

Characterization of air quality —

Part 3: Workplace atmospheres —

Section 3.4 Method for the determination of vaporous aromatic hydrocarbons by charcoal tube/solvent desorption/gas chromatography

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Environment and Pollution Standards Policy Committee (EPC/-) to Technical Committee EPC/35, upon which the following bodies were represented:

- Association of Consulting Scientists
- British Coal Corporation
- British Gas plc
- British Steel plc
- Department of Health
- Department of the Environment (Her Majesty's Inspectorate of Pollution)
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Department of Trade and Industry (Warren Spring Laboratory)
- Electricity Supply Industry in England and Wales
- European Resin Manufacturers' Association
- GAMBICA (BEAMA Ltd.)
- Health and Safety Executive
- Institute of Occupational Hygienists
- Institute of Petroleum
- Institution of Environmental Health Officers
- Institution of Gas Engineers
- Lead Development Association
- National Society for Clean Air
- Royal Society of Chemistry
- Society of Chemical Industry

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

- Asbestos Information Centre Ltd.
- Asbestosis Research Council
- British Occupational Hygiene Society
- Chemical Industries Association
- Council for Environmental Conservation
- Engineering Equipment and Materials Users' Association
- Fibre Cement Manufacturers' Association Limited
- Institute of Energy
- Institute of Occupational Medicine
- London Regional Transport

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National foreword

This Section of BS 6069, which has been prepared under the direction of the Environment and Pollution Standards Policy Committee, is identical with ISO 9487:1991 “*Workplace air — Determination of vaporous aromatic hydrocarbons — Charcoal tube/solvent desorption/gas chromatographic method*”. The International Standard was prepared by Technical Committee 146, Air quality, of the International Organization for Standardization (ISO) with the active participation and approval of the UK.

BS 6069 is being published in a series of Parts and Sections that will generally correspond to particular International Standards arising from the UK participation in the work of ISO/TC 146. This Section of BS 6069 is one of several relating to workplace atmospheres that are being published as Sections of Part 3. Methods concerning stationary sources are being published as Sections of Part 4 of BS 6069. Topics relating to other aspects of air quality characterization will be published as further Parts or Sections of BS 6069.

The following Parts of BS 6069 are being published.

- *Part 1: Units of measurement (complete);*
- *Part 2: Glossary (complete);*
- *Part 3: Workplace atmospheres;*
- *Part 4: Stationary source emissions.*

Methods for the determination of particular constituents of ambient air are being published as Parts of BS 1747 “*Methods for measurement of air pollution*”.

Cross-references

International Standard	Corresponding British Standard
ISO 5725:1986	BS 5497 <i>Precision of test methods</i> Part 1:1987 <i>Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests</i> (Identical) BS 4559 <i>Methods for preparation of calibration gas mixtures</i>
ISO 6145-1:1986	Section 5.1:1987 <i>Review of methods of calibration</i> (Identical)
ISO 6349:1979	Part 7:1981 <i>Permeation method</i> (Identical)

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This International Standard specifies a charcoal tube/gas chromatographic method for the determination of the concentration of vaporous aromatic hydrocarbons in workplace air.

The method is valid for the measurement of the concentrations of airborne vapours of any of the following compounds:

- a) benzene;
- b) toluene;
- c) ethylbenzene;
- d) *m*-xylene;
- e) *o*-xylene;
- f) *p*-xylene;
- g) styrene (vinylbenzene);
- h) cumene (isopropylbenzene);
- i) 1,2,3-trimethylbenzene;
- j) 1,2,4-trimethylbenzene;
- k) 1,3,5-trimethylbenzene;
- l) 4-*tert*-butyltoluene;
- m) 3-methylstyrene;
- n) 4-methylstyrene;
- o) isopropenylbenzene.

The method is valid for concentrations of airborne vapours of these compounds in the range from approximately 1 mg/m³ to 1 000 mg/m³ (about 0,2 ml/m³ to 200 ml/m³; see 8.1) when sampling 10 litres of air.

NOTE 1 The upper limit of the useful range is set by the adsorptive capacity of the first section of the charcoal tube (5.1) used. This capacity is measured as a breakthrough volume of air, which should not be exceeded during sampling (see clause 6 and Annex A).

The lower limit is set by a number of parameters, including the noise level of the detector (5.9), blank concentrations due to the contamination of the charcoal and carbon disulfide by the substance analysed, desorption efficiency (see Annex B) and interference of the solvent peak in the gas chromatographic analysis.

The method is also valid for the measurement of airborne concentrations of mixtures of these compounds. In such cases, the unique properties of each compound have to be considered when determining the volume of air to be sampled and the gas chromatographic conditions to be used. The method may be applicable for other substituted monocyclic aromatic hydrocarbons, but its validity has to be tested.

NOTE 2 When analysing aromatic hydrocarbon mixtures with very large differences in concentrations and in which several compounds are present, the reproducibility and repeatability of the compounds of minor importance might be influenced.

The method has been validated for a selection of typical aromatic hydrocarbons[1].

This procedure is compatible with low flow rate personal sampling equipment, and can be used for personal and fixed location sampling for obtaining time-weighted-average concentrations of aromatic hydrocarbon solvent vapours in air. It cannot be used to measure instantaneous or short-term fluctuations in concentrations. Alternative on-site procedures, such as gas chromatography or infrared spectrometry, shall be used to measure rapidly changing concentrations.

Organic components which have the same or nearly the same retention time as the substance analysed during the gas chromatographic analysis will interfere. Interferences can be minimized by proper selection of gas chromatographic columns and programme conditions.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

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

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

3 Principle

A known volume of air sample is passed through a glass or metal tube packed with activated charcoal. The organic vapours are adsorbed onto the charcoal. The collected vapours are desorbed using a suitable solvent and analysed with a gas chromatograph equipped with a flame ionization detector or other suitable detector.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 *Aromatic hydrocarbons*, listed in clause 1, a) to o).

WARNING — Benzene is a recognized human carcinogen.

Avoid any exposure by inhalation or skin contact.

4.2 Carbon disulfide (CS_2), chromatographic quality, previously checked for compounds coincident with the substance analysed of interest. If necessary, remove interfering compounds by percolation through silica gel (dried at 180 °C for 8 h under nitrogen) contained in a small glass column (600 mm $\times$ Ø int. 20 mm). This size of column will clean about 50 ml of carbon disulfide.

WARNING — Carbon disulfide vapour is toxic and highly flammable. Avoid any exposure by inhalation or skin contact. Use only in a well-ventilated fume cupboard. A carbon-dioxide fire extinguisher should be available at all times.

Dispose of small waste quantities of carbon disulfide in accordance with local regulations and accepted practices.

NOTE 3 Other desorption solvents may be used if their blanks and desorption efficiencies are adequate.

4.3 Activated coconut shell charcoal, particle size between 0,4 mm and 0,8 mm. Before packing the tubes, the charcoal shall be heated in an inert atmosphere, e.g. high purity nitrogen at 600 °C for 1 h. To prevent recontamination of the charcoal, it shall be kept in a clean atmosphere during its cooling to room temperature, storage and loading into the tubes.

NOTE 4 Suitable tubes containing pretreated coconut charcoal are commercially available (see 5.1).

4.4 Aromatic hydrocarbons, standard solutions for the preparation of the calibration graph (see 7.1).

Prepare standard solutions of aromatic hydrocarbons (4.1) gravimetrically and make serial dilutions from them to cover the range of the analysis. The concentration range of the analysis shall exceed the concentration range of the desorbed samples (see 7.2). As an example, a typical procedure for toluene is described below.

Prepare standard solutions in carbon disulfide (4.2).

If the standard solution (4.4.1) has to be stored (for not longer than 1 week), prepare it in *n*-heptane.

4.4.1 Toluene, standard solution, about 50 µg/ml.

Accurately weigh approximately 500 mg of toluene in a 10 ml one-mark volumetric flask. Dilute to the mark with carbon disulfide and mix well.

4.4.2 Toluene, standard solution, about 5 mg/ml.

Pipette 1 ml of the standard solution (4.4.1) into a 10 ml one-mark volumetric flask. Dilute to the mark with carbon disulfide and mix well.

4.4.3 Toluene, standard solution, about 500 µg/ml.

Pipette 1 ml of the standard solution (4.4.2) into a 10 ml one-mark volumetric flask. Dilute to the mark with carbon disulfide and mix well.

4.4.4 Toluene, standard solution, about 50 µg/ml.

Pipette 1 ml of the standard solution (4.4.3) into a 10 ml one-mark volumetric flask. Dilute to the mark with carbon disulfide and mix well.

4.4.5 Toluene, standard solution, about 5 µg/ml.

Pipette 1 ml of the standard solution (4.4.4) into a 10 ml one-mark volumetric flask. Dilute to the mark with carbon disulfide and mix well.

4.5 Toluene, standard matching solutions for checking the calibration graph.

If the standard solutions (4.5.1 and 4.5.2) have to be stored (for not longer than 1 week); prepare them in *n*-heptane.

4.5.1 Toluene, standard matching solution, about 25 mg/ml.

Accurately weigh approximately 250 mg of toluene in a 10 ml one-mark volumetric flask. Dilute to the mark with carbon disulfide and mix well.

4.5.2 Toluene, standard matching solution, about 5 mg/ml.

Accurately weigh approximately 50 mg of toluene in a 10 ml one-mark volumetric flask. Dilute to the mark with carbon disulfide and mix well.

5 Apparatus

Ordinary laboratory apparatus, and the following.

5.1 Charcoal tube, made of a glass tube with both ends flame sealed, 70 mm long with an outside diameter of 6 mm and an inside diameter of 4 mm, containing two sections of 0,4 mm to 0,8 mm of activated charcoal. The adsorbing section contains 100 mg of charcoal and the back-up section, 50 mg. The sections are separated and their contents are held in place with an inert material, e.g. glass wool plugs (preferably silanized).

The pressure drop across the tube shall not exceed 3 kPa (25 mmHg) at the maximum flow rate recommended for sampling (in this International Standard, 200 ml/min).

The glass tubes shall be held in suitable protective holders to prevent breakage. The desorption efficiency (*D*) for each batch of tubes shall be checked by one of the methods described in Annex B. If the desorption efficiency is lower than 0,75 (75 %), the tubes shall not be used.

Clips shall be provided to hold the charcoal tube and connecting tubing to the wearer's lapel area.

IMPORTANT — Do not use charcoal tubes with any tubing upstream of the charcoal, as this may interfere with sampling and/or analysis.

NOTE 5 Instead of a commercial two-section tube, two single-section tubes in series may be used. This arrangement has the advantage that it is not necessary to store tubes at sub-ambient temperatures after sampling, to prevent migration of the trapped compounds from one section to the other.