  is the combined uncertainty given by pooling the uncertainty components.

NOTE 3 Therefore, occupational or environmental exposure limits lower than the value corresponding to a method's LOQ may indicate that improvement in the method is required.

NOTE 4 The interpretation of LOD and LOQ given here closely relates to the concept of the tolerance interval. Detection assertion always refers to the confidence in the method evaluation.

## Annex C (informative)

### Method evaluation example

Table C.1 gives an example of typical results for the experiment to determine system measurement uncertainty. Columns correspond to the collection substrate number of a given batch, and rows to separate batches. For illustration purposes, masses (in micrograms) were simulated using mean mass gain (over all the batches) equal to 5 µg, batch variability equal to 5 µg, and uncorrelated weighing uncertainty equal to 5 µg. This means that the mass-difference standard deviation, the quantity to be estimated from the data, is  $\sigma = \sqrt{2} \times 5 \mu\text{g} = 7,07 \mu\text{g}$ .

**Table C.1 — Collection substrate mass difference measurements**

Batch	Collection substrate µg						Estimated variance ( $s_b^2$ )
	1	2	3	4	5	6	
1	21	21	15	18	14	18	8,6
2	-4	-11	2	2	-6	2	30
3	9	22	-12	0	12	12	140
4	-2	6	20	6	8	6	51
5	-11	11	4	5	0	1	54
Mean variance estimate ( $s^2$ )							56

Taking the square root of the mean variance estimate gives the estimated mass-difference standard deviation:

$$u = \sqrt{56} \mu\text{g} = 7,5 \mu\text{g} \text{ with 25 degrees of freedom,}$$

which compares well with the true value 7,07 µg.

Now suppose that in actual application, three blanks are to be used. Then, Equation (A.5) indicates that the mass-determination uncertainty component  $u_w$  is given by

$$\begin{aligned} u_w &= s_w \\ &= u\sqrt{1+1/3} \\ &= 8,6 \mu\text{g} \end{aligned}$$

The limits of detection and quantification, the values of LOD and LOQ [Equations (A.6) and (A.7)], would be

$$\text{LOD} = 26 \mu\text{g}$$

$$\text{LOQ} = 86 \mu\text{g}$$



## Annex D (normative)

### Test of transportation integrity

#### D.1 General

The purpose of this test is to help minimize mass measurement uncertainty due to either material loss or collection substrate contamination while transporting aerosol samples from field to laboratory. The test proposed here is similar to the method of EN 13205:2001, Annex E. As it is the responsibility of the weighing laboratory to design how the transportation of samples and blanks from and back to the lab should be done, the transportation procedure shall be conveyed by the weighing laboratory to the customers.

#### D.2 Procedure

**D.2.1** Weigh at least 30 collection substrates plus blanks, as required by the written procedures of the laboratory.

**D.2.2** Deposit dust onto at least 3 groups of 10 collection substrates, each with dust masses equal to

- a) the limit of quantification of the weighing system,
- b) the maximum dust deposit for which the laboratory intends the method to be used, and
- c) a mid-range value between the previous extreme values of dust deposits.

The dust could preferably be deposited onto the substrates at workplaces, so that a 'real' test dust is used in the test.

**D.2.3** Instead of monodisperse particles, use dust with size distributions expected in application after passing through the sampler of interest. Use a non-volatile, non-reactive, non-sticky and non-hygroscopic dust. The dust composition may be as important as size. Sampled lead or beryllium, for example, may be only loosely held by the substrate, especially if heavily loaded, whereas diesel soot is often tightly bound to a substrate comprised of a quartz fibre filter. It is not necessary to deposit the dust in a laboratory. The dust may be deposited onto the collection substrates at the workplaces. However, extra care shall then be taken to safeguard those aerosol sample particles which are most easily lost in transport to the laboratory.

**D.2.4** Weigh the collection substrates according to the written procedures of the laboratory.

**D.2.5** Pack the collection substrates according to the written procedures of the laboratory.

**D.2.6** Dispatch the transport containers by mail, consigned delivery system or by the end-user's own vehicle (whatever is most common/appropriate) to a trusted addressee/consignee at a remote location. Ask the addressee/consignee to return the package containing the test collection substrates, with the same transport system, without opening it.

**D.2.7** Weigh the active collection substrates plus blanks according to the written procedures of the laboratory.

**D.2.8** Determine the relative transport losses for each of the three levels of dust deposit.

#### D.3 Requirements

**D.3.1** The relative dust loss due to transport shall be less than 5 %.

**D.3.2** The collection substrate mass range for which this requirement is met shall be documented.

## Annex E (informative)

### Check on weighing uncertainty

#### E.1 General

Weighing uncertainty may be tested by loading aerosol collection substrates with a known amount of a salt dissolved in a liquid. The following procedure has been found to be useful for measuring inter-laboratory variation. For details, see Reference [40]. Sodium tetraborate pentahydrate,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ , dissolved in ultra-pure water, may be used for this purpose.

#### E.2 Procedure

**E.2.1** Dissolve up to 100 mg of sodium tetraborate pentahydrate per cubic centimetre of ultra-pure water. Make sure all solids have dissolved.

**E.2.2** Determine the amount of liquid needed on each collection substrate in order to obtain deposits in the required range.

**E.2.3** Mount the collection substrates in such a way that the liquid may be deposited onto a collection substrate without causing breakthrough or liquid contact with a surface beneath the collection substrate.

**E.2.4** Verify that the required amount of liquid may be deposited without loss of liquid.

**E.2.5** Pre-weigh the number of collection substrates needed, including blanks.

**E.2.6** For each required amount of liquid, deposit the liquid onto at least seven collection substrates. Deposit the same amount of liquid (without any dissolved salt) onto the blank collection substrates. Leave the deposits to dry.

**E.2.7** Post-weigh the number of collection substrates used, including blanks. Determine the weight increase of the collection substrates.

#### E.3 Analysis

For each deposited amount of salt, determine the relative difference between collection substrate loading (according to the weighing) and nominal loading, and the ratio of the standard deviation of collection substrate loading (according to the weighing) to the nominal loading. If any of these exceeds 0,025 (2,5 %), there is a problem either with the weighing of the laboratory or with its execution of this test procedure.

## Annex F (informative)

### Balance uncertainty

#### F.1 General

The uncertainty components resulting from the balance calibration as effected by the manufacturer and from the inexactness of the calibration masses themselves are generally negligible in comparison to variations in collection substrate mass during the process of weighing. This can be seen as follows. Suppose the calibration mass uncertainty specified by the balance manufacturer is as given in Table F.1. Suppose further that the uncertainty in weighing the calibration masses is approximately equal to their uncertainty as reported by the manufacturer.

**Table F.1 — Calibration masses and typical manufacturer-provided uncertainty (standard deviation)**

Calibration mass μg	Calibration mass uncertainty ( $u_m$ ) μg
10 000	2
100 000	3
500 000	4
1 000 000	5
2 000 000	6
5 000 000	8

#### F.2 Analysis

**F.2.1** When aerosol samples are to be weighed, the tare weight of the collection substrate is always many times larger than the weight of the aerosol sample. Weighing is therefore always differential weighing, where the weight of the aerosol sample is the difference between the post-weight and the pre-weight, corrected for the weight change of the aerosol collection substrate. Thus, the uncertainty  $u_{\text{cal}}$  relative to the aerosol sample mass due to calibration inexactness is related to the uncertainty in the slope  $k$  of the calibration line of the balance. Specifically,

$$u_{\text{cal}} = \sqrt{2 \times V(k)} \quad (\text{F.1})$$

where the factor 2 accounts for the uncertainty in the calibration masses and in their measurement at calibration, according to the simplifying assumption in F.1, and  $V(k)$  is the variance of  $k$ .

**NOTE** Calibration uncertainty is strictly speaking a bias uncertainty, since uncertainty in the calibration manifests itself as a systematic error in all balance uses between calibrations. Bias uncertainty is pooled in with other uncertainty components associated with variation at each measurement. The resulting combined uncertainty can be interpreted in a prediction sense, as in Reference [39], or simply as an abstract performance index.

**F.2.2** The simplest way to compute  $V(k)$  is through simulation by generating many sets of mass measurements, using the values  $u_m$  in Table F.1. For each set of six masses, weighted linear regression, with weight equal to  $1/u_m^2$  where  $u_m$  is found in Table F.1, provides a value of  $k$ . A set of about 1 000 values of  $k$  allows the variance to be easily and accurately computed.

### F.3 Result

The procedure described in F.2 gives the following result:

$$u_{\text{cal}} = \sqrt{2 \times V(k)} = 2 \times 10^{-4} \% \quad (\text{F.2})$$

Therefore, this uncertainty is negligible, even when multiplied by 1,960 providing coverage at the 95 % level.

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