Dichlorobenzene isomers (1,2-dichlorobenzene, 1,3-dichlorobenzene and 1,4 dichlorobenzene)

Method number	1
Application	Air analysis
Analytical principle	Gas chromatography (thermal desorption)
Completed in	December 2010

Summary

This analytical method permits the determination of gaseous dichlorobenzene isomers (1,2-dichlorobenzene, 1,3-dichlorobenzene and 1,4-dichlorobenzene) present in the workplace air in a concentration range from a tenth to twice the currently valid limit value in air [1, 2] by means of adsorption on Tenax TA™ and subsequent thermal desorption.

Sampling is carried out by drawing a defined volume of ambient air through an adsorption tube packed with Tenax TA™ using a suitable sampling pump. After thermal desorption, the dichlorobenzene isomers are determined by gas chromatography with a flame ionisation detector (FID). The quantitative evaluation is based on calibration functions, whereby the concentrations of dichlorobenzene in the reference standards are plotted versus the resulting peak areas.

Characteristics of the method

Precision:

1,2-Dichlorobenzene
Standard deviation (rel.): $s = 1.8$ to 7.9%
Expanded uncertainty: $U = 11.9$ to 21.8%
in the concentration range from 3.3 to 129.8 mg/m³ and $n = 6$ determinations

1,3-Dichlorobenzene
Standard deviation (rel.): $s = 2.2$ to 7.1%
Expanded uncertainty: $U = 12.0$ to 19.8%

in the concentration range from 6.4 to 254.3 mg/m³ and $n = 6$ determinations

1,4-Dichlorobenzene

Standard deviation (rel.): $s = 2.1$ to 7.4 %
Expanded uncertainty: $U = 12.2$ to 21.0 %

in the concentration range from 5.3 to 212.1 mg/m³ and $n = 6$ determinations

Limit of quantification:	1,2-Dichlorobenzene	0.38 mg/m³
	1,3-Dichlorobenzenel	0.21 mg/m³
	1,4-Dichlorobenzenel	0.90 mg/m³

at an air sample volume of 200 mL, a relative uncertainty of 33% ($k = 3$) and a statistical certainty (P) of 99%

Recovery: $\eta > 0.99$ (> 99%)

Sampling recommendation:	Air sample volume:	0.2 L
	Flow rate:	0.3 L/h

## Description of the substances

1,2-Dichlorobenzene [CAS No. 95-50-1]
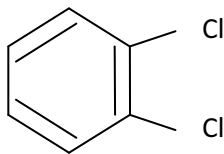
Synonyms: *o*-Dichlorobenzene
   o-DCB

1 mL/m³ $\cong$ 6.12 mg/m³

1,2-Dichlorobenzene is a yellowish, flammable, viscous liquid with a characteristic aromatic odour (molar mass 147.0 g/mol, melting point –17°C, boiling point 180.5°C). It is insoluble in water, but is readily dissolved in organic solvents. It has a density of approx. 1.3 g/cm³, which means that its density is higher than that of water. Explosive vapour/air mixtures can be formed at temperatures above 66°C. Heating causes decomposition to form toxic, corrosive vapours (hydrogen chloride). 1,2-Dichlorobenzene is widely used, e.g. in the production of paints and pesticides. Moreover, it is used as a solvent for lacquers, rubber, waxes, resins and disinfectants. Most of the 1,2-dichlorobenzene that is produced is further processed by nitration to 1,2-dichloro-4-nitrobenzene.

The currently valid MAK value and the occupational exposure limit (OEL) is 61 mg/m^3 (10 mL/m^3) [1, 2] The short-term exposure value of 1,2-dichlorobenzene is assigned to Peak Limitation Category II with an excursion factor of 2. The detailed documentation of the toxicity of 1,2-dichlorobenzene is found in the "MAK Value Documentations" [3].

### 1,3-Dichlorobenzene [CAS No. 541-73-1]
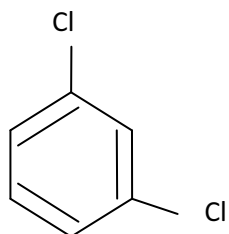
Synonyms: *m*-Dichlorobenzene
m-DCB

1 mL/m^3 ≙ 6.12 mg/m^3

1,3-Dichlorobenzene is a colourless, flammable liquid with a characteristic aromatic odour (molar mass 147.0 g/mol, melting point −24.7°C, boiling point 173°C). It decomposes on contact with hot objects or flames. It is insoluble in water, but is readily dissolved in organic solvents. In Germany 1,3-dichlorobenzene is exclusively produced as an intermediate product for further processing.

The currently valid MAK value is 12 mg/m^3 and the occupational exposure limit (OEL) is 20 mg/m^3 (3 mL/m^3) [1, 2]. The short-term exposure value of 1,3-dichlorobenzene is assigned to Peak Limitation Category II with an excursion factor of 2 in the List of MAK and BAT Values [1] and is assigned to Peak Limitation Category II with an excursion factor of 4 in TRGS 900 [2]. The detailed documentation of the toxicity of 1,3-dichlorobenzene is found in the "MAK Value Documentations" [4].

1,4-Dichlorobenzene [CAS No. 106-46-7]
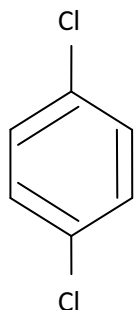
Synonyms: *p*-Dichlorobenzene
p-DCB

1 mL/m^3 ≙ 6.12 mg/m^3

1,4-Dichlorobenzene is a white, crystalline solid with a camphor-like odour (molar mass 147.0 g/mol, melting point 53°C, boiling point 174°C, vapour pressure 1.7 hPa at 20°C, flash point 66°C). 1,4-Dichlorobenzene is flammable; the vapours are heavier than air and form explosive mixtures with air at temperatures above 66°C. When it is ignited, corrosive, toxic vapours are formed e.g. hydrogen chloride and phosgene. It is insoluble in water. In contrast, it is readily dissolved by alcohol, ether or benzene. 1,4-Dichlorobenzene is used as an intermediate product in the manufacture of pigments and pesticides as well as a solvent for lacquers, rubber, waxes, resins and disinfectants. Furthermore, it is used to purify water, to render synthetic silk matt. It is also employed as a moth control agent and to improve air quality (e.g. deodoriser blocks).

1,4-Dichlorobenzene is assigned to Carcinogen Category 2 in the List of MAK and BAT Values [1]; the occupational exposure limit (OEL) is 120 mg/m^3 (20 mL/m^3) [2]. For short-term exposure assessment it is classified in Peak Limitation Category II with an excursion factor of 2 [2]. The detailed documentation of the toxicity of 1,4-dichlorobenzene is found in the "MAK Value Documentations" [5].

The method described here was examined by checking for plausibility [6] *by experts of the "Air Analyses" working group of the Deutsche Forschungsgemeinschaft's Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area.*

Author: *M. Tschickardt*
Examiner: *M. Ball*

Dichlorobenzene isomers (1,2-dichlorobenzene, 1,3-dichloro-benzene and 1,4 dichlorobenzene)

Method number	1
Application	Air analysis
Analytical principle	Gas chromatography (thermal desorption)
Completed in	December 2010

Contents

1
General principles

This analytical method permits the determination of gaseous dichlorobenzene isomers (1,2-dichlorobenzene, 1,3-dichlorobenzene and 1,4-dichlorobenzene) present in the workplace air in a concentration range from a tenth to twice the currently valid limit value in air [1, 2] by means of adsorption on Tenax TA™ and subsequent thermal desorption.

Sampling is carried out by drawing a defined volume of ambient air through an adsorption tube packed with Tenax TA™ using a suitable sampling pump. After thermal desorption, the dichlorobenzene isomers are determined by gas chromatography with a flame ionisation detector (FID). The quantitative evaluation is based on calibration functions, whereby the concentrations of dichlorobenzene in the reference standards are plotted versus the resulting peak areas.

2
Equipment, chemicals and solutions

2.1
Equipment

— Adsorption tubes made of stainless steel (6.3 mm × 90 mm, 5 mm inner diameter), packed with 200 mg of Tenax TA™, 60 to 80 mesh (e.g. PerkinElmer LAS, Rodgau, Germany)
— Pump, suitable for a flow rate of 5 mL/min (e.g. PP-1, Gilian, USA)
— Gas chromatograph with thermal desorber and flame ionisation detector (e.g. Turbomatrix ATD, PerkinElmer LAS, Rodgau, Germany)
— Sealing caps (e.g. Swagelok® with PTFE seals, PTFE or aluminium)
— Capillary column, (e.g. Wax-ETR: 30 m, 0.5 µm film thickness; 0.25 mm inner diameter (PerkinElmer LAS, Rodgau, Germany)
— Gas meter or stopwatch and soap bubble flowmeter
— Adjustable piston pipette 1 to 10 µL (e.g. Microman CP10, Gilson International B.V., Limburg-Offheim, Germany)
— Volumetric flask, 100 mL
— Volumetric pipette, 10 mL

2.2

Chemicals

- 1,2-Dichlorobenzene PESTANAL®, purity > 99.9%, Sigma-Aldrich, Order No. 36707
- 1,3-Dichlorobenzene PESTANAL®, purity > 99.4%, Sigma-Aldrich, Order No. 36708
- 1,4-Dichlorobenzene PESTANAL®, purity > 99.9%, Sigma-Aldrich, Order No. 35775
- Methanol, p.a., purity > 99.9%, Merck, Order No. 6009

Gases for operation of the gas chromatograph/thermal desorber:

- Helium 4.6, purity 99.996%
- Purified or synthetic air (free of hydrocarbons, dew point ≤ –50°C)
- Hydrogen 5.0, purity 99.999%

2.3

Pre-treatment of the adsorption tube

Before use, the adsorption tubes packed with Tenax TA™ are heated for 10 minutes in the thermal desorber at 280°C and then tested for blank values. Swagelok® or aluminium sealing caps are used to seal the tubes for storage.

2.4

Solutions

To prepare Calibration Solution I the masses of 1,2-dichlorobenzene, 1,3-dichlorobenzene and 1,4-dichlorobenzene stated in Table 1 are weighed into a 100 mL volumetric flask that is then filled to the mark with methanol and shaken.

Calibration Solution II (1:10) and Calibration Solution III (1:100) are prepared from Calibration Solution I by dilution with methanol.

The adsorption tubes are spiked directly with aliquots of these calibration solutions, and then the loaded tubes are used for calibration.

Table 1 Concentration of the calibration solutions

Substance	Mass	Calibration Solution		
	[mg]	I [mg/L]	II [mg/L]	III [mg/L]
1,2-Dichlorobenzene	250	2500	250	25
1,3-Dichlorobenzene	100	1000	100	10
1,4-Dichlorobenzene	500	5000	500	50

2.5

Preparation of the calibration standards

Aliquots of Calibration Solutions I to III are applied to the adsorption tubes for calibration. The methanol is then removed by purging in a stream of nitrogen. As a rule a nitrogen flow rate of 30 mL per minute for 10 minutes is sufficient to purge the methanol. The amounts of analyte added to the tubes are shown in Table 2.

  The calibration standards (see Table 2) are equivalent to the air concentrations shown in Table 3, based on an air sample volume of 200 mL.

Table 2 Calibration standards

Calibration Solution No.	Volume of calibration solution [μL]	Mass per sample in μg		
		1,2-Dichloro-benzene	1,3-Dichloro-benzene	1,4-Dichloro-benzene
III	1	0.025	0.01	0.05
III	5	0.125	0.05	0.25
III	10	0.25	0.1	0.5
II	5	1.25	0.5	2.5
II	10	2.5	1	5
I	2.5	6.25	2.5	12.5
I	5	12.5	5	25
I	7.5	18.75	7.5	37.5
I	10	25	10	50

Table 3 Calculated air concentrations in the calibration standards

Calibration Solution No.	Volume of calibration solution [µL]	Air concentrations in mg/m³		
		1,2-Dichloro-benzene	1,3-Dichloro-benzene	1,4-Dichloro-benzene
III	1	0.125	0.05	0.25
III	5	0.625	0.25	1.25
III	10	1.25	0.5	2.5
II	5	6.25	2.5	12.5
II	10	12.5	5	25
I	2.5	31.25	12.5	62.5
I	5	62.5	25	125
I	7.5	93.75	37.5	187.5
I	10	125	50	250

3
Sampling and sample preparation

Sampling can be carried out as stationary or personal sampling. The important parameters for the determination of the concentration of the dichlorobenzene isomers in the air samples (temperature, air pressure, relative humidity) are to be documented in a sampling record. They are measured in the breathing zone in the case of personal sampling. It is important to ensure that the inlet aperture of the adsorption tube is freely accessible.

Prior to the start of sampling the conditioned adsorption tube is opened and connected with the pump. The sampling pump continuously draws the air to be analysed through the adsorption tube over a period of 40 minutes at a flow rate of 5 mL/min. In the case of longer sampling times the flow rate must be adjusted in such a way that the air sample volume does not exceed 200 mL. On completion of sampling, PTFE caps are used to seal both ends of the loaded adsorption tube. The samples should be analysed as soon as possible after the end of sampling. If the samples are to be stored over a longer period before analysis (see Section 8.4), then they must be tightly sealed with Swagelok® screw caps with PTFE seals.

4
Operational parameters

The thermal desorber is connected to the gas chromatograph via a heated transfer line equipped with a deactivated fused silica capillary. An example of the operating conditions is given below.

4.1

Operating conditions for thermal desorption

Apparatus:	Turbomatrix ATD (PerkinElmer LAS)
Desorption temperature:	280°C
Desorption time:	10 min
Valve temperature:	200°C
Temperature of transfer line:	200°C
Length of transfer line:	1.5 m
Cold trap (adsorption):	−30°C
Cold trap (injection):	300°C
Heating rate:	40°C/s
Packing of cold trap:	20 mg of Tenax TA™
Carrier gas:	Helium 4.6
Carrier gas pressure:	100 kPa (1.6 mL/min)
Input split:	40 mL/min
Desorb flow:	10 mL/min
Outlet split:	30 mL/min
Purge drying phase:	1 min at room temperature at 30 mL/min

4.2

Operating conditions for gas chromatography

Apparatus:	Gas Chromatograph Autosystem XL, PerkinElmer LAS	
Column:	Material:	Fused silica capillary
	Stationary phase:	WAX-ETR
	Length:	30 m
	Inner diameter:	0.25 mm
	Film thickness:	0.5 µm
Detector:	Flame ionisation detector (FID)	
Detector temperature:	320°C	
Detector gases:	Hydrogen 5.0 (45 mL/min)	
	Synthetic air (450 mL/min)	
Temperature program:	Initial temperature: 40°C, 10 min isothermal	
	Heating rate: 8°C/min	
	Final temperature: 120°C, 10 min isothermal	

Figure 1 shows a chromatogram of the substance mixture of the dichlorobenzene isomers recorded under the conditions given above. Under these conditions 1,3-dichlorobenzene elutes after 23.0 min, 1,4-dichlorobenzene after 23.6 min and 1,2-dichlorobenzene after 24.4 min.
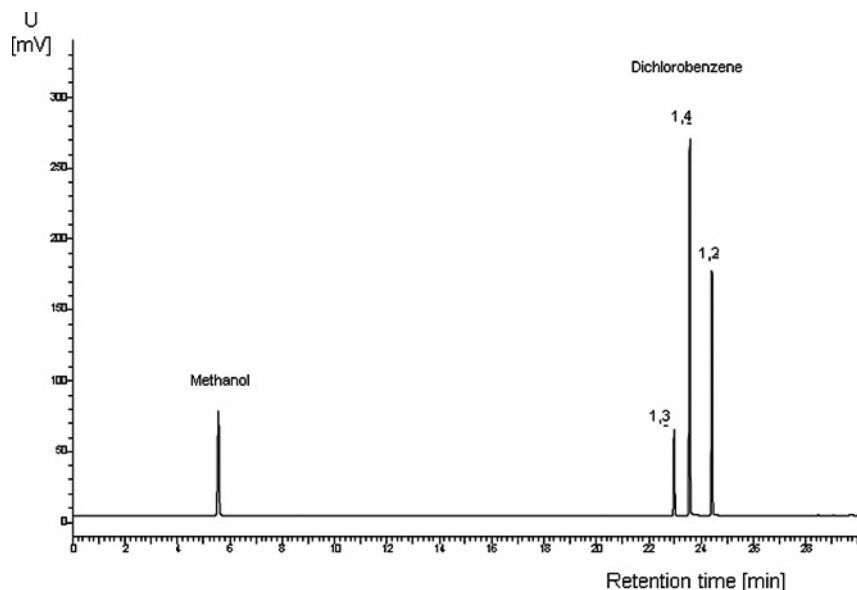
Fig. 1 Example of a chromatogram for the gas chromatographic separation of the dichloroben-zene isomers (cf. Section 4 for the chromatographic conditions)

5
Analytical determination

After preparation of the thermal desorber and the gas chromatograph (see Section 4), the calibration standards and the analytical samples are measured.

For gas chromatographic analysis, the Tenax TA™ tubes prepared as described in Section 3 are heated to 280°C in the thermal desorber, whereby the dichloroben-zene isomers are transferred to a cold trap by means of a carrier gas. After complete desorption, the split outlet is opened and the cold trap is heated to 300°C. The sample enters to the separation column as a narrow substance plug. After separation, the individual dichlorobenzene isomers are detected by means of the FID.

6
Calibration

The calibration standards prepared as described in Section 2.5 are used to obtain the calibration functions. The calibration standards should be spiked to give con-

centrations of the selected dichlorobenzene isomers of between 0.1 and 2 times their air limit values [7]. The loaded Tenax TA™ tubes are prepared and analysed as described in Sections 3, 4 and 5.

The calibration functions are obtained by plotting the resulting peak areas versus the respective loaded masses in μg (see Table 2). The calibration functions are linear in the stated ranges and should be regularly checked during routine analysis. A calibration standard of known concentration must be analysed in each analytical series for this purpose. The graph of the calibration function of 1,2-dichlorobenzene is shown as an example in Figure 2.
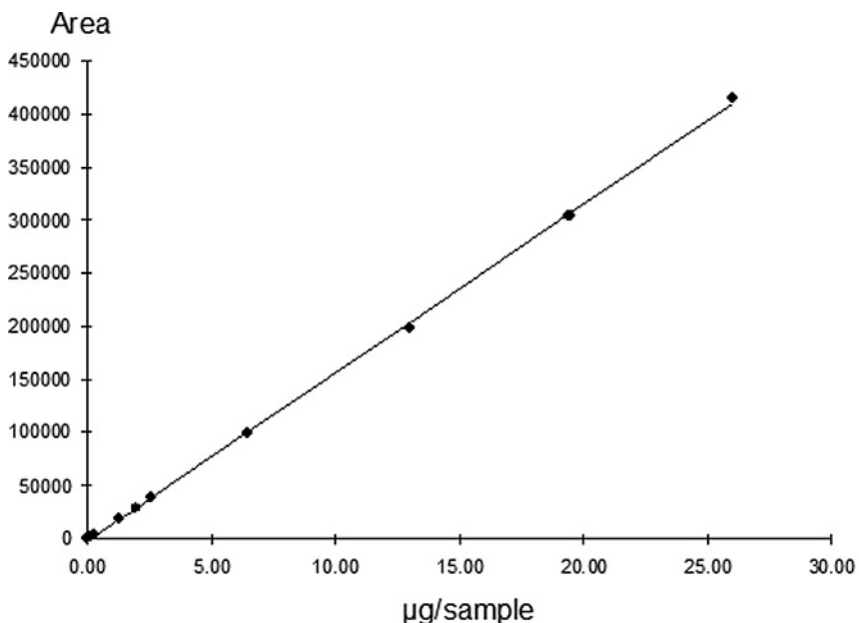

Fig. 2 Graphic illustration of the calibration function for 1,2-dichlorobenzene in the concentration range from 0.2 to 212 mg/m³.

7
Calculation of the analytical result

The concentration of the dichlorobenzene isomers in the workplace air is calculated on the basis of a calibration function (cf. Section 6). Based on the determined peak areas, the corresponding mass X in μg is obtained from the appropriate cali-

bration function. The corresponding concentration by weight (ρ) is calculated according to the following equation (1):

$$\rho = \frac{X}{V} \tag{1}$$

Equation (2) enables calculation of the value at 20 °C and 1013 hPa (standard conditions):

$$\rho_0 = \rho \times \frac{273 + t_a}{293} \times \frac{1013}{p_a} \tag{2}$$

The corresponding concentration by volume σ – independent of the pressure and temperature – is calculated according to equation (3) and (4).

$$\sigma = \rho_0 \times \frac{V_m}{M} \tag{3}$$

$$\sigma = \rho \times \frac{273 + t_a}{p_a} \times \frac{1013}{293} \times \frac{V_m}{M} \tag{4}$$

The concentration by volume σ in mL/m³ is calculated at $t_a = 20°C$ and $p_a = 1013$ hPa for each dichlorobenzene isomer with the following equation (5):

$$\sigma = 0.164 \times \rho_0 \tag{5}$$

where:

ρ	is the concentration by weight of a dichlorobenzene isomer in mg/m³
ρ_0	is the concentration by weight of a dichlorobenzene isomer in mg/m³ at 20°C and 1013 hPa
X	is the mass of a dichlorobenzene isomer in the sample in μg
t_a	is the temperature during sampling in °C
p_a	is the atmospheric pressure during sampling in hPa
V	is the air sample volume in L
V_m	is the molar volume in L/mol
M	is the molar mass in g/mol

8
Evaluation of the method

The characteristics of the method were calculated as stipulated in EN 482 [7].

The precision and the accuracy of the total analytical procedure were determined for each dichlorobenzene isomer at three different concentrations.

8.1
Reliability of the method

8.1.1   Precision and expanded uncertainty

Three reference series, each with six samples, were prepared by spiking the Tenax TA™ tubes with the calibration solutions shown in Table 1 in order to determine the precision and the expanded uncertainty. Then 500 mL of ambient air was drawn through the tubes with a pump at a flow rate of 50 mL/min. The Tenax TA™ tubes were prepared and analysed as described in Sections 3, 4 and 5. Table 4 shows the results that were obtained.

Table 4   Standard deviation (rel.) and expanded uncertainty U for n = 6 determinations

Substance	Concentration [mg/m³]	Standard deviation (rel.) [%]	Expanded uncertainty *U* [%]
1,2-Dichlorobenzene	3.3	7.9	21.8
	64.9	2.7	12.8
	129.8	1.8	11.9
1,3-Dichlorobenzene	6.4	7.1	21.0
	127.2	2.3	12.2
	254.3	2.2	12.2
1,4-Dichlorobenzene	5.3	7.4	19.8
	106.0	2.4	12.5
	212.1	2.1	12.0

8.1.2   Accuracy

Recovery experiments were performed for the three dichlorobenzene isomers to check the accuracy of the method. For this purpose three reference series, each with six samples, were prepared by spiking with the calibration solutions shown in

Table 1 and 500 mL of ambient air was drawn through the tubes at a flow rate of 50 mL/min. Then the samples were prepared and analysed as described in Sections 3, 4 and 5. The results obtained are shown in Table 5.

Table 5   Mean recoveries for the dichlorobenzene isomers

Substance	Spiked mass [µg]	Recovered mass [µg]	Recovery [%]
1,2-Dichlorobenzene	0.649	0.592	91.2
	12.98	13.428	103
	25.96	26.675	103
1,3-Dichlorobenzene	0.242	0.219	90.4
	4.845	4.969	103
	9.690	9.957	103
1,4-Dichlorobenzene	1.060	0.991	93.5
	21.205	21.891	103
	42.410	43.413	102

The mean recoveries are:

1,2-dichlorobenzene = 99.1%
1,3-dichlorobenzene = 98.8%
1,4-dichlorobenzene = 99.5%

8.2
Limit of quantification

The limit of quantification for the dichlorobenzene isomers was determined from a 10-point calibration as stipulated in DIN 32645 [8]. For this purpose, aliquots of 1 to 10 µL of Calibration Solution III (cf. Table 3) were applied to the Tenax TA™ tubes and then the solvent (methanol) was purged for 10 minutes at a flow rate of 30 mL of nitrogen per minute. The results are shown in Table 6.

Table 6 Limits of quantification for the dichlorobenzene isomers determined according to DIN 32645 (P = 99%, k = 3)

Substance	Limit of quantification	
	Absolute	Relative
	µg per sample	mg/m^3 (for an air sample volume of 200 mL)
1,2-Dichlorobenzene	0.075	0.38
1,3-Dichlorobenzene	0.042	0.21
1,4-Dichlorobenzene	0.179	0.90

8.3
Recovery

Direct determination of the recovery is not possible in the case of thermal desorption procedures. The desorption rates of the individual dichlorobenzene isomers were checked as part of comparative investigations by two-fold heating of various adsorption tubes. They were greater than 99% at the concentrations of the dichlorobenzene isomers used in each case and they can be regarded as a measure of the recovery.

8.4
Storage stability

Investigations of the storage stability of the loaded sample carriers were carried out over a period of four weeks. For this purpose ten Tenax TA™ tubes were each spiked with 5 µL of Calibration Solution I and then 500 mL of ambient air was drawn through them. The adsorption tubes were subsequently sealed with Swage-lok® caps, stored at room temperature and analysed after a period of four weeks. The results of the storage stability tests are documented in Table 7.

Table 7 Recoveries after a storage period of 4 weeks

Substance	Spiked mass [µg]	Recovered mass [µg]	Recovery [%]
1,2-Dichlorobenzene	12.98	13.08	100.8
1,3-Dichlorobenzene	4.85	4.84	99.8
1,4-Dichlorobenzene	21.21	21.36	100.7

8.5
Capacity of the adsorbent

To check the capacity of the Tenax TA™ tubes used, the first of two Tenax TA™ tubes connected in series was spiked with 20 µL of Calibration Solution I (corresponding to about four times the concentration of the MAK value of each dichlorobenzene isomer) and then 500 mL of ambient air was drawn through the tubes at a flow rate of 50 mL per minute. No breakthrough was observed.

8.6
Interference

Even when appropriate gas chromatographic analytical procedures are used, interference is principally possible due to components with the same retention times on account of the low specificity of flame ionisation detectors.

Another column of different polarity or, if necessary, a mass spectrometric detector can also be used to confirm the analytical results.

9
Discussion

Firstly, it is essential to ensure that adsorption tubes without blank values are used.

This method was tested using stainless steel adsorption tubes packed with 200 mg of Tenax TA™. However, another adsorption material may be used under certain circumstances. If other adsorbents and tubes of deviating dimensions are used, then the breakthrough and retention volumes as well as the analytical characteristics must be checked. The procedure has been tested and is usable at a relative air humidity up to 70%.

Loaded collection phases that have been sealed with PTFE caps should be analysed without delay. As part of the validation experiments, samples sealed with Swagelok® caps were stored for up to four weeks at room temperature without measurable losses occurring.

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