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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

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Foreword

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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 [3], 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 [8], 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^3, 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]).