



SLOVENSKI STANDARD

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NfU_`bUXYcj bYa `a Yghi `!`8]Z n]`g_]`j ncf Yj Ub_]`nUXc`c Yj Ub`Y`d`]bcj `]b`dUf`!
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Workplace atmospheres - Diffusive samplers for the determination of gases and vapours
- Requirements and test methods

Luftbeschaffenheit am Arbeitsplatz - Diffusionssammler für Gase und Dämpfe -
Anforderungen und Prüfung

Atmospheres des lieux de travail - Echantillonneurs par diffusion pour la détermination
des gaz et vapeurs - Prescriptions et méthodes d'essai

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ICS:

13.040.30 Kakovost zraka na delovnem mestu Workplace atmospheres

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EUROPEAN STANDARD

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English version

**Workplace atmospheres - Diffusive samplers for
the determination of gases and vapours -
Requirements and test methods**

Atmosphères des lieux de travail -
Echantillonneurs par diffusion pour la
détermination des gaz et vapeurs -
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Diffusionssammler für Gase und Dämpfe -
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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
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Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 137 "Assessment of workplace exposure" of which the secretariat 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of an identical text or by endorsement, at the latest by May 1996, and conflicting national standards shall be withdrawn at the latest by May 1996.

According to the CEN/CENELEC Internal Regulations, 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.

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0 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

1.1 General

This European Standard specifies performance requirements and test methods under prescribed laboratory conditions for a diffusive sampler used for the determination of gases or vapours in workplace atmospheres.

Additional tests designed to establish whether the performance characteristics of the diffusive sampler 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 diffusive sampler for measuring a particular chemical agent which meets the requirements of this European Standard, it is recommended to use a diffusive sampler whose performance is nearest to the specified requirements.

1.2 Field of application

The European Standard is applicable to:

- type A samplers: diffusive samplers which are used for the direct determination of concentrations, for example, length-of-stain detector tubes;
- type B samplers: diffusive samplers which are used for the indirect determination of concentration by sampling and analysis in separate stages.

Type B samplers may be further divided into:

- type B 1 samplers: diffusive samplers which rely on sorption onto a solid, desorption with solvent, and subsequent analysis of the desorbate;
- type B 2 samplers: diffusive samplers which rely on sorption onto a solid, desorption by heat, and analysis of the desorbate;
- type B 3 samplers: diffusive samplers which rely on sorption into a liquid, and subsequent analysis of the solution.

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

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1.3 Object of standard

This European Standard should enable manufacturers and users of diffusive samplers 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 diffusive samplers 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 diffusive samplers should be cautioned that interferences may occur especially for type A samplers. Such information shall be included in the instructions for use.

If it is known in advance that a certain type of diffusive sampler is unaffected by an environmental influence, then the relevant tests in 7.7 to 7.9, or the equivalent in annex E shall be modified to examine only the factors likely to have an influence.

NOTE 1: 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 may significantly affect sampler performance.

NOTE 2: Because of the known effect of pressure on diffusion coefficients, a pressure test is 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

Workplace atmospheres - General requirements for the performance of procedures for the measurement of chemical agents

3 Definitions

For the purposes of this standard, the following definitions apply.

3.1 bias, limit value, measuring procedure, overall uncertainty, precision, true value, validation

Definitions for these terms are as in EN 482.

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

3.3 diffusive sampler: A device which is capable of taking samples of gases or vapours from the atmosphere at a rate controlled by a physical process such as gaseous diffusion through a static air layer or permeation through a membrane, but which does not involve the active movement of air through the sampler.

3.4 diffusive uptake rate: The rate at which the diffusive sampler collects a particular gas or vapour from the atmosphere, expressed in nanograms per parts per million (volume/volume) per minute ($\text{ng ppm}^{-1}(\text{V/V}) \text{ min}^{-1}$) or cubic centimetres per minute ($\text{cm}^3 \text{ min}^{-1}$).

3.5 exposure dose: The product of exposure concentration expressed in parts per million, (volume/volume) or milligrams per cubic metre (ppm (V/V) , or mg m^{-3}) and exposure time expressed in minutes or hours.

4 Symbols and abbreviations

- A the cross-sectional area of sorption surface, in square centimetres;
- C the observed concentration in milligrams per cubic metre;
- C' the observed concentration in parts per million (volume/volume);
- D the diffusion coefficient of analyte, in square centimetres per minute;
- D₁ the diffusion coefficient of analyte 1;
- D₂ the diffusion coefficient of analyte 2;

- d the desorption efficiency (types B 1 and B 2 only);
- l the length of static air layer in sampler (or equivalent for permeation types), in centimetres;
- LV the limit value;
- m_b the mass of the analyte which is desorbed from the blank sampler, in nanogramms;
- m_d the mass of the analyte which is desorbed, in nanogramms;
- m_s the mass of the analyte which is sorbed by diffusion, in nanogramms;
- m_1 the mass loss from permeation tube, in milligrams per minute;
- m_2 the mean observed exposure dose (type A samplers) or mean mass uptake (type B samplers) of samplers exposed to a 30 min pulse (see 7.2.2);
- m_3 the mean observed exposure dose (type A samplers) or mean mass uptake (type B samplers) of samplers exposed to a 30 min pulse plus 7,5 h of clean air (see 7.2.2);
- M the molar mass of analyte, in grams per mol;
- p the actual pressure of the atmosphere sampled, in kilopascals;
- t the exposure time, in minutes;
- T the temperature of the atmosphere sampled, in Kelvin;
- U the diffusive uptake rate, in cubic centimetres per minute;
- U_1 the diffusive uptake of analyte 1;
- U_2 the diffusive uptake of analyte 2;
- U' the diffusive uptake rate, in nanogramms per parts per million (volume/volume) per minute ($\text{ng ppm}^{-1} \text{min}^{-1}$);
- \dot{V} the volumetric flow of air, in cubic metres per minute;
- δ the bias;
- ρ the delivered concentration, in milligrams per cubic metre;
- ρ_1 the concentration of the given analyte at the beginning of the diffusion layer ($l = 0$), in milligrams per cubic meter (see annex B.1);
- ρ_2 the concentration of the given analyte at the end of the diffusion layer, in milligrams per cubic meter (see annex B.1);
- ϕ the delivered concentration in parts per million (volume/volume);
- 24,0 the molar volume in litres per mol at 293 K and 101 kPa.

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5 Requirements

5.1 Desorption efficiency (for type B 1 samplers)

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

5.2 Desorption efficiency (for type B 2 samplers)

When tested in accordance with 7.1.2, the desorption efficiency shall be $\geq 0,95$ with a coefficient of variation of $\leq 0,1$ at each loading.

5.3 Storage (for type B samplers)

When tested in accordance with 7.9.1, the mean value of the recovery after storage shall not differ more than 10 % from the value before storage.

5.4 Overall uncertainty

When tested in accordance with 7.2, 7.7 and 7.8 the overall uncertainty calculated in accordance with 7.13 and 7.14 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 7.4, the blank value shall be less than one-third of the calculated mass uptake by the sampler for 30 min exposure to 0,1 LV.

5.6 Sampler leak test (only samplers not thermally sealed)

When tested in accordance with 7.10, 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 Uptake rate value (for type B samplers)

If it is possible to calculate the steady-state value in accordance with 7.3.2, the nominal uptake rate value, determined in accordance with 7.3.1, shall be within ± 25 % of the ideal steady-state value.

5.8 Effect of air velocity/sampler orientation

The minimum air velocity and influence of sampler orientation given in the manufacturer's instructions for use shall be verified in accordance with 7.6.

5.9 Shelf life (for type A samplers)

The shelf life of the diffusive sampler in the original package shall be as specified by the manufacturer. At the end of the shelf life the results obtained in accordance with 7.11 shall not differ by more than 10 % from the original values.

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5.10 Mechanical strength (for type A samplers)

Following the tests in 7.12, the diffusive samplers shall maintain their physical integrity and the results obtained in accordance with 7.13 and 7.14 shall not differ by more than 10 % from the original values.

5.11 Instructions for use

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

- a) uptake rate (measured or calculated) for a specific analyte;
- b) designated use including level of evaluation;

- c) direction for proper handling of the diffusive sampler including opening and closing;
- d) general information on the system e. g. sorbent;
- e) 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;
- f) response time;
- g) information on the calculation of results including evaluation of reading and correction factors for temperature and desorption efficiency, if applicable;
- h) information on storage and transport;
- i) minimum air velocity;
- j) desorption efficiency;
- k) information on the desorption method.

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

5.12 Sample identification

The diffusive sampler 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 polytetrafluoroethylene (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.

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

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 pumped sorbent tube, a bubbler method, or a different diffusive sampler method; or

b) by using an independently calibrated on-line instrument, e. g. a flame-ionization detector, an infrared analyzer or mass spectrometer.

6.4 Generation of a 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 7.3, 7.7 and 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 should not be taken to imply a preference for permeation systems for generating calibration gas mixtures of gases and vapours.

This value is the calculated inlet value of the exposure chamber concentration.

Make a correction to take account of the analyte removed by the exposed diffusive samplers, as these will remove amounts of analyte proportional to their number and sampling rate. The reduced value is the calculated outlet value of the exposure chamber concentration.

If the sampling rate of the diffusive samplers is not known in advance, so that the above correction cannot be made, then the delivered concentrations at the inlet and outlet of the exposure chamber shall be determined experimentally as in 6.5.

Determine whether the calculated or determined outlet concentration differs by more than 5 % from the inlet concentration. If it does, then the generation system shall be changed, e. g. by increasing the flow rate or chamber volume, until the difference is less than 5 %.

When the difference is less than 5 %, calculate the mean calculated value either from the mean of the calculated inlet and outlet values, or from the mean calculated inlet value adjusted for (half of) the experimentally determined depletion.

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 ± 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 7.7.2 and 7.8.2.

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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

If not otherwise specified in the test procedure, the sampler orientation shall be as specified by the manufacturer.

Set up a calibration gas mixture of the test analyte as in 6.4.1 but include at least six test (diffusive) samplers, as well as the sampler(s) of the independent method. In addition, measure the concentration of the generated atmosphere using two additional samplers of the independent method located one at the inlet and one at the outlet of the exposure chamber.

NOTE 1: The two additional samplers are unnecessary if the diffusive samplers are calculated to deplete the atmosphere by less than 5 % of the delivered atmosphere concentration.

NOTE 2: In each case, where a "nominal exposure time" of 8 h is specified, 8 h is a guideline and may be reduced to ≥ 6 h if circumstances demand.

7 Test methods

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

7.1 Desorption efficiency

7.1.1 Tube spiking method (for type B 1 samplers)

7.1.1.1 Determination

Conduct the determination at four different loadings, corresponding to exposure doses within the range 0,1 LV (30 min exposure) to 2 LV (8 h exposure). Add a known mass of analyte to the sorbent in the diffusive sampler. Use a calibrated syringe in the range 1 μ l to 10 μ l, diluting in a non-interfering solvent if necessary. The analyte shall either be applied directly to the sorbent or be allowed to diffuse from a spiked glass-fibre filter in a closed system. 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 Calculation

Calculate the exposure loadings by multiplying the exposure dose by the diffusive uptake rate (see 7.3.2) or use a manufacturer's value.

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 Tube spiking method (for type B 2 samplers)

7.1.2.1 Determination

Add a known mass of analyte to the sorbent as in 7.1.1 but prepare three tubes at each level. Thermally desorb the analyte, either as recommended by the manufacturer, or using any other suitable conditions.

7.1.2.2 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.

7.1.3 Phase equilibrium method (for type B 1 samplers)

7.1.3.1 Determination

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

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