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Workplace air — Determination of vinyl chloride — Charcoal tube/gas chromatographic method

*Air des lieux de travail — Détermination du chlorure de vinyle — Méthode par tube à charbon
actif/chromatographie en phase gazeuse*

Reference number
ISO 8762 : 1988 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8762 was prepared by Technical Committee ISO/TC 146, *Air quality*.

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

Workplace air — Determination of vinyl chloride — Charcoal tube/gas chromatographic method

1 Scope

This International Standard specifies a charcoal tube/gas chromatographic method for the determination of the vinyl chloride monomer concentration in workplace air.

The method is valid for vinyl chloride concentrations in the range from $250 \mu\text{g}/\text{m}^3$ ($\approx 100 \mu\text{l}/\text{m}^3$) to $25 \text{ mg}/\text{m}^3$ ($\approx 10 \text{ ml}/\text{m}^3$) when sampling 30 litres of air^[1].

NOTE — The upper limit of the useful range is set by the adsorptive capacity of the charcoal tube used. This capacity is measured as a break-through volume of air, which volume should not be exceeded during sampling. The lower limit is set by a number of parameters, including the noise level of the detector, blank concentrations due to contamination of the charcoal and carbon disulfide by vinyl chloride, poor desorption efficiency at very low sample loadings, and interference of the solvent peak in the gas chromatographic analysis.

This procedure is compatible with low flow-rate personal sampling equipment and can be used for personal and fixed location sampling. It cannot be used to measure instantaneous or short-term fluctuations in vinyl chloride concentrations. Alternative on-site procedures, such as gas chromatography or infra-red spectrometry, should be used to measure rapidly changing concentrations. Organic components which have the same or nearly the same retention time as vinyl chloride during the gas chromatographic analysis will interfere. Interferences can be minimized by proper selection of gas chromatographic columns and programme conditions.

For certain applications, alternative detectors such as the photo-ionization detector may offer greater selectivity and sensitivity to vinyl chloride.

Water mists, high relative humidity, and other vapours at high concentrations reduce the adsorptive capacity of the charcoal for vinyl chloride.

The sampling method gives a time-weighted average result.

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 listed 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 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 vinyl chloride is adsorbed onto the charcoal. The collected vinyl chloride is desorbed using carbon disulfide and analysed with a gas chromatograph equipped with a flame ionization detector.

4 Reagents

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

4.1 Vinyl chloride liquid in a small gas cylinder.

WARNING — Vinyl chloride is recognized as a human carcinogen. Avoid any exposure by inhalation or skin contact. Personal protection (e.g. an effective respirator) shall be available in all cases where exposure to vinyl chloride is possible.

Vinyl chloride vapour is highly flammable. Usage shall be restricted to a well-ventilated fume cupboard. An effective fire extinguisher shall be permanently available.

4.2 Carbon disulfide (CS_2), chromatographic quality, previously checked for compounds coincident with the vinyl chloride peak. The CS_2 shall not contain impurities that co-elute with vinyl chloride giving a response greater than 0,06 ng of vinyl chloride at an injection volume of 1 μl . This requirement shall be checked for each batch of CS_2 .

WARNING — Carbon disulfide vapour is toxic. Avoid any exposure by inhalation or skin contact. Carbon disulfide is highly flammable. Usage shall be restricted to a well-ventilated fume cupboard. An effective fire extinguisher shall be permanently available.

Small waste quantities of carbon disulfide shall be disposed of only in accordance with local regulations and accepted practices.

4.3 Activated coconut shell charcoal, particle size 0,35 mm to 0,85 mm. Before packing the tubes, the charcoal shall be heated in an inert atmosphere, e.g. high-purity nitrogen, at approximately 600 °C for 1 h. To prevent recontamination of the charcoal, it shall be kept in a clean atmosphere during cooling to room temperature, storage and loading into the tubes.

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

4.4 Vinyl chloride standard solutions for the preparation of the calibration graph.

4.4.1 Vinyl chloride standard solution, containing 260 mg of $\text{C}_2\text{H}_3\text{Cl}$ per litre.

Pipette 10,0 ml of the carbon disulfide (4.2) into a vial (5.10), cap the vial and cool in dry ice. Obtain pure vinyl chloride vapour at atmospheric pressure by partially filling a previously evacuated gas bag (5.11) with pure gas from the small gas cylinder (4.1).

Fill a syringe (5.8) with 1,0 ml of the vinyl chloride vapour from the gas bag and close the valve of the syringe. Insert the tip of the needle through the septum cap into the carbon disulfide. Open the valve and withdraw the plunger slightly to allow the carbon disulfide to enter the syringe. The action of the vinyl chloride dissolving in the carbon disulfide creates a vacuum and the syringe becomes filled with the solvent. Return the solution to the vial. Flush the syringe twice with the solution and return the flushings to the vial.

1 ml of this standard solution contains 260 μg of $\text{C}_2\text{H}_3\text{Cl}$.

NOTE — The vinyl chloride concentrations are given for the case in which the vinyl chloride vapour is injected at 20 °C and 101,3 kPa.

4.4.2 Vinyl chloride standard solution containing 52 mg of $\text{C}_2\text{H}_3\text{Cl}$ per litre.

Add approximately 5 ml of the carbon disulfide (4.2) to a 10 ml one-mark volumetric flask. Pipette 2 ml of the standard solution (4.4.1) into this flask, dilute to the mark with the carbon disulfide and mix well.

1 ml of this standard solution contains 52 μg of $\text{C}_2\text{H}_3\text{Cl}$.

4.4.3 Vinyl chloride standard solution containing 5,2 mg of $\text{C}_2\text{H}_3\text{Cl}$ per litre.

Add approximately 5 ml of the carbon disulfide (4.2) to a 10 ml one-mark volumetric flask. Pipette 1 ml of the standard solution (4.4.2) into this flask, dilute to the mark with the carbon disulfide and mix well.

1 ml of this standard solution contains 5,2 μg of $\text{C}_2\text{H}_3\text{Cl}$.

4.4.4 Vinyl chloride standard solution containing 0,52 mg of $\text{C}_2\text{H}_3\text{Cl}$ per litre.

Add approximately 5 ml of the carbon disulfide (4.2) to a 10 ml one-mark volumetric flask. Pipette 1 ml of the standard solution (4.4.3) into this flask, dilute to the mark with the carbon disulfide and mix well.

1 ml of this standard solution contains 0,52 μg of $\text{C}_2\text{H}_3\text{Cl}$.

4.4.5 Vinyl chloride standard solution containing 0,052 mg of $\text{C}_2\text{H}_3\text{Cl}$ per litre.

Add approximately 5 ml of the carbon disulfide (4.2) to a 10 ml one-mark volumetric flask. Pipette 1 ml of the standard solution (4.4.4) into this flask, dilute to the mark with the carbon disulfide and mix well.

1 ml of this standard solution contains 0,052 μg of $\text{C}_2\text{H}_3\text{Cl}$.

4.5 Vinyl chloride control solutions for checking the calibration graph.

4.5.1 Vinyl chloride control solution, containing 130 mg of $\text{C}_2\text{H}_3\text{Cl}$ per litre.

Prepare this solution as described in 4.4.1, but inject 0,5 ml instead of 1,0 ml of the vinyl chloride vapour.

1 ml of this control solution contains 130 μg of $\text{C}_2\text{H}_3\text{Cl}$.

4.5.2 Vinyl chloride control solution, containing 52 mg of $\text{C}_2\text{H}_3\text{Cl}$ per litre.

Use the solution prepared as described in B.3.1.4.

1 ml of this control solution contains 52 μg of $\text{C}_2\text{H}_3\text{Cl}$.