



# SLOVENSKI STANDARD

## SIST EN 1076:1998

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Workplace atmospheres - Pumped sorbent tubes for the determination of gases and vapours - Requirements and test methods

Arbeitsplatzatmosphäre - Pumpenbetriebene Sammelröhrchen zur Bestimmung von Gasen und Dämpfen - Anforderungen und Prüfverfahren

Atmospheres des lieux de travail - Tubes a adsorption avec pompage pour la détermination des gaz et vapeurs - Exigences et méthodes d'essai

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EUROPEAN STANDARD

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English version

**Workplace atmospheres - Pumped sorbent tubes  
for the determination of gases and vapours -  
Requirements and test methods**

Atmosphères des lieux de travail - Tubes à  
adsorption avec pompage pour la détermination  
des gaz et vapeurs - Exigences et méthodes  
d'essai

Arbeitsplatzatmosphäre - Pumpenbetriebene  
Sammelröhrchen zur Bestimmung von Gasen und  
Dämpfen - Anforderungen und Prüfverfahren

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European Committee for Standardization  
Comité Européen de Normalisation  
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

**Contents**

	Page
Foreword .....	2
Introduction .....	3
1 Scope .....	3
2 Normative references .....	4
3 Definitions .....	4
4 Symbols and abbreviations .....	4
5 Requirements .....	5
6 General test conditions .....	6
7 Test methods .....	7
8 Levels of evaluation .....	11
9 Test report .....	12
10 Marking .....	12
Annex A (informative)	
Examples for the determination of the breakthrough volume .....	13
Annex B (normative)	
Analysis of experimental data (two-factor experiment) .....	14
Annex C (informative)	
Field tests - Paired comparisons .....	19
Annex D (informative)	
Field tests - Multiple comparisons .....	20
Annex E (normative)	
Environmental influences - Multifactor test .....	22
Annex F (informative)	
Typical tube dimensions .....	22
Annex G (informative)	
Bibliography .....	23

**Foreword**

This European Standard has been prepared by Technical Committee CEN/TC 137 "Assessment of workplace exposure" the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 1997, and conflicting national standards shall be withdrawn at the latest by November 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.



## Introduction

CEN/TC 137 "Assessment of workplace exposure" has proposed general performance criteria that methods of determining the concentration of chemical agents in workplace atmosphere should meet (see EN 482). These performance criteria include maximum values of overall uncertainty (a combination of precision and bias) achievable under prescribed laboratory conditions for the methods to be used. In addition, the performance criteria should also be met under a wider variety of environmental influences, representative of workplace conditions.

## 1 Scope

This European Standard specifies performance requirements and test methods under prescribed laboratory conditions for a pumped sorbent tube used in conjunction with an air sampling pump for the determination of gases and vapours in workplace atmospheres.

Additional tests designed to establish whether the performance characteristics of the pumped sorbent tube are affected by the wider range of environmental influences that may be encountered in field use are described in annexes C and D.

If there is no pumped sorbent tube for measuring a particular chemical agent which meets the requirements of this European Standard, it is recommended to use a pumped sorbent tube whose performance is nearest to the specified requirements.

Some of the test methods described in this European Standard require the use of an air sampling pump. The specification for such a pump is covered by EN 1232.

The European Standard is applicable to pumped sorbent tubes which are used for the indirect determination of concentrations, by sampling and analysis in separate stages.

These tubes can be divided into:

- type A samplers which rely on sorption onto a solid or onto a support impregnated with a reagent, desorption with solvent, and subsequent analysis of the desorbate;
- type B samplers which rely on sorption onto a solid or onto a support impregnated with a reagent, desorption by heat, and analysis of the desorbate.

NOTE 1: Type A samplers usually consist of two beds of sorbent in series, i.e. with a back-up section, and contain an active sorbent (e. g. charcoal) or a support impregnated with reagent. The back-up section is not used in the breakthrough tests, but in field use is a protection against unexpected breakthrough.

NOTE 2: Type B samplers usually consist of a single bed of active sorbent (e. g. porous polymer resin).

Specific aspects of the use of reagent impregnated systems will be covered in additional parts of this standard.

The European Standard does not apply to:

- pumped sorbent tubes 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).

This European Standard should enable manufacturers and users of pumped sorbent tubes to adopt a consistent approach to sampler validation and provide a framework for the assessment of sampler performance against criteria specified in EN 482. It is the responsibility of the manufacturer or of those who assemble the pumped sorbent tubes to ensure that the sampler complies with the overall uncertainty requirements under the specified laboratory conditions given in this European Standard including such environmental influences (e. g. temperature and humidity) that may be expected to affect performance.

No useful performance requirements can be given for the effect of interferents (with the exception of water vapour). However, the user of pumped sorbent tubes should be cautioned that interferences may occur (see 5.8.e)).

NOTE 3: Annexes C and D relating to field tests are not obligatory as part of this European Standard but are directed primarily to the users of such devices, who are in the best position to know the specific factors that can significantly affect sampler performance.

NOTE 4: Because of the known relationship between atmosphere pressure and atmospheric concentrations, a pressure test is normally not necessary.

## 2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

- EN 482 : 1994 Workplace atmospheres - General requirements for the performance of procedures for the measurement of chemical agents
- EN 838 Workplace atmospheres - Diffusive samplers for the determination of gases and vapours - Requirements and test methods
- EN 1232 Workplace atmospheres - Pumps for personal sampling of chemical agents - Requirements and test methods

## 3 Definitions

For the purposes of this standard, the definitions for bias, limit value, measuring procedure, overall uncertainty, precision, true value and validation given in EN 482, as well as the following definitions apply.

**3.1 breakthrough volume:** The volume of a known atmosphere that can be passed through the tube before the concentration of vapour eluting from the tube reaches 5 % of the applied test concentration.

**3.2 retention volume:** The elution volume at peak maximum of a small aliquot of a vapour eluted from the tube by atmosphere (or chromatographic carrier gas).

**3.3 desorption efficiency:** The ratio of the mass of analyte desorbed from a sampling device to that applied.

**3.4 pumped sampler:** A device which is capable of taking samples of gases and vapours from the atmosphere and consisting of a sampling medium, such as a sorbent tube, and an air sampling pump.

NOTE: The sampling pump to be used should conform to the specification as laid down in EN 1232.

**3.5 sorbent tube:** A tube, usually made of metal or glass, containing an active sorbent or a reagent-impregnated support, through which sampled atmosphere is passed at a rate controlled by an air sampling pump.

**3.6 loading:** The product of concentration expressed in parts per million (volume per volume) or milligrams per cubic metre (ppm (V/V) or  $\text{mg} \cdot \text{m}^{-3}$ ) and the sampled atmosphere volume (flow rate x sampling time).

## 4 Symbols and abbreviations

- C* the measured concentration of analyte, in milligrams per cubic metre;
- C'* the measured concentration of analyte, in parts per million (volume/volume) or volume per volume;
- d* the desorption efficiency;
- LV* the limit value;
- m<sub>b</sub>* the mass of analyte which is desorbed from the tube blank, in micrograms;
- m<sub>d</sub>* the mass of analyte desorbed from the spiked tube, in micrograms ;
- m<sub>1</sub>* the mass loss from permeation tube, in milligrams per minute;
- M* the molar mass of the analyte, in grams per mol;
- p* the actual pressure of the atmosphere sampled, in kilopascals;
- T* the temperature of the atmosphere sampled, in Kelvin;
- v* the volumetric flow of air, in cubic metres per minute;
- V* the volume of atmosphere sampled, in litres.

- $\rho$  the delivered concentration, in milligrams per cubic metre;  
 $\phi$  the delivered concentration, in parts per million (volume/volume)

## 5 Requirements

### 5.1 Desorption efficiency (for type A samplers)

When tested in accordance with 7.1.1 or 7.1.2, the desorption efficiency shall be  $\geq 0,75$  with a coefficient of variation  $\leq 0,1$  at each loading.

### 5.2 Desorption efficiency (for type B samplers)

When tested in accordance with 7.1.3, the desorption efficiency shall be  $\geq 0,95$  with a coefficient of variation  $\leq 0,1$  at each loading.

### 5.3 Storage

When tested in accordance with 7.7.1, the mean value of the recovery after storage shall not differ by more than 10 % from the value before storage.

### 5.4 Overall uncertainty

When tested in accordance with 7.5 and 7.6 the overall uncertainty, calculated in accordance with 7.12, shall be as specified in EN 482.

The manufacturer shall state the range of temperature and humidity over which the overall uncertainty requirement is met. The temperature range should be from 5 °C to 40 °C, but shall be at least from 10 °C to 30 °C. In this temperature range, and at relative humidity between 20 % and 80 % the overall uncertainty requirement shall be met without the use of correction factors. Outside the temperature range of 10 °C to 30 °C the requirement may be fulfilled by the use of correction factors.

### 5.5 Blank value

When tested in accordance with 7.3 the blank value shall be less than one-third of the calculated mass collected by the sampler during 30 min sampling at 0,1 LV at the minimum flow rate specified in 7.5.1.

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### 5.6 Sampler leak test (only samplers not thermally sealed)

When tested in accordance with 7.8, any additional analyte determined above the blank value (see 5.5) shall be less than one-third of the calculated mass uptake by the sampler for 30 min exposure to 0,1 LV.

### 5.7 Flow resistance

When tested in accordance with 7.9, before and after the test in 7.10 the pressure drop of at least 19 out of 20 tested tubes shall not exceed 5 kPa at a flow rate of 300 ml/min.

### 5.8 Instructions for use

The instructions for use supplied with the pumped sorbent tube shall be in the language(s) of the country where the pumped sorbent tube is to be marketed. It shall contain at least the following information:

- breakthrough volume for a specific analyte;
- designated use including measuring range and level of evaluation;
- direction for proper handling of the pumped sorbent tube including opening and closing;
- general information on the system e. g. sorbent;
- the levels at which other typical gases and vapours, including water, are likely to interfere to the extent of increasing the overall uncertainty above the level specified in this European Standard;
- information on the calculation of results including the evaluation of reading and correction factors for temperature, pressure and desorption efficiency, if applicable;
- information on storage and transport;
- desorption efficiency;



i) information on the desorption method.

The general information on the system, d), can be given in additional literature.

## 5.9 Sample identification

The pumped sorbent tube shall have a suitable area for sample identification by the user.

## 6 General test conditions

### 6.1 Reagents

Use only analytical grade reagents.

### 6.2 Apparatus

6.2.1 Usual laboratory apparatus and resources and:

6.2.2 A dynamic system for generating, pre-mixing and delivering a known concentration of a test gas or vapour in air (see annex G for corresponding ISO standards).

6.2.3 An exposure chamber constructed of inert materials such as glass or polytetrafluorethylene (PTFE), through which the generated atmosphere is passed, and of sufficient capacity to accommodate simultaneously at least six test samplers and six samplers of one independent method (see 6.3) positioned in such a manner that there is no interference between each sampler.

6.2.4 Provisions for measuring, controlling and varying the rate of air flow through the chamber and the concentration, temperature and relative humidity of the calibration gas mixture.

6.2.5 An appropriate sampling pump which meets the performance requirements of EN 1232.

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

6.2.7 Flow-meter (as in A.1.1.2 but with a range from 10 ml/min to 350 ml/min)

### 6.3 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, e. g. a diffusive sampler method, bubbler method or a different pumped sorbent tube; or
- b) by using an independently calibrated on-line instrument, e. g. a flame-ionization detector, an infrared analyzer or mass spectrometer.

If a diffusive sampler is used as the independent method, consult EN 838 for any special provisions, e. g. minimum air velocity requirements.

### 6.4 Generation of the calibration gas mixture

#### 6.4.1 General

Using the apparatus detailed in 6.2, set up a calibration gas mixture of the test analyte at the concentration and values of temperature, humidity, etc. specified in 7.5 to 7.8. Ensure that the flow rate through the exposure chamber exceeds the combined sampling rate of all the test and independent method samplers.

#### 6.4.2 Calibration gas mixture

Calculate the concentration of the calibration gas mixture from the atmosphere generation parameters; e. g. for a permeation cell system, the delivered concentration is:

$$\rho = \frac{m_1}{V} \quad (1)$$

NOTE: The example given does not give a preference for permeation systems for generating calibration



gas mixtures of gases and vapours.

Determine the mean concentration of the atmosphere within the exposure chamber experimentally as in 6.5, using the results from the samplers of the independent method. A correction may be applied for any known bias in the independent method. Compare this value with the mean calculated value.

If the experimentally determined value is within  $\pm 10\%$  of the calculated value of the mean concentration of the delivered atmosphere, take the calculated value as the true value of the delivered concentration, as used in 7.4, 7.5.2 and 7.6.2.

If this requirement is not met, then adjustments shall be made or an alternative generation method shall be used or the independent method shall be verified.

If it is not possible to calculate a concentration of the calibration gas, e. g. for reactive gases, the value of the independent method shall be used as the true value.

## 6.5 Test conditions

Set up a calibration gas mixture of the test analyte as in 6.4.1 and allow at least six test (pumped) samplers, as well as the sampler(s) of the independent method to withdraw vapour from the exposure chamber for a known period of time. For the pumped samplers, use representative air sampling pumps as in 6.2.5, such that the volume of atmosphere sampled does not exceed the breakthrough capacity of the tubes (see 7.2). The pumps shall be operated such that the flow rate is within the range recommended by the manufacturer.

NOTE: In each case, where a "nominal exposure time" of 8 h is specified, 8 h is a guideline and can be reduced to  $\geq 6$  h if circumstances demand. It can be further reduced to  $< 6$  h only if the breakthrough volume (see 7.2) would otherwise be exceeded.

## 7 Test methods

If it is known in advance that a certain type of pumped sorbent tube is unaffected by an environmental influence, then the relevant tests in 7.5 to 7.7, or the equivalent in annex E, shall be modified to examine only the factors likely to have an influence.

Where significance tests are employed, testing should be done at a 5 % level.

### 7.1 Desorption efficiency

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#### 7.1.1 Tube spiking method (for type A samplers)

##### 7.1.1.1 Determination from the gas phase

Conduct the determination at four different loadings, within the range 0,1 LV and 10 ml/min flow rate to 2 LV and 200 ml/min flow rate each for a nominal 8 h exposure. Add a known mass of analyte to the sorbent tube by passing a known volume of atmosphere (using the apparatus in 6.2) at a known concentration (the "calibration gas mixture") under conditions where breakthrough (see 7.2) does not occur. This atmosphere shall be generated at  $(20 \pm 2)^\circ\text{C}$  and  $(50 \pm 5)\%$  relative humidity. The nominal 8 h exposure value should be reduced if overload of the sorbent tube occurs. Calculate the exposure loadings from the concentrations of the vapour or solution standards and the volumes applied. Desorb the analyte (or a reaction product, if appropriate), using a desorption solvent recommended by the manufacturer, or any other suitable solvent. Analyze the solution in triplicate by reference to liquid standards prepared directly.

##### 7.1.1.2 Determination from the liquid phase

Alternatively determine the desorption efficiency by adding a known mass of analyte to the sorbent tube using the same loadings as in 7.1.1.1. Use a calibrated microlitre syringe in the range  $1\ \mu\text{l}$  to  $10\ \mu\text{l}$ , diluting in a non-interfering solvent if necessary. The analyte may be applied directly to the sorbent or be introduced by means of the injection facility.

##### 7.1.1.3 Calculation

Calculate the desorption efficiency by dividing the mean mass recovered at each level from the spiked samples by the mass applied, and the coefficient of variation of replicates.

## 7.1.2 Phase equilibrium method (for type A samplers)

### 7.1.2.1 Determination

Prepare four pairs of solutions corresponding to the different loadings in 7.1.1.1 in volumes of desorption solvent as used for the desorption of sorbent tubes. Add the sorbent from an unspiked sorbent tube to one solution of each pair and allow to equilibrate for at least 30 min. Analyze in triplicate all solutions.

### 7.1.2.2 Calculation

Calculate the desorption efficiency ( $d$ ) by dividing the concentrations of the solutions to which sorbent has been added by the concentrations of the corresponding solutions without added sorbent. If the mean desorption efficiency measured by the phase equilibrium method is less than 95 % or the desorption efficiency measured at any one level is less than 90 %, only the test given in 7.1.1.1, shall be used.

## 7.1.3 Type B samplers

### 7.1.3.1 Procedure

Add a known mass of analyte to the sorbent as in 7.1.1.2. Thermally desorb the analyte, as recommended by the manufacturer, or using any other suitable conditions.

### 7.1.3.2 Calculation

Calculate the desorption efficiency by dividing the mean mass recovered at each level from the spiked samples by the mass applied and the coefficient of variation of replicates.

## 7.2 Determination of breakthrough volume

The breakthrough volume is determined either by a direct method using a vapour atmosphere or by an indirect chromatographic method (examples are given in annex A) or another suitable method.

### 7.2.1 Direct method

The breakthrough volume shall be measured by sampling from a standard vapour atmosphere (using the apparatus in 6.2) at a concentration of approximately 2 LV and at 200 ml/min at  $(20 \pm 2)$  °C and  $(80 \pm 5)$  % relative humidity or as otherwise recommended by the manufacturer, whilst monitoring the effluent with a flame ionisation or equivalent detector. A suitable way of doing this is described in A.1.

If it is proved that the breakthrough volume is not affected by humidity, the determination can be conducted at a lower humidity.

For two-bed type A tubes, use only the first (primary) bed or use specially prepared single-section tubes.

### 7.2.2 Chromatographic method

For porous polymers and similar chromatographic sorbents, instead of the procedure described above and in A.1, the breakthrough volume may be predicted from the chromatographic retention volume. A suitable procedure is described in A.2.

NOTE: Breakthrough volumes determined by the chromatographic method do not take account of humidity or very high concentrations ( $> 500$  g/m<sup>3</sup>). Measurements by the direct method indicate that breakthrough volumes at high (95 %) humidity are about a factor of two lower for porous polymers.

The chromatographic method is not suitable for reagent impregnated sorbents.

## 7.3 Determination of the blank value

Analyze six unspiked samplers, to determine the blank value of analyte.

## 7.4 Calculation of concentration

Calculate the concentration from the mass of analyte recovered from the sorbent tube and the sampled atmosphere volume as follows:

$$C = \frac{m_d - m_b}{d \cdot V} \quad (2)$$

NOTE: If it is desired to express concentrations reduced to specified conditions, e. g., 25 °C and 101 kPa, then,

$$C_{corr} = C \cdot \frac{101}{P} \cdot \frac{T}{298} \quad (3)$$

The volume fraction of the analyte in atmosphere is then:

$$C' = C_{corr} \cdot \frac{24,5}{M} \quad (4)$$

## 7.5 Effect of exposure concentration and atmosphere volume (loading) on sampler performance

### 7.5.1 Procedure

Using each of at least six pumped samplers withdraw a known volume from an atmosphere of the test analyte (see 6.5) under the following exposure combinations:

- concentration: approximately 0,1 LV, 2 LV, plus one intermediate value;
- flow rate: 10 ml/min, 200 ml/min plus one intermediate value;
- time: 8 h (nominal);
- relative humidity: (50 ± 5) %;
- temperature: (20 ± 2) °C.

NOTE: The recommended procedure is to start with the four combinations 0,1 LV, 2 LV, 10 ml/min, 200 ml/min. If the statistical analysis (see 7.5.2) shows no significant effects, the other combinations can be omitted.

As specified in 7.2, the sample volume shall not exceed the breakthrough capacity of the sorbent tube for the test analyte. If necessary, the exposure time shall be reduced accordingly.

Analyze the sorbent tubes by reference to standard solutions or to standard tubes spiked with known amounts of analyte.

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### 7.5.2 Calculation

For each exposure combination, calculate the concentration (see 7.4) for each of the six (or more) replicate pumped sorbent tubes. Divide each by the relevant calibration gas mixture concentration (true value, see 6.4;  $\rho$  or  $\phi$ ). Conduct an analysis of variance on the result according to annex B to determine whether there are any significant main effects or interactions with respect to the factors concentration and flow rate/atmosphere volume.

## 7.6 Effect of atmosphere temperature and atmosphere humidity on sampler performance

An alternative procedure for the determination of the environmental influences is described in annex E.

### 7.6.1 Procedure

Using each of at least six pumped samplers withdraw a known volume from an atmosphere of the test analyte (see 6.5) for all the following exposure combinations:

- concentration: 2 LV;
- flow rate: 200 ml/min;
- time: 8 h (nominal);
- relative humidity: (20 ± 5) %, (80 ± 5) %;
- temperature: (5 ± 2) °C, (40 ± 2) °C.

NOTE: The high and low values of the variables are guidelines; if it is known that the samplers are to be used in wider, or more restricted, ranges, the values can be adjusted accordingly.

As specified in 7.2, the sample volume shall not exceed the breakthrough capacity of the sorbent tube for the test analyte. If necessary, the exposure time shall be reduced accordingly.

Analyze the sorbent tubes by reference to standard solutions or to standard tubes spiked with known amounts of analyte.