

# Pentachlorophenol and Lindane

<b>Method number</b>	1
<b>Application</b>	Air analysis
<b>Analytical principle</b>	Gas chromatography/mass spectrometry
<b>Completed in</b>	June 2001

## Summary

In June 2000 guideline VDI 4301 Part 2 appeared. It contains instructions for measuring the concentrations of pentachlorophenol (PCP) and lindane ( $\gamma$ -hexachlorocyclohexane,  $\gamma$ -HCH) in the air inside buildings.

The air to be analysed is drawn via a sampling head through a glass fibre filter and then through polyurethane (PU) foam as adsorbent. This enables the determination of the particle bounded and the filter passing pentachlorophenol and lindane. Pentachlorophenol and lindane are extracted from the filter and PU foam with a solvent, pentachlorophenol is derivatised and both substances are identified and quantified using gas chromatography/mass spectrometry (GC/MS). Evaluation is carried out using a  $^{13}\text{C}$ -labelled internal standard.

The procedure has been tested for a sampling period of up to eight hours. The minimum sampling period is half an hour. Recommended is a sampling period of an hour with a sampled air volume of  $2.7\text{ m}^3$  to  $2.8\text{ m}^3$ .

## Characteristics of the method

Precision:	Standard deviation (rel.)	$s = 11\%$ (PCP)
		$s = 11\%$ ( $\gamma$ -HCH)
	Mean variation	$u = 24\%$ (PCP)
		$u = 23\%$ ( $\gamma$ -HCH)
	in the concentration range around $0.2\text{ }\mu\text{g/m}^3$ and $n = 14$ and $17$ determinations	
Quantification limits:	$0.02\text{ }\mu\text{g/m}^3$ (PCP)	
	$0.02\text{ }\mu\text{g/m}^3$ ( $\gamma$ -HCH)	

Sampling recommendation: Sampling time: 1 hour  
Sample volume: 2800 L

**Pentachlorophenol [CAS No.: 87-86-5]**  
**Lindane [CAS No.: 58-89-9]**

Pentachlorophenol (Fig. 1) and lindane (Fig. 2) can be found indoors e.g. because they have been used as wood preservatives. Lindane was and still is used as a household insecticide and in agriculture and is found, for example, in textiles and carpets.

Two guidelines were established in 1997 for pentachlorophenol in the ambient air [7]:

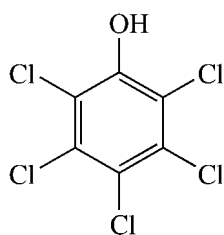
- Reference value 1:  $0.1 \mu\text{g}/\text{m}^3$
- Reference value 2:  $1 \mu\text{g}/\text{m}^3$

The molecular weight of pentachlorophenol ( $\text{C}_6\text{HCl}_5\text{O}$ ) is 266.3383. Its solubility in water is 0.0014 g/100 mL. The melting point and boiling point are  $174^\circ\text{C}$  and  $310^\circ\text{C}$ . Pentachlorophenol is classified as a substance considered to be carcinogenic for man (Carcinogenicity category 2) [8].

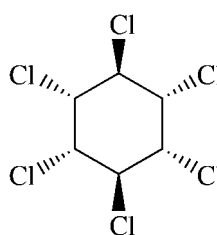
The molecular weight of lindane ( $\text{C}_6\text{H}_6\text{Cl}_6$ ) is 290.8314. Its solubility in water is 0.00073 g/100 mL. The melting point and boiling point are  $112.9^\circ\text{C}$  and  $323.4^\circ\text{C}$ . The current threshold value in workplace air for lindane is  $0.1 \text{ mg}/\text{m}^3$  for the inhalable fraction (I). The current BAT value is  $25 \mu\text{g}/\text{L}$ . Lindane is classified as a substance with carcinogenic potential for which genotoxicity plays no or at most a minor part (Carcinogenicity category 4) [8].

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**Fig. 1.** Pentachlorophenol.



**Fig. 2.** Lindane.

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## 1 General principles

The air to be analysed is drawn via a sampling head through a glass fibre filter and then through a PU foam adsorbent. This enables both the particulate pentachlorophenol and lindane to be determined and that not deposited on the filter.

Pentachlorophenol and lindane are extracted from the filter and PU foam with a solvent, pentachlorophenol is derivatised and both substances are identified and quantified using gas chromatography/mass spectrometry (GC/MS). Evaluation is carried out using a  $^{13}\text{C}$ -labelled internal standard.

The procedure has been tested for a sampling period of up to eight hours. The minimum sampling period is half an hour. Recommended is a sampling period of an hour with a sampled air volume of  $2.7\text{ m}^3$  to  $2.8\text{ m}^3$ .

## 2 Equipment, chemicals and solutions

### 2.1 Equipment

Pump with a flow rate of  $2.7\text{ m}^3/\text{h}$  to  $2.8\text{ m}^3/\text{h}$  under sampling conditions

Sampling head and transport vessels (Fig. 3 a and b)

Gasmeter

Hygrometer

Thermometer

Barometer

Soxhlet extractor

Heating device e. g. sand bath, heating mantle

10, 50 and 100  $\mu\text{L}$  Syringes

Gas chromatograph with mass spectrometer as detector

Capillary column of low polarity (e. g. OV 1, DB 5, SE 54, HP 1)

### 2.2 Chemicals

Pentachlorophenol (e. g. Aldrich, Ehrenstorfer, Promochem)

Lindane (e. g. Aldrich, Ehrenstorfer, Promochem)

All solvents listed must be of sufficient purity (e. g. nanograde)

Ethyl acetate

*n*-Hexane

Toluene

Acetic anhydride, analytical grade

Potassium carbonate solution 0.5 mol/L

Sulfuric acid 0.1 mol/L

Sodium sulfate, anhydrous, analytical grade

Glass fibre filter  $\varnothing$  50 mm

PU foam  $\varnothing$  50 mm, length 25 mm

Purification guidelines: Extraction for 24 hours with toluene in the Soxhlet extractor, extraction for 24 hours with acetone in the Soxhlet extractor, drying in a desiccator under vacuum and with a low flow rate of highly pure nitrogen (e.g. from the evaporation of liquid nitrogen) or in a vacuum-drying cabinet at 40 °C.

During extraction and purification commercially available PU foam may shrink. It must be checked that it still fits exactly after the purification process.

## 2.3 Solutions

$^{13}\text{C}$ -pentachlorophenol e.g. as 100 mg/L solution (e.g. Promochem, Cambridge Isotope Laboratories)

$^{13}\text{C}$ -lindane e.g. as 100 mg/L solution (e.g. Promochem, Cambridge Isotope Laboratories)

Internal standard solutions:

The concentrations of  $^{13}\text{C}$ -pentachlorophenol and  $^{13}\text{C}$ -lindane in the internal standard solutions are 100 mg/L. Solutions of this concentration are used as obtained from the supplier or prepared as follows: 50.0 mg of each of  $^{13}\text{C}$ -pentachlorophenol and  $^{13}\text{C}$ -lindane are dissolved in 50 mL toluene. This stock solution is diluted with toluene in the ratio 1 : 10 (1 + 9).

Stock solutions of pentachlorophenol and lindane:

The solutions are prepared as follows: 50.0 mg of pentachlorophenol and lindane are each dissolved in 50 mL toluene (concentration 1 g/L). These stock solutions are diluted with toluene in the ratio 1 : 10 (1 + 9) resulting in solutions with a concentration of 100 mg/L.

Calibration solutions:

The calibration solutions are prepared in toluene according to the following scheme (Table 1) from the internal standard solutions and pentachlorophenol and lindane stock solutions (amounts in  $\mu\text{L}$ ).

**Table 1.** Preparation of the calibration solutions.

Calibration solution (concentrations of the compounds to be analysed)	PCP $\mu\text{L}$	$\gamma$ -HCH $\mu\text{L}$	$^{13}\text{C}$ -PCP $\mu\text{L}$	$^{13}\text{C}$ - $\gamma$ -HCH $\mu\text{L}$
0 $\mu\text{g/L}$	0	0	20	20
50 $\mu\text{g/L}$	5	5	20	20
100 $\mu\text{g/L}$	10	10	20	20
250 $\mu\text{g/L}$	25	25	20	20
500 $\mu\text{g/L}$	50	50	20	20
1000 $\mu\text{g/L}$	100	100	20	20

The six calibration solutions are each made up to 10 mL with toluene. The concentrations of the internal standards in all solutions are 200 µg/L.

The same internal standard solution is to be used for the preparation of the calibration solutions and for the spiking of the PU foam later.

### 3 Sample collection and preparation

The sampling apparatus set-up is shown in Figure 4. A detailed description of the equipment required can be found in guideline VDI 2463 Part 7 [14].

#### 3.1 Sample collection

The  $^{13}\text{C}$ -labelled standards are placed on the PU foam by injecting a total volume of 10 µL of the internal standard solution into various parts of the foam (e.g. with a microlitre syringe). The standard solutions are usually placed on the inlet side of the filter. At the sampling site, the PU foam is fitted into the sampling head as shown in Figure 1. The glass fibre filter is placed in the filter holder using tweezers and fixed in place with a Seeger retaining ring. When sampling is continued for more than two hours, to be on the safe side a second piece of PU foam should be placed behind the first and included in the analytical procedure. The filter holder is attached to the frame in such a way that the sampled air flows vertically from the bottom to the top. The bottom edge (inlet) of the inlet tube is about 1.2 m to 1.5 m above the ground. The sampling time is normally one hour. The hourly air sample volume should not exceed 10% of the ventilation rate. If this is not known, the hourly air sample volume should not be greater than 10% of the air volume in the room. The flow rate should be 2.7 m<sup>3</sup> to 2.8 m<sup>3</sup> per hour and not differ at the beginning and end of sampling by more than 5% (this must be documented). The temperature of the gas in the gasmeter must be taken into account when determining the volume. Further details for planning measurements can be found in guidelines VDI 4300 Parts 1 and 4 [1, 2]. During the determination, the ambient temperature, the relative humidity and the air pressure must be recorded, in order, if necessary, to be able to compare the current values with results determined under other conditions. The cartridge with the PU foam and the glass fibre filter is removed from the sampling head after sampling, closed at both ends and stored in the dark if preparation cannot take place immediately. The shelf-life of the loaded foam is at least four weeks.

#### 3.2 Sample preparation

For sample preparation the PU foam and the glass fibre filter are treated separately. The glass fibre filter is placed e.g. in a small beaker, 1 mL sulfuric acid and 5 mL toluene are added and the substances are extracted for 15 minutes in an ultrasonic bath.

Extraction of the PU foam is carried out with toluene in the Soxhlet extractor over a period of 16 hours. At least 50 extraction cycles should be performed.

The extracts are combined and concentrated at reduced pressure to about 5 mL. 1 mL of the solution is shaken twice with 1 mL potassium carbonate solution. The remaining organic extract is analysed for lindane in a gas chromatograph. 1 mL hexane and 0.1 mL acetic anhydride are added to the combined potassium carbonate solutions and shaken intensively for 2 minutes. The mixture is left to stand for 10 minutes and then 0.1 mL acetic anhydride is added. After the solution has been shaken for another two minutes and left to stand for ten minutes, the supernatant hexane extract is removed and used for the gas chromatographic determination of pentachlorophenol.

*Note:* To evaluate the extraction efficiency of the internal standards or for additional evaluation via external standard compounds, the solution must be concentrated to a little less than 5 mL, transferred to a 5 mL volumetric flask and then made up to the mark.

#### 4 Operating conditions for gas chromatography

Analysis is carried out by gas chromatography/mass spectrometry (GC/MS). The following devices are given as examples; if other devices are used, the parameters must be adjusted accordingly.

Gas chromatograph:	Hewlett-Packard 5890 II
Column:	Quartz capillary column
	Internal diameter: 0.25 mm
	Length: 30 m
	Stationary phase: Methyl silicone with 5% phenyl (e.g. HP-5 MS)
	Film thickness: 0.25 µm
Injector:	Deactivated glass insert
Injection volume:	1 to 2 µL (splitless)

##### GC conditions:

Carrier gas:	Helium with a linear carrier gas velocity of about 20 cm/s (column pressure: about 70 hPa corresponding to about 10 psi)
Injector temperature:	260 °C
MS transfer line:	280 °C
Temperature programme:	130 °C $\xrightarrow{5\text{ °C/min}}$ 200 °C $\xrightarrow{10\text{ °C/min}}$ 280 °C (8 min)

## 5 Mass spectrometric conditions

Mass spectrometer:	Hewlett-Packard 5971 A
Ionization:	EI
Detector mode:	SIM
Setting:	Autotune
Resolution:	Low resolution
Dwell time	100 ms

Under these conditions, for pentachlorophenol (as pentachlorophenol acetate) a retention time of about 11 to 13 minutes is to be expected and for lindane a retention time of about 9 to 11 minutes. Figures 5 and 6 show examples of ion chromatograms obtained during calibration with pentachlorophenol and lindane.

## 6 Analytical determination

1 to 2  $\mu\text{L}$  of the extracts are injected into the system. The mass fragmentograms are evaluated according to the procedure described below.

## 7 Calibration

### 7.1 Selection of the fragment ions for quantitative evaluation

For mass spectrometric evaluation, two fragment ion traces should be used for each substance. Usually the fragment ion trace with the higher intensity is used for the calculation, while the other ion trace serves to confirm its identity.

**Pentachlorophenol:** To improve the chromatography of pentachlorophenol, it is derivatized and is then present as pentachlorophenol acetate. The fragmentation of this molecule yields a fragment ion cluster, formed by cleavage of  $[\text{CH}_2=\text{C}=\text{O}]$  chains, which produce fragment ions with  $m/z = 264, 266, 268, 270, 272$  and  $274$  in the mass spectrum. These are the most intensive fragment ions of this mass spectrum. The intensities of the individual ions are determined by the chlorine isotope ratio, naturally  $^{35}\text{Cl}:^{37}\text{Cl} = 3:1$ . The fragment ion with  $m/z = 266$  has the highest signal intensity in this fragment ion cluster, the signals of the ions  $m/z = 264$  and  $268$  are only about half or  $2/3$  as high.

The internal standard ( $^{13}\text{C}_6$ -pentachlorophenol) yields the same fragmentation pattern as the “native” pentachlorophenol, but in this case the mass numbers are six mass units higher. The corresponding fragment ion cluster, that for  $^{13}\text{C}_6$ -pentachlorophenol-acetate, therefore has fragment ions with  $m/z = 270, 272, 274, 276, 278$  and  $280$ .

As ions with  $m/z = 270, 272$  and  $274$  are fragment ions of both “native” pentachlorophenol-acetate, i.e. that to be analysed, and also  $^{13}\text{C}_6$ -pentachlorophenol-acetate (the



internal standard), only the fragment ions with  $m/z = 264$ , 266 and 268 can be used for the determination of pentachlorophenol. For  $^{13}\text{C}_6$ -pentachlorophenol-acetate, in addition to the fragment ion traces with  $m/z = 276$ , 278 and 280, also the fragment ion trace with  $m/z = 274$  can be used, as this is the fragment ion with the most intensive signal.

If interference occurs with the ion traces with  $m/z = 264$ , 266 and 268 for pentachlorophenol or with  $m/z = 276$ , 278 and 280 for  $^{13}\text{C}_6$ -pentachlorophenol, also the ion traces with  $m/z = 306$ , 308 and 310, with about  $1/5$  of the intensity, which belong to the molecule ion of pentachlorophenol acetate and the ion traces with  $m/z = 318$ , 320 and 322, which belong to the molecule ion of  $^{13}\text{C}_6$ -pentachlorophenol-acetate, can be used for quantitative evaluation.

**Lindane:** For the analysis of lindane, similar considerations apply as for the analysis of pentachlorophenol. As the molecule ion with the molecule ion cluster with  $m/z = 288$ , 290, 292, 294, 296, 298 and 300 has only a low intensity, the fragment ion cluster formed by cleavage of HCl and additional cleavage of Cl, with  $m/z = 217$ , 219, 221, 223 and 225, is suitable for the analysis of lindane. Also suitable are the fragment ion traces with  $m/z = 181$ , 183 and 185 formed by further Cl cleavage. Also here the intensity of the fragment ion trace is determined by the chlorine isotope ratio (in the fragment ion: 3 or 4 Cl atoms); the  $^{13}\text{C}_6$ -lindane (of the internal standard) which has 6 heavy C atoms yields therefore the fragment ion cluster with  $m/z = 187$ , 189, 191 or  $m/z = 223$ , 225, 227, 229 and 231.

As the fragment ions 223 and 225 belong to both the "native" lindane and also  $^{13}\text{C}_6$ -lindane, these fragment ion traces cannot be used for the quantitative determination of lindane. For the evaluation, the fragment ion traces with  $m/z = 181$ , 183 and 185 or  $m/z = 217$ , 219 and 221 are therefore used for lindane and  $m/z = 227$ , 229 and 231 for  $^{13}\text{C}_6$ - $\gamma$ -HCH.

## 7.2 Drawing up the calibration curve

### 7.2.1 Pentachlorophenol

For calibration, the calibration solutions are prepared and derivatized like the sample extracts. After the analysis, for each calibration solution the ratio of the peak areas of the fragment ion trace for pentachlorophenol (e.g.  $m/z = 266$ ) to the peak areas of the fragment ion trace for the internal standard (e.g.  $m/z = 274$ ) is calculated.

The calibration must be checked for each analysis or analytical series!

The following results give an example of the peak area ratios obtained for the calibration of pentachlorophenol (c.f. Fig. 7):

Concentrations [ $\mu\text{g/L}$ ]	0	50	100	250	500	1000
Ratio of the peak areas for $m/z = 266$ and $m/z = 274$	0	0.566	1.118	2.833	5.257	10.832

Evaluation using linear regression analysis, where the  $y$  values represent the peak area ratios and the  $x$  values the concentrations, yields:

$$\begin{aligned} a &= 0.02676 \ (s_a = 0.05451) & r &= 0.9998 & n &= 6 \\ b &= 0.01076 \ (s_b = 0.00012) & s_{y,x} &= 0.09866 \end{aligned}$$

where:

$a$  is the axis intercept

$s_a$  is the standard deviation of the axis intercept

$b$  is the slope

$s_b$  is the standard deviation of the slope

$r$  is the correlation coefficient

$s_{y,x}$  is the standard deviation of the regression (standard deviation of the estimation)

$n$  is the number of points measured

### 7.2.2 Lindane

The calibration solutions are treated in the same way as the sample extracts. After analysis, the ratio of the peak area of the fragment ion trace of lindane (e.g.  $m/z = 183$ ) and the fragment ion trace of the internal standard (e.g.  $m/z = 227$ ) is formed.

The following results give an example of the peak area ratios obtained for the calibration of lindane (c.f. Fig. 8):

Concentrations in $\mu\text{g/L}$	0	50	100	250	500	1000
Ratio of the peak areas for $m/z = 266$ and $m/z = 274$	0	0.723	1.474	3.765	7.142	14.921

Evaluation using linear regression analysis, where the  $y$  values represent the peak area ratios and the  $x$  values the concentrations, yields:

$$\begin{aligned} a &= -0.03198 \ (s_a = 0.07929) & r &= 0.9997 & n &= 6 \\ b &= 0.01485 \ (s_b = 0.00017) & s_{y,x} &= 0.1435 \end{aligned}$$

where:

$a$  is the axis intercept

$s_a$  is the standard deviation of the axis intercept

$b$  is the slope

$s_b$  is the standard deviation of the slope

$r$  is the correlation coefficient

$s_{y,x}$  is the standard deviation of the regression (standard deviation of the estimate)

$n$  is the number of points measured

### 7.3 Calculation of the concentrations in the analysed solutions

To calculate the concentrations of pentachlorophenol and lindane in the analysed solutions, the peak area ratio is calculated for the fragment ion traces which were also used for the calibration. Using the calculated peak area ratio ( $v_{PF}$ ) and the regression coefficient, the concentration  $c$  in the sample solution is calculated as follows:

$$c = \frac{v_{PF} - a}{b} \quad (1a)$$

If the value for the axis intercept  $a$  is not significantly different from zero, the concentration can also be calculated as follows:

$$c = \frac{v_{PF}}{b} \quad (1b)$$

To check whether the axis intercept  $a$  is significantly different from zero, a t-test with the null hypothesis  $H_0: a = \alpha$  (with  $\alpha = 0$ ) is used. The value is calculated here according to the following equation:

$$\hat{t} = \frac{|a|}{s_a} \quad (2)$$

where:

$\hat{t}$  is the calculated value

$a$  is the axis intercept

$s_a$  is the standard deviation of the axis intercept

The calculated value  $\hat{t}$  is then compared with the tabulated  $t$  value:  $t_{\text{tab}} = t_{95\%, n-2}$ . If the calculated value  $\hat{t}$  exceeds the tabulated  $t$  value, the null hypothesis (axis intercept is zero) must be rejected, i.e. the axis intercept must be taken into account in the calculation of the concentration from the peak area ratio  $v_{PF}$  (equation 1 a).

For the calibration curve, whose regression coefficient and standard deviations are given above for the example of pentachlorophenol,  $\hat{t}$  is given by:

$$\hat{t} = 0.02656/0.05435 = 0.489.$$

This value is *smaller* than the tabulated  $t$  value ( $t_{95\%, 4} = 2.78$  (see Table 2). The null hypothesis “the axis intercept is not different from zero” can be retained and equation 1 b can be used for the calculation.

**Table 2.** Values for  $t$  distribution.

Number of degrees of freedom $f$	$P(90)$	$P(95)$	$P(99)$
1	6.31	12.71	63.66
2	2.92	4.30	9.92
3	2.35	3.18	5.84
4	2.13	2.78	4.60
5	2.01	2.57	4.03
6	1.94	2.45	3.71
7	1.89	2.36	3.50
8	1.86	2.31	3.36
9	1.83	2.26	3.25
10	1.81	2.23	3.17
$\infty$	1.64	1.96	2.58

For a sample whose peak area ratio (pentachlorophenol/ $^{13}\text{C}_6$ -pentachlorophenol) has the value ( $v_{\text{PF}}$ ) of 1.455, a concentration in the solution of  $c = 1.455/0.06081 = 135.22 \mu\text{g/L}$  is calculated.

The ion chromatogram for this toluene extract of an air sample containing pentachlorophenol is shown in Figure 9.

An example of an ion chromatogram for a toluene extract of an air sample containing lindane is shown in Figure 10.

**Estimation of the recovery of the internal standards:** The absolute areas of the fragment ion traces of the internal standards  $^{13}\text{C}_6$ -pentachlorophenol-acetate and  $^{13}\text{C}$ -lindane in the sample can be used, by comparison with those of the calibration solutions, to estimate the recovery of the internal standards and thus the efficiency of sample preparation. The recovery of the internal standards should be more than 80% for lindane and more than 50% for pentachlorophenol.

**Blank value over the whole procedure:** It must be checked that the PU foam, equipment, solvents, filters etc. used for sample preparation, are not contaminated with either pentachlorophenol or lindane. This can be ascertained by carrying out the whole analytical procedure with an unloaded filter and PU foam.

## 8 Calculation of the analytical result

### 8.1 Weight of pentachlorophenol and lindane in the sample

The weight of pentachlorophenol and lindane in the sample (filter and PU foam) is calculated using equation (3) from the concentrations of pentachlorophenol and lindane in the sample solutions calculated as described in Section 7.3:

$$m = \frac{cm_{IS}}{c_{IS}} \quad (3)$$

where:

$m$  is the weight of pentachlorophenol and lindane in the sample in ng

$c$  is the concentration in the sample solution in  $\mu\text{g/L}$

$m_{IS}$  is the weight of  $^{13}\text{C}_6$ -pentachlorophenol and  $^{13}\text{C}_6$ -lindane in the internal standard in ng

$c_{IS}$  is the concentration of  $^{13}\text{C}_6$ -pentachlorophenol and  $^{13}\text{C}_6$ -lindane in the internal standard in  $\mu\text{g/L}$

If the above example in which a concentration  $c = 135.22 \mu\text{g/L}$  for pentachlorophenol was obtained in the sample solution is taken further, the weight of pentachlorophenol in this sample would be  $m = (135.22 \times 1000)/200 = 676.1 \text{ ng}$ .

## 8.2 Concentration of pentachlorophenol and lindane in the ambient air

The concentration of pentachlorophenol and lindane in the ambient air is calculated according to Equation 4:

$$c_L = \frac{m}{V_L} \quad (4)$$

where:

$c_L$  is the concentration of pentachlorophenol or lindane in the ambient air in  $\text{ng/m}^3$

$m$  is the weight of pentachlorophenol or lindane in the sample in ng

$V_L$  is the air sample volume in  $\text{m}^3$

If the above example in which the weight of pentachlorophenol of  $m = 676.1 \text{ ng}$  was obtained in the sample is again taken further, the concentration of pentachlorophenol in the ambient air for an air sample volume of  $2.8 \text{ m}^3$  would be:

$$c_L = 676.1/2.8 = 241.464 \text{ ng/m}^3$$

The result should be rounded to two significant figures, i.e. here  $240 \text{ ng/m}^3$ .

## 9 Reliability of the method

### 9.1 Precision

In comparative studies in which four different laboratories used the procedure described in these guidelines, the standard deviations listed in Tables 3 and 4 were found. The detection limits for pentachlorophenol and lindane were approximately  $0.01 \mu\text{g/m}^3$ .

**Table 3.** Standard deviation and coefficient of variation ( $v$ ) for different concentrations of pentachlorophenol in the ambient air.

$c_L$ $\mu\text{g}/\text{m}^3$	$s_L$ $\mu\text{g}/\text{m}^3$	$v$ %	$n$
1.60	0.133	8	20
0.29	0.025	9	16
0.21	0.023	11	14
0.14	0.030	21	20

**Table 4.** Standard deviation and coefficient of variation with different concentrations of lindane in the ambient air.

$c_L$ $\mu\text{g}/\text{m}^3$	$s_L$ $\mu\text{g}/\text{m}^3$	$v$ %	$n$
0.41	0.040	10	20
0.31	0.018	6	20
0.30	0.025	8	15
0.21	0.023	11	17

## 9.2 Detection and quantification limits

The detection limit of the procedure is dependent not only on the yield of the extraction step, but also on the matrix effects, which, depending on the composition of the ambient air tested, can lead to interference and overlapping of the fragment ion chromatograms intended for quantification. The detection limit is defined as 3 times the background noise of the fragment ion chromatogram used for evaluation.

It should be ensured that, under the conditions used, concentrations as low as  $20 \text{ ng}/\text{m}^3$  in the ambient air can be quantified with the necessary certainty; this is to the quantification limit.

## 9.3 Shelf-life

In several intercomparison programmes, PU foam loaded with pentachlorophenol and lindane was dispatched by post. The samples were packed in aluminium cans with no further packing. After storage at ambient temperatures ( $15\text{--}30^\circ\text{C}$ ), individual samples were analysed during a period of 6 weeks. The shelf-life of loaded PU foam was found to be at least 4 weeks.

## 9.4 Sources of error

The quality of the PU foam used must be carefully checked for each batch, as residual free amines can prevent the complete extraction of pentachlorophenol.

As a result of the isotope-labelled standards, the analytical method is insensitive to interference. The analysis of lindane, however, often requires, a specific selection of the masses used for evaluation for each sampling site.

## 10 Discussion of the method

The preparation and derivatization places high demands on the laboratory personnel. Experience in several laboratories has shown that with this procedure, too, pentachlorophenol can be successfully analysed only after intensive training.

The working range of the procedure is designed for air inside buildings and covers the concentrations 20 to 2000 ng/m<sup>3</sup>.

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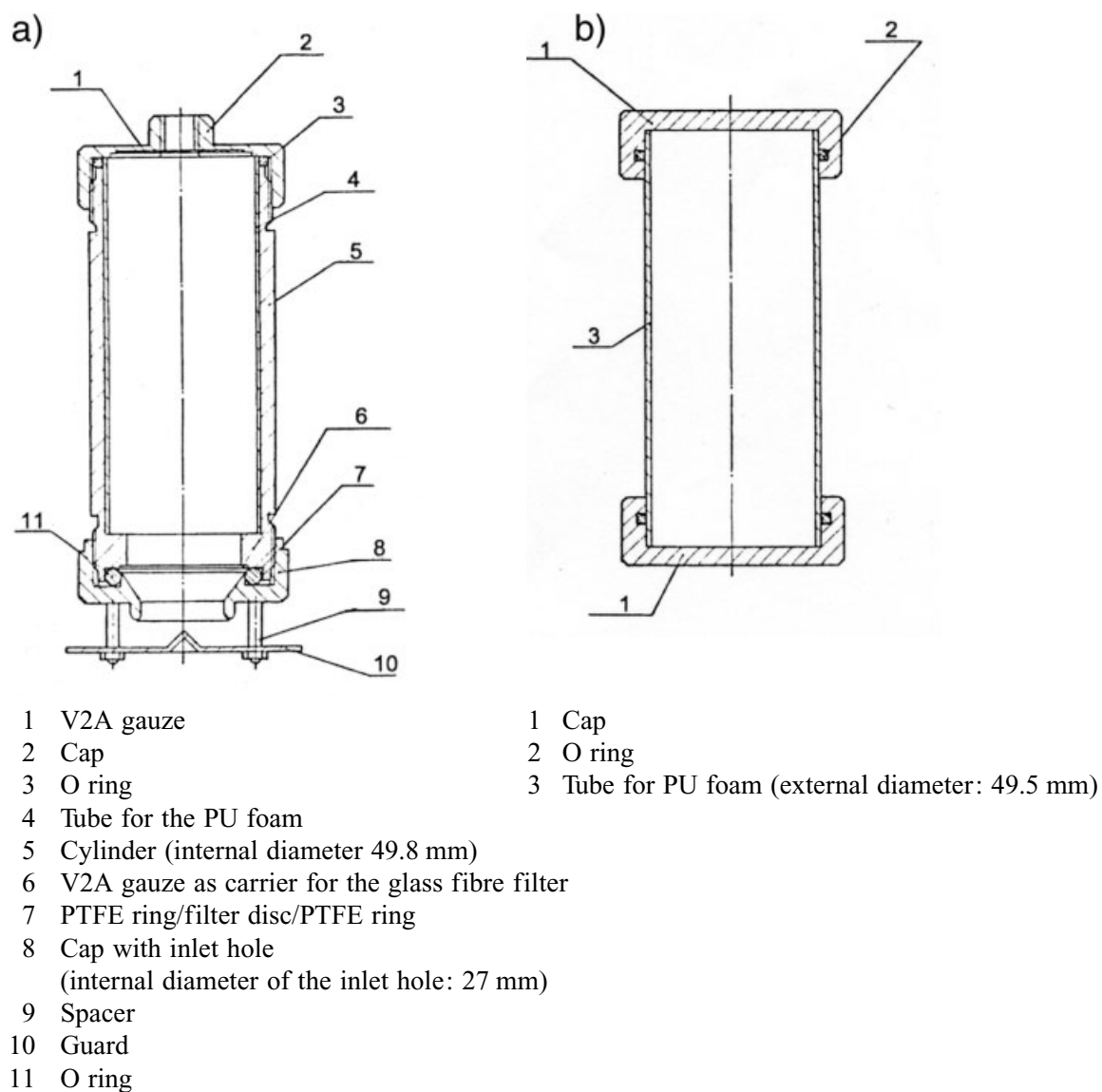
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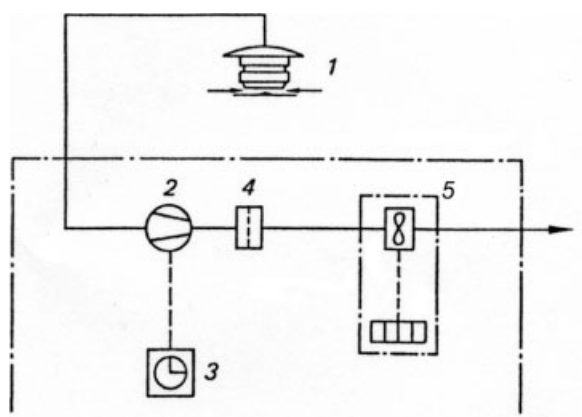
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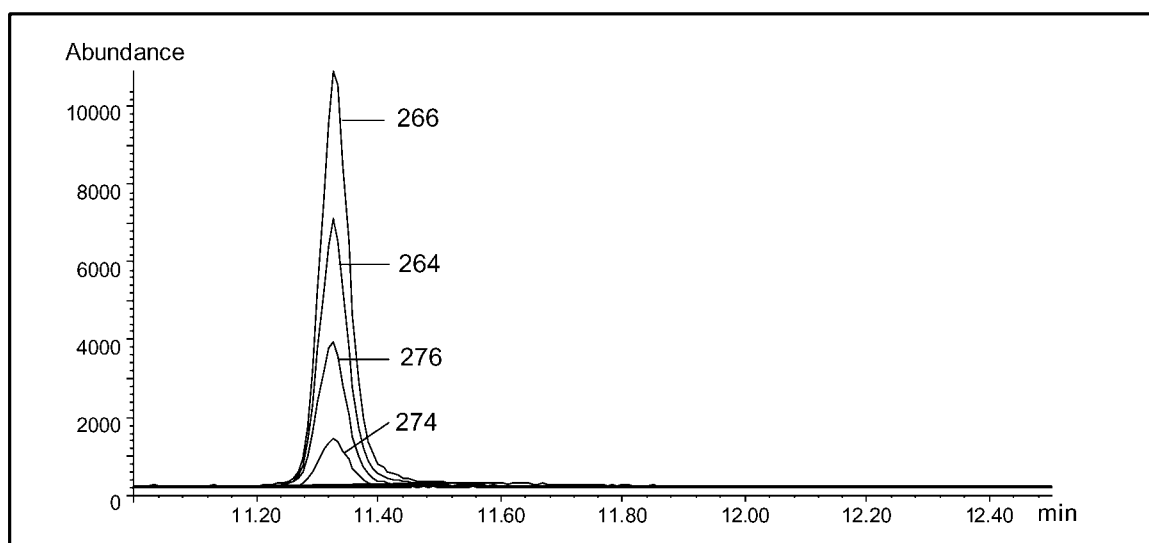


**Fig. 3.** a) Sampling head; b) tube for PU foam with caps (aluminium).



- 1 Sampling head
- 2 Pump
- 3 Timer
- 4 Collector for rubbings
- 5 Volume meter

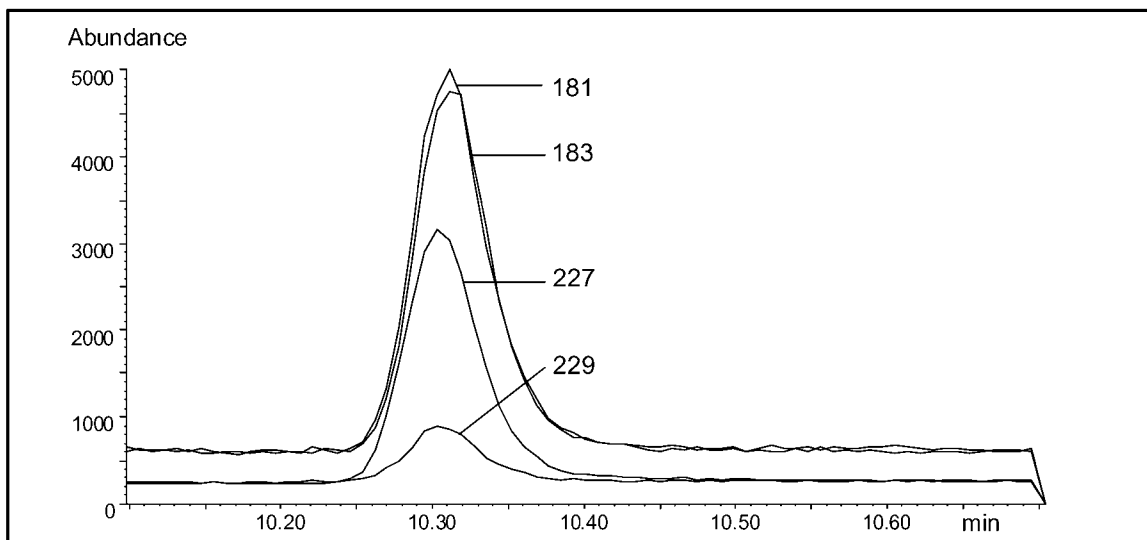
**Fig. 4.** Sampling apparatus set-up.



**Fig. 5.** Ion chromatograms of a pentachlorophenol standard (as acetate).

$m/z$ : 264, 266 pentachlorophenol acetate (100  $\mu\text{g/L}$ )

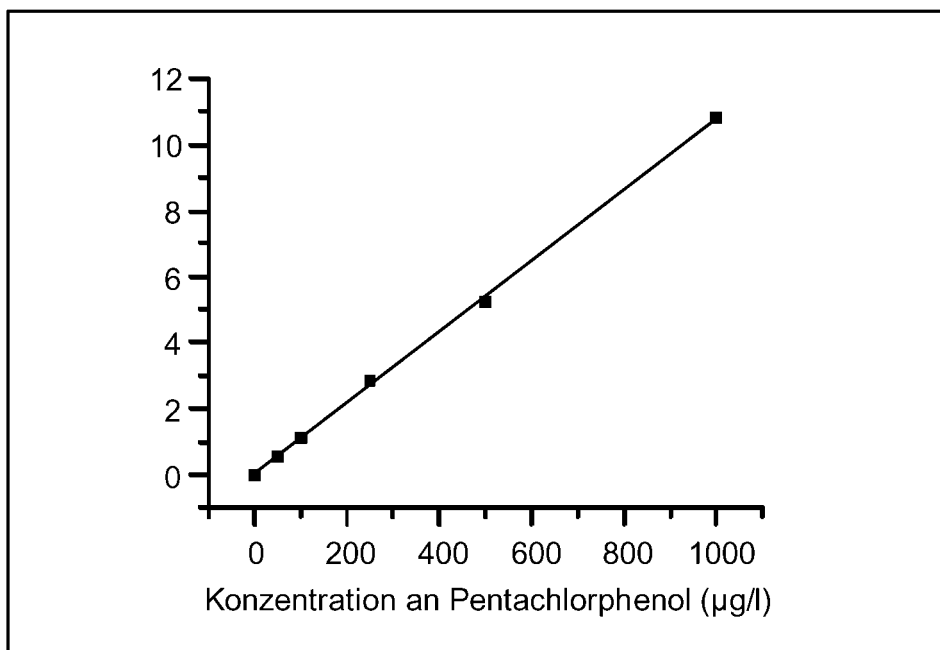
$m/z$ : 274, 276  $^{13}\text{C}$ -pentachlorophenol acetate (internal standard, 200  $\mu\text{g/L}$ )



**Fig. 6.** Ion chromatograms of a lindane standard.

*m/z*: 181,183 lindane (100 µg/L)

*m/z*: 227, 229 <sup>13</sup>C-lindane (internal standard, 200 µg/L)



**Fig. 7.** Six-point calibration curve for pentachlorophenol.

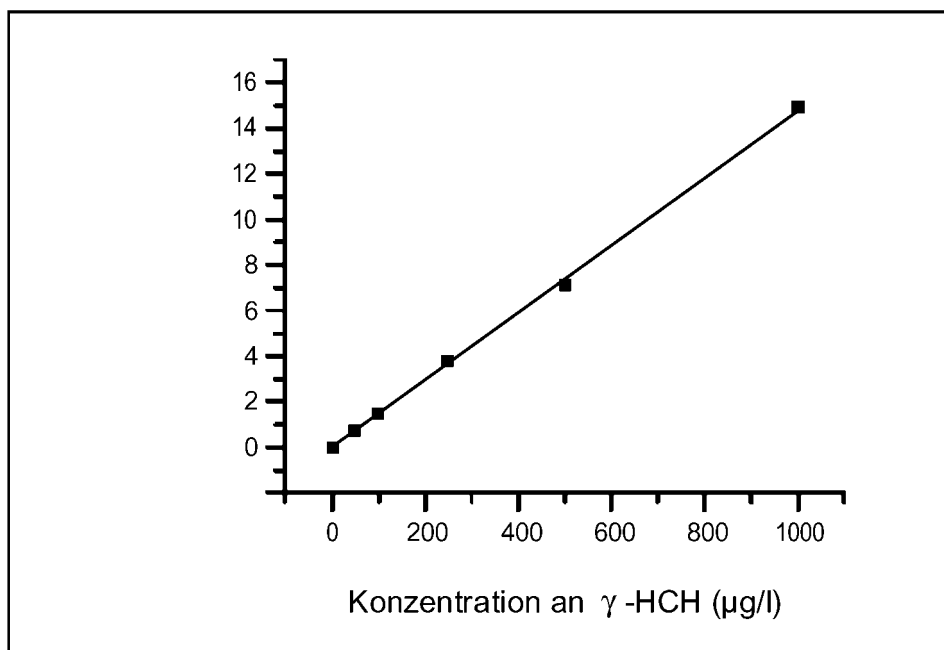


Fig. 8. Six-point calibration curve for lindane.

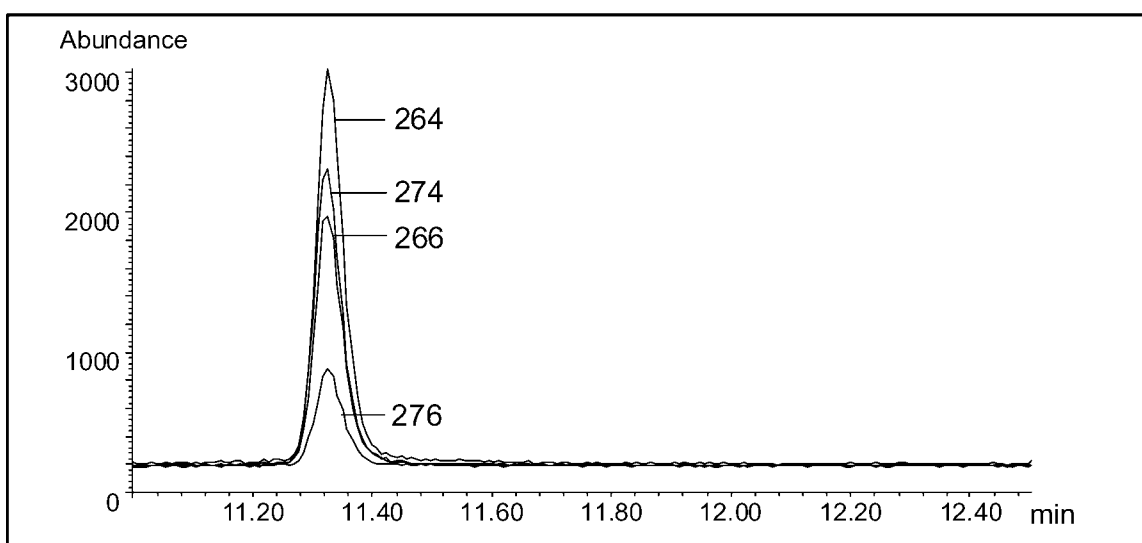
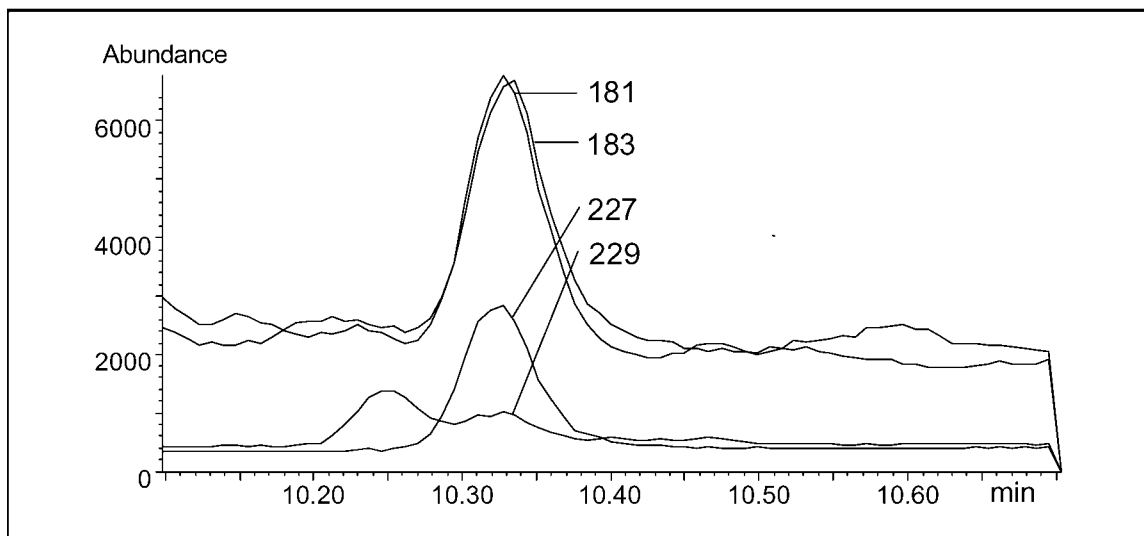


Fig. 9. Ion chromatograms of a toluene extract of an air sample  
here: analysis of pentachlorophenol (as acetate)  
 $m/z$ : 264, 266 pentachlorophenol acetate  
 $m/z$ : 274, 276  $^{13}\text{C}_6$ -pentachlorophenol acetate (internal standard)  
(concentration of pentachlorophenol about 130  $\mu\text{g/L}$ )



**Fig. 10.** Ion chromatograms of a toluene extract of an air sample  
here: analysis of lindane  
 $m/z$ : 181, 183 lindane  
 $m/z$ : 227, 229  $^{13}\text{C}_6$ -lindane (internal standard)  
(concentration of lindane about 180  $\mu\text{g/L}$ )