INTERNATIONAL STANDARD

ISO 16017-1

First edition
2000-11-15

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

Part 1: Pumped 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 1: Échantillonnage par pompage

Reference number
ISO 16017-1:2000(E)

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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 3.

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 part of ISO 16017 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 16017-1 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*

Annexes A and B form a normative part of this part of ISO 16017. Annexes C through F are for information only.

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

Part 1:
Pumped 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 ambient, indoor and workplace atmospheres and the assessment of emissions from materials in small- or full-scale test chambers.

This part of ISO 16017 is appropriate for 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 will generally require derivatization, very low boiling compounds will only be partially retained by the sorbents, depending on ambient temperature, and can only be estimated qualitatively. Semi-volatile compounds will be fully retained by the sorbents, but may only be partially recovered. Compounds for which this part of ISO 16017 has been tested are given in tables. This part of ISO 16017 may be applicable to compounds not listed, but in these cases it is advisable to use a back-up tube containing the same or a stronger sorbent.

This part of ISO 16017 is applicable to the measurement of airborne vapours of VOCs in a concentration range of approximately 0,5 μg/ m^3 to 100 mg/m^3 individual compound.

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 sorptive capacity is measured as a breakthrough volume of air, which determines the maximum air volume that shall not be exceeded when sampling.

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 and molecular sieves such as Spherocarb, or pure charcoal; at low nanogram levels for Tenax TA and at 5 ng to 50 ng levels for other porous polymers such as Chromosorbs and Porapaks. Sensitivity is typically limited to 0,5 μg/m^3 for 10-litre air samples with this latter group of sorbents because of their inherent high background.

The procedure specified in this part of ISO 16017 is applicable to low flowrate personal sampling pumps and gives a time-weighted average result. It is not applicable to the measurement of instantaneous or short-term fluctuations in concentration.

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1) The sorbents listed in annex C and elsewhere in this International Standard 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.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 16017. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 16017 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 6141:2000, *Gas analysis — Requirements for certificates for calibration gases and gas mixtures.*

ISO 6145-1:1986, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 1: Methods of calibration.*

ISO 6145-3:1986, *Gas analysis — Preparation of calibration gas mixtures — Dynamic volumetric methods — Part 3: Periodic injections into a flowing gas stream.*

ISO 6145-4:1986, *Gas analysis — Preparation of calibration gas mixtures — Dynamic volumetric methods — Part 4: Continuous injection method.*

ISO 6145-5:—[2)], *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 5: Capillary calibration devices.*

ISO 6145-6:—[2)], *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 6: Critical orifices.*

ISO 6349:1979, *Gas analysis — Preparation of calibration gas mixtures — Permeation method.*

EN 1076:1997, *Workplace atmospheres — Pumped sorbent tubes for the determination of gases and vapours — Requirements and test methods.*

3 Terms and definitions

For the purposes of this part of ISO 16017, the following terms and definitions apply.

3.1
breakthrough volume
volume of test atmosphere that can be passed through a sorbent tube before the concentration of eluting vapour reaches 5 % of the applied test concentration

NOTE 1 The breakthrough volume varies with the vapour and the sorbent type.

NOTE 2 See reference [4]. 3.2

retention volume
elution volume at peak maximum of a small aliquot of an organic vapour eluted from a sorbent tube by air or chromatographic carrier gas

2) To be published.

4 Principle

A measured volume of sample air is drawn through one (or more) sorbent tubes in series; an appropriate sorbent (or sorbents) being selected for the compound or mixture to be sampled. Provided suitable sorbents are chosen, volatile organic components are retained by the sorbent tube and thus are removed from the flowing air stream. 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. Analytical calibration is achieved by means of liquid or vapour spiking onto a sorbent tube.

5 Reagents and materials

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

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

5.1 Volatile organic compounds, for calibration purposes, using either liquid spiking (5.7 and 5.8) or vapour spiking (5.4 to 5.6) onto sorbent tubes.

5.2 Dilution solvent, for preparing calibration blend solution for liquid spiking (5.7). This should be of chromatographic quality. It shall be free from compounds co-eluting with the compound or compounds of interest (5.1).

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.

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

Each sorbent should be preconditioned under a flow of inert gas by heating it overnight (= 16 h) at a temperature at least 25 °C below the published maximum for that sorbent before packing the tubes. To prevent recontamination of the sorbents, 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.

NOTE 1 Sorbent particle sizes larger than 0,18 mm to 0,25 mm may be used but the breakthrough characteristics given in Tables 1 to 6 may be affected. Smaller sorbent particle size ranges are not recommended because of back-pressure problems.

NOTE 2 A description of sorbents is given in annex C and a guide for sorbent selection is given in annex D. Equivalent sorbents may be used. A guide to sorbent conditioning and analytical desorption parameters is given in annex E.

5.4 Calibration standards, preferably prepared by loading required amounts of the compounds of interest on sorbent tubes from standard atmospheres (see 5.5 and 5.6), 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 5.7 and 5.8), provided that the accuracy of the spiking technique is either:

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

b) confirmed by comparison with reference materials, if available, standards produced using standard atmospheres, or results of reference measurement procedures.

NOTE The loading ranges given in 5.6, 5.7 and 5.8 are not mandatory and approximate to the application range given in clause 1 for a 2-litre sample. For specific applications in which larger volumes are used to measure lower concentrations, other loading ranges may be more appropriate.

5.5 Standard atmospheres.

Prepare standard atmospheres of known concentrations of the compound(s) of interest by a recognized procedure. Methods described in ISO 6141, the appropriate part of ISO 6145 and ISO 6349 are suitable. 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.

5.6 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 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.

5.7 Solutions for liquid spiking.

5.7.1 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 (5.2), stopper and shake to mix.

5.7.2 Solution 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 5.7.1 Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.7.3 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 (5.2), stopper and shake to mix.

5.7.4 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 described in 5.7.3. Make up to 100 ml with dilution solvent, stopper and shake to mix.

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

For gases, e.g. ethylene oxide, a high level calibration solution may 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, but correct for any non-ideality of the particular pure gas compound.

5.7.6 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, but correct for any non-ideality of the particular pure gas compound.

5.8 Standard sorbent tubes loaded by liquid spiking.

Prepare loaded sorbent tubes by injecting aliquots of standard solutions onto clean sorbent tubes as follows. Fit a sorbent tube into the injection unit (6.10) through which inert purge gas and a 1 µl to 4 µl aliquot of an appropriate standard solution, injected through the septum, are passed. After an appropriate time, disconnect and seal the tube. Prepare fresh standards with each batch of samples. For workplace air, load sorbent tubes with 1 µl to 5 µl of solutions 5.7.1, 5.7.2 or 5.7.5. For ambient and indoor air, load sorbent tubes with 1 µl to 5 µl of solutions 5.7.3, 5.7.4 or 5.7.6.

NOTE In the case of methanol, a purge gas flowrate of 100 ml/min and a 5 min purge time have been found to be appropriate to eliminate most of the solution solvent from the tube. If other dilution solvents are used, the conditions should be determined experimentally.

6 Apparatus

Use ordinary laboratory apparatus and the following.

6.1 Sorbent tubes, compatible with the thermal desorption apparatus to be used (6.9).

Typically, but not exclusively, sorbent tubes are constructed of stainless steel tubing, 6,3 mm (1/4 inch) OD, 5 mm ID and 90 mm long. Tubes of other dimensions may be used but the safe sampling volumes (SSV) given in Tables 1 to 6 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. One end of the tube is marked, for example by a scored ring about 10 mm from the sampling inlet end. The tubes are packed with one or more preconditioned sorbents (5.3) so that the sorbent bed will be within the desorber heated zone and a gap of at least 14 mm is retained at each end to minimize errors due to diffusive ingress at very low pump flowrates. Tubes contain between 200 mg and 1 000 mg sorbent, depending on sorbent density (typically about 250 mg porous polymer or 500 mg carbon molecular sieve or graphitized carbon). The sorbents are retained by stainless steel gauzes and/or unsilanized glass wool plugs. If more than one sorbent is used in a single tube, the sorbents should be arranged in order of increasing sorbent strength and separated by unsilanized glass wool, with the weakest sorbent nearest to the marked sampling inlet end of the tube.

Do not pack sorbents with widely different (> 50 °C) maximum desorption temperatures into a single tube, or it will be impossible to condition or desorb the more stable sorbent(s) sufficiently thoroughly without causing degradation of the least stable sorbent(s).

6.2 Sorbent tube end caps.

The tubes shall be sealed according to the requirements of EN 1076:1997, subclause 5.6, or equivalent, e.g. with metal screw-cap fittings with polytetrafluoroethylene (PTFE) seals.

6.3 Sorbent tube unions.

Two sorbent tubes may be connected in series during sampling with metal screw-cap couplings with PTFE seals.

6.4 Syringes, including 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.

6.5 Sampling pump

The pump should fulfil the requirements of EN 1232 [10] or equivalent.

The sampling pump shall be in accordance with local safety regulations.

6.6 Plastic or rubber tubing, about 90 cm long, of appropriate diameter to ensure a leak-proof fit to both pump and sample tube or tube holder, if used. Clips should be provided to hold the sample tube and connecting tubing.

Sampling tubes shall not be used with plastic or rubber tubing upstream of the sorbent. The use of such tubing may introduce contaminants or sorbed sampled VOCs.

6.7 Soap-bubble meter or other suitable device for calibrating pump.

The flow meter shall be traceably calibrated to a primary flow standard.

NOTE The use of an uncalibrated integral flow meter for the calibration of pump flowrates may result in systematic errors of several tens of percent.

6.8 Gas chromatograph, fitted with a flame ionization, 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.

The gas chromatograph shall have a capillary column capable of separating the analytes of interest from other components.

6.9 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 flowrate. 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 (10.2). The desorbed sample, contained in the purge gas, is routed to the gas chromatograph and capillary column via a heated transfer line.

6.10 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.

7 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 E). Typical conditioning time is 10 min with carrier gas flowrate of 100 ml/min. The carrier gas flow should be in a direction opposite to that used during sampling. 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 used for 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.

8 Calibration of pump

Calibrate the pump with a representative sorbent tube assembly in-line, using an appropriate external calibrated meter.

One end of the calibrated flow meter should be at atmospheric pressure to ensure proper operation.

9 Sampling

Select a sorbent tube (or tube combination) appropriate for the compound or mixture to be sampled. Guidance on suitable sorbents is given in annex D.

If more than one tube is to be used, prepare a tube assembly by joining the tubes with a union (6.3).

Attach the pump to the sorbent tube or tube assembly with plastic or rubber tubing, so that the tube containing the stronger sorbent is nearest the pump.

When used for personal sampling, to minimize channelling the tube assembly should be mounted vertically in the breathing zone. The pump is attached as appropriate to minimize inconvenience. When used for fixed location sampling, a suitable sampling site is chosen.

Turn the pump on and adjust the flowrate so that the recommended sample volume is taken in the available time. The recommended air sample volume for the volatile organic compounds covered by this standard is between 1 litre and 10 litres. If the total sample is likely to exceed 1 mg (i.e. 1 mg on each tube), the sample volume shall be reduced accordingly, or overload may occur.

NOTE 1 Sampling efficiency is 100 % (quantitative), provided the sampling capacity of the sorbents is not exceeded. If this capacity is exceeded, breakthrough of vapour from the tube assembly will occur. The breakthrough volume may be measured by sampling from a standard vapour atmosphere, whilst monitoring the effluent air with a flame ionization or equivalent detector (a suitable method is described in annex A). Alternatively, instead of determining the breakthrough volume directly, the mathematically related retention volume may be determined. The retention volume is determined chromatographically at elevated temperatures and subsequent extrapolation to room temperature. A suitable method is described in annex B.

The breakthrough volume of porous polymers vary with ambient air temperature, reducing by a factor of about 2 for each 10 °C rise in temperature. It also varies with sampling flowrate, being reduced substantially at flowrates below 5 ml/min or above 500 ml/min. The breakthrough volumes of carbon molecular sieves are less affected by temperature and flowrate, but are substantially reduced at high concentrations of volatile organic vapour or high relative humidity. To allow a suitable margin of safety, a safe sampling volume (SSV) is defined such that it is a volume of not more than 70 % of the 5 %-breakthrough volume (see A.1.1 in annex A) or 50 % of the retention volume (see B.1 in annex B). Tables 1 to 6 give typical values for retention volumes and safe sampling volumes. These values have been determined by the chromatographic method (annex B).

NOTE 2 The safe sampling volumes in Tables 1 to 6 have been determined by the chromatographic method (annex B). Measurements by the direct method (annex A) [4] indicate that the chromatographic method is a reliable indication of the true breakthrough capacity except under conditions of high concentrations or very high humidity. These measurements [4] indicate that breakthrough volumes at high (80 %) humidity are about a factor of two lower for porous polymers and a factor of ten lower for carbonaceous sorbents than the low humidity value. If high concentrations [> 300 mg/m^3 (100 ppm)] are also anticipated, the breakthrough volumes for carbonaceous sorbents should be further reduced by a factor of two.

If safe sampling volumes for compounds are estimated which are not listed in Table 1, this estimation is only possible for such compounds which are situated between the two listed compounds of homologues of a chemical group. In all other cases the safe sampling volume shall be tested experimentally with appropriate trials (e.g. similar sampling media in-line and separate analysis).

Note and record the times, temperature, flowrate or register reading if appropriate and the barometric pressure when the pump was turned on. At the end of the sampling period, note and record the flowrate or register reading, turn the pump off, and note and record the time, temperature and barometric pressure.

Disconnect the sample tube assembly and seal both ends of each tube with compression seals. Tighten these seals securely. The tubes should be uniquely labelled. 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, refrigerated sealed metal or glass container. If possible the sampler should be refrigerated during transportation.

Record air temperature and barometric pressure periodically during sampling if it is desired to express concentrations reduced to specific conditions (11.1).

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. Label these as blanks.

NOTE 3 Since this method uses thermal desorption, unless the TD apparatus has the facility to retrap the sample after analysis, there will generally only be one opportunity to analyse the sample. If the sample is important and the chance of overload and/or sample breakthrough is a possibility, a second sample at a lower flowrate should be taken.

10 Procedure

10.1 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.

10.2 Desorption and analysis

Place the sorbent tube in a compatible thermal desorption apparatus. Purge the air from the tube to avoid chromatographic artefacts arising from the thermal oxidation of the sorbent or gas chromatographic stationary phase. Then heat the tube to displace the organic vapours which are passed to the gas chromatograph by means of a carrier gas stream. The gas flow direction at this stage should be the reverse of that used during sampling, i.e. the marked end of the tube should be nearest the gas chromatograph column inlet. Typically the gas flowrate through the tube should be in the order of 30 ml/min to 50 ml/min for optimum desorption efficiency.

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. During the purge period, care should be taken to minimize heating of the tube.

The desorbed sample occupies a volume of several millilitres of gas, so that pre-concentration is essential prior to capillary GC analysis. This can 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, can be used to pre-concentrate 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 shall be completely eliminated from the sample tube prior to desorption in order to prevent ice formation blocking the capillary tubing and stopping the thermal desorption process.

NOTE 1 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 will typically be 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:

