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Method for the determination of chlorinated hydrocarbons in workplace air using gas chromatography after thermal desorption

Air Monitoring Method

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German Social Accident Insurance
Expert Committee Raw Materials and Chemical Industry
Subcommittee Hazardous Substances
Analytical Subcommittee of the Chemistry Board of Experts*

Recognized analysis procedures for carcinogenic mutagenic or substances toxic to reproduction

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This method has been tested and recommended for the determination of chlorinated hydrocarbons in the air at workplaces by the German Social Accident Insurance.

Both personal and stationary sampling can be performed for the assessment of workplaces.

Sampling is carried out with a pump and adsorption onto Chromosorb 106. Analysis is performed by gas chromatography after thermal desorption.

The method is validated for the following substances:

Name	CAS No.	Molar mass
Dichloromethane	75-09-2	84.93
Tetrachloroethylene	127-18-4	165.83
1,1,1-Trichloroethane	71-55-6	133.42
Trichloroethylene	79-01-6	131.39

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Summary

This procedure permits the determination of the concentration of chlorinated hydrocarbons (CHCs) such as dichloromethane, tetrachloroethylene, 1,1,1-trichloroethane and trichloroethylene in working areas averaged over the sampling duration after personal or stationary sampling.

Principle: A defined volume of air is drawn through an adsorption tube filled with Chromosorb 106 using a suitable pump. The adsorbed chlorinated hydrocarbons are then thermally desorbed from the sample tube and determined by means of gas chromatography fitted with a flame ionisation detector (FID). The calibration is carried out with test gases or methanolic calibration solutions containing the corresponding analytes.

Limit of quantification: Absolute: 0.17 to 0.86 µg per CHC
Relative: 0.85 to 4.3 mg/m^3 of CHC for an air sample volume of 200 mL.

Selectivity: Due to the presence of interfering components, too high values are possible. Interferences can be eliminated by using a mass selective detector (MSD) for evaluation.

Advantages: Personal and selective measurements are possible.

Disadvantages: No indication of peak concentrations.

Apparatus: Pump, flow meter, adsorption tube filled with 300 mg Chromosorb 106, thermodesorber, gas chromatograph with FID

Detailed description of the method

Contents

1 Equipment, chemicals and solutions

1.1 Equipment

For sampling:

- Pump, suitable for a flow rate of 5 mL/min (e.g. PP1 from Gilian, supplied by DEHA Haan & Wittmer, 71296 Heimsheim, Germany)
- Volume flow meter, e.g. Gilibrator, Gilian
- Adsorption tubes made of stainless steel (90 mm × 6.3 mm, 5 mm internal diameter), filled with 300 mg of Chromosorb 106 (e.g. from PerkinElmer LAS, 63110 Rodgau, Germany).
  The tube is fixed between two metal sieves. Before use, the adsorption tubes filled with Chromosorb 106 are heated for 10 min in the thermodesorber at 170 °C and tested for blank values. For storage, the tubes are sealed with Swagelok caps.
- Sealing caps (e.g. PTFE or Swagelok with PTFE seals)

For analytical determination:

- Adjustable piston pipette (e.g. MICROMAN M1000, supplied by Gilson International, 65555 Limburg an der Lahn, Germany)
- Microlitre syringe, 10 µL (e.g. Hamilton, supplied by Duratec, 68766 Hockenheim, Germany)
- Volumetric flasks, nominal volume 100 mL
- Dynamic test gas facility
- Syringe pump (diluter/dispenser) (e.g. Microlab M, from Hamilton, supplied by Duratec)
- Thermodesorber, gas chromatograph with flame ionisation detector (FID)

1.2 Chemicals

- Dichloromethane, purity ≥ 99,8%, e.g. Merck, 64293 Darmstadt, Germany
- Tetrachloroethylene, purity ≥ 99,8%, e.g. Merck
- 1,1,1-Trichloroethane, purity ≥ 99,0%, e.g. Merck
- Trichloroethylene, purity ≥ 99,5%, e.g. Merck
- Toluene, purity ≥ 99,9%, e.g. Merck
- Methanol, purity ≥ 99,9%, e.g. Merck

Gases for operation of the gas chromatograph/thermodesorber:

- Helium 4.6 (purity 99,996%)
- Hydrogen 5.0 (purity 99.999%)
- Synthetic air (free of hydrocarbons, dew point lower than –40 °C)
- Nitrogen 5.0 (purity 99.999%)

1.3 Solutions

Stock solution: Mixture of chlorinated hydrocarbons

The chlorinated hydrocarbons listed in Table 1 are weighed exactly to the nearest 0.1 mg into a 100 mL volumetric flask. The flask is then filled to the mark with toluene, shaken and stored in the refrigerator. The stock solution is either used undiluted for producing test gases or used diluted with methanol for direct spiking of the adsorption tubes.

In the refrigerator at approx. 4 to 8 °C the solution is stable for at least 6 months. The date of preparation must be noted on the flask.

Table 1 Masses and concentrations of the chlorinated hydrocarbons in the stock solution

Substance	Mass [g]	Concentration [g/L]
Dichloromethane	20.1503	201.50
Tetrachloroethylene	9.6109	96.11
1,1,1-Trichloroethane	79.6979	796.98
Trichloroethylene	4.3484	43.48

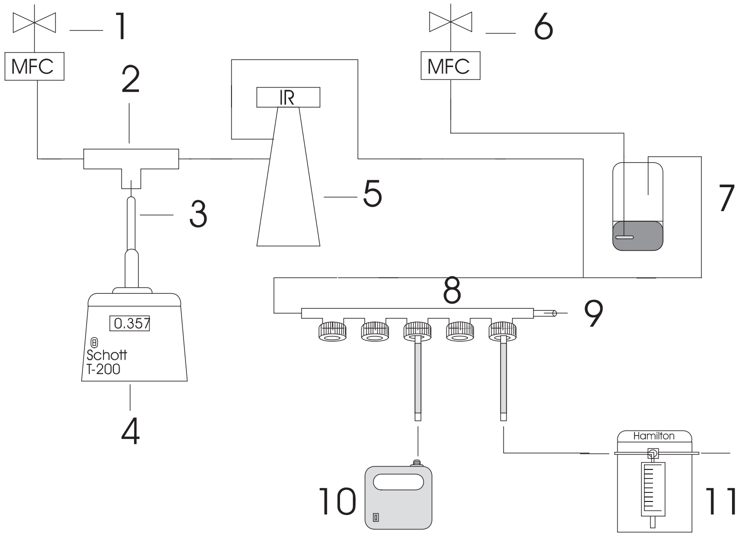
Table 2 Concentrations of the chlorinated hydrocarbons in the calibration solutions in g/L

Substance	Calibration solution [g/L]		
	I	II	III
Dichloromethane	20.2	1.01	0.101
Tetrachloroethylene	9.61	0.481	0.0481
1,1,1-Trichloroethane	79.7	3.98	0.398
Trichloroethylene	4.35	0.217	0.0217

A dilution series is prepared for direct spiking. For that purpose, the stock solution is diluted in methanol in the ratios of 1:10 (calibration solution I), 1:200 (calibration solution II) and 1:2000 (calibration solution III). The concentrations of the calibration solutions are shown in Table 2.

1.4 Test gases and calibration standards

In the case of thermal desorption it is advisable to calibrate with test gases. There are different techniques for preparing test gases [1]. One option for generating test gases is continuous injection (see Figure 1).



1	Basic gas regulator	7	Humidifier
2	Injector	8	Withdrawal manifold
3	Piston	9	Excess test gas
4	Piston burette	10	Sampling pump
5	IR cuvette or buffer vessel	11	Diluter/dispenser
6	Dilution gas regulator		

Figure 1 Schematic illustration of the dynamic test gas facility

The stock solution of the chlorinated hydrocarbons (see Section 1.3 and Table 1) is continuously injected into a dynamic test gas facility at a rate of 120 µL/h into a basic stream of gas with a flow rate of 400 mL/min. The desired test gas concentration is subsequently adjusted with an additional gas flow (dilution gas flow of 1100 mL/min). Adsorption tubes filled with Chromosorb 106 are connected to the suction side of the syringe pump and aliquots from 1 to 500 mL are drawn through the tubes from this calibration atmosphere. The resulted test gas concentrations and calibration masses for a total volume flow of 1500 mL/min are shown in Table 3 and 4.

For calibration by means of direct injection, aliquots of the calibration solutions I, II, and III are applied onto the sieve in sampling direction (see Section 1.3). Immediately, with the help of nitrogen the calibration substances were transferred and adsorbed onto the Chromosorb 106. For this purpose a flow rate of 30 mL/min over

Table 3 Test gas concentrations in mg/m^3

Substance	Dichloro-methane	Tetrachloro-ethylene	1,1,1-Trichloro-ethane	Trichloro-ethylene
Test gas concentration [mg/m^3]	269	128	1062	58

Table 4 Calibration masses resulting from the test gas concentrations

Sample volume [mL]	Mass per sample [µg]			
1	0.27	0.13	1.06	0.06
5	1.34	0.64	5.31	0.29
25	6.72	3.2	26.6	1.45
50	13.4	6.41	53.1	2.90
100	26.9	12.8	106	5.80
150	40.3	19.2	159	8.70
200	53.7	25.6	213	11.6
500	134	64.1	531	29.0

Table 5 Calibration masses for calibration by means of liquid spiking

Spiked volume [µL]	Calibration solution	Dichloro-methane [µg]	Tetrachloro-ethylene [µg]	1,1,1-Trichloro-ethane [µg]	Trichloro-ethylene [µg]
2	III	0.202	0.096	0.797	0.043
5	III	0.504	0.240	1.99	0.109
10	III	1.01	0.481	3.99	0.217
2	II	2.02	0.961	7.97	0.435
5	II	5.04	2.40	19.9	1.09
10	II	10.1	4.81	39.9	2.17
2	I	40.3	19.2	159	8.70
5	I	101	48.1	399	21.7
10	I	202	96.1	797	43.5

Table 6 Concentrations of the chlorinated hydrocarbons in the calibration samples in the case of liquid spiking (with regard to a sample volume of 200 mL)

Spiked volume [µL]	Calibration solution	Dichloromethane [mg/m³]	Tetrachloroethylene [mg/m³]	1,1,1-Trichloroethane [mg/m³]	Trichloroethylene [mg/m³]
2	III	1.01	0.48	3.99	0.218
5	III	2.52	1.20	9.95	0.545
10	III	5.05	2.41	20.0	1.09
2	II	10.1	4.81	39.9	2.18
5	II	25.2	12.0	99.9	5.45
10	II	50.5	24.1	200	10.9
2	I	202	96.0	795	43.5
5	I	505	241	2000	109
10	I	1010	481	3990	218

a period of 20 min is sufficient. The calibration masses obtained for the doped samples are given in Table 5.

The calibration samples (based on an air sample volume of 200 mL) correspond to the concentrations shown in Table 6.

2 Sampling

Pre-treated adsorption tubes are used for sampling (see Section 1.2). At the beginning of sampling the sealing caps of a tube are removed and the tube is connected to the pump. Air is drawn through the adsorption tube at a flow rate of 5 mL/min. The desired sampling duration is 40 min which correspond to an air sample volume of 200 mL. It is necessary to ensure, that an air sample volume of 300 mL may not be exceeded. In cases where a higher flow rate is to be set, the sampling time must be reduced accordingly. The important parameters for the determination of the concentration in air (air sampling volume, temperature, air pressure, relative humidity) are documented in the sampling record.

The pump and adsorption tube are either worn by a person during work or stationary sampling is carried out. Air from the breathing zone is sampled, whereby the adsorption tube opening should be freely accessible. After sampling is complete, the loaded adsorption tube is sealed with Swagelok caps with PTFE seals at both ends. After sampling, the flow rate must be checked for constancy. If the deviation from the adjusted flow rate is greater than ± 5%, it is advisable to reject the measurement and repeat the sampling (for details see DGUV Information 213-500 “General Part“, Section 3 [6]).

3 Analytical determination

3.1 Sample preparation and analysis

After preparing the thermodesorber and the gas chromatograph the calibration and analysis samples are measured. If other thermodesorber devices are used, then the device conditions must be adapted accordingly. The adsorption tubes are sealed with desorption caps and heated in the thermodesorber, whereby the analytes are transferred into the cold trap filled with Tenax TA by means of the carrier gas. After complete desorption, the split outlet is opened and the cold trap is heated. Thus the sample reaches the separation column as a small substance drop.

The thermodesorber is set to the following device conditions:

Apparatus:	Turbomatrix 650 (PerkinElmer LAS)
Desorption temperature:	170 °C
Desorption time:	20 min
Valve temperature:	220 °C
Temperature of transfer line:	200 °C
Transfer line:	1.5 m deactivated blank capillary (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 5.0
Carrier gas flow:	1.7 mL/min
Input split:	200 mL/min
Desorption flow over the cold trap:	10 mL/min
Output split:	20 mL/min
Purge drying phase:	1 min at room temperature at 50 mL/min

3.2 Operating conditions for gas chromatography

Apparatus:	Clarus 600 gas chromatograph with flame ionisation detector (FID) and a Clarus 600T mass selective detector (optional for the purpose of verification the results of the identification), from PerkinElmer LAS.	
Column:	Material:	Fused silica capillary
	Stationary phase:	DB-624 (6% cyanopropylphenyl polysiloxane and 94% dimethylpolysiloxane)
	Length:	30 m
	Internal diameter (ID):	0.25 mm
	Film thickness:	1.4 µm

Temperature program:	10 min at 35 °C, increase by 10 °C/min to 240 °C, 10 min	
Detector temperature:	FID:	320 °C
Detector gases:	Hydrogen (45 mL/min), synthetic air (450 mL/min)	

MS operational conditions

Column flow splitter:	GRAPHPACK 3D/2 flow splitter order No. GC 08240 40 from Gerstel, 45473 Mülheim an der Ruhr, Germany.	
	Split ratio:	1 : 1 (FID/MSD)
Temperatures:	Ion source:	180 °C
	Transfer line:	200 °C
Ionisation mode:	Electron impact (EI)	
Ionisation energy:	70 eV	
Mass range:	35 to 520 amu	

4 Evaluation

4.1 Calibration

The calibration samples prepared according to Section 1.4 are analysed as described in Section 3. The calibration functions are obtained by plotting the calculated peak areas of the FID signals versus the respective masses loaded onto the tubes (cf. Table 3, 4 or 5). The calibration functions are linear in the investigated concentration range and should be regularly checked during routine analysis. For that purpose, a calibration standard of known concentration should be analysed in each analytical series.

4.2 Calculation of the analytical result

On the basis of the peak areas obtained, the corresponding mass m of each sample in µg is determined from the calibration curve. The corresponding mass concentration (c) is calculated according to Equation (1) as follows:

$$c = \frac{m}{V \cdot \eta} \tag{1}$$

where

c is the mass concentration of a component in the air in mg/m^3
m is the mass of the component in the analytical sample in µg
V is the sample volume in litres (calculated from the flow rate and the sampling duration)
η is the recovery (see Section 5.1)

5 Reliability of the method

5.1 Precision and recovery

The precision in the minimum measuring range according to DIN EN 482 [2] and the recovery was determined for four concentrations (see Table 7). For this purpose, a test gas with a relative humidity of 40 to 60% was used. The test gas was generated with the stock solution prepared as described in Section 1.3.

In each case six samples with aliquots of 10, 50, 100, and 200 mL test gas were taken with a syringe pump and drawn through the adsorption tubes (filled with 300 mg Chromosorb 106) with a flow rate of 25 mL/min at an ambient temperature of 23 °C. Then 200 mL clean air with a relative humidity of 50% was drawn through the loaded tube. The preparation and analytical determination was carried out as described in Section 3. The results are shown in Table 7.

Table 7 Concentrations of the chlorinated hydrocarbons in the test gases for validation (based on an air sample volume of 200 mL)

Substance	Test gas concentration [mg/m³]	Validation concentration [mg/m³]			
		c_1	c_2	c_3	c_4
Dichloromethane	546	27.3	137	273	546
Tetrachloroethylene	261	13.0	65.1	130	261
1,1,1-Trichloroethane	2160	108	540	1080	2160
Trichloroethylene	118	5.90	29.5	59.0	118

The results of the recoveries and rel. standard deviations in conducting this experiment in sixfold repetition are given in Tables 8 and 9. Quantitative evaluation is based on a calibration line using liquid doped reference standards.

As part of a comparative measurement two test gases were produced at a relative humidity of 50% and a temperature of 22 °C. From this test gases each three samples were taken from two laboratories and analysed there. The results of the comparative measurement are summarised in Tables 10 and 11.

Table 8 Relative standard deviations for $n = 6$ determination

Substance	Relative Standard deviation [%]				
	s (c_1)	s (c_2)	s (c_3)	s (c_4)	mean s
Dichloromethane	0.90	2.7	1.8	2.1	1.9
Tetrachloroethylene	1.4	1.3	1.7	0.64	1.3
1,1,1-Trichloroethane	0.57	0.83	1.4	1.1	0.98
Trichloroethylene	3.2	0.57	1.7	0.94	1.6

Table 9 Recoveries for n = 6 determination

Substance	Recovery				
	η (c_1)	η (c_2)	η (c_3)	η (c_4)	mean η
Dichloromethane	0.90	0.95	0.99	1.03	0.97
Tetrachloroethylene	0.95	0.94	0.96	0.99	0.96
1,1,1-Trichloroethane	0.94	0.97	1.00	1.00	0.96
Trichloroethylene	0.96	0.96	0.98	1.01	0.98

Table 10 Results of the comparative measurements (test gas 1)

Substance	Test gas concentration [mg/m³]	Laboratory 1 [mg/m³]	Laboratory 2 [mg/m³]	mean recovery η
Dichloromethane	479	458	462	0.96
Tetrachloroethylene	229	225	228	0.99
1,1,1-Trichloroethane	1900	2010	1870	1.02
Trichloroethylene	103	101	103	0.99

Table 11 Results of the comparative measurements (test gas 2)

Substance	Test gas concentration [mg/m³]	Laboratory 1 [mg/m³]	Laboratory 2 [mg/m³]	mean recovery η
Dichloromethane	86.0	82.4	81.6	0.95
Tetrachloroethylene	41.1	40.0	40.4	0.98
1,1,1-Trichloroethane	341	318	340	0.96
Trichloroethylene	18.6	19.2	18.9	1.02

5.2 Limit of quantification

The limits of quantification of the individual chlorinated hydrocarbon were determined in accordance with DIN 32645 [3] using 10-point calibrations with air sample volumes from 1 to 10 mL. The results are given in Table 12.

Table 12 Limits of quantification according to the calibration line method (P = 95% und k = 3)

Substance	Test gas concentration [mg/m³]	Mass range [µg]	Limit of quantification	
			absolute [µg]	relative* [mg/m³]
Dichloromethane	183	0.18 – 1.83	0.19	0.95
Tetrachloroethylene	129	0.13 – 1.29	0.38	1.9
1,1,1-Trichloroethane	1070	1.07 – 10.7	0.85	4.3
Trichloroethylene	58.3	0.06 – 0.58	0.17	0.85

* based on an air sample volume of 200 mL

5.3 Selectivity

The selectivity depends above all on the used separation column. The given separation column (see Section 3.2) has proven successful in practice. The determination can be interfered by substances of the same retention time. If such interferences are recognised by the calculation of the mass spectrometric data, the corresponding samples are determined by the use of the individual substance masses.

5.4 Expanded uncertainty

The expanded uncertainties were estimated taking all relevant influencing factors into consideration as stipulated in DIN EN 482 [2] and DIN EN 1076 [4] and calculated according to [5]. 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 uncertainties ranged from 13 to 23% over the entire measuring range. The results are given in Table 13.

Table 13 Expanded uncertainties for measurement of the chlorinated hydrocarbons

Substance					
Dichloromethane	Concentration [mg/m³]	27.3	137	273	546
	Expanded uncertainty [%]	22.9	21.4	20.7	20.9
Tetrachloroethylene	Concentration [mg/m³]	13.0	65.1	130	261
	Expanded uncertainty [%]	13.4	13.9	14.0	12.6
1,1,1-Trichloroethane	Concentration [mg/m³]	108	540	1080	2160
	Expanded uncertainty [%]	14.5	14.2	13.9	13.7
Trichloroethylene	Concentration [mg/m³]	5.90	29.5	59.0	118
	Expanded uncertainty [%]	14.5	14.2	13.9	13.7

5.5 Storage stability

The storage stability was performed with 12 loaded adsorption tubes with test gas concentrations in the concentration range from a tenth to twice the Occupational Exposure Limits (OELs) at a rel. humidity of approx. 75%. The tubes were sealed with Swagelok caps and stored at room temperature. The experiment was carried out over a period of two and four weeks. The results are shown in Table 14. During a storage period of four weeks no losses were detected, therefore storage stability is ensured over a period of at least four weeks.

Table 14 Results of the storage stability tests

Substance	Recovery [η]				
	Storage period [week]	**Concentration [mg/m^3]**		**η (n = 3)**	**mean η**
Dichloromethane	2	39.6	35.3	0.89	0.90
	4		34.5	0.87	
	2	625	539	0.95	
	4		565	0.90	
Tetrachloroethylene	2	18.9	17.7	0.94	0.94
	4		286	0.96	
	2	289	17.7	0.94	
	4		274	0.92	
1,1,1-Trichloroethane	2	157	145	0.92	0.94
	4		147	0.94	
	2	2470	2370	0.96	
	4		2320	0.94	
Trichloroethylene	2	8.6	8.0	0.93	0.93
	4		7.6	0.97	
	2	135	130	0.89	
	4		124	0.92	

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2 DIN EN 482 (2012) Workplace atmospheres – General requirements for the performance of procedures for the measurement of chemical agents. Beuth Verlag, Berlin, Germany

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4 EN 1076 (2010) Workplace exposure – Procedures for measuring gases and vapours using pumped samplers – Requirements and test methods. Beuth Verlag, Berlin

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