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Workplace atmospheres — Controlling and characterizing errors in weighing collected aerosols

Atmosphères des lieux de travail — Contrôle et caractérisation des erreurs de pesée des aérosols collectés

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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

Introduction

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 hazardous material in the air. The accuracy of 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 errors 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, 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 International Standard describes such potential errors and provides solutions for their minimization.

Workplace atmospheres — Controlling and characterizing errors 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 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 errors associated with given aerosol capture methods are quantified where possible. Recommendations as to materials to be used are given. Means of controlling or correcting errors arising from instability are provided. Recommendations for the weighing procedure are given. A procedure for estimating weighing errors is described. Finally, recommendations are given for the reporting of measured masses.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

EN 482, *Workplace atmospheres — General requirements for performance of procedures for the measurement of chemical agents*

EN 13205:2001, *Workplace atmospheres — Assessment of performance of instruments for measurement of airborne particle concentrations*

3 Terms and definitions

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

3.1
substrate
aerosol sampling filter, foam, etc., together with whatever mounting is weighed as a single item

NOTE As an example of the converse, the 25-mm 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.

3.2
equilibration time
time constant 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 mass loss or gain rate as measured over a finite time interval.

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

NOTE 3 Equilibration time is expressed in seconds.

3.3
field blank
blank substrate that undergoes the same handling as the sample substrate, generally including conditioning and, often, loading into the samplers or transport containers, as well as transportation between lab and sampling site, but without being exposed to sampling

3.4
lab blank
blank substrate that undergoes the same handling as the sample substrate in the laboratory, including conditioning and loading into the samplers or transport containers if this is done in the laboratory

3.5
blank substrate
collection medium or substrate taken from the same batch as the sampling medium, but unexposed to sampling

3.6
limit of detection
LOD
three times the estimated standard deviation of the mass of the sample, accounting for the double weighing (exposed vs. unexposed) and for the uncertainty associated with any correction blanks used

NOTE The value LOD can be used as a threshhold value to assert the presence of a substance with confidence in the method. Annex B describes how to estimate, on the basis of the method evaluation, the false positive rate in such assertions.

3.7
limit of quantitation
LOQ
ten times the estimated standard deviation of the mass of the sample

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

4 Weight instability — Causes and minimization

4.1 General

Weight instability of sampling substrates can be attributed to several causes [1] to [11]. The following subclauses address the more important of these.

4.2 Moisture sorption

4.2.1 Moisture sorption is the most common cause of weight instability. Water can be directly collected by the filter or foam or other 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 error [1].

4.2.2 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 sampling substrates with different water adsorption features.

Table 1 — Water sorption characteristics of some aerosol sampling media

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 substrate		*	*	
Greased aluminium foil impaction 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 substrates, such as greasing, can also affect water sorption.

4.3 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. See also reference [7].

4.4 Effects of volatile compounds (other than water)

4.4.1 Volatile compounds can be present in unused collection media [3], or can be adsorbed onto media during sampling.

4.4.2 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 5).

4.4.3 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.

4.4.4 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. Non-sorptive media should preferably be used.

4.5 Handling damage

4.5.1 If friable substrates are used, procedures are needed to avoid mechanical damage.

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

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

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

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

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

4.6 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 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 in order to weigh down to 0,1 mg if pressure changes of 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.

5 Correcting for weight instability by use of blanks

5.1 General

Many approaches to controlling weight instability exist [12] to [20]. 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 mass change from the mass change 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. Blanks shall be matched to samples, i.e. if the sample consists of a filter within a cassette which is weighed, the blank shall be the same type of filter within the same type of cassette.

NOTE The effect of filter variations due to their manufacture is generally eliminated through the use of blanks.

5.2 Minimum number of blanks

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

5.3 Weighing times and sequence

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

5.4 Conditioning times

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

5.5 Storage stability

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

Archived 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.

6 Transport of samples to laboratory

6.1 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 the test method given in Annex D.

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

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

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

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

— Dust can migrate from sampling cassette to substrate.

NOTE Transportation losses are discussed in references [12] and [13].

6.2 Recommended packaging

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

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

6.2.3 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.

6.2.4 The 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.