
Zrak na delovnem mestu - Določevanje vinil klorida - Cevka z aktivnim ogljem/metoda plinske kromatografije

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

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

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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*

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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.

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.

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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 $\text{C}_2\text{H}_3\text{Cl}$.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Charcoal tube, in two sections, 5 mm to 7 mm internal diameter, and packed with activated coconut shell charcoal (4.3), the front section containing 750 mg to 800 mg and the back-up section 200 mg to 250 mg. The sections are separated and their contents held in place with an inert material, e.g. glass wool plugs (preferably silicated).

The pressure drop across the charcoal tube shall not exceed 3 kPa at the maximum flow rate used for sampling (in this International Standard, 200 ml/min). Glass tubes shall be kept in holders. 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.

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

NOTES

- 1 Instead of a commercial two-section tube, two single-section tubes in series may be used with at least 750 mg of charcoal in the first tube. This arrangement has the advantage that it is not necessary to store tubes at sub-ambient air temperatures after sampling.
- 2 Tubes meeting these requirements are commercially available, but they may also be made by the user.

5.2 Polyethylene end caps, for capping charcoal tubes. 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 (between 50 ml/min and 200 ml/min, see clause 6). The volume of air sampled by the pump over the recommended sampling period shall be within 10 % of the calculated volume. The flow rate shall be constant within 5 % over the period of sampling.

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

The pump shall comply with local safety regulations.

5.4 Plastics or rubber tubing, of length about 90 cm and of appropriate diameter to ensure a leak-proof fit to both pump and sample tube or tube holder, if used.

5.5 Soap bubble flowmeter, for calibration of the pump (5.3), capacity 50 ml to 100 ml.

5.6 Syringe, capacity 10 µl, graduated to 0,1 µl.

5.7 Syringe, capacity 50 µl, graduated to 1 µl.

5.8 Syringe, capacity 1 ml, graduated to 0,05 ml, gas-tight.

5.9 Syringe, capacity 20 ml, graduated to 0,5 ml, gas-tight.

5.10 Vials, fitted with septum caps, or glass-stoppered, nominal capacity 10 ml.

5.11 Gas bags, capacity 1 litre or 5 litres, for preparing calibration mixtures. Bags made of inert material, e.g. polyester laminated with aluminium foil, are suitable. Because of the significant solubility of vinyl chloride in polytetrafluorethene (PTFE), bags made of PTFE are unsuitable.

NOTES

- 1 Standard concentrations of vinyl chloride in air may be purchased. Standard concentrations may also be prepared in the laboratory, e.g. dynamically with permeation tubes (see ISO 6349).
- 2 For an alternative method for generating standard atmospheres, see bibliographic reference [1] (annex C).

5.12 Gas chromatograph, with flame ionization detector, capable of detecting 0,25 ng of vinyl chloride with a signal-to-noise ratio of at least 5 to 1.

5.13 Gas chromatography column capable of separating vinyl chloride from other components.

The column suitability shall be verified by testing with two or more columns of dissimilar packing to ensure the absence of interferences. Examples of gas chromatography columns that have been found suitable are presented in table 1. This list is not exhaustive. Capillary columns may also be suitable.

6 Sampling

Attach the pump (5.3) to a charcoal sampling tube (5.1) with the rubber or plastics tubing (5.4), placing the back-up section nearest to the pump.

When a relative humidity above 80 % or high concentrations of other interferences are suspected, use a second charcoal sampling tube connected in series and identical to the first tube.

When using a second charcoal sampling tube in series, ensure that the calibration of the pump has been previously carried out with two tubes in series.

Attach the charcoal sampling tube in a vertical position to the lapel of the operator for personal sampling. Then attach the pump to the operator as appropriate to minimize inconvenience. For fixed location sampling choose a suitable sampling site.

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Table 1 — Characteristics of gas chromatography columns capable of separating vinyl chloride from other components

Column	1	2	3	4	5	6	7
Length m	3	6	1,8	1,8	1,8	2	1,5
Internal diameter mm	2,2	2,2	2,2	2,2	2,2	2,2	2,2
Stationary phase	DC 200	SE 30	Porapak Q	Chromosorb 102	Carbowax 1500	picric acid	Porapak S Porapak T 80 : 20
Support	Chromosorb W/HP	Chromosorb W/AW/DMCS			Carbopak A	Carbopak C	
Particle size μm	135 to 177	135 to 177	—	135 to 177	—	80 to 100	80 to 100
Stationary phase on support %	10	10	—	—	0,4	0,19	—
Column temperature $^{\circ}\text{C}$	75	60	135	145	ambient	50	110
Bibliographic reference	[3]	[4]	[5]	[6]	[7]	[8]	[9]

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Turn the pump on and adjust the flow rate to provide a sampling rate of 100 ml/min to 200 ml/min for a 4 h sampling period or 50 ml/min to 100 ml/min for an 8 h sampling period. Note and record the time, temperature, flow rate and barometric pressure when the pump was turned on.

If the samples are not to be analysed within 8 h, store them in a sealed metal or glass container placed either in dry ice or in a freezer maintained at -20°C or lower.

NOTES

NOTES

1 The adsorption efficiency does not significantly change at flow rates lower than 200 ml/min.

2 The sampling efficiency will be 100 % provided that the sampling capacity of the charcoal is not exceeded. If this capacity is exceeded, break-through of the vapour from the front section to the back-up section (or second charcoal tube, if used) will occur. The break-through volume is defined and may be determined as specified in annex A. The break-through volume varies with ambient air temperature, relative humidity, concentration of vinyl chloride and of other contaminants, and with the sampling flow rate. Increases in any of these parameters cause a reduction of the break-through volume. A typical break-through volume for coconut shell charcoal at a concentration of vinyl chloride of $2,6 \text{ mg/m}^3$ and low relative humidity (less than 50 %) is about 100 l/g, e.g. about 75 litres for the front section.

3 A sample volume of about 30 litres is sufficient for determining vinyl chloride concentrations in the range of $250 \mu\text{g/m}^3$ to 25 mg/m^3 without break-through.

At the end of the sampling period, note and record the flow rate, turn the pump off, and note and record the time, temperature and barometric pressure.

Disconnect the sampling tube and seal both ends with the polyethylene end caps (5.2). Place identifying labels on each tube.

NOTE 4 — If a second charcoal tube is used, this should be stored separately.

NOTES

5 Storage for up to 10 days under these conditions has no influence on the results of analysis^[1].

6 Storage at -20°C is recommended in order to retard migration and to prevent loss of the sample.

Select one charcoal tube from the same batch of tubes used for sampling and subject it to the same procedure as the sample tubes except that no air shall be drawn through it. Label this as the blank.

7 Procedure

7.1 Calibration

Analyse each vinyl chloride standard solution (4.4) in triplicate by gas chromatography.

Introduce in a reproducible way a known aliquot in the range of $1 \mu\text{l}$ to $5 \mu\text{l}$ of the standard solution into the gas chromatograph (5.12). The volume of the aliquot shall be the same for each sample standard solution.

In the case of overloading the chromatographic system, smaller sample volumes shall be used. The lower limit of detection will then be increased accordingly.

Prepare a calibration graph by plotting the areas of the vinyl chloride peaks, corrected for the blank value (carbon disulfide), against the mass concentrations of the vinyl chloride standard solutions, in micrograms per millilitre.

Check the calibration graph by analysing the control solutions (4.5). If the difference is more than 5 %, carry out a new calibration.

7.2 Determination

Pipette exactly 5,0 ml of the carbon disulfide (4.2) into a 10 ml vial (5.10) and cap the vial immediately. Cool in dry ice for about 2 min.

Score the charcoal tube containing the sample in the front (larger) section of charcoal and break open the tube. Remove the glass wool and discard it. Open the vial and transfer the charcoal from the front section to this vial and close the vial. Repeat the same procedure for the back-up section, using a different vial. Allow the contents to desorb for 30 min, with occasional swirling.

Inject the same volume of the sample as used for preparing the calibration graph into the gas chromatograph.

Determine the area of the vinyl chloride peak. Read from the calibration graph the corresponding mass concentration, in micrograms per millilitre, of vinyl chloride in the injected sample.

If the back-up section contains more than 10 % of the vinyl chloride contained in the front section, discard the sample as unreliable.

Analyse the blank tube in the same manner. Analyse a freshly prepared standard solution (4.4) with each batch of samples.

8 Expression of results

8.1 Calculation

The mass concentration of vinyl chloride in the air sample $\varrho(\text{VC})$, in milligrams per cubic metre, is given by the equation

$$\varrho(\text{VC}) = \frac{(\varrho_1 + \varrho_2 - \varrho_3) V_d}{D \cdot V}$$

where

ϱ_1 is the mass concentration of vinyl chloride, in micrograms per millilitre, in the solution prepared from the front section of the charcoal tube;

ϱ_2 is the mass concentration of vinyl chloride, in micrograms per millilitre, in the solution prepared from the back-up section of the charcoal tube ($\varrho_2 < 0,1 \varrho_1$);

ϱ_3 is the mass concentration of vinyl chloride, in micrograms per millilitre, in the solution prepared from both sections of the blank;

V_d is the volume of carbon disulfide, in millilitres, used for desorption (5 ml);

D is the desorption efficiency as read from the D -curve, taking $(\varrho_1 + \varrho_2 - \varrho_3) V_d$ as the mass of vinyl chloride found (B.2.6);

V is the volume, in litres, of the air sample.

NOTES

1 If the vinyl chloride concentration has to be expressed at reference conditions (293 K and 101,3 kPa), use the following equation:

$$\varrho(\text{VC})_{\text{corr}} = \varrho(\text{VC}) \times \frac{101,3 (t + 273)}{p \times 293}$$

where

$\varrho(\text{VC})$ is the concentration of vinyl chloride, in milligrams per cubic metre, as calculated above;

t is the temperature, in degrees Celsius, during sampling;

p is the atmospheric pressure, in kilopascals, during sampling;

101,3 is the standard atmospheric pressure, in kilopascals;

273 is the standard temperature, in kelvins;

293 is the temperature in kelvins corresponding to 20 °C.

2 The volume fraction $\phi(\text{VC})$ of vinyl chloride in the air sample, in millilitres per cubic metre (ppm), is given by the equation:

$$\begin{aligned} \phi(\text{VC}) &= \varrho(\text{VC})_{\text{corr}} \times \frac{24,0}{62,5} \\ &= \varrho(\text{VC})_{\text{corr}} \times 0,384 \end{aligned}$$

$\varrho(\text{VC})_{\text{corr}}$ is the vinyl chloride concentration, in milligrams per cubic metre, at reference conditions (8.1, note 1);

24,0 is the molar volume of an ideal gas, in litres per mole, at 20 °C and 101,3 kPa;

62,5 is the relative molar mass, in grams per mole, of vinyl chloride.

8.2 Precision

Preliminary investigations by a collaborative study^[1] in the range of 0,2 mg/m³ to 18 mg/m³ indicated a repeatability of about 22 % (a coefficient of variation of about 8 %) and a reproducibility of about 45 % calculated as described in ISO 5725.

9 Test report

The test report shall include at least the following information:

- a complete identification of the sample;
- a reference to this International Standard;
- the place and period of sampling;
- the atmospheric pressure and temperature;
- the test result;
- any unusual features noted during the determination;
- any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.