

Ambient air quality — Standard method for measurement of benzene concentrations —

Part 1: Pumped sampling followed by thermal desorption and gas chromatography

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National foreword

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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:

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Pumpe mit anschließender Thermodesorption und
Gaschromatographie

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Foreword

This European Standard (EN 14662-1:2005) 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of an identical text or by endorsement, at the latest by November 2005, and conflicting national standards shall be withdrawn at the latest by November 2005.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive 2000/69/EC and EU Directive 96/62 EC.

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1 Scope

This part of EN 14662 gives general guidance for the sampling and analysis of benzene in air by pumped sampling, thermal desorption and capillary gas chromatography.

This part of EN 14662 is in accordance with the generic methodology selected as the basis of the European Union reference method for the determination of benzene in ambient air [1] for the purpose of comparison of measurement results with limit values with a one-year reference period.

This part of EN 14662 is valid for the measurement of benzene in a concentration range of approximately $0,5 \mu\text{g}/\text{m}^3$ to $50 \mu\text{g}/\text{m}^3$ in an air sample typically collected over a period of 24 hours.

The upper limit of the useful range is set by the sorptive capacity of the sorbent and by the linear dynamic range of the gas chromatograph column and detector or by the sample splitting capacity 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 benzene and/or interfering artefacts on the sorbent. Artefacts are typically sub ng for sorbents, but higher levels of aromatic hydrocarbons have been noted in other sorbents. The detection limit will be approximately 1/10 of the lower concentration range.

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.

ENV 13005, *Guide to the expression of uncertainty in measurements*

EN ISO 16017-1:2000, *Indoor, ambient and workplace air – Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography – Part 1: Pumped sampling (ISO 16017-1:2000)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)*

3 Terms and definitions

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

NOTE Attention is drawn to the fact that the terms Ambient Air and Limit Value are defined in Directive 96/62/EC [2].

3.1

Certified reference material

A reference material [3.5], accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

[ISO Guide 30:1992]

3.2

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 13005:1999]

3.3

Desorption efficiency

Ratio of the mass of analyte desorbed from a sampling device to that applied. [EN 1076:1997]



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3.4**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. [ENV 13005:1999]

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 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 13005:1999.

3.5**Reference material**

A material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

[ISO Guide 30:1992]

3.6**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 3534-1:1993]

3.7**Sampling efficiency**

Ratio of the mass of analyte collected by a sampling device to that applied

3.8**Standard uncertainty**

Uncertainty of the result of a measurement expressed as a standard deviation [ENV 13005:1999]

3.9**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 13005:1999].

Attention is drawn to the fact that the terms Ambient Air and Limit Value are defined in Directive 96/62/EC [2].

4 Method description**4.1 Principle**

A measured volume of sample air is drawn through a sorbent tube. [EN –ISO 16017-1] Provided suitable sorbents are chosen, benzene is retained by the sorbent and thus is 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 ionisation detector or other suitable detector, where it is analysed. The analysis is calibrated by means of liquid or vapour spiking onto a sorbent tube.

4.2 Reagents and materials

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

Use only volumetric glassware and syringes that are calibrated to ensure traceability of volume to primary standards.

4.2.1 Benzene

Benzene is required as a reagent for calibration purposes, using either liquid spiking (4.2.7 to 4.2.9) or vapour spiking (4.2.4 to 4.2.6) onto sorbent tubes. The benzene used shall be of a minimum purity of 99.5%.

4.2.2 Dilution solvent

A dilution solvent is required for preparing calibration blend solution for liquid spiking (4.2.7).

NOTE Methanol is frequently used. Alternative solvents may be used provided they do not interfere with the gas chromatographic analysis, either by co-elution or by altering detector response.

4.2.3 Sorbents

A particle size 0,18 mm to 0,25 mm (60 to 80 mesh) is recommended. Sorbent particle sizes larger than 0,18mm to 0,25 mm may be used but the breakthrough characteristics given in Annex A may be affected. Smaller sorbent particle size ranges are not recommended because of back pressure problems. Each sorbent should be preconditioned under a flow of inert gas by heating it at a temperature at least 25 °C below the published maximum for that sorbent overnight 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 A description of sorbents is given in Annex B and 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.

4.2.4 Calibration standards

Calibration standards are preferably prepared by loading the required amount benzene on the sorbent tubes from standard atmospheres (see 4.2.5 and 4.2.6) as this procedure most closely resembles the practical sampling situation.

NOTE The loading ranges given in 4.2.6, 4.2.7 and 4.2.9 are not mandatory and approximate to the application range given in the scope for a 10 litre sample. For specific applications where larger volumes are used to measure lower concentrations other loading ranges may be more appropriate.

If this way of preparation is not practicable, standards may be prepared by a liquid spiking procedure (see 4.2.7 to 4.2.9) 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.

To minimise matrix effects, calibration tubes should be closely matched to the sample tubes with respect to sorbent type, particle size and mass of sorbent.

4.2.5 Standard atmospheres

Prepare standard atmospheres of known concentrations of benzene by a recognised procedure. Methods described in ISO 6144 and ISO 6145 are suitable. If the procedure is not applied under conditions that will allow the establishment of full traceability of the generated concentrations to primary standards of mass and/or volume the concentrations need to be confirmed using an independent procedure.

4.2.6 Standard sorbent tubes loaded by spiking from standard atmospheres

Prepare loaded sorbent tubes by passing an accurately known volume of the standard 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 sorbent. After loading the tube is disconnected and sealed. Prepare fresh standards representing benzene levels in the samples corresponding to the concentration range of $0,5 \mu\text{g}/\text{m}^3$ to $50 \mu\text{g}/\text{m}^3$ with each batch of samples or use spiked control samples to ensure consistency of detector response.

4.2.7 Preparation of standard solutions for liquid spiking

4.2.7.1 Solution containing approximately 100 $\mu\text{g}/\text{ml}$ of benzene.

Weigh a 100 ml volumetric flask and introduce approximately 75 ml of dilution solvent (4.2.2) into a 100 ml volumetric flask. Then weigh approximately 10 mg benzene into the flask. Make up to 100 ml with dilution solvent, stopper, weigh and shake to mix.

NOTE In order to ensure an uncertainty of the mass of benzene introduced appropriate to the application the weighing uncertainty of the equipment used ($k=2$) shall be less than $\pm 70 \mu\text{g}$.

4.2.7.2 Solution containing approximately 50 $\mu\text{g}/\text{ml}$ benzene

Introduce 30 ml of dilution solvent into a preweighed 100 ml volumetric flask. Weigh in 50 ml of solution described in 4.2.7.1. Make up to 100 ml with dilution solvent, stopper, weigh and shake to mix.

4.2.7.3 Solution containing approximately 10 $\mu\text{g}/\text{ml}$ benzene

Introduce 50 ml of dilution solvent into a preweighed 100 ml volumetric flask. Weigh in 20 ml of solution described in 4.2.7.2. Make up to 100 ml with dilution solvent, stopper, weigh and shake to mix.

4.2.7.4 Solution containing approximately 5 $\mu\text{g}/\text{ml}$ benzene

Introduce 50 ml of dilution solvent into a preweighed 100 ml volumetric flask. Weigh in 10 ml of solution described in 4.2.7.2. Make up to 100 ml with dilution solvent, stopper, weigh and shake to mix.

4.2.7.5 Solution containing approximately 1 $\mu\text{g}/\text{ml}$ benzene

Introduce 50 ml of dilution solvent into a preweighed 100 ml volumetric flask. Weigh in 10 ml of solution described in 4.2.7.3. Make up to 100 ml with dilution solvent, stopper, weigh and shake to mix.

4.2.8 Stability of standard solutions

Fresh standard solutions should be prepared weekly, or more frequently if evidence is noted of deterioration.

4.2.9 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 to a T piece of which one end is fitted with a septum, or injection facility of a gas chromatograph through which inert purge gas is passed at 100ml/min. Inject a $5\mu\text{l}$ aliquot of an appropriate standard solution through the septum and purge for 5 minutes. The tube is then disconnected and sealed. Prepare fresh standards with each batch of samples

NOTE 1 In the case of methanol, a purge gas flow 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 packed with Tenax. If other dilution solvents and sorbents are used, the conditions should be determined experimentally.

NOTE 2 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.

4.3 Apparatus

The following specific items of laboratory equipment are required.

4.3.1 Sorbent tubes

These tubes shall be compatible with the thermal desorption apparatus to be used (4.3.10). Typically, but not exclusively, they are constructed of stainless steel tubing, 6,4 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 Annex A are based on these tube dimensions. 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 preconditioned sorbent 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 minimise errors due to diffusive ingress at very low pump flow rates. Tubes contain between 200 mg and 1000 mg sorbent, depending on sorbent density - typically about 250 mg porous polymer. The sorbents are retained by stainless steel gauzes and/or unsilanised glass wool plugs.

4.3.2 Sorbent tube end caps

The tubes shall be sealed, according to the requirements of EN ISO 16017-1 or equivalent. e.g. with metal screw cap fittings with PTFE seals.

4.3.3 Syringes

A precision 10 µl liquid syringe readable to 0,1 µl. The volume of the solvent delivered shall be calibrated by gravimetry.

4.3.4 Sampling device

Sampling device capable of maintaining a preset flow rate of 5 ml/min – 200 ml/min within $\pm 5\%$ during the required sampling period (typically 24 h). As such, the following devices may be used:

- a pump with an adjustable flow rate
- a vacuum pump with a critical orifice
- a mass-flow controller operated by a vacuum pump
- a vacuum pump with a constant pressure drop over a restriction.

NOTE The sampling device should be in accordance with local safety regulations, if any.

4.3.5 Tubing

Tubing of appropriate length and internal diameter to ensure a leak-proof fit to both pump and sample tube or tube holder, if used.

4.3.6 Flow calibration device for calibrating pump

A flow meter that is traceably calibrated to a primary flow standard over the desired flow range (4.3.5). The uncertainty in the calibration of the flow meter shall be $\leq 2\%$.

NOTE The use of an uncalibrated integral flow meter for the calibration of pump flow rates may result in large systematic errors.

4.3.7 Precipitation shield

A protective cover to prevent the entrance of particles or water droplets into the sampling tube during the sampling.

NOTE EN 13528-3 describes various shields for diffusive samplers that may also be suitable for pumped samplers.

4.3.8 Support

A device capable of positioning the sampling device and sorbent tube at the appropriate height and distance from obstacles to warrant undisturbed sampling.

4.3.9 Gas chromatograph

A gas chromatograph fitted with a flame ionisation, photo ionisation detector, mass spectrometric or other suitable detector, capable of detecting an injection of 0,5 ng benzene with a signal-to-noise ratio of at least 5 to 1.

A gas chromatograph column capable of separating benzene from other components.

4.3.10 Thermal desorption apparatus

Apparatus for the two-stage thermal desorption of the sorbent tubes and transfer of the desorbed vapour via an inert gas flow into a gas chromatograph shall be required. 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 (4.7.2). The desorbed sample, contained in the purge gas, is routed to the gas chromatograph and capillary column via a heated transfer line.

4.4 Sample tube conditioning

Prior to use, tubes shall be conditioned by desorbing them at a temperature at or just above the analytical desorption temperature (see Annex D). Typical conditioning time is 10 min with a carrier gas flow of at least 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 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 screw caps with combined PTFE ferrule fittings and stored in an airtight container when not sampling or being conditioned.

4.5 Setting of sampling flow rate

Calibrate the flow of the sampling device with a representative sorbent tube assembly in line, using an appropriate external calibrated meter (4.3.4).

Determine the flow rate by taking the average of a minimum of 3 consecutive measurements. The uncertainty in the measured flow shall be $\leq 2,5\%$.

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

4.6 Sampling

Select a sorbent tube appropriate for sampling benzene. Guidance on suitable sorbents is given in Annex C.

Attach the sampling device to the non marked end of the sorbent tube or tube assembly with plastic or rubber tubing. Turn the device on and measure the flow rate and/or adjust the flow rate so that the recommended sample volume is taken in the available time. The typical air sample volume for benzene is 10 litres.

NOTE Sampling efficiency will be 100 % (quantitative), provided the sampling capacity of the sorbents is not exceeded. If this capacity is exceeded, breakthrough of benzene 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 ionisation or equivalent detector (a suitable method is described in Annex E). Alternatively, for relatively hydrophobic sorbents, 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 F.

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 flow rate, being reduced substantially at flow rates below 5 ml/min or above 500 ml/min. 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 Annex E.1.1) or 50 % of the retention volume (see Annex C.1). Annex A gives typical values for retention volumes and safe sampling volumes. These values have been determined by the chromatographic method (Annex F).

NOTE 1 The safe sampling volumes in Annex A have been determined by the chromatographic method (Annex F). Measurements by the direct method (Annex E) [3] 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 [3] indicate that breakthrough volumes at high (80 %) humidity are about a factor of two lower for porous polymers.

NOTE 2 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 flow rate should be taken.

Record the sampling period for each sample taken. Measure the flow rate again at the end of the period of unattended operation as described in 4.4.

Knowledge of the average temperature and barometric pressure of the sampled air is required in order to express concentrations reduced to standard conditions. This information may be obtained from measurements on the site using a traceably calibrated thermometer and barometer. Alternatively, information from a nearby weather station may be used.

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 hours, they should be stored in such a way that contamination cannot occur.

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.

4.7 Procedure

4.7.1 Safety precautions

This part of EN 14662 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 standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

4.7.2 Desorption and analysis

The sorbent tube is placed in a compatible thermal desorption apparatus and a leak test performed. 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 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. The gas flow through the tube should be typically 30 ml/min to 50 ml/min for optimum desorption efficiency.

NOTE 1 For the air purge described above it is usually necessary to use 10 x 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 used, it may be necessary to employ a larger purge volume to reduce sorbed air and water to prevent ice formation blocking the cold trap. During the purge period, care should be taken to minimise heating of the tube.