

# Vinyl chloride – Determination of vinyl chloride in workplace air using gas chromatography

## Air Monitoring Method

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## Abstract

This analytical method is a validated measurement procedure for the determination of vinyl chloride [75-01-4] in workplace air averaged over the sampling period after personal or stationary sampling. Sampling is performed by drawing a defined volume of air through an adsorption tube filled with carbon black and a carbon molecular sieve using a suitable flow-regulated pump. The flow rate is set to 20 ml/min with a recommended air sample volume of 1.2 l (which corresponds to a sampling period of 60 min). After thermal desorption at 300 °C the sample is analysed by means of GC with a flame ionisation detector (FID) or preferably with a mass spectrometric detector (MSD). The relative limit of quantification (LOQ) is 0.00023 mg/m<sup>3</sup> vinyl chloride for an air sample volume of 1.2 l. The mean recovery was 93% for an evaluation with MSD and the expanded uncertainty for vinyl chloride was 15% over the entire measurement range. This method has been tested and recommended for the determination of vinyl

chloride in workplaces by the German Social Accident Insurance. Both personal and stationary sampling can be performed for measurements for the evaluation of work areas.

## 1 Summary

The mean concentration of vinyl chloride at the workplace over the sampling period can be determined with this method using personal or stationary sampling.

Measurement Principle:	<p>A pump draws a defined volume of air through an adsorption tube filled with carbon black and a carbon molecular sieve. A desiccant tube is connected upstream to capture interfering moisture in the air.</p> <p>The deposited vinyl chloride is then thermally desorbed. Vinyl chloride can be determined using a mass spectrometric detector (MSD) and flame ionisation detector (FID) after gas chromatographic separation and splitting the eluate by means of the outlet flow splitter. In this case determination by means of MSD is preferable over FID, as MSD has a lower limit of quantification and a measurement signal that is largely free of interference.</p> <p>Calibration is carried out using test gases according to the internal (MSD) standard method.</p> <p>The method is developed for an air sample volume of 1.2 l, which allows detection of vinyl chloride concentrations of up to 4.4 mg/m<sup>3</sup>. If higher concentrations of vinyl chloride are present, then vinyl chloride concentrations up to 18 mg/m<sup>3</sup> can be captured by reducing the air sample volume to 0.3 l.</p>
Limit of quantification:	<p>Absolute: 0.000275 µg</p> <p>Relative: 0.00023 mg/m<sup>3</sup> for an air sample volume of 1.2 l.</p>
Measurement range:	From 0.00023 mg/m <sup>3</sup> to 4.5 mg/m <sup>3</sup> for an air sample volume of 1.2 l or 0.00091 mg/m <sup>3</sup> to 17.8 mg/m <sup>3</sup> for an air sample volume of 0.3 l.
Selectivity:	High due to the use of a mass spectrometer.
Advantages:	Personal and selective measurements are possible.
Disadvantages:	No indication of concentration peaks.
Apparatus:	<p>Pump</p> <p>Flow meter</p> <p>SVI type multi-bed tubes</p> <p>Thermal desorber, gas chromatograph with FID and MSD</p>

## 2 Equipment and chemicals

### 2.1 Equipment

For sampling:

- Sampling pump, suitable for a flow rate of 5 to 40 ml/min, e.g. PP1, from Gilian, supplied by DEHA Haan & Wittmer, 71296 Heimsheim, Germany
- Flow meter, e.g. Gilibrator, from Gilian
- TC-20 tube conditioner, Markes International GmbH, 63065 Offenbach am Main, Germany

- Multi-bed Soil Vapour Intrusion (SVI) tubes made of stainless steel (6.3 mm x 90 mm, 5 mm inner diameter), filled with carbon black and a carbon molecular sieve e.g. from PerkinElmer LAS, 63110 Rodgau, Germany, Order No. N9306277 (The adsorption tubes contain a sequence of adsorbents with increasing adsorption power, therefore it is important to ensure that sampling is carried out in the correct direction)
- SVI tube configuration: 125 mg of Carbograph 2, 125 mg of Carbograph 1 plus 65 mg of Carboxen 1003, the adsorbent layers are separated by 3 mm glass wool plugs, Carbograph 2 is equivalent to Carbopack C; Carbograph 1 is equivalent to Carbopack B.
- Empty glass tubes (6.3 mm x 90 mm, inner diameter of 4 mm), e.g. from PerkinElmer LAS, Order No. L4071594
- Stoppers for tubes with an inner diameter of 4 mm, from Gerstel, 45473 Mühlheim an der Ruhr, Germany, Order No. 008980-010-00
- Sealing caps made of brass for transportation and storage, ¼-inch Swagelok with PTFE seals, e.g. from PerkinElmer, Order No. 0990-8851
- Sealing caps made of PTFE for analysis, e.g. from PerkinElmer, Order No. N620-0119
- ¼-inch Swagelok fitting with PTFE cones for connection of desiccant tubes and adsorption tubes, from Swagelok, 63477 Maintal-Dörnigheim, Germany, Order No. SS-400-6

For analysis:

- Syringe pump (diluter/dispenser), e.g. Microlab M, from Hamilton, supplied by DURATEC Analysetechnik GmbH, 68766 Hockenheim, Germany
- Thermal desorber with a gas dosing loop (for the internal standard), gas chromatograph with flame ionisation detector (FID) and/or mass-selective detector (MSD)
- Graphpack-3D/2-eluate flow splitter, from Gerstel, Order No. GC 08194-40, split ratio: 1:1 (FID/MSD)

## 2.2 Chemicals

- Lithium chloride, p.a., e.g. from Merck KGaA, 64293 Darmstadt, Germany, Order No. 105679
- Chromosorb W-AW (60-80 mesh), e.g. from CS, 52379 Langerwehe, Germany
- Distilled water
- Silanised glass wool, e.g. from PerkinElmer LAS, Order No. 0009 798
- Vinyl chloride test gas in nitrogen, approx. 10 ppm, e.g. from AirLiquide Deutschland GmbH, 40235 Düsseldorf, Germany
- 4-Bromofluorobenzene test gas in nitrogen, approx. 10 ppm (BFB, internal standard), e.g. from Praxair Deutschland GmbH, 64584 Biebesheim, Germany

Gases for the operation of the gas chromatograph and the tube conditioner:

- Helium, purity 99.996%
- Hydrogen, purity 99.999%
- Synthetic compressed air, free of hydrocarbons, dew point lower than -40 °C
- Nitrogen, purity 99.999%

## 2.3 Preparation of the desiccant tubes

6.5 g of lithium chloride are dissolved in 10 ml of distilled water and mixed with 10 g of Chromosorb W-AW in a porcelain sample dish. After homogenisation, it is dried at 120 °C in the drying cabinet.

165 mg of the mixture, equivalent to 65 mg of lithium chloride, are filled into an adsorption tube made of glass. Plugs made of silanised glass wool and fixing plugs are introduced on either side in order to hold the lithium chloride in place. At a flow rate of 60 ml/min the flow resistance for the desiccant tube/adsorption tube combination is less than 10 hPa, if the desiccant tube is not too densely packed.

Before sampling, the desiccant tubes are prepared in a stream of nitrogen at 50 ml/min for 20 min at 120 °C in the tube conditioner. They are then tightly sealed with Swagelok caps.

## 2.4 Test gases and calibration standards

A test gas with a concentration of approx. 10 ppm of vinyl chloride in nitrogen is used for calibration. This is either used undiluted (Test Gas 1) or used mixed dynamically with synthetic compressed air (Test Gas 2) (DIN 2020).

**Tab.1** Test gas concentrations and calibration masses

Test Gas	1	2
Dilution factor	1	231
Concentration [mg/m <sup>3</sup> ] at 20 °C and 1013 hPa	26.7	0.116
Sample volume [ml]	Mass per sample [µg]	
1	0.0267	0.000116
2	0.0533	0.000231
3	0.0800	0.000347
4	0.107	0.000462
5	0.133	0.000578
6	0.160	0.000693
7	0.187	0.000809
8	0.213	0.000924
9	0.240	0.00104
10	0.267	0.00116
20	0.533	
30	0.800	
40	1.07	
50	1.33	
60	1.60	
70	1.87	
80	2.13	
90	2.40	
100	2.67	
150	4.00	
200	5.33	

SVI tubes are connected to the suction side of the syringe pump and different aliquots of Test Gas 1 are drawn through these in the range of 1 to 200 ml. The test gas concentrations shown in [Table 1](#) result in the corresponding calibration masses.

The limit of quantification was determined by drawing aliquots of 1 to 10 ml of Test Gas 2 through the SVI tubes.

The calibration samples of Test Gas 1 are equivalent to the concentrations shown in [Table 2](#), based on an air sample volume of 1.2 l or 0.3 l.

**Tab. 2** Concentrations of vinyl chloride in the calibration samples prepared from Test Gas 1 in mg/m<sup>3</sup> (based on an air sample volume of 1.2 l or 0.3 l)

Volume of Test Gas 1 [ml]	Vinyl chloride mass per sample [µg]	Vinyl chloride concentration at 1.2 l [mg/m <sup>3</sup> ]	Vinyl chloride concentration at 0.3 l [mg/m <sup>3</sup> ]
1	0.0267	0.0223	0.0890
2	0.0533	0.044	0.178
3	0.0800	0.067	0.267
4	0.107	0.089	0.357
5	0.133	0.111	0.443
6	0.160	0.133	0.533
7	0.187	0.156	0.623
8	0.213	0.178	0.710
9	0.240	0.200	0.800
10	0.267	0.223	0.890
20	0.533	0.444	1.78
30	0.800	0.667	2.67
40	1.07	0.892	3.57
50	1.33	1.11	4.43
60	1.60	1.33	5.33
70	1.87	1.56	6.23
80	2.13	1.78	7.10
90	2.40	2.00	8.00
100	2.67	2.23	8.90
150	4.00	3.33	13.3
200	5.33	4.44	17.8

### 3 Sampling

Pre-treated adsorption tubes with desiccant tubes connected upstream are used for sampling (see [Sections 2.1](#) and [2.3](#)). The sealing caps are removed at the beginning of sampling and the desiccant tube is connected to the adsorption tube by means of the Swagelok screw fitting, which is in turn connected to the sampling pump.

The recommended sampling period is 60 minutes at a flow rate of 20 ml/min, which is equivalent to an air sample volume of 1.2 l. Differing sampling periods for shorter or longer periods, such as 30 minutes or 4 hours, are possible. However, the flow rate must be adapted to the intended sampling period. If vinyl chloride concentrations greater than 4.5 mg/m<sup>3</sup> are anticipated, then the intended air sample volume should be reduced to 0.3 l. Thus, concentrations of up to 17.8 mg/m<sup>3</sup> can be determined.

The important parameters for the determination of the concentration (sample volume, temperature, air pressure and relative humidity) are documented in the sampling record.

The pump and combined tubes are either worn by the person while performing their activities or stationary sampling is carried out. On completion of sampling, Swagelok screw caps are used to seal both ends of the loaded adsorption tube. The desiccant tube is also sealed and must be conditioned at 120 °C in a stream of nitrogen (see [Section 2.3](#)) before it can be reused.

After sampling, the flow rate must be tested for constancy. If the deviation from the adjusted flow rate is greater than 5%, it is advisable to repeat sampling (see DGUV Information 213-500 “General Part”, Section 3 (DGUV 2015)).

## 4 Analytical determination

### 4.1 Thermal desorption conditions

The adsorption tubes are fitted with desorption caps and transferred into the autosampler. In this case it is important to ensure that the multi-bed tubes are heated in the opposite direction in the thermal desorber than that for sampling. Before desorption, the internal standard is transferred to the previously loaded tubes by means of a gas dosing loop. Then a carrier gas transfers the adsorbed components to a cold trap packed with Tenax TA. After desorption from the adsorption tube, the cold trap is heated so that the vinyl chloride and the internal standard reach the separation column.

The thermal desorber is set to the following instrumental conditions:

Apparatus:	Turbomatrix 650 (PerkinElmer LAS)
Desorption temperature:	300 °C
Desorption period:	10 min
Valve temperature:	220 °C
Temperature of transfer line:	200 °C
Transfer line:	1.5 m of deactivated blank capillaries, ID 0.32 mm
Cold trap (adsorption):	-30 °C
Cold trap (injection):	280 °C
Cold trap filling:	20 mg of Tenax TA
Heating rate:	40 °C/s
Carrier gas:	Helium
Carrier gas flow:	1.7 ml/min
Split (before the cold trap):	0 ml/min (input split)
Flow over the cold trap:	30 ml/min (desorption flow)
Split (after the cold trap):	10 ml/min (output split)
Purge drying phase:	1 min at room temperature at 50 ml/min
Gas dosing loop (accessory for the internal standard):	
Volume:	5 ml
Filling period:	2 min
Equilibration period:	2 min
Flooding period:	2 min
Rinse flow:	50 ml/min

## 4.2 Operating conditions for chromatography

### GC conditions

Apparatus:	Clarus 600 gas chromatograph with flame ionisation detector (FID), Clarus 600T mass-selective detector (MSD), from PerkinElmer LAS
Column:	Material: Fused silica capillary Stationary phase: DB-624 (6% cyanopropylphenylpolysiloxane and 94% dimethylpolysiloxane) Length: 30 m Inner diameter: 0.25 mm Film thickness: 1.4 µm
Eluate flow splitter:	Graphpack-3D/2-eluate flow splitter, Order No. GC 08194-40 from Gerstel, split ratio: 1:1 (FID/MSD)
Detector temperature (FID):	320 °C
Detector gases:	Hydrogen (45 ml/min), synthetic air (450 ml/min)
Rate of heating:	10 min at 35 °C, increased by 10 °C/min up to 240 °C, 10 min

### MS conditions

Temperatures:	Ion source: 180 °C Transfer line: 200 °C
Type of ionisation:	Electron impact (EI)
Ionisation energy:	70 eV
Mass range:	
Internal standard (BFB):	Full-scan measurement 35 – 520 amu (the sum of the masses 95, 174 and 176 are used for evaluation)
Vinyl chloride:	SIM mode: m/z 62 Dwell time (sec): 0.400 Interchannel delay (sec): 0.005

## 5 Evaluation

### 5.1 Calibration

The calibration standards prepared according to [Section 2.4](#) are analysed as described in [Sections 4](#). The calibration function is obtained by plotting the resulting peak area ratios of the SIM signal of vinyl chloride to the area of the sum of the masses 95, 174 and 176 of the full-scan signals of BFB versus the respective loaded masses (see [Table 1](#)). The calibration function is linear in the investigated concentration range and should be checked regularly during routine analysis.

### 5.2 Calculation of the analytical result

Based on the determined peak area ratios and taking the mass of internal standard into consideration, the corresponding mass  $m$  in µg per sample is obtained from the calibration curve. The corresponding mass concentration  $c$  is calculated according to the following [Equation 1](#):

$$c = \frac{m}{V \times \eta} \quad (1)$$

where:

- $c$  is the mass concentration of vinyl chloride in the air in  $\text{mg}/\text{m}^3$   
 $m$  is the mass of vinyl chloride in the analytical sample in  $\mu\text{g}$   
 $V$  is the air sample volume in litres  
 $\eta$  is the recovery (see Section 6.1)

## 6 Reliability of the method

### 6.1 Precision and recovery

The precision in the lowest measurement range according to EN 482 (DIN 2015) as well as the recovery for 4 concentrations (see Table 4) was determined. In this case Test Gas 1 (see Section 2.4) was used.

The syringe pump is used to draw 6 samples in each case with test gas aliquots (see Table 3) through the combined desiccant tube/multi-bed tube at a flow rate of 25 ml/min and at an ambient temperature of 23 °C. Then 1.2 l of clean air with a relative humidity of 80% were drawn through the tube. The preparation and analytical determination were carried out as described in Section 4. Due to a problem with blank values arising in the FID, evaluation was carried out subsequently by means of the MSD.

**Tab.3** Vinyl chloride masses for the validation with Test Gas 1

Sample series	Sampling conditions	Test gas aliquot [ml]	Vinyl chloride mass [ $\mu\text{g}$ ]	Vinyl chloride concentration based on 1.2 l [ $\text{mg}/\text{m}^3$ ]
1	24 °C, 999 hPa	10	0.260	0.216
2	22 °C, 999 hPa	60	1.57	1.31
3	23 °C, 1000 hPa	100	2.61	2.17
4	22 °C, 1026 hPa	200	5.37	4.47

**Tab.4** Precision and recovery (MSD)

Vinyl chloride mass [ $\mu\text{g}$ ]	Relative standard deviation [%]	Recovery [%]
0.243	8.0	93
1.46	3.3	93
2.43	2.7	93
5.37	9.3	94

The mean recovery is 93% when evaluation is carried out using the MSD.

### 6.2 Breakthrough volume

The SVI tube type used for the adsorption of vinyl chloride was developed specifically for the determination of air pollutants according to the TO-17 EPA method (Varisco et al. 2009). In this case, the adsorbent combination was selected to ensure that, if an air sample volume of no more than 10 l is adhered to, then no breakthrough of the adsorbed vinyl chloride should be anticipated.

During laboratory experiments with air volumes of up to 5 l no breakthrough was observed.



### 6.3 Limit of quantification

The limit of quantification for the MSD was determined as stipulated in DIN 32645 (DIN 2008) according to the calibration line method with 10 test gas standards in each case. Test Gas 2 was used in this case. The result is shown in Table 5.

**Tab.5** Limit of quantification according to the calibration line method (P=95% and k=3)

Substance	Test gas concentration [mg/m <sup>3</sup> ]	Mass range from – to [µg]	Limit of quantification	
			Absolute [µg]	Relative <sup>a)</sup> [mg/m <sup>3</sup> ]
Vinyl chloride	0.116	0.000116 – 0.00116	0.00027	0.00023

<sup>a)</sup> based on an air sample volume of 1.2 l

### 6.4 Storage stability

Storage stability was determined using six sample carriers loaded with Test Gas 1 (see Section 2.4). For this purpose, 10 ml, 100 ml and 200 ml of the test gas were drawn through the combined tubes at a flow rate of 25 ml/min, followed by 1.2 l of clean air at a flow rate of 20 ml/min and at a relative humidity of approx. 50%. Under the sampling conditions (T=23 °C, p=995 hPa) 10 ml of the test gas at a theoretical air sample volume of 1.2 l is equivalent to a vinyl chloride concentration of 0.216 mg/m<sup>3</sup>. 100 ml of the test gas is equivalent to a vinyl chloride concentration of 2.16 or 8.66 mg/m<sup>3</sup> at an air sample volume of 1.2 or 0.3 l (T=23 °C, p=997 hPa). 200 ml of the test gas is equivalent to a vinyl chloride concentration of 4.44 or 17.8 mg/m<sup>3</sup> at an air sample volume of 1.2 or 0.3 l (T=23 °C, p=1023 hPa).

The multi-bed tubes were then sealed at both ends with Swagelok screw caps. The loaded tubes were stored at room temperature.

The preparation and analytical determination were carried out as described in Section 4.

The investigations for the aliquots of 10 and 100 ml were carried out over time periods of 2 and 4 weeks, for the experimental series with 200 ml of test gas a time period of 2 weeks was selected. The results are shown in Table 6.

**Tab.6** Storage stability

Substance	Recovery			
	Storage period [weeks]	Mass [µg]	η (n=3)	mean η
Vinyl chloride	2	0.26	0.246	0.95
	4		0.205	
	2	2.6	2.64	1.02
	4		2.26	
	2	5.3	5.1	0.96
	4			

The mean recovery was 97% for a storage period of 2 weeks without losses.

### 6.5 Selectivity

The selectivity depends above all on the type of separation column used. The specified column has proved successful in practice. Interference can arise from substances with the same retention time when the FID is used for determination. These interferences are largely eliminated when the mass spectrometric detector is used for evaluation.

## 6.6 Uncertainty

The expanded uncertainty was determined taking all relevant influencing factors into consideration as stipulated in DIN EN 482 (DIN 2015). The uncertainty of the entire method and thus also of the analytical result consists principally of the uncertainty contributions of the air sampling (e.g. air sample volume, deviation from the sampling convention) and the analytical preparation (complete desorption, scatter of the calibration function, fluctuations in the recovery and the reproducibility). The expanded uncertainty was 15% over the entire measurement range (see Table 7).

**Tab. 7** Expanded uncertainties

Vinyl chloride concentration [mg/m <sup>3</sup> ]	0.216	1.31	2.17	4.47
U (%)	15	15	15	15

## 6.7 Remarks

The method was developed for the purpose of evaluating ambient air, which may be affected by ground contamination due to chlorinated hydrocarbons. In this case other volatile chlorinated hydrocarbons were also taken into consideration in addition to vinyl chloride.

Substance-specific measurement parameters were selected in the SIM mode for the measurement of individual volatile chlorinated hydrocarbons. The resulting limits of quantification are shown in Table 8.

The use of the desiccant tube meant that the majority of the air humidity was deposited in the desiccant tube. The desiccant tube itself allows the volatile components to pass unhindered, the remaining relative humidity of the air sample is less than 11% relative humidity at the outlet of the desiccant tube during sampling.

**Tab. 8** Limits of quantification and detection of volatile chlorinated hydrocarbons at an air sample volume of 2.4 l and detection with MSD

Component <sup>a)</sup>	Detection limit [µg/m <sup>3</sup> ]	Limit of quantification [µg/m <sup>3</sup> ]
1,1-Dichloroethene	0.07	0.24
Dichloromethane	0.09	0.30
trans-1,2-Dichloroethene	0.08	0.27
1,1-Dichloroethane	0.07	0.24
cis-1,2-Dichloroethene	0.10	0.33
Trichloromethane	0.08	0.28
1,1,1-Trichloroethane	0.12	0.38
Carbon tetrachloride	0.11	0.37
1,2-Dichloroethane	0.10	0.34
Trichloroethene	0.12	0.38
1,1,2-Trichloroethane	0.12	0.38
Tetrachloroethene	0.11	0.35

<sup>a)</sup> Listed by the sequence of elution

## Notes

### Competing interests

The established rules and measures of the commission to avoid conflicts of interest ([https://www.dfg.de/en/dfg\\_profile/statutory\\_bodies/senate/health\\_hazards/conflicts\\_interest/index.html](https://www.dfg.de/en/dfg_profile/statutory_bodies/senate/health_hazards/conflicts_interest/index.html)) ensure that the content and conclusions of the publication are strictly science-based.

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