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Workplace air — Determination of vaporous chlorinated hydrocarbons — Charcoal tube/solvent desorption/gas chromatographic method

*Air des lieux de travail — Détermination des hydrocarbures chlorés
vaporeux — Méthode d'analyse par tube à charbon actif/désorption des
solvants/chromatographie en phase gazeuse*



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Foreword

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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.

International Standard ISO 9486 was prepared by Technical Committee ISO/TC 146, *Air quality*, Sub-Committee SC 2, *Workplace atmospheres*.

Annexes A, B and C form an integral part of this International Standard. Annex D is for information only.

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Workplace air — Determination of vaporous chlorinated hydrocarbons — Charcoal tube/solvent desorption/gas chromatographic method

1 Scope

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

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

- a) dichloromethane;
- b) chloroform;
- c) carbon tetrachloride;
- d) 1,1-dichloroethane;
- e) 1,2-dichloroethane;
- f) 1,1-dichloroethene;
- g) 1,2-dichloroethene;
- h) 1,1,1-trichloroethane;
- i) 1,1,2-trichloroethane;
- j) trichloroethene;
- k) 1,1,2,2-tetrachloroethane;
- l) tetrachloroethene;
- m) 1,2-dichloropropane;
- n) chlorobenzene;
- o) *o*-dichlorobenzene.

The method is valid for concentrations of airborne vapours of these compounds in the range from ap-

proximately 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 tube 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.

NOTE 2 When analysing chlorinated 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 chlorinated 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 chlorinated 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.

NOTE 3 For certain applications, in particular the analysis of carbon tetrachloride and chloroform, alternative detectors such as the electron capture detector may offer greater selectivity and sensitivity.

4 Reagents

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

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

WARNING — Carbon tetrachloride and chloroform are suspect human carcinogens.

Avoid any exposure by inhalation or skin contact.

4.2 Carbon disulfide (CS₂), chromatographic quality, previously checked for compounds coincident with the substance analysed. 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 × 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 4 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 5 Suitable tubes containing pretreated coconut charcoal are commercially available (see 5.1).

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

Prepare standard solutions of chlorinated 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 trichloroethene is described below.

Prepare standard solutions in carbon disulfide (4.2).

Prepare standard solutions of 1,2-dichloropropane in 15 % (V/V) acetone in cyclohexane.

4.4.1 Trichloroethene, standard solution, about 50 mg/ml.

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

4.4.2 Trichloroethene, 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 Trichloroethene, 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 Trichloroethene, 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 Trichloroethene, 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 Trichloroethene, standard matching solutions for checking the calibration graph (see 7.1).

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

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

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

Accurately weigh approximately 50 mg of trichloroethene 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.

NOTES

6 The breakthrough volumes of dichloromethane, 1,2-dichloroethene and 1,2-dichloropropane are lower than for the other substances analysed. Consequently, a larger tube containing (400 + 200) mg charcoal is recommended for long-term sampling when these compounds are present.

7 1,1,2,2-Tetrachloroethane and 1,2-dichloropropane are unstable on coconut shell charcoal; petroleum-based charcoal should be used instead.

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.

NOTES

8 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.

9 Tubes meeting these requirements are commercially available, however they may also be made by the user.

5.2 Polyethylene end caps, for capping charcoal tubes (5.1). The caps shall fit the tubes tightly to prevent leakage.

5.3 Pump, with adjustable flow rate, capable of being worn by a person while carrying out his normal work, and capable of operating continuously for 8 h at the flow rate used (see clause 6). The volume of air sampled by the pump during the recommended sampling period shall be within 10 % of the calculated volume. The flow rate shall be constant within 5 % during the sampling period.

Calibrate the pump with a representative charcoal tube (5.1) in line, using the soap bubble meter (5.5).

The pump shall be in accordance with local safety regulations.

5.4 Plastics or rubber tubing, of length about 90 cm and of a diameter which is suitable for ensuring a leak-proof fit to both the pump and sample tube or tube holder, if used.