BRITISH STANDARD

BS EN 13528-2:2002

Ambient air quality — Diffusive samplers for the determination of concentrations of gases and vapours — Requirements and test methods —

Part 2: Specific requirements and test methods

The European Standard EN 13528-2:2002 has the status of a British Standard

ICS 13.040.20

British Standards

National foreword

This British Standard is the official English language version of EN 13528-2:2002.

The UK participation in its preparation was entrusted by Technical Committee EH/2, Air quality, to Subcommittee EH/2/3, Ambient atmospheres, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

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Contents

Foreword

This document (EN 13528-2:2002) has been prepared by Technical Committee CEN/TC 264 “Air Quality”, 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of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.

This European Standard is a multi-part standard having the following parts:

- Part 1: (Ambient Air Quality) General requirements;
- Part 2: (Ambient Air Quality) Specific requirements and test methods;
- Part 3: (Ambient Air Quality) Guide to selection, use and maintenance.

The annexes A and B are normative, annex C is informative.

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

Introduction

This European Standard specifies requirements and test methods for the determination of performance characteristics of diffusive samplers used for the determination of concentrations of gases and vapours in ambient atmospheres.

With regard to air quality, the objectives fixed in the 5th Action Program of the European Union are for the effective protection of all people against recognised risks from air pollution and the establishment of permitted concentration levels of air pollutants, which should take into account the protection of the environment. These objectives include monitoring and control of concentrations with regard to standards.

Successive programs of action of the European Union on the protection of the environment have stressed the need to find a balance between the use of different tools: product standards, emission limits and environmental objectives/standards.

The implementation of existing Directives has highlighted the existence of various problems, which are being addressed in the Council Directive on Ambient Air Quality Assessment and Management [1]. These include:

- the different monitoring strategies in comparable situations between and within Member States;
- the harmonisation of measuring methods;
- the quality of the measurements which depends on the calibration and quality assurance procedures.

Diffusive samplers used to measure air quality have to fulfil some general requirements, which are given in EN 13528-1. These requirements include unambiguity, selectivity and Data Quality Objectives, including uncertainty.

Such general requirements can also be appropriate for other measurement procedures used in the assessment of ambient air quality.

In addition, diffusive samplers used to measure ambient air quality have to also fulfil some specific requirements in addition to those specified in EN 13528-1. These specific requirements are given in this part of EN 13528 (see clause 5). prEN 13528-3 gives guidance on the selection, use and maintenance of diffusive samplers used to measure ambient air quality.

It is the user's primary responsibility to choose appropriate procedures or devices that meet the requirements of this European Standard. One way of doing this is to obtain information or confirmation from the manufacturer. Type testing, or more generally, the assessment of performance criteria of procedures or devices, can be undertaken by the manufacturer, user, test house or research and development laboratory, as is most appropriate.

Although this standard specifically addresses ambient air, diffusive sampling is also relevant to the assessment of air quality in indoor air. Both pumped and diffusive sampling procedures are considered appropriate for such measurements, depending on circumstances (particularly any requirement for time resolution) [2]. prEN 14412 gives guidance on the selection, use and maintenance of diffusive samplers used to measure indoor air quality.

This part of EN 13528 is similar in content to EN 838 and EN 13528-1 is similar in content to EN 482. The series of standards on the use of diffusive samplers for ambient air has been created in addition to those for workplace air because the underlying European Directives are different and the consequent definitions and practical applications of the estimation of the uncertainty of measurements are different.

1 Scope

This European Standard specifies specific performance requirements and test methods under prescribed laboratory and field conditions for diffusive samplers used for the determination of the concentration of gases or vapours in ambient air.

Such requirements apply to all diffusive samplers, irrespective of the physical nature of the rate-controlling process and irrespective of the nature of the sorption process and the analytical determination.

This standard applies to all stages of the measuring procedure, including transportation and storage of the sample where appropriate.

This standard applies to measuring procedures with separate sampling and analysis stages, and also to direct-reading devices.

This European Standard is applicable to diffusive samplers according to 3.6 of EN 13528-1:2002.

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 13528-1. It is the responsibility of the manufacturer or of those who assemble the diffusive samplers to ensure that the sampler complies with the data quality objectives given in this European Standard.

For the purpose of demonstrating compliance with data quality objectives prescribed for methods to be used in support of the Council Directive on Ambient Air Quality Assessment and Management, this part of EN 13528 provides the relevant requirements and test methods for the determination of measurement uncertainty. As indicated in 6.5.2 to 6.5.4, some of these procedures are to be undertaken by a single representative laboratory, some by several laboratories in an interlaboratory trial, and some by the laboratory implementing the method. This division of responsibility is in accordance with Table 1 of CEN Report CR 14377:2002 [3].

Where only one laboratory is involved in establishing the performance characteristics of a method, as in a manufacturer’s assessment, the reproducibility elements of the uncertainty budget shall be estimated under within-laboratory reproducibility conditions, i.e. by performing the relevant experiments independently, as far as reasonably practicable. This type of assessment is identified as level 2 (see clause 8).

This standard can encourage the development of new types of diffusive sampler. Prior to conducting a full evaluation according to this standard, it would be useful to conduct laboratory or field intercomparisons with established samplers. These intercomparisons should be conducted by experienced institutes.

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 (including amendments).

ENV 13005:1999, *Guide to the expression of uncertainty in measurement.*

ISO 5725, *Accuracy (trueness and precision) of measurement methods and results (all parts).*

3 Terms and definitions

For the purpose of this European Standard the following terms and definitions apply.

3.1
ambient air
outdoor air in the troposphere, excluding indoor air and workplaces

3.2
averaging time
period of time for which the measuring procedure yields a single value [EN 482]

3.3
bias
difference between the expectation of the test results and an accepted reference value [ISO 3534-1]

3.4
combined standard uncertainty
standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities [ENV 13005]

3.5
desorption efficiency
ratio of the mass of analyte desorbed from a sampling device to that applied [EN 838]

3.6
diffusive sampler
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 a porous material and/or permeation through a membrane, but which does not involve the active movement of air through the device

NOTE 1 Active normally refers to the pumped movement of air.

NOTE 2 This definition differs from that in EN 838:1995 by the addition of the words “or a porous material”.

3.7
diffusive uptake rate
rate at which the diffusive sampler collects a particular gas or vapour from the atmosphere, expressed in picograms per parts per billion[1] per minute (pg ppb^{-1} min^{-1}) or cubic centimetres per minute (cm^3 min^{-1})

NOTE 1 pg ppb^{-1} min^{-1} are equivalent to ng ppm^{-1} min^{-1}.

NOTE 2 This definition differs from that in EN 838:1995 by the substitution of “picograms per parts per billion” for “nanograms per parts per million”. The expression is numerically the same, but ambient concentrations are usually in the ppb range.

3.8
expanded uncertainty
quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand

NOTE 1 The fraction may be viewed as the coverage probability or level of confidence of the interval.

NOTE 2 To associate a specific level of confidence with the interval defined by the expanded uncertainty requires explicit of implicit assumptions regarding the probability distribution characterised by the measurement result and its combined standard

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[1] ppb is volume fraction, $(\phi)=10^{-9}$; ppm is volume fraction, $(\phi)=10^{-6}$.

uncertainty. The level of confidence that can be attributed to the interval can be known only to the extent to which such assumptions may be justified.

NOTE 3 Expanded uncertainty is termed *overall uncertainty* in ENV 13005.

[ENV 13005]

3.9
measuring procedure
procedure for sampling and analysing one or more pollutants in ambient air and including storage and transportation of the sample

3.10
nominal uptake rate
the diffusive uptake rate determined experimentally under specified conditions

3.11
repeatability conditions
conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time [ISO 3534-1]

3.12
reproducibility conditions
conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment [ISO 3534-1]

3.13
standard uncertainty
uncertainty of the result of a measurement expressed as a standard deviation [ENV 13005]

3.14
uncertainty (of measurement)
parameter, associated with the results of a measurement, that characterises the dispersion of values that could reasonably be attributed to the measurand

NOTE 1 The parameter may be, for example, a standard deviation (or given multiple of it), or the half width of an interval having a stated level of confidence.

NOTE 2 Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of a series of measurements and can be characterised by experimental standard deviations. The other components, which can also be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information.

NOTE 3 It is understood that the result of a measurement is the best estimate of the value of a measurand, and that all components of uncertainty, including those arising from systematic effects, such as components associated with corrections and reference standards, contribute to this dispersion [ENV 13005].

Attention is drawn to the fact that the terms Assessment, Limit Value and Pollutant are defined in Directive 96/62/EC [1].

4 Symbols and abbreviations

A	cross-sectional area of the diffusion path, or equivalent sorption surface, in square centimetres;
C	observed concentration, in micrograms per cubic meter;
C'	observed concentration, in parts per billion (volume fraction = 10^{-9});
D	diffusion coefficient of analyte, in square centimetres per minute;

D_1	diffusion coefficient of analyte 1, in square centimetres per minute;
D_2	diffusion coefficient of analyte 2, in square centimetres per minute;
b_i	blank level, expressed as a percentage of the concentration level, *i;*
d	desorption efficiency;
l	length of static air layer in sampler (or equivalent for permeation types), in centimetres;
m_b	mass of the analyte which is desorbed from the blank sampler, in picograms;
m_d	mass of the analyte which is desorbed from exposed samplers, in picograms;
m_s	mass of the analyte which is sorbed by diffusion, in picograms;
m_1	mass loss from permeation tube, in micrograms per minute;
m_2	mean exposure dose in parts per billion (volume fraction = 10^{-9}) minutes (direct-reading samplers) or mean mass uptake in picograms (indirect-reading samplers) of samplers exposed continuously to an intermediate concentration (see 7.3);
m_3	mean exposure dose in parts per billion (volume fraction = 10^{-9}) minutes (direct-reading samplers) or mean mass uptake in picograms (indirect-reading samplers) of samplers exposed to alternately 2 x the intermediate concentration and clean air (see 7.3);
P	pressure of the sampled atmosphere during sampling, in kilopascals;
t	exposure time, in minutes;
U	diffusive uptake rate, in cubic centimetres per minute;
U_1	diffusive uptake rate of analyte 1, in cubic centimetres per minute;
U_2	diffusive uptake rate of analyte 2, in cubic centimetres per minute;
U'	diffusive uptake rate, in picograms per parts per billion (volume fraction = 10^{-9}) per minute (pg ppb^{-1} min^{-1});
V	volumetric flow rate of air, in cubic meters per minute;
x_i	mean result for test (subscript) at concentration level i*;*
δ	bias;
ρ	delivered concentration, in micrograms per cubic meter;
ρ_i	test concentration, at level i, in micrograms per cubic meter;
σ_i	standard deviation for test (subscript) at concentration level *i;*
ϕ	delivered concentration in parts per billion (volume fraction = 10^{-9}).

5 Requirements

5.1 Desorption efficiency (for samplers involving solvent desorption)

For measurements above the upper assessment threshold (see Directive [1] or equivalent), the desorption efficiency shall be $d \geq 0{,}75$ and its repeatability standard deviation shall be better than 0,1 at each loading when tested in accordance with 7.2.1. For measurements below the upper assessment threshold (see annex A of EN 13528-1:2002 or equivalent), the desorption efficiency shall be $d \geq 0{,}5$ and its repeatability standard deviation shall be better than 0,2 at each loading when tested in accordance with 7.2.1.

5.2 Desorption efficiency (for samplers involving thermal desorption)

The desorption efficiency shall be $d \geq 0{,}95$ and its repeatability standard deviation shall be better than 0,1 at each loading when tested in accordance with 7.2.2.

5.3 Effect of air velocity/sampler orientation

The acceptable range of air velocity and the influence of sampler orientation given in the manufacturer's instructions for use shall be verified in accordance with 7.4.

NOTE 1 Samplers can be used in situations where the wind speed may periodically be lower than the minimum required velocity or above the maximum permissible velocity, provided that the actual wind speed is measured, but the measurement should be regarded as semi-quantitative. If the combined periods below and above the required velocities are less than 10 % of the whole averaging time, then the result is likely to be close to the true value. If the combined periods are more than 50 % of the whole averaging time, then the results should be discarded.

NOTE 2 Information on the potential effects of air velocity and sampler orientation are given in 7.5.4 and clause 8 of prEN 13528-3.

5.4 Storage (indirect-reading samplers)

The mean value of the recovery after storage shall not differ more than 10 % from the value before storage when tested in accordance with 7.6.

5.5 Shelf life

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 7.7 shall not differ by more than 10 % from the original values.

5.6 Sampler integrity

When tested in accordance with 7.8, any analyte determined above its blank value (see 5.9) shall be less than one-third of the calculated mass uptake by the sampler as calculated in 5.9.

5.7 Blank value

When tested in accordance with 7.9 (and 7.10), the blank value shall be less than one-third of the calculated mass uptake by the sampler for an exposure at the lowest concentration specified in 6.10 of EN 13528-1:2002 and for a typical averaging time. In addition, the standard deviation of the blank value shall be less than one-tenth of the calculated mass uptake by the sampler the same exposure conditions.

NOTE The limit of detection is normally expressed as three times the standard deviation of the blank value and the limit of determination is normally expressed as ten times the standard deviation of the blank value.

5.8 Field tests

If the measurement uncertainty under field conditions is significantly greater than that determined under laboratory repeatability conditions, then the results of the laboratory tests should be reviewed to identify any possible sources of additional uncertainty.

If these additional sources of uncertainty can be identified, further tests shall be conducted to estimate the additional uncertainty components.

If these additional sources of uncertainty cannot be identified, then the additional field uncertainty shall be added to the uncertainty budget calculated according to 8.9.

5.9 Uncertainty

Measurement uncertainty is determined according to the tests in 7 and calculated (at a 95 % confidence interval) in accordance with clause 8. The specification of uncertainty shall be as required by the measurement task. Measurement tasks which are appropriate to the implementation of the Council Directive on Ambient Air Quality Assessment and Management [1] are given in annex A of EN 13528-1:2002.

5.10 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 (manufacturer’s value, or measured according to A.1, or calculated according to A.2) for the substance of interest and any known dependence on concentration and/or averaging time;

b) designated use;

c) directions 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 uncertainty above the level specified in this European Standard;

f) response time, where appropriate;

g) information on the calculation of results including use of correction factors, if applicable, and evaluation of reading for direct-reading samplers;

h) information on storage and transport;

i) minimum and maximum air velocity;

j) desorption efficiency and any known dependence on concentration and/or loading;

k) information on the desorption method.

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

5.11 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

6.2.2 A dynamic system for generating, pre-mixing and delivering a known concentration of a test gas or vapour in air.

NOTE Information on suitable procedures is given in [4].

6.2.3 An exposure chamber, constructed of inert materials such as glass, polytetrafluoro-ethylene (PTFE) or stainless steel, depending on the substance to be measured, through which the generated atmosphere is passed and of sufficient capacity to accommodate simultaneously at least twenty-four test samplers and six samplers of an independent method (6.3) or one independently calibrated instrument (6.3) positioned in such a manner that there is no interference between each sampler.

NOTE Information on the construction of a suitable chamber is given in prEN 13528-3.

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 and the concentration of any added interferent.

6.2.5 Instruments for analysing the collected samples from 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 traceable to national standards or shall be verified as follows:

a) by a reference method described in a CEN or ISO standard ; or

b) by any other method which can be demonstrated to give results equivalent to the reference method.

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 6.5.1. Ensure that the flow rate through the exposure chamber exceeds the combined sampling rate of all the test and independent method samplers and that equilibrium conditions have been achieved.

NOTE Generation of standard atmospheres is considered impractical below 5 °C and above 80 % relative humidity and provision for testing outside the above ranges (if appropriate for the intended use of the sampler) is included in 6.5.5.

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:

$$\rho = \frac{\dot{m}_1}{\dot{V}} \qquad (1)$$

NOTE The example given does not imply a preference for permeation systems for generating calibration mixtures of gases and vapours.

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

Determine the concentrations at the inlet and outlet of the exposure chamber using the independent method 6.3, with all samplers within the test chamber, including both the test and independent methods, functioning.

Determine whether the outlet concentration differs by more than 5 % from the inlet concentration. If a decision is not possible, due to inaccuracies in the independent measurement, then the difference shall be calculated from the amounts of the analyte removed by the exposed test samplers, as these will remove amounts of analyte proportional to their number and sampling rate.

If the difference is greater than 5 %, then the generation system shall be modified until the difference is less than 5 %.

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

Determine the mean concentration of the atmosphere within the exposure chamber experimentally as in 6.5.1, 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 accepted reference value of the delivered concentration.

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 accepted reference value.

6.5 Test conditions

6.5.1 General

If not otherwise specified in the test procedure, the tests shall be conducted at the concentration levels specified in 6.10 of EN 13528-1:2002.

If not otherwise specified in the test procedure, the tests shall be conducted over a range of exposure times appropriate for the practical application of the device. For example, the minimum, the intermediate and the maximum values of the exposure time could be 1 week, 2 weeks and 4 weeks respectively.

If not otherwise specified in the test procedure, the sampler orientation shall be as specified by the manufacturer and the air velocity shall be in the range of the plateau area obtained in test 7.4.

If not otherwise specified in the test procedure, the temperature shall be (20 ± 2) °C, the relative humidity shall be (50 ± 5) %.

The ranges of concentration, temperature and the other varied parameters are guidelines. If it is known that the test device is to be used in wider or more restricted ranges, the test ranges shall be adjusted accordingly.

6.5.2 Laboratory tests under repeatability conditions involving standard atmospheres (7.3, 7.4, 7.7, 7.8 and annex A)

Tests 7.3, 7.4, 7.7 and 7.8 are conducted only by one representative laboratory. If the nominal uptake rate is determined experimentally (annex A) this shall be done by all laboratories. Set up a calibration gas mixture of the test analyte as in 6.4.1 but include at least six test samplers, as well as six samplers of the independent method (6.3) or one independently calibrated instrument (6.3).

6.5.3 Laboratory test under reproducibility conditions involving standard atmospheres (7.5)

Set up a calibration gas mixture of the test analyte as in 6.4.1 but include at least twenty-four test samplers, as well as six samplers of the independent method (6.3) or one independently calibrated instrument (6.3). The test samplers shall be provided by at least four different representative laboratories, which shall analyse their own samplers after exposure in the calibration gas mixture.

6.5.4 Laboratory tests not involving standard atmospheres (7.2, 7.6, 7.9)

Tests 7.2 and 7.9 are conducted by all laboratories implementing this standard. Test 7.6 is conducted by one representative laboratory. All tests are based on concentrations and other exposure parameters equivalent to those in the tests 6.5.2 and 6.5.3.

6.5.5 Field tests

Select an appropriate test site or sites representative of the intended field of application of the diffusive sampler. Consideration should be given to location (urban, rural, background), topography (soil covering, degree of afforestation), environmental conditions (temperature, humidity, wind speed) and likely chemical interferences. Within the possible ranges of these variables, at least two tests, conducted by one laboratory, should be employed which are representative of the practical extremes likely to be encountered in the intended field of application of the diffusive sampler.

For each test, expose for an intermediate time (6.5.1) at least six samplers in an array such that so far as is practicable, they all sample from the same atmosphere. Also subject six samplers to all procedures except for the actual period of exposure and label as field blanks.

NOTE Guidance on the likely effects of environmental conditions on sampler performance is given in 7.5 of prEN 13528-3:2002. The field tests are designed to identify additional errors arising from the use of the in the field that are not adequately covered by the laboratory tests.

7 Test methods

7.1 General

If it is known in advance that a certain type of diffusive sampler is unaffected by an environmental influence, then the relevant tests in 7.2 to 7.10, shall be modified to examine only the factors likely to have an influence.

NOTE Because it is known that diffusive uptake rates (when expressed in $pg.ppb^{-1}.min^{-1}$) are independent of atmospheric pressure, and (when expressed in $ml.min^{-1}$) are nearly independent of atmospheric pressure, a pressure test is not necessary.

See clause 5 for performance requirements.

7.2 Desorption efficiency

7.2.1 Spiking method for samplers involving solvent desorption

7.2.1.1 Determination

Conduct the determination at loadings corresponding to the exposure concentrations and the intermediate averaging time as specified in 6.5.1. Prepare 6 samplers at each loading.

Add a known mass of analyte to the sorbent in the test sampler. Use a calibrated syringe in the range 1 µl to 10 µl, 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 auxiliary support such as 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. Analyse the solution in triplicate by reference to liquid standards prepared directly.

NOTE The effect of interferents on desorption efficiency is not tested directly, but, if important, will become apparent in test 7.5.1.

7.2.1.2 Calculation

Calculate the desorption efficiency, by dividing the mean mass recovered at each loading from the spiked samples by the mass introduced. Also calculate the standard deviation of replicates.

7.2.2 Spiking method for samplers involving thermal desorption

7.2.2.1 Determination

Add a known mass of analyte to the sorbent as in 7.2.1. Thermally desorb the analyte, either as recommended by the manufacturer, or using any other suitable conditions.

7.2.2.2 Calculation

Calculate the desorption efficiency, by dividing the mean mass recovered at each loading from the spiked samples by the mass applied, and the standard deviation of replicates.

7.2.3 Phase equilibrium method (samplers involving solvent desorption)

7.2.3.1 Determination

Prepare two identical sets of solutions corresponding to the loadings of 7.2.1 in volumes of desorption solvent as used for the desorption of samples. To each solution in the second set add the sorbent from an unspiked sampler and allow to equilibrate for at least 30 min. Analyse both sets of solutions in triplicate by reference to liquid standards prepared directly.

7.2.3.2 Calculation

Calculate the desorption efficiency by dividing the concentrations of 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 given in 7.2.3 is less than 95 % or if the desorption efficiency measured at any one level is less than 90 %, only the test given in 7.2.1 shall be used.

NOTE A desorption efficiency of less than 95 % frequently occurs with mixtures of polar and non-polar compounds [9].

7.3 Bias due to the selection of a non-ideal sorbent (for samplers relying on reversible sorption)

7.3.1 Procedure

Expose test samplers in a set of at least six replicates to a calibration gas mixture under the exposure conditions specified in 6.5.1 but for an intermediate concentration and intermediate exposure time only. Expose a second set of identical samplers alternately to a concentration double that of the first set and clean air for the same total exposure time. The second exposure regime should cycle every 24 hours, i.e. 12 hours at 2 times the intermediate concentration and 12 hours in clean air.

NOTE Diffusive samplers will normally be unbiased, since they are calibrated against a calibration gas mixture. However, bias can result from the use of non-ideal sorbents [10]. This test estimates the magnitude of any bias due to back diffusion.

7.3.2 Calculation

Calculate the measurement bias, δ, using the following equation:

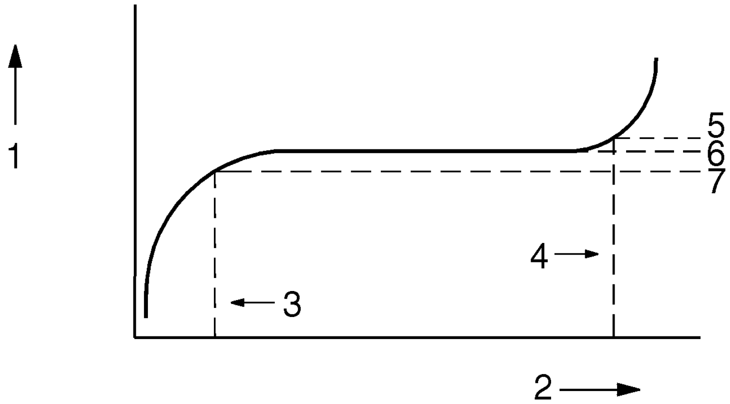
$$\partial = \frac{m_2 - m_3}{m_2} \qquad (2)$$

Also determine the standard deviations of the estimations of m_2 and m_3.

7.4 Test on influence of air velocity and sampler orientation

Expose at least six test samplers in a calibration gas mixture of a suitable analyte (see 6.4) over a range of air velocities and sampler orientations. Vary the air velocity between 0,1 ms^{-1} and 2,0 ms^{-1} and the orientation to be the positions that offer the maximum and minimum resistance to the airflow. Use the other conditions as in annex A.

Analyse the test samplers by reference to standard solutions prepared gravimetrically or to standard samplers spiked with known amounts of analyte.



Key
1 Measured concentration *c*
2 Air velocity across sampler
3 Minimum velocity
4 Maximum velocity
5 110 % of C_{plateau}
6 C_{plateau}
7 90 % of C_{plateau}

Figure 1 — Typical relationship between air velocity and measured concentration for diffusive samplers

Calculate the measured concentration (see annex B) and plot the mean value against air velocity, assuming linear flow. Figure 1 gives a typical relationship, with a plateau at mid-range air velocities (see EN 13528-3 for more details). Determine the air velocity corresponding to measured concentrations of 90 % and 110 % of its plateau value for each sampler orientation.

Test the samplers and use under conditions where air velocities are in the range of the plateau area.

As the influence of air movement on diffusive sampler performance is dependent on sampler geometry and not on the analyte selected, it is necessary to perform this test only on a given diffusive sampler with one typical analyte.

7.5 Effect of environmental parameters on sampler performance (uptake rate)

7.5.1 Procedure

Two sets of samples shall be generated. Each set involves the generation of traceable or verified standard atmospheres (6.4) at concentrations as specified in 6.5.1. For each set, the values of temperature, humidity, concentration, averaging time, orientation of the diffusive sampler and presence/absence of interferent are set at the extremes of the ranges specified below. For one set (series A) the values of these parameters are set such that a low measurement result is anticipated. For the other set (series B) the values of these parameters are set such that a high measurement result is anticipated. For example, if it is known that the diffusive sampling rate increases with ambient temperature, set A is set at 10 °C and set B at 30 °C.

NOTE For a typical diffusive sampler, a lower uptake rate will be observed with increasing concentration, averaging time, relative humidity or interferent and with decreasing temperature.

— concentration range:	as specified in 6.5.1
— averaging time:	maximum, minimum, value;
— relative humidity:	(20 ± 5) %, (80 ± 5) %;
— temperature:	(10 ± 2) °C, (30 ± 2) °C;
— air velocity:	acceptable (‘plateau’) range as specified in 7.4;
— interferent:	presence, absence of interferent.

Analyse the test samplers by reference to standard solutions prepared gravimetrically or to standard samplers spiked with known amounts of analyte.

7.5.2 Calculation

For each exposure combination, and for the samples of each participating laboratory, calculate the measured concentration (see annex B) for each of the six (or more) replicate test samplers. Divide each by the relevant calibration gas mixture concentration (accepted reference value, see 6.4; ρ or ϕ).

7.6 Storage (for indirect-reading samplers)

7.6.1 Procedure

Spike two sets of six samplers directly with an equivalent dose as in 7.2.1. Add an amount of liquid water equivalent to an exposure to air at 80 % relative humidity at a temperature of 20 °C for the appropriate time. Analyse one set within one day and the other set after two weeks' storage at room temperature, or as otherwise directed by the manufacturer. Also store and analyse six blank tubes in the same way.

The amount of liquid water to be added may be calculated from data supplied by the manufacturer. In the absence of such data or if there is prior evidence that the effect of liquid water on sampler storage is different to that of water vapour, samplers shall be exposed to clean air at 20 °C and a relative humidity of 80 % before spiking with the test analyte.

7.6.2 Calculation

For each pair of tests calculate the means for the two sets of test results. For the blank test, also calculate the standard deviation of the blank for the stored samples. The unstored blank samples are the same test as 7.5.

7.7 Shelf life

The test sampler shall be stored at the maximum storage temperature specified by the manufacturer. At the nominal end of the specified shelf life, that is, within three months of the end in order to allow time for tests, the sampler is tested according to 7.5, but only for the series B set of conditions and under repeatability conditions.

7.8 Sampler leak test

Expose sealed (unopened) samplers in a set of at least six replicates in an atmosphere of test analyte corresponding to ten times the intermediate concentration specified in 6.10 of EN 13528-1:2002 for an intermediate averaging time (6.5.1).

Analyse the set to determine the sampler integrity.

It is only necessary to perform this test on a given diffusive sampler for one typical analyte.

7.9 Determination of the blank value

Analyse unexposed samplers, in replicates of six, to determine the mean blank value of analyte. Also determine the standard deviation of the blank value.

7.10 Field tests

Expose samplers as in 6.5.5. Analyse the samplers and calculate the measured exposure concentration for each sampler. For diffusive samplers, use the calculation given in annex B. Also analyse the field blanks and determine the mean value and the standard deviation.

8 Calculation of uncertainty

8.1 General

The combined standard uncertainty shall be calculated as a combination of individual standard uncertainties, both of Type A and Type B as appropriate (see 3.7 of EN 13528-1:2002), according to ENV 13005.

The expanded uncertainty shall be calculated from the combined uncertainty, according to ENV 13005 for a level of confidence of 95 %. In most instances, the coverage factor may be assumed to be 2. The more general case is described in ENV 13005:1999, 6.3.

Contributions to the uncertainty budget are given in 8.2 to 8.9 below.

8.2 Desorption efficiency

For each concentration level, ρ_i, the mean desorption efficiency, d, is d_i with a standard deviation of σ_{di}.

If a correction is made for the desorption efficiency at the appropriate load level then the contribution to the uncertainty budget is σ_{di}

If no correction is made for the desorption efficiency at the appropriate load level, then, assuming a rectangular distribution, as in ENV 13005, Figure 2(a), the contribution to the uncertainty budget is $\surd[(1 - d_i)^2 /3 + \sigma_{di}^2]$.

8.3 Uptake rate

For each concentration level, ρ_i , and for the samples of each participating laboratory, j, then, in test 5.5:

- the mean low value, relative to the accepted reference value, is $(1 - x_{ij})$ with a standard deviation of σ_{xij};

- the mean high value, relative to the accepted reference value, is $(1 + y_{ij})$ with a standard deviation of σ_{yij};

Then, assuming a rectangular distribution, as in ENV 13005:1999, Figure 2(a), the contribution to the uncertainty budget is $0{,}5\surd[(x_{ij} - y_{ij})^2 /3 + \sigma_{xij}^2 + \sigma_{yij}^2]$.

In addition, the results of each laboratory (either the low results or the high results set) shall be evaluated according to ISO 5725-2 to determine, at each concentration level, ρ_i, the repeatability variance, s_{rij}^2, and the reproducibility variance, s_{Rij}^2. The contribution to the uncertainty budget is then the between-laboratory variance, $s_{Rij}^2 - s_{rij}^2$.

In addition, if a bias, δ, results from the test 5.2, with standard deviations σ_{m3} and σ_{m2}, the contribution to the uncertainty budget is $\surd[\delta^2 /3 + \sigma_{m3}^2 - \sigma_{m2}^2]$.

8.4 Storage

For each concentration level, ρ_i, the mean recovery after storage is x_{si}, with a standard deviation of σ_{si}. The analytical precision at zero time is σ_{0i}.

If the storage period is known, and a correction is made for the recovery at the appropriate load level, then the contribution to the uncertainty budget is $\surd(\sigma_{si}^2 - \sigma_{0i}^2)$

If the storage period is not known, and no correction is made for the recovery at the appropriate load level, then, assuming a rectangular distribution, as in ENV 13005:1999, Figure 2(a), the contribution to the uncertainty budget is $\surd[(1 - x_{si})^2 /3 + \sigma_{si}^2 - \sigma_{0i}^2]$.

8.5 Shelf life

For each concentration level, ρ_i, the mean difference between the results before and after storage is $(1 +/- x_{li})$ with a standard deviation of σ_{li}.

If the storage period is known, and a correction is made for the recovery at the appropriate load level, then the contribution to the uncertainty budget is $\surd(\sigma_{li}^2 - \sigma_{0i}^2)$.

If the storage period is not known, and no correction is made for the recovery at the appropriate load level, then, assuming a rectangular distribution, as in ENV 13005:1999, Figure 2(a), the contribution to the uncertainty budget is $\surd[(1 – x_{li})^2 /3 + \sigma_{li}^2 - \sigma_{0i}^2]$.

8.6 Sampler integrity

For each concentration level, ρ_i, the mean collected analyte, relative to the concentration level, assuming a typical uptake rate and averaging time, is $(1 + x_{qi})$ with a standard deviation of σ_{qi}.

If the storage conditions are known, and a correction is made for the ingress of analyte at the appropriate load level, then the contribution to the uncertainty budget is $\surd(\sigma_{qi}^2 - \sigma_{0i}^2)$.

If the storage period is not known, and no correction is made for the recovery at the appropriate load level, then, assuming a rectangular distribution, as in ENV 13005:1999, Figure 2(a), the contribution to the uncertainty budget is $\surd[(1 – x_{qi})^2 /3 + \sigma_{qi}^2 - \sigma_{0i}^2]$.

8.7 Blank value

For each concentration level, ρ_i, the mean blank value, expressed as a percentage of the concentration level, assuming a typical averaging time, is b_i % with a standard deviation of σ_{bi}.

If a correction is made for the blank at the appropriate load level, then the contribution to the uncertainty budget is $\sqrt{(\sigma_{bi}^2 - \sigma_{0i}^2)}$.

If no correction is made for the blank at the appropriate load level, then, assuming a rectangular distribution, as in ENV 13005:1999, Figure 2(a), the contribution to the uncertainty budget is $\sqrt{[(1 - x_{bi})^2 /3 + \sigma_{bi}^2 - \sigma_{0i}^2]}$.

8.8 Type B errors

Uncertainties due to the standard atmosphere generation and purity of reagents shall be estimated from available information.

8.9 Combined uncertainty

The combined uncertainty is calculated from a root-sum-of-squares of the appropriate individual uncertainties (8.2 to 8.8).

A worked example is in annex C.

For measuring procedures involving several component steps (6.2 of EN 13528-1:2002), the combined uncertainty is calculated from a root-sum-of-squares of the individual uncertainties of the component steps.

8.10 Field tests

If, in the field tests (6.5.5), any additional uncertainty calculated according to 5.10 cannot be identified, then the additional field uncertainty shall be added to the uncertainty budget calculated according to 8.9.

9 Levels of evaluation

There are three levels of evaluation. To comply with the minimum requirements of this European Standard evaluation to level 3 is sufficient. These levels are defined as follows:

a) Level 1: an evaluation according to the main text of this European Standard;

b) Level 2: an evaluation using only one laboratory, but maintaining reproducibility conditions where possible (see Scope);

c) Level 3: an evaluation of an analogue within an homogeneous series both upper and lower members of which have previously been tested and shown to comply to level 1 or 2. Such an evaluation shall be based on the determination of desorption efficiency (see 7.2) and diffusive uptake rate (see annex A) as a minimum.

10 Test report

The test report shall include at least the following information:

d) complete identification of the atmosphere for testing including the independent method used;

e) the type of test sampler used;

f) a reference to the standard and the method used, i.e. EN 13528-2;

g) detailed test conditions;

h) the level of evaluation;

i) the determined values for desorption efficiency and diffusive uptake rate;

j) the test results, including any statistical analysis;

k) whether the acceptance criteria are met for each test;

l) any unusual features noted during the determinations;

m) any operation not included in this European Standard that may have influence on the results;

n) the technical justification of omitting any tests (see 1);

o) details of analytical procedure.

11 Marking

Diffusive samplers shall be marked. The marking shall contain at least the following:

- manufacturer's identification;
- type identification of the diffusive sampler;
- date of expiry;
- number of this European Standard;
- batch identification;
- unique sampler identification number.

The marking may be placed on the packaging of the diffusive sampler if this is necessary due to limited space. However, manufacturer's identification, type identification and unique sampler identification number shall be indicated on the diffusive sampler.

Annex A
(normative)

Determination of diffusive uptake rates (indirect-reading samplers)

A.1 Nominal uptake rates

A.1.1 General

The uptake rate shall be a nominal one, determined experimentally under typical conditions (7.3.1), or a calculated one (7.3.2), or a manufacturer's value. For the determination of the performance characteristics of the diffusive sampler, any of these can be used in the calculation of exposure concentration (7.5.2). However, for type testing, the manfacturer's value of the uptake rate shall be used. A calculated value of the uptake rate is normally only useful for level 3 evaluations (8).

A.1.2 Procedure

Expose test samplers in an atmosphere of the test analyte under the exposure conditions specified in 6.5.1, except that only one concentration level and exposure time are required, which shall be the intermediate value of those specified:

Analyse the test samplers by reference to standard solutions prepared gravimetrically or to standard samplers spiked with known amounts of analyte.

A.1.3 Calculation

Calculate the nominal diffusive uptake rate *U*, using the following equation:

$$U = \frac{m_d - m_b}{d \times \rho \times t} \qquad (3)$$

NOTE If concentration is expressed in parts per billion, use U' and ϕ instead of U and ρ.

A.2 Calculation of uptake rates from diffusion coefficients

Calculate the mass uptake of a diffusive sampler (see prEN 13528-3) using the following equation:

$$m_s = \frac{A \times D \times \rho \times t}{l} \qquad (4)$$

Calculate the diffusive uptake rates, either from a knowledge of the physical parameters of the diffusion barrier (equation 5) or by comparison with another analyte for which the uptake rate is known (equation 6).

$$U = \frac{m_s}{\rho \times t} = \frac{A \times D}{l} \qquad (5)$$

and

$$U_1 = \frac{D_1 \times U_2}{D_2} \qquad (6)$$

NOTE If no diffusion coefficient is available, see [11] and annex C of prEN 14412.

Annex B
(normative)

Calculation of exposure concentration

B.1 Direct-reading samplers

Read the response of the sampler to the collected analyte. Convert the response to exposure dose (concentration multiplied by exposure time), if necessary by using a manufacturer's calibration graph and/or colour code. Then convert to concentration by dividing by the exposure time.

B.2 Indirect-reading samplers

Calculate the measured concentrations C, using the nominal value of the diffusive uptake rate (annex A).

$$C = \frac{m_\mathrm{d} - m_\mathrm{b}}{d \times U \times t} \qquad (7)$$

NOTE If concentration is expressed in parts per billion, use C' and U' instead of C and U.

Annex C
(informative)

Calculation of uncertainty components

Source of uncertainty	**Test / Requirement**	**Uncertainty characteristics**	**Uncertainty component**	**Typical value %**
Sampling				
Uptake rate as f(ρ)	7.5 / 5.4	8.3 Deviate < 20 %; σ < 3 %	$0,5\sqrt{[(x_{ij} - y_{ij})^2/3 + \sigma_{xi}^2 + \sigma_{yi}^2]}$.	6,15
		8.3 Between-lab < 5 %	$s_{Rij}^2 - s_{rij}^2$.	5
Back diffusion	7.3 / 5.2	8.3 Bias < 10%; σ < 3%	$\sqrt{[\delta^2/3 + \sigma_{m3}^2 - \sigma_{m2}^2]}$.	5,8
Time	-	8.8 (Type B) < 0.5 %	$\sqrt{[0,5^2/3]}$	0,3
Standard atmosphere generation	-	8.8 (Type B) < 5 %	$\sqrt{[5^2/3]}$	2,9
Air velocity	7.4 / 5.3	Qualitative		
Storage				
Recovery as f(ρ)	7.6 / 5.5	8.4 Recovery > 95 %; σ < 3 %	Correcting: $\sqrt{(\sigma_{si}^2 - \sigma_{0i}^2)}$; Not correcting: $\sqrt{[(1-x_{si})^2/3 + \sigma_{si}^2 - \sigma_{0i}^2]}$	Correcting: 0; Not correcting: 2,9
Shelf life	7.7 / 5.6	8.5 Recovery > 95 %; σ < 3 %	Correcting: $\sqrt{(\sigma_{li}^2 - \sigma_{0i}^2)}$; Not correcting: $\sqrt{[(1 - x_{li})^2/3 + \sigma_{li}^2 - \sigma_{0i}^2]}$	Correcting: 0; Not correcting: 2,9
Leak test	7.8 / 5.7	8.6 relative to mass uptake	Correcting: $\sqrt{(\sigma_{qi}^2 - \sigma_{0i}^2)}$	0
Blank	7.9 / 5.8	8.7 relative to mass uptake	Correcting: $\sqrt{(\sigma_{bi}^2 - \sigma_{0i}^2)}$	0
Extraction				
Efficiency	7.2 / 5.1	8.2 Recovery > 95 %; σ < 3 %; or Recovery	Correcting: σ_{di}; Not correcting: $\sqrt{[(1 - d_i)^2/3 + \sigma_{di}^2]}$	Not correcting: not recommended Correcting: 3 (5)

Source of uncertainty	Test / Requirement	Uncertainty characteristics	Uncertainty component	Typical value %
		> 75 %; σ < 3 %, or Recovery > 50 %; σ < 5 %		
Calibration				
Standards (reagent purity)	-	8.6 (Type B) < 1 %	$\sqrt{[1^2/3]}$	0,6
Lack of fit	-	8.6 (Type B) < 3 %	$\sqrt{[3^2/3]}$	1,7
Analysis				
Precision	(7.6)	Part of storage test σ < 3 %	σ_{0i}	1,7
Combined uncertainty (root sum of squares)				12

NOTE 1 The combined uncertainty, based on typical values of individual sources of uncertainty, is given in the table as the 'worst case', i.e. for not correcting for recovery and shelf life and for 5 % uncertainty in efficiency. According to 8.1, this gives an expanded uncertainty of 24 %. This can be compared with the minimum requirement of 25 % for fixed measurements or 30 % for indicative measurements of benzene [12].

NOTE 2 Actual individual values of uncertainty can be greater or less than the typical values. Minimum requirements for particular uncertainty sources are given in clause 5.

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