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Workplace air — Gases and vapours — Requirements for evaluation of measuring procedures using diffusive samplers

*Air des lieux de travail — Gazes et vapeurs — Exigences pour
l'évaluation des procédures pour le mesurage à l'aide de dispositifs de
prélèvement par diffusion*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document provides a framework for assessing the performance of procedures for measuring gases and vapours against the general requirements for the performance of procedures for measuring chemical agents in workplace atmospheres as specified in ISO 20581. These performance criteria include maximum values of expanded uncertainty achievable under prescribed laboratory conditions for the methods to be used.

This document enables manufacturers and users of diffusive samplers and developers and users of procedures for measuring gases and vapours to adopt a consistent approach to method validation.

This document is based on EN 838:2010, published by the European Committee for Standardization (CEN) and is also complementary to ISO 16107.

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Workplace air — Gases and vapours — Requirements for evaluation of measuring procedures using diffusive samplers

1 Scope

This document specifies performance requirements and test methods under prescribed laboratory conditions for the evaluation of diffusive samplers (see Reference [1]) and of procedures using these samplers for the determination of gases and vapours in workplace atmospheres (see Reference [2]).

This document is applicable to diffusive samplers and measuring procedures using these samplers, such as ISO 16200-2 and ISO 16017-2, in which sampling and analysis are carried out in separate stages.

This document is not applicable to

- diffusive samplers which are used for the direct determination of concentrations, and
- diffusive samplers which rely on sorption into a liquid.

This document addresses requirements for method developers and/or manufacturers.

NOTE For the purposes of this document a manufacturer can be any commercial or non-commercial entity.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 20581, *Workplace air — General requirements for the performance of procedures for the measurement of chemical agents*

ISO 22065, *Workplace air — Gases and vapours — Requirements for evaluation of measuring procedures using pumped samplers*

ISO 18158, *Workplace air — Terminology*

ISO 8655-2, *Piston-operated volumetric apparatus — Part 2: Piston pipettes*

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric methods for the determination of measurement error*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 and ISO 20581 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Symbols and abbreviated terms

NOTE See 8.4 and Annex C for symbols used in conjunction with uncertainty of measurement only.

A	cross-sectional area of sorption surface, in square centimetres (cm ²)
CRM	certified reference material
D_a	diffusion coefficient of an analyte, in square centimetres per minute (cm ² · min ⁻¹)
D_{a1}	diffusion coefficient of analyte 1, in square centimetres per minute (cm ² · min ⁻¹)
D_{a2}	diffusion coefficient of analyte 2, in square centimetres per minute (cm ² · min ⁻¹)
l	length of static air layer in sampler (or equivalent for permeation types), in centimetres (cm)
m_b	mass of analyte desorbed from blank sampler, in nanograms (ng)
m_d	mass of analyte desorbed, in nanograms (ng)
m_s	mass of the analyte which can diffuse to a suitable sorbent within a certain time, i.e. the mass uptake of a diffusive sampler, in nanograms (ng)
$\dot{m}_l$	mass loss from permeation tube, in micrograms per minute (µg · min ⁻¹)
M_a	molar mass of analyte, in grams per mole (g · mol ⁻¹)
n	number of replicate samples
OELV	occupational exposure limit value
p_{at}	actual pressure of the test atmosphere sampled, in kilopascals (kPa)
R	recovery
R_{an}	analytical recovery
RH	relative humidity of the test atmosphere sampled, in percent (%)
t_e	exposure time, in minutes (min)
T_{at}	temperature of the test atmosphere sampled, in Kelvin (K)
$\dot{U}_d$	uptake rate, in cubic centimetres per minute (cm ³ · min ⁻¹)
$(\dot{U}_d)'$	uptake rate, in nanograms per parts per million (volume fraction) per minute (ng · ppm ⁻¹ · min ⁻¹)
$\dot{U}_{d1}$	uptake rate of analyte 1, in cubic centimetres per minute (cm ³ · min ⁻¹)
$\dot{U}_{d2}$	uptake rate of analyte 2, in cubic centimetres per minute (cm ³ · min ⁻¹)
$\dot{V}$	flow rate into the exposure chamber, for example, in litres per minute (l · min ⁻¹)
β_a	mass concentration of the analyte in the calibration gas mixture, in milligrams per cubic metre (mg · m ⁻³)
$(\beta_a)'$	mass concentration in parts per million (ppm);
β_{a1}	mass concentration of the given analyte at the beginning of the diffusion layer (i.e. at the distance l from the surface of the sorbent), in milligrams per cubic metre (mg · m ⁻³)

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β_{a2}	mass concentration of the given analyte at the end of the diffusion layer (i.e. at the surface of the sorbent), in milligrams per cubic metre ($\text{mg} \cdot \text{m}^{-3}$)
$\bar{\beta}_{a,R}$	mean mass concentration of the analyte recovered from the test gas atmosphere, in milligrams per cubic metre ($\text{mg} \cdot \text{m}^{-3}$);
β_{cg}	mass concentration of the calibration gas mixture, in milligrams per cubic metre ($\text{mg} \cdot \text{m}^{-3}$)
ϑ_{at}	temperature of the test atmosphere sampled, in degree Celsius ($^{\circ}\text{C}$)
K_v	coefficient of variation (CV)(The predecessor term "relative standard deviation" is deprecated and has been replaced by the term "coefficient of variation". See also ISO 3534-1:2006, 2.38, Note 2.)
$\varnothing_a$	volume fraction of the analyte, in microlitres per litre ($\mu\text{l} \cdot \text{l}^{-1}$)

5 Types of samplers

Samplers for gases and vapours can be divided into type A samplers and type B samplers:

Type A samplers rely on sorption onto a solid or onto a support impregnated with a reagent, desorption with solvent, and subsequent analysis of the desorbate. They are usually made of polypropylene or glass and consist of one or more sorbent layers and contain an active sorbent (e.g. activated carbon) or a support impregnated with reagent.

Type B samplers rely on sorption onto a solid or onto a support impregnated with a reagent, thermal desorption, and analysis of the desorbate. They are usually made of glass or metal, are sealed with removable fittings and consist of one or more beds of sorbent (e.g. porous polymer resin).

6 Requirements

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6.1 General

Some requirements (see 6.2) shall be initially verified by the manufacturer once for each type of sampler. Other requirements (see 6.3) shall be verified for each combination sampler/chemical agent.

Measuring procedures shall meet the requirements for measuring procedures specified in 6.3. When use of a sampler for measurement of a particular gas or vapour is claimed, the sampler shall meet the requirements specified in 6.2.

NOTE 1 No useful performance requirements can be given for the effect of interferents (with the exception of relative humidity). The effect of interferents is difficult to predict for a non-ideal sorbent without adsorption isotherm data on mixed systems which is normally unavailable. However, the user of diffusive samplers is cautioned that the adsorption of water vapour on certain sorbents, e.g. activated carbon and silica gel, can have a large effect on sampler capacity and analytical recovery.

NOTE 2 Because of the known effect of pressure on diffusion coefficients, a pressure test is not necessary.

6.2 Sampler requirements

6.2.1 Nominal uptake rate

The nominal uptake rate and the coefficient of variation shall be provided by the manufacturer in accordance with 8.2.1 and Annex A.

6.2.2 Air velocity/sampler orientation

The manufacturer shall test the working range of air velocity and the influence of sampler orientation in accordance with 8.2.2.

6.2.3 Sampler leak test

When tested in accordance with 8.2.3, any additional analyte determined above the blank value (see 6.3.2.3) shall be less the one-third of the calculated mass uptake by the sampler for 30 min exposure to a concentration of 0,1 OELV.

6.2.4 Shelf life

The manufacturer shall specify the shelf life of the diffusive sampler when stored in its original package. During this period the sampler shall fulfil all requirements.

6.2.5 Sampler identification (for commercially available diffusive samplers)

Samplers shall be uniquely identified.

6.2.6 Marking

Diffusive samplers shall be marked with at least the following:

- a) manufacturer's name;
 - b) product identification;
 - c) batch identification;
 - d) shelf life (if applicable);
 - e) number of this document.
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If required due to limited space, the marking may be placed on the packaging of the diffusive sampler. However, the manufacturer's name and product identification shall be indicated on the diffusive sampler.

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6.2.7 Instructions for use

The instructions for use supplied with the diffusive sampler shall be written in the principal language(s) used in the countries where the diffusive sampler is to be marketed. Where appropriate, the instructions for use shall contain directly and by reference to an online document, at least the following information:

- a) designated use (general purpose for a number of gases and vapours or, specific, for a particular gas or vapour, see 6.1);
- b) blank value (only when used for a particular gas or vapour, see 6.1);
- c) nominal uptake rate for the substances for which the diffusive sampler is intended to use;
- d) directions for proper handling of the diffusive sampler, including opening and closing;
- e) general information on the principle of use, for example, sorbent type, reaction of the reagent impregnated solid, desorption method;
- f) information on storage and transport;
- g) air velocity range in which the sampler can be used;
- h) orientation;
- i) information on health or environmental hazards and method of disposal.

The general information on the principle of use can be given in additional literature.

6.3 Measuring procedure requirements

6.3.1 Sampling procedure requirements

6.3.1.1 Sampling time

Sampling time shall be established according to concentration range of the compounds of interest over which measurements are to be made, i.e. up to two times the OELV, see ISO 20581, and taking into account the nominal or theoretical uptake rate.

6.3.1.2 Bias due to the selection of a non-ideal sorbent (back diffusion)

When tested in accordance with [8.3.1.1](#), the bias shall be $\leq 10\%$.

6.3.1.3 Uptake rate

If it is possible to calculate the ideal steady-state value in accordance with [8.2.1](#), the nominal uptake rate, determined in accordance with [8.2.1](#), shall be within $\pm 25\%$ of the steady-state value.

6.3.1.4 Storage conditions after sampling

The storage conditions after sampling shall be specified. When tested in accordance with [8.3.1.3](#), the mean value of the recovery after storage shall not differ by more than 10 % from the value before storage.

6.3.2 Analytical procedure requirements

6.3.2.1 Limit of quantification

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The limit of quantification as determined in [8.3.2.1](#) for long-term OELVs shall be lower than the mass collected by the sampler at a concentration of 0,1 OELV for 8 h.

The limit of quantification for short-term OELVs shall be lower than the mass collected by the sampler at a concentration of 0,5 OELV for 15 min.

If the sampler is to be used for shorter reference periods, the limit of quantification also shall be able to be measured at those periods.

6.3.2.2 Analytical recovery

When tested in accordance with [8.3.2.2](#) the analytical recovery R_{an} shall be

for type A samplers: $R_{an} \geq 75\%$ with $K_v \leq 10\%$ at each loading, and

for type B samplers: $R_{an} \geq 95\%$ with $K_v \leq 10\%$ at each loading.

Where the analytical recovery cannot be achieved the user shall take care that the measuring procedure meets all other requirements of this document and ISO 20581.

6.3.2.3 Blank value

In order to obtain acceptable values for the quantification limit of the method, the blank value of the sampling media should be as low as technically possible.

The blank value as tested in [8.3.2.3](#) shall be less than the limit of quantification as determined in accordance with [8.3.2.1](#) or otherwise it can be subtracted from the result, but the standard deviation of the blank value will contribute to the expanded uncertainty of the measuring procedure.

Where it is known that the blank value is significant and varies between batches of samplers, it shall be checked on each batch of samplers.

To eliminate any contamination which could occur during storage before use Type B samplers should be cleaned before sampling by taking them through the thermal desorption procedure.

This cleaning process should be carried out as close as possible to the time when the samplers will be used.

6.3.3 Expanded uncertainty

When tested in accordance with 8.3 the expanded uncertainty calculated in accordance with 8.4 shall meet the requirements given in ISO 20581.

The expanded uncertainty requirement shall be met from 10 °C to 40 °C and at relative humidities from 20 % to 80 %. Above 30 °C the use of correction factors is permitted to meet this requirement.

In addition, the performance criteria should also be met under a wider variety of environmental influences, representative of workplace conditions.

6.3.4 Method description

6.3.4.1 Scope of the measuring procedure

The scope of the measuring procedure shall include information on the following:

- a) principle of the method;
- b) chemical agents covered by the measuring procedure;
- c) analytical technique used;
- d) working ranges;
- e) chemical agents for which the measuring procedure is known to be adequate but not completely validated according to this document, especially in case of compounds of the same chemical family or homologous series;
- f) chemical agents for which the measuring procedure is known to be inadequate;
- g) any known interferences.

6.3.4.2 Method performance

The measuring procedure shall comprise information about method performance, including the following:

- a) the chemical agents for which measuring procedure has been shown to be effective;
- b) the range of concentrations of chemical agents in air, sample volume, uptake rates, exposure time and range of environmental conditions over which the measuring procedure has been shown to meet the performance criteria for expanded uncertainty prescribed in ISO 20581;
- c) the limit of quantification of the measuring procedure for chemical agents of interest;
- d) full details of any known interferences, including suitable and sufficient information on how to minimise their effects.

6.3.4.3 Apparatus and reagents

With regard to apparatus and reagents the measuring procedure shall

- a) specify that the diffusive sampler used complies with the provisions of this document,
- b) specify the required characteristics of analytical instruments to be used,
- c) specify the quality of the reagents to be used.

6.3.4.4 Safety information

The measuring procedure shall provide suitable and sufficient information on the safety hazards associated with the reagents and equipment used.

7 General test conditions

7.1 Reagents

Use reagents of analytical grade, where possible.

7.2 Apparatus

Usual laboratory apparatus and the following:

7.2.1 Dynamic system for generating, pre-mixing and delivering a known concentration of a test gas or vapour in air (see ISO 6145-1, ISO 6145-4, ISO 6145-6 and ISO 6145-10), including at least:

- an exposure chamber constructed of inert materials such as glass or polytetrafluorethylene (PTFE), through which the generated test atmosphere is passed, of sufficient capacity to accommodate simultaneously at least six test samplers and six samplers of one independent method (see 7.3) positioned in such a manner that there is no interference between each sampler;
- provisions for measuring, controlling and varying the air flow rate through the chamber and the concentration, temperature and relative humidity of the calibration gas mixture.

NOTE It is also possible to use a smaller exposure chamber and to carry out repeat experiments to obtain at least six pairs of data.

7.2.2 Micropipettes or syringes, for applying known volumes of standard solutions, conforming with the requirements of ISO 8655-2 and with a calibration checked in accordance with ISO 8655-6.

7.2.3 Instruments for analysing the gas, vapour or a characteristic reaction product collected by either the test sampler or an independent sampling method.

7.3 Independent method

The concentration of the generated calibration gas mixture in the exposure chamber shall be verified as follows:

- a) by an independent method, which has been validated using an established protocol, for example a pumped sampler method, bubbler method, or a different diffusive sampler method; or
- b) by using an independently calibrated on-line instrument, e.g. a flame ionization detector, or an infrared spectrometer.

If a pumped sampler procedure is used as the independent method, the method shall conform with all requirements of ISO 22065.