INTERNATIONAL STANDARD

ISO 16017-2

First edition
2003-05-15

Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography —

Part 2: Diffusive sampling

Air intérieur, air ambiant et air des lieux de travail — Échantillonnage et analyse des composés organiques volatils par tube à adsorption/désorption thermique/chromatographie en phase gazeuse sur capillaire —

Partie 2: Échantillonnage par diffusion

Reference number
ISO 16017-2:2003(E)

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Published in Switzerland

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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16017-2 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

ISO 16017 consists of the following parts, under the general title *Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography*:

— *Part 1: Pumped sampling*

— *Part 2: Diffusive sampling*

Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography —

Part 2: Diffusive sampling

1 Scope

This part of ISO 16017 gives general guidance for the sampling and analysis of volatile organic compounds (VOCs) in air. It is applicable to indoor, ambient and workplace air.

This part of ISO 16017 is applicable to a wide range of VOCs, including hydrocarbons, halogenated hydrocarbons, esters, glycol ethers, ketones and alcohols. A number of sorbents[1] are recommended for the sampling of these VOCs, each sorbent having a different range of applicability. Very polar compounds generally require derivatisation; very low boiling compounds are only partially retained by the sorbents and can only be estimated qualitatively. Semi-volatile compounds are fully retained by the sorbents, but may only be partially recovered.

This part of ISO 16017 is applicable to the measurement of airborne vapours of VOCs in a mass concentration range of approximately 0,002 mg/m^3 to 100 mg/m^3 individual organic for an exposure time of 8 h, or 0,3 µg/m^3 to 300 µg/m^3 individual organic for an exposure time of four weeks.

The upper limit of the useful range is set by the sorptive capacity of the sorbent used and by the linear dynamic range of the gas chromatograph column and detector or by the sample splitting capability of the analytical instrumentation used. The lower limit of the useful range depends on the noise level of the detector and on blank levels of analyte and/or interfering artefacts on the sorbent tubes. Artefacts are typically sub-nanogram for well-conditioned Tenax GR and carbonaceous sorbents such as Carbopack/Carbotrap type materials, carbonized molecular sieves such as Spherocarb and pure charcoals. Artefacts are typically at low nanogram levels for Tenax TA and at 5 ng to 50 ng levels for other porous polymers such as Chromosorbs and Porapaks.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16000-1, *Indoor air — Part 1: General aspects of sampling strategy*

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1) The sorbents listed in Annex B and elsewhere in this part of ISO 16017 are those known to perform as specified under this part of ISO 16017. Each sorbent or product that is identified by a trademarked name is unique and has a sole manufacturer; however, they are widely available from many different suppliers. This information is given for the convenience of users of this part of ISO 16017 and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

3 Principle

The diffusive sampler (or samplers) is exposed to air for a measured time period. The rate of sampling is determined by prior calibration in a standard atmosphere (see 8.6). The organic vapour migrates down the tube by diffusion and is collected on the sorbent (see Annex A). The collected vapour (on each tube) is desorbed by heat and is transferred under inert carrier gas into a gas chromatograph equipped with a capillary column and a flame ionization detector or other suitable detector, where it is analysed. The analysis is calibrated by means of liquid or vapour spiking onto a sorbent tube.

Information on possible saturation of the sorbent bed, the effect of transients and the effect of face velocity is given in Annex A. Annex A also explains the dependence of effective uptake rates on the concentration level of pollutants and the time of diffusive sampling, for non-ideal sorbents, which results in different values being given in Tables 1 and 2. Further detailed information on the theory of performance of diffusive samplers is given in prEN 13528-3 [1].

4 Reagents and materials

During the analysis, use only reagents of recognized analytical reagent grade.

Fresh standard solutions should be prepared weekly, or more frequently if evidence is noted of deterioration, e.g. condensation reactions between alcohols and ketones.

4.1 Volatile organic compounds.

A wide range of VOCs are required as reagents for calibration purposes, using either liquid spiking (4.7 and 4.8) or vapour spiking (4.4 to 4.6) onto sorbent tubes.

4.2 Dilution solvent, for preparing calibration blend solutions for liquid spiking (4.7).

The solvent should be of chromatographic quality. It shall be free from compounds co-eluting with the compound(s) of interest (4.1).

NOTE Methanol is frequently used. Alternative dilution solvents, e.g. ethyl acetate or cyclohexane, can be used, particularly if there is no possibility of reaction or chromatographic co-elution.

4.3 Sorbents, preferably of particle size 0,18 mm to 0,25 mm (60 mesh to 80 mesh).

Each sorbent should be preconditioned under a flow of inert gas by heating it overnight at a temperature at least 25 °C below the published maximum for that sorbent before packing the tubes. They shall be kept in a clean atmosphere during cooling to room temperature, storage, and loading into the tubes. Wherever possible, analytical desorption temperatures should be kept below those used for conditioning. Tubes prepacked by the manufacturer are also available for most sorbents and as such only require conditioning. Care should be taken with the disposal of the sorbents, using normal laboratory practice.

NOTE A guide for sorbent selection is given in Annex C. Equivalent sorbents may be used. A guide to sorbent conditioning and analytical desorption parameters is given in Annex D. In most cases the sorbents can be used for indoor air measurements as well as for ambient air and workplace atmosphere measurements.

4.4 Calibration standards.

Calibration standards are preferably prepared by loading required amounts of the compounds of interest on sorbent tubes from standard atmospheres (see 4.5 and 4.6), as this procedure most closely resembles the practical sampling situation.

If this way of preparation is not practicable, standards may be prepared by a liquid spiking procedure (see 4.7 and 4.8) provided that the accuracy of the spiking technique is established by one of the following methods:

a) by using procedures giving spiking levels fully traceable to primary standards of mass and/or volume;

b) confirmed by comparison with reference materials, if available;

c) confirmed by comparison with standards produced using standard atmospheres;

d) confirmed by comparison with results of reference measurement procedures.

4.5 Standard atmospheres, of known concentrations of the compound(s) of interest.

Prepare standard atmospheres by an independent method. Methods described in ISO 6141 and several parts of ISO 6145 are suitable (see Bibliography). If the procedure is not applied under conditions that allow the establishment of full traceability of the generated concentrations to primary standards of mass and/or volume, or if the chemical inertness of the generation system cannot be guaranteed, the concentrations shall be confirmed using an independent procedure.

4.6 Standard sorbent tubes, loaded by spiking from standard atmospheres.

Prepare loaded sorbent tubes by passing an accurately known volume of the calibration atmosphere through the sorbent tube, e.g. by means of a pump. The volume of atmosphere sampled shall not exceed the breakthrough volume of the analyte-sorbent combination. After loading, disconnect and seal the tube. Prepare fresh standards with each batch of samples. Prepare standard atmospheres of mass concentrations equivalent to 10 mg/m^3 and 100 µg/m^3. For workplace air, load sorbent tubes with 100 ml, 200 ml, 400 ml, 1 l, 2 l, or 4 l of the 10 mg/m^3 atmosphere. For ambient or indoor air load sorbent tubes with 100 ml, 200 ml, 400 ml, 1 l, 2 l, 4 l or 10 l of the 100 µg/m^3 atmosphere.

4.7 Solutions for liquid spiking.

4.7.1 Solution containing approximately 10 mg/ml of each liquid component.

Accurately weigh approximately 1 g of substance or substances of interest into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with dilution solvent (4.2), stopper and shake to mix.

4.7.2 Solutions containing approximately 1 mg/ml of liquid components.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution 4.7.1 Make up to 100 ml with dilution solvent, stopper and shake to mix.

4.7.3 Solution containing approximately 100 µg/ml of each liquid component.

Accurately weigh approximately 10 mg of substance or substances of interest into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with dilution solvent (4.2), stopper and shake to mix.

4.7.4 Solution containing approximately 10 µg/ml of liquid components.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution 4.7.3. Make up to 100 ml with dilution solvent, stopper and shake to mix.

4.7.5 Solution containing approximately 1 mg/ml of gas components.

For gases, e.g. ethylene oxide, a high-level calibration solution can be prepared as follows. Obtain gas at atmospheric pressure by filling a small plastic gas bag from a gas cylinder containing pure gas. Fill a 1 ml gas-tight syringe with 1 ml of the pure gas and close the valve of the syringe. Using a 2 ml septum vial, add 2 ml dilution solvent and close with the septum cap. Insert the tip of the syringe needle through the septum cap into the dilution solvent. Open the valve and withdraw the plunger slightly to allow the dilution solvent to enter the syringe. The action of the gas dissolving in the dilution solvent creates a vacuum, and the syringe fills with solvent. Return the solution to the flask. Flush the syringe twice with the solution and return the washings to the flask. Calculate the mass of gas added using the gas laws, i.e. 1 mole of gas at STP (standard temperature and pressure: 273,15 K and 1 013,25 hPa) occupies 22,4 litres.

4.7.6 Solution containing approximately 10 µg/ml of gas components

For gases, e.g. ethylene oxide, a low level calibration solution may be prepared as follows. Obtain pure gas at atmospheric pressure by filling a small plastic gas bag from a gas cylinder. Fill a 10 µl gas-tight syringe with 10 µl of the pure gas and close the valve of the syringe. Using a 2 ml septum vial, add 2 ml dilution solvent and close with the septum cap. Insert the tip of the syringe needle through the septum cap into the dilution solvent. Open the valve and withdraw the plunger slightly to allow the dilution solvent to enter the syringe. The action of the gas dissolving in the dilution solvent creates a vacuum, and the syringe fills with solvent. Return the solution to the flask. Flush the syringe twice with the solution and return the washings to the flask. Calculate the mass of gas added using the gas laws, i.e. 1 mole of gas at STP occupies 22,4 litres.

4.8 Standard sorbent tubes loaded by liquid spiking

Loaded sorbent tubes are prepared by injecting aliquots of standard solutions onto clean sorbent tubes as follows. A sorbent tube is fitted into the injection unit (5.7) through which inert purge gas is passed at 100 ml/min and a 1 µl to 4 µl aliquot of an appropriate standard solution injected through the septum. After 5 min, the tube is then disconnected and sealed. Prepare fresh standards with each batch of samples. For workplace air, load sorbent tubes with 1 µl to 5 µl of solution 4.7.1, 4.7.2 or 4.7.5. For ambient and indoor air, load sorbent tubes with 1 µl to 5 µl of solution 4.7.3, 4.7.4 or 4.7.6.

5 Apparatus

Use ordinary laboratory apparatus and the following devices.

5.1 Sorbent tubes.

These tubes shall be compatible with the thermal desorption apparatus to be used (5.6). Typically, but not exclusively, they are constructed of stainless steel tubing of dimensions 6,3 mm (1/4 in) OD, 5 mm ID and 90 mm long. Tubes of other dimensions may be used, but the uptake rates given in Table 1 are based on these tube dimensions. For labile analytes, such as sulfur-containing compounds, glass-lined or glass tubes (typically 4 mm ID) should be used. Mark one end of the tube, for example by a scored ring, about 10 mm from the (diffusive) sampling end. Pack the tubes with preconditioned sorbents so that the sorbent bed will be within the desorber heated zone and a consistent gap of about 14 mm is retained at the marked (diffusive) end of the tube.

Uptake rates in Table 1 are given for tubes with a nominal air gap (between sorbent bed and diffusive end cap) of at least 14 mm. In practice, packed tube dimensions vary[2], and tubes should be rejected where the air gap (between stainless steel screen retaining the sorbent bed and the end of the tube) is outside the range 14,0 mm to 14,6 mm.

Tubes contain between 200 mg and 1 000 mg sorbent, depending on sorbent density, which is typically about 250 mg porous polymer, or 500 mg carbon molecular sieve or graphitized carbon. The sorbents are retained by a stainless steel gauze at the diffusion end and an unsilanized glass wool plug and/or a second stainless gauze at the other end.

5.2 Sorbent tube end caps.

The tubes shall be sealed, e.g. with metal screw cap fittings with PTFE seals.

5.3 Sorbent tube end caps for sampling.

The diffusive end cap is similar to 5.2, but allows the ingress of vapour through a metal gauze, the size of the opening being the same as the cross-section of the tube.

Some versions of the end cap incorporate a silicone membrane next to the gauze.

5.4 Syringes.

A precision 10 µl liquid syringe readable to 0,1 µl, a precision 10 µl gas-tight syringe readable to 0,1 µl and a precision 1 ml gas-tight syringe readable to 0,01 ml.

5.5 Gas chromatograph, fitted with a flame ionization detector, photoionization detector, mass spectrometric or other suitable detector capable of detecting an injection of 0,5 ng toluene with a signal-to-noise ratio of at least 5 to 1, and including a gas chromatograph capillary column capable of separating the analytes of interest from other components.

5.6 Thermal desorption apparatus, for the two-stage thermal desorption of the sorbent tubes and transfer of the desorbed vapours via an inert gas flow into a gas chromatograph.

A typical apparatus contains a mechanism for holding the tubes to be desorbed whilst they are heated and purged simultaneously with inert carrier gas. The desorption temperature and time is adjustable, as is the carrier gas flow rate. The apparatus should also incorporate additional features, such as automatic sample tube loading, leak-testing, and a cold trap in the transfer line to concentrate the desorbed sample (8.2). The desorbed sample, contained in the purge gas, is routed to the gas chromatograph and capillary column via a heated transfer line.

5.7 Injection facility for preparing standards by liquid spiking.

A conventional gas chromatographic injection port may be used for preparing sample tube standards. This can be used *in situ*, or it can be mounted separately. The carrier gas line to the injector should be retained. The back of the injection port should be adapted if necessary to fit the sample tube. This can be done conveniently by means of a compression coupling with an O-ring seal.

6 Sample tube conditioning

Prior to use, tubes should be reconditioned by desorbing them at a temperature at or just above the analytical desorption temperature (see Annex D) for 10 min with a carrier gas flow of at least 100 ml/min. The carrier gas flow should be towards the diffusive sampling end to prevent recontamination of the sorbents. Tubes should then be analysed, using routine analytical parameters, to ensure that the thermal desorption blank is sufficiently small. If the blank is unacceptable, tubes should be reconditioned by repeating this procedure. Once a sample has been analysed, the tube may be reused to collect a further sample immediately. However, it is advisable to check the thermal desorption blank if the tubes are left for an extended period before reuse, or if sampling for a different analyte is envisaged. Tubes should be sealed with metal screwcaps with combined PTFE ferrule fittings and stored in an airtight container when not sampling or being conditioned.

NOTE The sorbent tube blank level is acceptable if interfering peaks are no greater than 10 % of the typical areas of the analytes of interest.

7 Sampling

Select a sorbent tube (or tubes) appropriate for the compound or mixture to be sampled. Guidance on suitable sorbents is given in Tables 1 and 2 and Annex B.

Immediately before sampling, remove the storage end cap from the marked end of the sample tube and replace it with a diffusion end cap. Make sure the diffusion cap is properly seated and the other end cap is in place.

When used for personal sampling, the tube(s) should be mounted in the breathing zone. When used for fixed-location sampling, choose a suitable site; for indoor air in accordance with ISO 16000-1. For ambient air, recommendations for site selection and for the protection of samples from adverse environmental conditions are given in Annex A and prEN 13528-3 [1]. Attention shall be paid to three main considerations: air velocity, protection from precipitation, and security. More information is given in the next paragraph, in A.5 and reference [1].

Expose the sampling tubes only under conditions where the face velocity requirement can be expected to be satisfied. For the tubes specified in 5.1 with end caps 5.3, wind speed (air velocity) has no influence. Other devices may have different requirements, including also a minimum wind speed.

Instruments to measure wind speeds as low as 0,007 m/s are not commonly available, so the wind speed may have to be measured indirectly. The user is also cautioned about the possible influence of very high wind speeds (above 12 m/s) for which performance characteristics are currently unavailable.

The recommended exposure time for the VOCs covered by this part of ISO 16017 is 8 h for workplace monitoring and four weeks for ambient and indoor air monitoring. Sampling over shorter periods is possible, down to 30 minutes for workplace monitoring and one week for ambient and indoor air monitoring, but the monitored concentration range will be changed accordingly. For example, for a 4-h sampling period, the working range is approximately 0,004 mg/m^3 to 200 mg/m^3.

NOTE The working ranges specified in the Scope (see Clause 1) for 8 h and 4 weeks are not equivalent because they depend on the choice of sorbent, different diffusive uptake rates and different practical applications.

Note and record the times, temperature and the barometric pressure at the start of the sampling period. At the end of the sampling period, again note and record the time, temperature and barometric pressure.

Replace the diffusion end cap with a storage end cap and tighten the seal securely. The tubes should be uniquely labeled. Solvent containing paints and markers or adhesive labels should not be used to label the tubes.

If samples are not to be analysed within 8 h, place them in a clean, uncoated, sealed metal or glass container.

Record air temperature and barometric pressure periodically during sampling, if it is desired to express concentrations reduced to specific conditions (91).

Field blanks should be prepared by using tubes identical to those used for sampling and subjecting them to the same handling procedure as the sample tubes except for the actual period of sampling. For the field blanks end caps are used instead of diffusion caps. Label these as blanks.

8 Procedure

8.1 Safety precautions

This part of ISO 16017 does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this part of ISO 16017 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

8.2 Desorption and analysis

8.2.1 Desorption

The sorbent tube is placed in a compatible thermal desorption apparatus. Air is purged from the tube to avoid chromatographic artefacts arising from the thermal oxidation of the sorbent or gas chromatographic stationary phase. The tube is then heated to displace the organic vapours which are passed to the gas chromatograph by means of a carrier gas stream. The gas flow at this stage shall be towards the diffusive sampling end, i.e. the marked end of the tube should be nearest the gas chromatograph column inlet. The gas flowrate through the tube should be on the order of 30 ml/min to 50 ml/min for optimum desorption efficiency. During the purge period, care should be taken to minimize heating of the tube.

NOTE 1 For the initial air purge, it is usually necessary to use 10 × the tube volume (i.e. 20 ml to 30 ml) of inert gas to completely displace the volume of air (2 ml to 3 ml) in the tube. However, if strongly hydrophilic sorbents are needed, it may be necessary to employ a larger purge to reduce sorbed air and water to prevent ice formation blocking the cold trap.

The desorbed sample occupies a volume of several millilitres of gas, so that pre-concentration is essential prior to capillary GC analysis. This may be achieved using a small, cooled, secondary sorbent trap, which can be desorbed sufficiently rapidly at low flowrates (< 5 ml/min) to minimize band broadening, and produce capillary-compatible peaks. Alternatively, an empty secondary trap, or one containing an inert material such as glass beads, may be used to preconcentrate the sample, but such traps typically require cooling to below – 100 °C. Alternatively, the desorbed sample can be passed directly to the gas chromatograph (single-stage desorption) where it shall be refocused. This typically requires a high phase ratio column (e.g. 5 µm film thickness, 0,2 mm to 0,32 mm ID) and a sub-ambient starting temperature.

If a secondary sorbent cold trap is not available and if sub-zero capillary cryofocusing temperatures are used to preconcentrate the analytes, water should be completely eliminated from the sample tube prior to desorption in order to prevent ice formation which can block the capillary tubing and stop the thermal desorption process.

NOTE 2 If a secondary cold trap is not available and optimum sample tube desorption flowrates of 30 ml/min to 50 ml/min are used, a minimum split ratio of 30:1 to 50:1 is typically required for operation with high-resolution capillary columns. Single-stage thermal desorption may thus limit sensitivity.

Desorption conditions should be chosen such that desorption from the sample tube is complete, and no sample loss occurs in the secondary trap, if used. Typical parameters are:

Desorption temp	250 °C to 325 °C
Desorption time	5 min to 15 min
Desorption flow rate	30 ml/min to 50 ml/min
Cold trap low	+ 20 °C to – 180 °C, depending on type of cold trap
Cold trap high	250 °C to 350 °C
Cold trap sorbent	typically same as tubes, 40 mg to 100 mg, if used
Carrier gas	helium
Split ratios	Split ratios between the sample tube and secondary trap and between the secondary trap and analytical column (if applicable) should be selected dependent on expected atmospheric concentration. (See guidance from respective manufacturers of the thermal desorption apparatus.)

NOTE 3 The desorption temperature depends on the analyte and the sorbent used. Recommendations for maximum desorption temperatures for particular sorbents are given in Annexes A and C. Due to their potential thermal instability, secondary and tertiary volatile amines and some polyhalogenated compounds having one or two carbon atoms specially brominated compounds may suffer some thermal degradation.

8.2.2 Analysis

Set the sample flowpath temperature (transfer line temperature) high enough to prevent analyte condensation but not so high as to cause degradation. Analytes sufficiently volatile to be present in the vapour phase in air at ambient temperature do not usually require flowpath temperatures above 150 °C. However, some types of apparatus may require higher temperatures.

Set up the gas chromatograph for the analysis of volatile organic compounds. A variety of chromatographic columns may be used for the analysis of these compounds. The choice depends largely on which compounds, if any, are present that might interfere in the chromatographic analysis. Typical examples are 50 m × 0,22 mm fused silica columns with thick-film (1 µm to 5 µm) dimethylsiloxane or a 50 m 7 % cyanopropyl, 7 % phenyl, 86 % methyl siloxane stationary phase. Typical operating conditions for these columns are a temperature programme from 50 °C to 250 °C at 5 °C/min, with an initial hold time of 10 min at 50 °C.

The capillary column, or preferably a length of uncoated, deactivated fused silica, should be threaded back through the transfer line from the thermal desorption apparatus to the gas chromatograph such that it reaches as close as possible to the sorbent in the cold trap, or as close as possible to the tube in a single-stage desorber. Internal tubing shall be inert and dead volumes shall be minimized. A split valve(s) is conveniently placed at the inlet and/or outlet of the secondary trap. The split valve on the outlet of the secondary trap may be located either at the inlet or the outlet of the transfer line. Split ratios depend on the application.

NOTE Lower split ratios are suitable for ambient (typically 1:1 to 10:1) and indoor air measurements (typically 1:1 to 20:1); higher split ratios for workplace air measurements (typically 100:1 to 1000:1).

Correspondence of retention times on a single column should not be regarded as proof of identity.

8.3 Calibration

Analyse each sorbent tube standard (4.6 or 4.8) by thermal desorption and gas chromatography.

Prepare a calibration graph by plotting the base-ten logarithm of the areas of the analyte peaks corrected for blank levels on the vertical scale against the base-ten logarithm of the mass of the analyte, in micrograms, on the sorbent tube standard corresponding to the solutions in 4.7 or atmospheres in 4.5.

8.4 Determination of sample concentration

Analyse the samples and sample blanks as described for the calibration standards in 8.3. Determine the peak area and read from the calibration graph the mass of the analyte in the desorbed sample [3].

8.5 Determination of desorption efficiency

The efficiency of desorption should be checked by comparing the chromatographic response of a sorbent tube standard (8.3) with that obtained by injecting aliquots of the standard solutions or the atmosphere directly into the gas chromatograph. Thus, prepare a second calibration graph of peak area against mass of analyte as in 8.3, but using solutions 4.7 or atmosphere 4.6. This calibration should be the same, or nearly the same, as that in 8.3. The desorption efficiency is the response of a tube standard divided by that of the corresponding liquid standard injected directly. If the desorption efficiency is less than 95 %, change the desorption parameters accordingly.

Some makes of thermal desorber do not have a direct liquid injection facility. In these cases, and when loaded tubes are prepared from a calibration blend atmosphere, desorption efficiency should be checked by comparing the calibration graph of the substance of interest (4.1) with that of n-hexane. The ratio of the slope of the calibration graph of the substance of interest relative to that of n-hexane should be the same as the relative response factor for that compound. Response factors for other compounds may be calculated approximately from effective carbon numbers[1]. If the ratio of the slopes of the calibration graphs do not agree with the relative response factor within 10 %, change the desorption parameters accordingly.

8.6 Calibration of uptake rate

The uptake rates given in Tables 1 and 2 are for tubes with the dimensions given in 5.1 without a membrane in the diffusion end cap (5.3). For other specifications, it may be necessary to follow EN 838[4] or EN 13528-2 [5] to determine the uptake rate.

NOTE Diffusive uptake rate is sometimes dependent on the choice of sorbent (see prEN 13528-3[1]).

9 Calculations

9.1 Mass concentration of analyte

Calculate the mass concentration of the analyte in the sampled air, in micrograms per cubic metre (μg/m^3), by means of Equation (1):

$$\rho = \frac{m_a - m_b}{q_V \cdot t} \times 10^6 \quad (1)$$

where

ρ is the mass concentration of analyte in the air sampled, in micrograms per cubic metre;

m_a is the mass of analyte present in the actual sample as found in 8.4, in micrograms;

m_b is the mass of analyte present in the blank tube, in micrograms;

q_V is the diffusive uptake rate, in cubic centimetres per minute (Table 1 or 8.6);

t is the exposure time, in minutes.

NOTE 1 If m_a and m_b are expressed in milligrams, the resultant mass concentration ρ will be in milligrams per cubic metre.

NOTE 2 If it is desired to express mass concentrations reduced to specified conditions, e.g. 25 °C and 101 kPa, then:

$$\rho_c = \rho \cdot \frac{101}{p} \cdot \frac{T + 273}{298} \quad (2)$$

where

ρ_c is the mass concentration of the analyte in air sampled, reduced to specified conditions, in micrograms per cubic metre;

p is the actual pressure of the air sampled, in kilopascals;

T is the actual temperature of the air sampled, in degrees Celsius.

9.2 Volume fraction of analyte

Alternatively, calculate the content (volume fraction) of the analyte in air, in microlitres per cubic metre, by means of the following equation:

$$\varphi = \frac{m_a - m_b}{q_{V'} \cdot t} \times 10^6 \quad (3)$$

where

φ is the volume fraction of the analyte in air, in microlitres per cubic metre;

$q_{V'}$ is the diffusive uptake rate in ng·(μl/l)$^{-1}$·min^{-1} (Table 1 or 8.5)[2)];

2) μl/l is often expressed in the non-ISO units ppm (= 10^{-6}).