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

*Air des lieux de travail — Gaz et vapeurs — Exigences pour l'évaluation des procédures de mesure à l'aide de dispositifs de prélèvement par pompage*

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# Foreword

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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*.

This second edition cancels and replaces the first edition (ISO 22065:2019), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

- Editorial updates.

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. It enables manufacturers and users of pumped samplers and developers and users of procedures for measuring gases and vapours to adopt a consistent approach to method validation (see Annex B).

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

## 1 Scope

This document specifies performance requirements and test methods under prescribed laboratory conditions for the evaluation of pumped samplers used in conjunction with an air sampling pump and of procedures using these samplers for the determination of gases and vapours in workplace atmospheres.

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

NOTE 1 For the purposes of this document, a manufacturer can be any commercial or non-commercial entity.

NOTE 2 For the sampling of semi-volatile compounds which can appear as a mixture of vapours and airborne particles in workplace atmospheres see EN 13936.

This document is applicable to pumped samplers and measuring procedures using these samplers in which sampling and analysis are carried out in separate stages.

This document is not applicable to:

— pumped samplers which are used for the direct determination of concentrations, for example, length-of-stain detector tubes;

— samplers which rely on sorption into a liquid, and subsequent analysis of the solution (bubblers).

## 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 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*

ISO 13137:2013, *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

ISO 18158, *Workplace air — Terminology*

ISO 20581, *Workplace air — General requirements for the performance of procedures for the measurement of chemical agents*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 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 http://www.electropedia.org/

## 4 Symbols and abbreviated terms

For the purposes of this document, the following symbols and abbreviations apply.

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

| | |
|---|---|
| CRM | certified reference material |
| LV | limit value |
| $m_{\mathrm{a1}}$ | mass of analyte desorbed from tube blank, in micrograms (µg) |
| $m_{\mathrm{a2}}$ | mass of analyte desorbed from spiked tube, in micrograms (µg) |
| $m_{\mathrm{a,lt}}$ | maximum mass uptake of analyte in a leak test performed on a sealed sampler used for making measurements for comparison with a long-term limit value, in milligrams (mg) |
| $m_{\mathrm{a,st}}$ | maximum mass uptake of analyte in a leak test performed on a sealed sampler used for making measurements for comparison with a short-term limit value, in milligrams (mg) |
| $\dot{m}_1$ | mass loss from permeation tube, in micrograms per minute ($\mu g \cdot min^{-1}$) |
| $M_{\mathrm{a}}$ | molar mass of analyte, in grams per mole ($g \cdot mol^{-1}$) |
| $n$ | number of replicate samples |
| $p_{\mathrm{at}}$ | pressure of the test atmosphere sampled, in kilopascals (kPa) |
| $R_{\mathrm{me}}$ | method recovery |
| $R_{\mathrm{an}}$ | analytical recovery |
| RH | relative humidity of the test atmosphere sampled, in percent (%) |
| $t_{\mathrm{H}}$ | hold-up time of the unretained substance, in minutes (min) |
| $t_{\mathrm{s}}$ | sampling time, in minutes (min) |
| $T_{\mathrm{at}}$ | temperature of the test atmosphere sampled, in Kelvins (K) |
| $V_{\mathrm{at}}$ | volume of the test atmosphere sampled, in litres (l) |
| $V_{\mathrm{H}}$ | gas (vapour) hold-up volume (dead volume), in litres (l) |
| $V_{\mathrm{R}}$ | uncorrected retention volume, in litres (l) |
| $(V_{\mathrm{R}})'$ | corrected retention volume, in litres (l) |
| $\dot{v}$ | flow rate into the exposure chamber, for example, in litres per minute ($l \cdot min^{-1}$) |
| $v_{\mathrm{a}}$ | volumetric air flow rate through the sampler, for example, in litres per minute ($l \cdot min^{-1}$) |
| $\beta_{\mathrm{a}}$ | mass concentration of the analyte in the calibration gas mixture, in milligrams per cubic metre ($mg \cdot m^{-3}$) |
| $\bar{\beta}_{\mathrm{a,R}}$ | mean mass concentration of the analyte recovered from the test gas atmosphere, in milligrams per cubic metre ($mg \cdot m^{-3}$) |

| | |
|---|---|
| $\beta_{\mathrm{cg}}$ | mass concentration of the calibration gas mixture, in milligrams per cubic metre ($\mathrm{mg \cdot m^{-3}}$) |
| $\vartheta_{\mathrm{at}}$ | temperature of the test atmosphere sampled, in degrees Celsius (°C) |
| $K_{\mathrm{v}}$ | coefficient of variation (CV)<br>NOTE The predecessor term "relative standard deviation" is deprecated. See also ISO 3534-1:2006, 2.38, Note 2. |
| $\rho_{\mathrm{LV,lt}}$ | long-term limit value given as concentration, in milligrams per cubic metre ($\mathrm{mg \cdot m^{-3}}$) |
| $\rho_{\mathrm{LV,st}}$ | short-term limit value given as concentration, in milligrams per cubic metre ($\mathrm{mg \cdot m^{-3}}$) |
| $\phi_{\mathrm{a}}$ | volume fraction of the analyte, in microlitres per litre ($\mathrm{\mu l \cdot l^{-1}}$) |

## 5 Sampler types

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

- Type A samplers rely on sorption onto a collection substrate which can be impregnated with a reagent. The collection substrate is always desorbed with a solvent, and subsequently analysed.
- Type B samplers rely on sorption onto a solid, thermal desorption, and analysis of the desorbate.

## 6 Requirements

### 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.

Known or suspected interferences shall be noted as required in 6.3.4.1. The results of any tests performed to evaluate interferences, including suitable and sufficient information to minimize their effects shall be presented in the method report as required in 6.3.4.2.

NOTE No useful performance requirements can be given for the effect of interferents (with the exception of water vapour). The effect of interferents is difficult to predict for a non-ideal sorbent without adsorption isotherm data on mixed systems which is normally unavailable.

### 6.2 Sampler requirements

#### 6.2.1 Flow resistance

When tested in accordance with 8.2.1, at least 95 % of samplers shall have a back pressure less than the appropriate maximum value indicated in Table 1. A minimum of 20 samplers shall be tested.

NOTE Typical back pressure values for type A samplers and type B samplers are given in [1].

**Table 1 — Maximum back pressures**

| Sampler type | Maximum back pressure kPa |
|---|---|
| Type A (solvent desorption) | ≤10 |
| Type B (thermal desorption) | ≤3,5 |

### 6.2.2 Sampler leak test (for Type B samplers)

When tested in accordance with 8.2.2, for substances with a long-term limit value the maximum leakage, i.e. the maximum mass uptake of analyte above the blank value (see 6.3.2.3), shall be less than $m_{\mathrm{a,lt}}$ calculated according to Formula (1), in milligrams (mg), as follows:

$$m_{\mathrm{a,lt}} = \frac{1}{3}\left(0{,}1\rho_{\mathrm{LV,lt}} \times 240 \times 0{,}01 \times 10^{-3}\right) \tag{1}$$

where

$m_{\mathrm{a,lt}}$ is the maximum mass uptake of analyte in a leak test performed on a sealed sampler used for making measurements for comparison with a long-term limit value;

$\rho_{\mathrm{LV,lt}}$ is the long-term limit value of the substance given as concentration, in milligrams per cubic metre ($\mathrm{mg \cdot m^{-3}}$);

240 is the reference period, in minutes (min);

0,01 is the nominal minimum flow rate for type B samplers, in litres per minute ($\mathrm{l \cdot min^{-1}}$);

$10^{-3}$ is a factor applied to convert the nominal minimum flow rate from litres per minute (l/min) to cubic metres per minute ($\mathrm{m^3 \cdot min^{-1}}$);

1/3 is a factor applied to calculate the maximum permitted leakage.

When tested in accordance with 8.2.2, for substances with a short-term limit value the maximum leakage, i.e. the maximum mass uptake of analyte above the blank value (see 6.3.2.3), shall be less than $m_{\mathrm{a,st}}$ calculated according to Formula (2), in milligrams (mg), as follows:

$$m_{\mathrm{a,st}} = \frac{1}{3}\left(0{,}5\rho_{\mathrm{LV,st}} \times 15 \times 0{,}01 \times 10^{-3}\right) \tag{2}$$

where

$m_{\mathrm{a,st}}$ is the maximum mass uptake of analyte in a leak test performed on a sealed sampler used for making measurements for comparison with a short-term limit value;

$\rho_{\mathrm{LV,st}}$ is the short-term limit value of the substance given as concentration, in milligrams per cubic metre ($\mathrm{mg \cdot m^{-3}}$);

15 is the reference period, in minutes (min);

0,01 is the nominal minimum flow rate for type B samplers, in litres per minute ($\mathrm{l \cdot min^{-1}}$);

$10^{-3}$ is a factor applied to convert the nominal minimum flow rate from litres per minute ($\mathrm{l \cdot min^{-1}}$) to cubic metres per minute ($\mathrm{m^3 \cdot min^{-1}}$);

1/3 is a factor applied to calculate the maximum permitted leakage.

### 6.2.3 Shelf life

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

### 6.2.4 Sampler identification

Samplers shall be uniquely identified.

### 6.2.5 Marking

Samplers shall be marked with at least the following:

a) manufacturer's name,

b) product identification including batch identification, where available,

c) indication of the direction of air flow,

d) shelf life (or expiry date), and

e) number of this document.

NOTE The marking with the number of this document implies only that the sampler fulfils the requirements given in 6.2.

If required due to limited space, the marking may be placed on the packaging of the sampler. However, at least the product identification, batch identification, where available, and direction of air flow shall be indicated on the sampler.

### 6.2.6 Instructions for use

The instructions for use supplied with the sampler shall be written in the principal language(s) used in the countries where the sampler is to be marketed. They shall contain 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) assurance that blank value meets specifications, where necessary for a particular gas or vapour, (where a blank value is important),

c) directions for proper handling of the sampler, including opening and closing,

d) general information on the principle of use, for example, sorbent type, reaction of the reagent impregnated solid, desorption method,

e) information on the range of temperature where the sampler can be used,

f) information on storage and transport, and

g) information on health or environmental hazards and method of disposal.

## 6.3 Measuring procedure requirements

### 6.3.1 Sampling procedure requirements

#### 6.3.1.1 General

Sampling conditions (sample volume, flow rate and sampling time) shall be established according to the LV assigned to the compounds of interest, for example, short-term limit value, long-term limit value or both.

### 6.3.1.2 Sample volume

The recommended sample volume which is calculated from the recommended flow rate and the reference period should conform to the sampler capacity verification test (see 8.3.1.2).

### 6.3.1.3 Air flow rate

#### 6.3.1.3.1 Determination of the maximum air flow rate (only for impregnated filters)

When tested according to 8.3.1.4, the maximum air flow rate shall be 90 % of the flow rate at which the breakthrough volume drops by 5 %.

#### 6.3.1.3.2 Determination of the minimum air flow rate (only for thermal desorption)

A minimum air flow rate shall be established according to the test given in 8.3.1.5.

### 6.3.1.4 Storage conditions after sampling

The storage conditions after sampling shall be specified. When tested in accordance with 8.3.1.6, the mean value of the method 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

The limit of quantification shall be lower than or equal to the calculated mass of analyte that would be collected for the minimum air sample volume specified in the measuring procedure at the following concentrations:

- 0,1 LV for substances with long-term limit value,
- 0,5 LV for substances with short-term limit value only.

### 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.

The values given for analytical recovery are targets; lower values may be used provided equivalent precision is achieved.

### 6.3.2.3 Blank value

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

When tested in accordance with 8.3.2.3 the blank value shall be less than one-tenth of the calculated mass collected by the sampler during the recommended sampling time at the recommended air flow rate and at concentrations of

- 0,1 LV for substances with long-term limit value, and
- 0,5 LV for substances with short-term limit value only.

NOTE Higher blank values can be allowed provided the requirement of 6.3.2.1 is met.