

PCB 47, PCB 51 and PCB 68 – Determination of PCB 47, PCB 51 and PCB 68 in workplace air using gas chromatography (GC-ECD)

Air Monitoring Method – Translation of the German version from 2023

Keywords

polychlorinated biphenyls; air analyses; analytical method; workplace measurement; hazardous substance; gas chromatography; GC-ECD; Florisil; electron capture detection

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Abstract

This analytical method is a validated measurement procedure for the determination of the three tetrachlorinated biphenyls PCB 47 [2437-79-8], PCB 51 [68194-04-7] and PCB 68 [73575-52-7] in workplace air in a concentration range of 0.16 to 0.62 µg/m³. It was developed to detect PCBs that only may be generated during the manufacture of silicone products with peroxidic crosslinking with bis(2,4-dichlorobenzoyl) peroxide. By measurement in manufacturing plants it could be proven that the PCBs to be investigated are present exclusively in vapour form. For this reason, the method was only validated for vaporous samples. There are currently no valid evaluation criteria for these PCBs. Therefore, the German Occupational Exposure Limit Value for the sum of all PCBs (5 × sum of the 6 indicator PCBs [28, 52, 101, 138, 152, 180]) of 3 µg/m³ was used as the assessment standard for each congener. For sampling, a defined volume of air is drawn through a sorbent tube filled with Florisil. The flow rate is set to 1 l/min and sampling duration is 4 hours (which correspond to a sampling volume of 240 l). The PCBs are extracted with *n*-hexane at 40 °C in an ultrasonic bath and subsequently analysed using gas chromatography with electron capture detection. The quantitative determination is based on a calibration function. The limit of quantification is 0.11 µg/m³ based on an air sample volume of 240 l. The mean recovery is 96% and the expanded uncertainty for the validation range of 0.16 to 0.62 µg/m³ is 22 to 24%.

Method number	1
Application	Air analysis
Analytical principle	Gas chromatography with electron capture detection (GC-ECD)

1 Characteristics of the method

Precision:	Standard deviation (rel.):	
	PCB 47	$s = 1.1\text{--}4.1\%$
	PCB 51	$s = 5.0\text{--}7.2\%$
	PCB 68	$s = 2.9\text{--}4.8\%$
	Expanded uncertainty:	
	PCB 47	$U = 22\%$
	PCB 51	$U = 24\%$
	PCB 68	$U = 23\%$
	in the concentration range from $c = 0.16$ to $0.62 \mu\text{g}/\text{m}^3$ and for $n = 6$ determinations	
Limit of quantification:	$0.11 \mu\text{g}/\text{m}^3$ for an air sample volume of 240 l and a sampling period of 4 h	
Recovery:	PCB 47	$\eta = 90$ to 93%
	PCB 51	$\eta = 91$ to 93%
	PCB 68	$\eta = 96$ to 100%
Sampling recommendations:	Sampling period:	4 h
	Air sample volume:	240 l
	Volumetric flow rate:	1 l/min

2 Description of the substances

PCB 47 [2437-79-8], PCB 51 [68194-04-7], PCB 68 [73575-52-7]

The three investigated polychlorinated biphenyls (PCBs) 2,2',4,4'-tetrachlorobiphenyl (PCB 47), 2,2',4,6'-tetrachlorobiphenyl (PCB 51) and 2,3',4,5'-tetrachlorobiphenyl (PCB 68) are colourless, crystalline solids with only a slight odour. The physico-chemical data are listed in [Table 1](#) and the structural formulae are shown in [Figure 1](#).

The PCBs mentioned above can occur during the production of silicone products with peroxidic crosslinking (when bis(2,4-dichlorobenzoyl) peroxide [133-14-2] is used) (Hombrecher 2019).

Tab. 1 Predicted physico-chemical substance data of PCB 47, PCB 51 and PCB 68 according to WebTEST of the U.S. EPA (US EPA 2022)

	2,2',4,4'-Tetrachlorobiphenyl (PCB 47)	2,2',4,6'-Tetrachlorobiphenyl (PCB 51)	2,3',4,5'-Tetrachlorobiphenyl (PCB 68)
CAS No.	2437-79-8	68194-04-7	73575-52-7
Molar mass [g/mol]	291.98	291.98	291.98
Aggregate state at 20 °C	solid	solid	solid
Density at 25 °C [g/cm ³]	1.42	1.42	1.44
Vapour pressure at 25 °C [hPa]	$9.24 \cdot 10^{-5}$	$1.07 \cdot 10^{-4}$	$4.87 \cdot 10^{-5}$

Tab. 1 (continued)

	2,2',4,4'-Tetrachlorobiphenyl (PCB 47)	2,2',4,6'-Tetrachlorobiphenyl (PCB 51)	2,3',4,5'-Tetrachlorobiphenyl (PCB 68)
Melting point [°C]	103	94	97
Boiling point at 1013 hPa [°C]	340	334	351
Flash point [°C]	157	157	167

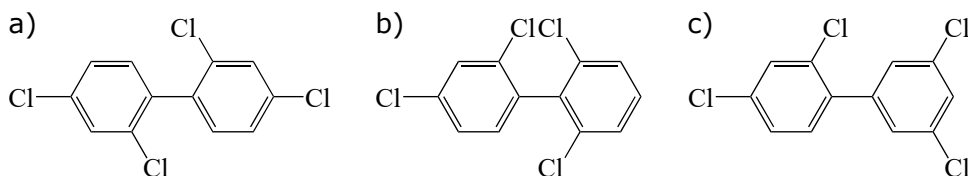


Fig. 1 Structural formulae for the polychlorinated biphenyls a) PCB 47, b) PCB 51 and c) PCB 68

3 General principles

The analytical method permits the determination of PCB 47, PCB 51 and PCB 68 in the workplace air. This method is suitable for measurement of PCB congeners 47, 51 and 68 in the range of 0.11 to approx. 0.62 µg/m³ in the workplace air. The cumulative limit value for all PCBs (5 × sum of the six indicator PCBs [28, 52, 101, 138, 152, 180]) of 3 µg/m³ (AGS 2022; DFG 2022) was used as the basis for devising this method.

A suitable pump draws a defined volume of air through a quartz glass tube filled with Florisil as well as through a sampling head (GGP-Mini) fitted with a 13 mm glass fibre filter connected upstream. PCBs contained in the air in vaporous and particulate form are deposited onto the sample carrier (glass fibre filter and Florisil tube). For analytical determination the PCBs are desorbed with *n*-hexane in an ultrasonic bath and transferred into a gas chromatograph with capillary column. After separation on a non-polar column, the qualitative and quantitative evaluation is carried out by means of electron capture detection (ECD). The internal standard method is used for quantitative evaluation.

During workplace measurements in production facilities carried out as part of the method development, it was verified that the PCBs to be investigated occur only in vaporous form. For this reason, the method was validated only for the sampling of vapour.

4 Equipment, chemicals and solutions

4.1 Equipment

For sampling:

- Sampling pump for personal or stationary sampling, suitable for a volumetric flow rate of 1 l/min (e.g. GilAir Plus, from DEHA Haan & Wittmer GmbH, 71296 Heimsheim, Germany)
- Quartz glass tube: length 100 mm, inner diameter 6 mm, outer diameter 8 mm, filled with 600 mg of Florisil 30–60 mesh (e.g. from VWR International GmbH, 64293 Darmstadt, Germany)
- Personal sampling head for the inhalable fraction (e.g. GGP-Mini, from GSA Messgerätebau GmbH, 40880 Ratingen, Germany)
- Glass fibre filter, Ø 13 mm (e.g. MN 85/90 BF type, from Macherey and Nagel GmbH, 52355 Düren, Germany)

- GGP-Mini Silicone adaptor (e.g. from Carmacon, 67574 Osthofen, Germany)
- Flow meter (e.g. TSI Flowmeter 4146, from TSI GmbH, 52068 Aachen, Germany)
- Screw-capped vials, with caps and sealing disks, nominal volume 10 ml (e.g. from LABC Labortechnik Zillger KG, 53773 Hennef, Germany)

For the sample preparation and the analytical determination:

- Volumetric flasks (glass), nominal volume 5 and 10 ml (e.g. from Brand GmbH + Co. KG, 97877 Wertheim, Germany)
- Variable piston pipettes, 10 to 100 μ l and 100 to 1000 μ l with 100-, 200- and 1000- μ l pipette tips (e.g. Eppendorf Multipette E3 with Combitips, from Eppendorf SE, 22366 Hamburg, Germany)
- Microlitre syringes, nominal volumes of 2, 5 und 10 μ l (e.g. from Hamilton Bonaduz AG, Bonaduz, Switzerland)
- Gas chromatograph with electron capture detection (GC-ECD) and data evaluation unit (e.g. from PerkinElmer LAS (Germany) GmbH, 63110 Rodgau, Germany)
- Disposable syringes, volume 2 ml with disposable hypodermic needles 0.9 \times 40 mm (e.g. from B. Braun SE, 34209 Melsungen, Germany)
- Syringe filters with PTFE membrane, \varnothing 13 mm, pore size 0.45 μ m (e.g. from VWR International GmbH, 64293 Darmstadt, Germany)
- Quartz wool, silanised (e.g. Supelco, Merck KGaA, 64293 Darmstadt, Germany)
- Autosampler vials, nominal volume 1.5 ml (e.g. from LABC Labortechnik Zillger KG, 53773 Hennef, Germany)
- Screw caps for the autosampler vials (e.g. from CS-Chromatographie Service GmbH, 52379 Langerwehe, Germany)
- Analytical balance (e.g. XP 20S Delta Range®, from Mettler-Toledo GmbH, 35396 Gießen, Germany)
- Non-polar separation column, e.g. Rxi-5-Sil MS, 60 m, inner diameter 2.5 mm and film thickness 1 μ m (e.g. from Restek GmbH, 61348 Bad Homburg, Germany)
- Heatable ultrasonic bath (e.g. Sonorex, from Bandelin electronic GmbH & Co. KG, 12207 Berlin, Germany)

4.2 Chemicals

- PCB 47 in isooctane (100 μ g/ml), 100% (e.g. from Cambridge Isotope Laboratories Inc., supplied by LGC Standards GmbH, 46469 Wesel, Germany)
- PCB 51 in isooctane (35 μ g/ml), 100% (e.g. from Cambridge Isotope Laboratories Inc., supplied by LGC Standards GmbH, 46469 Wesel, Germany)
- PCB 68 in isooctane (35 μ g/ml), 98% (e.g. from Cambridge Isotope Laboratories Inc., supplied by LGC Standards GmbH, 46469 Wesel, Germany)
- Pentachlorobenzene, 100% (e.g. from VWR International GmbH, 64293 Darmstadt, Germany)
- *n*-Hexane for gas chromatography, 100% (e.g. SupraSolv, from Merck KGaA, 64293 Darmstadt, Germany)
- Helium, 5.0
- Synthetic air, free of hydrocarbons

4.3 Solutions

The following solutions, which can be stored in the refrigerator at +4 °C for at least 3 months, are prepared from the chemicals listed in [Section 4.2](#):

Internal standard stock solution: (10 mg of pentachlorobenzene/ml in *n*-hexane):

Approx. 50 mg of pentachlorobenzene are weighed into a 5-ml volumetric flask, the flask is then filled to the mark with *n*-hexane and shaken.

Internal standard solution: (50 µg of pentachlorobenzene/ml in *n*-hexane):

The internal standard solution is prepared by dilution of the internal standard stock solution. For this purpose, 10 µl of the pentachlorobenzene stock solution is transferred into a 2-ml volumetric flask into which *n*-hexane has been previously placed. The flask is then filled to the mark with *n*-hexane and shaken.

Calibration stock solution: (1 µg of each PCB/ml in *n*-hexane):

A piston pipette is used to add 50 µl of the PCB 47 standard in isooctane and 150 µl each of the other PCB standards into a 5-ml volumetric flask, into which approx. 1.5 ml of *n*-hexane have been previously placed. The flask is then filled to the mark with *n*-hexane and shaken.

Control stock solution: (600 ng of each PCB/ml in *n*-hexane):

A piston pipette is used to add 30 µl of the PCB 47 standard in isooctane and 90 µl each of the other PCB standards into a 5-ml volumetric flask, into which approx. 1.5 ml of *n*-hexane have been previously placed. The flask is then filled to the mark with *n*-hexane and shaken.

4.4 Calibration standards

The calibration solutions are prepared by diluting the calibration stock solution. The volumes of the calibration stock solution given in Table 2 are each added into one 2-ml volumetric flask into which *n*-hexane has been previously placed. The volumetric flasks are then each filled to the mark with *n*-hexane. 2 µl of the internal standard solution are then added in each case with an appropriate microlitre syringe and the volumetric flasks are shaken.

The calibration solutions must be freshly prepared before each calibration.

Tab. 2 Preparation and concentrations of the calibration solutions

Volume of the calibration stock solution [µl]	Mass concentration [ng/ml]		
	PCB 47	PCB 51	PCB 68
20	10.0	10.5	10.5
45	22.5	23.6	23.6
70	35.0	36.8	36.8
95	47.5	49.9	49.9
120	60.0	63.0	63.0

4.5 Control solution

A control solution with a concentration in the intermediate concentration range must be analysed every working day by determination in triplicate under the conditions described in Section 6. It is prepared as follows:

Control solution: (75 ng of each PCB/ml in *n*-hexane):

A piston pipette is used to transfer 625 µl of the control stock solution into a 5-ml volumetric flask, into which approx. 1.5 ml of *n*-hexane have been previously placed. The flask is then filled to the mark with *n*-hexane and shaken. The solution is filled into the autosampler vials, stored in the refrigerator and a verification is carried out every working day.

5 Sampling and sample preparation

5.1 Pre-treatment of the sample carriers

The sample carrier consists of a quartz glass tube filled with approx. 600 mg of Florisil, stabilised at both ends with silanised quartz wool and secured with a PTFE ring. Before sampling, the Florisil tubes are loaded with 2 µl of internal standard solution (pentachlorobenzene) and air is drawn through the tube for 15 minutes at a volumetric flow rate of 1 l/min in order to eliminate the solvent. The tubes are sealed at both ends with plastic caps and the direction of sampling to be followed is marked with an arrow.

If glass fibre filters are used additionally, these are not loaded with internal standard.

5.2 Sampling

Sampling can be carried out as stationary or personal sampling. Measurements are taken in the breathing zone in the case of personal sampling. It is important to ensure that the inlet of the sampling head is freely accessible.

Suitable flow-regulated pumps are used for sampling. Using a representative sample carrier to simulate upstream resistance, the volumetric flow rate is set to 1 l/min (60 l/h).

Immediately before sampling, the plastic caps are removed from the Florisil tube and it is connected to the pump in the direction of the arrow. The other end of the tube is connected with the GGP-Mini sampling head via a silicone tube. The recommended sampling period is four hours. This is equivalent to an air sample volume of 240 l for this sampling period. The important parameters for the determination of the concentration in air (air sample volume, temperature, air pressure and relative humidity) are documented in the sampling record.

Immediately after sampling, the tube is resealed with the plastic caps. The sampling head of the GGP-Mini is unscrewed and tweezers are used to transfer the glass fibre filter into a 10-ml screw-capped vial, which is then sealed.

After sampling, the volumetric flow rate must be tested for constancy. If the deviation from the adjusted volumetric flow rate is $\geq \pm 5\%$, it is advisable to repeat the measurement. The hermetically sealed sample carriers are transported to the laboratory at room temperature and kept sealed in storage until sample preparation.

5.3 Sample preparation

The loaded Florisil sample carrier is opened. The anterior quartz wool as well as the entire Florisil are transferred into a 10-ml screw-capped vial. The vial is filled with 2 ml of *n*-hexane and extracted for 30 min in a heated ultrasonic bath at 40 °C. The extract is filtered through a PTFE syringe filter, transferred into an autosampler vial and analysed in the gas chromatograph.

If the glass fibre filter is also used to collect the particulate phase, it is processed in the same manner as the Florisil tube. For this purpose, the transport vessel is opened, 2 ml of *n*-hexane and 2 µl of the internal standard solution are added. The vial is then sealed and treated in the ultrasonic bath as described above.

6 Operating conditions

Apparatus:	Autosystem XL3 gas chromatograph, with ECD and autosampler, from PerkinElmer LAS (Germany) GmbH	
Separation column:	Stationary phase:	Rxi-5 Sil-MS
	Length:	60 m
	Inner diameter:	0.25 mm
	Film thickness:	1 µm

Injection volume:	1 µl
Injector temperature:	230 °C
Detector:	ECD with Ni 63 foil (555 MBq)
Detector temperature:	360 °C
Oven program:	Initial temperature 150 °C heat with 15 °C/min to 260 °C; maintain that temperature for 13 min
Carrier gas:	Helium 5.0
Column flow:	2 ml/min
Split:	10 ml/min

7 Analytical determination

By means of the autosampler, 1 µl of the prepared sample is injected into the gas chromatograph and analysed under the conditions described in [Section 6](#).

If the determined concentrations are above the calibration range, then a suitable dilution of the sample must be prepared with *n*-hexane and the analysis repeated.

8 Calibration

The calibration solutions described in [Section 4.4](#) are used to obtain the calibration functions.

In each case, 1 µl of the calibration solutions is injected and analysed in the same manner as the sample solutions. The quotients of the resulting peak areas of the PCB and internal standard are plotted versus the corresponding concentrations of the PCB. The calibration function is linear in the investigated concentration range.

A control solution must be analysed each working day to check the calibration function. The calibration must be performed anew if the analytical conditions change or the quality control results indicate that this is necessary.

9 Calculation of the analytical result

The concentration in the workplace air is obtained from the mass X_i of PCB per sample carrier calculated by the data evaluation unit. Taking the air sample volume V into account, the concentration of PCB in the workplace air is calculated according to [Equation 1](#):

$$\rho_i = \frac{X_i}{V \times \eta} \quad (1)$$

[Equation 2](#) enables for the calculation of the value at 20 °C and 1013 hPa:

$$\rho_{i,0} = \rho_i \times \frac{273 + t_a}{293} \times \frac{1013}{p_a} \quad (2)$$

where:

ρ_i is the mass concentration of the substance in the air sample in µg/m³ at t_a and p_a

- $\rho_{i,0}$ is the mass concentration of the substance in the air sample in $\mu\text{g}/\text{m}^3$ at 20 °C and 1013 hPa
- X_i is the mass of the substance in the analytical sample in ng
- V is the air sample volume in l (calculated from the volumetric flow rate and the sampling period)
- η is the recovery
- t_a is the temperature during sampling in °C
- p_a is the atmospheric pressure during sampling in hPa

10 Reliability of the method

The characteristics of the method were determined as stipulated in DIN EN 482 (DIN 2021 a), ISO 20581 (DIN 2016) and DIN EN ISO 22065 (DIN 2021 b). The limits of detection and limits of quantification were calculated as stipulated in DIN 32645 (DIN 2008) according to the blank value method. In this case, twice the limit value could not be covered, as only diluted standards and no pure substances were available for the validation.

10.1 Precision, recovery and expanded uncertainty

The characteristics of the method were determined by preparing the Florisil sample carriers as described in Section 5.1 and loading them with a spiking solution, to yield PCB spiking masses of 38 ng to 149 ng. This is equivalent to a concentration range of 0.16 to 0.62 $\mu\text{g}/\text{m}^3$ on the basis of an air sample volume of 240 l and an extraction volume of 2.0 ml. Conditioned air from a dynamic test gas facility was drawn through the spiked sample carriers for 4 hours at a volumetric flow rate of 1 l/min.

The experiments on the recovery and precision were carried out at a relative humidity of approx. 40%. The sample analysis was carried out as described in Sections 5 and 6.

Six sample carriers were loaded for each experiment to determine the recovery. The resulting variation coefficients as well as the calculated expanded uncertainties can be found in Table 3. The calculation was carried out as stipulated in DIN EN ISO 22065 (DIN 2021 b) using the IFA software calculation of the expanded uncertainty (IFA o.J.).

Tab. 3 Recoveries (η), relative standard deviations (s) and expanded uncertainties (U) according to DIN EN 482 (DIN 2021 a) and ISO 20581 (DIN 2016) for different concentration ranges

Substance	Concentration ^{a)} [$\mu\text{g}/\text{m}^3$]	η [%]	s [%]	U [%]
PCB 47	0.16	93	1.1	21.5
	0.31	91	3.1	21.6
	0.62	90	4.1	21.8
PCB 51	0.16	93	5.0	23.5
	0.31	91	7.2	23.9
	0.62	91	5.3	23.5
PCB 68	0.16	96	4.8	23.2
	0.31	100	3.5	22.7
	0.62	96	2.9	23.0

^{a)} on the basis of a four-hour sampling period at a volumetric flow rate of 1 l/min

The expanded uncertainty was obtained by estimating all relevant influencing parameters. The uncertainty of the result consists of two important contributions, the uncertainty components for sampling and for analysis.

The combination of all uncertainty contributions results in the concentration-dependent combined uncertainties of the entire method. The values for the expanded uncertainty, listed in [Table 3](#), are obtained by multiplying with the expansion factor $k = 2$.

10.2 Limit of quantification

The limit of quantification was determined according to the blank value method described in DIN 32654 (DIN 2008). The limits of quantification are 26 ng per sample carrier absolute for all three PCBs or $0.11 \mu\text{g}/\text{m}^3$ for an air sample volume of 240 litres (1 l/min and a sampling period of 4 h).

10.3 Influence of the humidity

The influence of the humidity was investigated at concentrations of $0.16 \mu\text{g}/\text{m}^3$ and $0.62 \mu\text{g}/\text{m}^3$ at relative humidities of approx. 20 and 80%. In this case, no evidence was found that recovery is dependent on the relative humidity.

10.4 Capacity of the sampling system

Two tubes were connected in series and the upstream tube was spiked with $1.5 \mu\text{g}$ of PCB 47 to determine the breakthrough behaviour of the sampling system used. This is equivalent to a concentration of $5 \mu\text{g}/\text{m}^3$ at an air sample volume of 300 l, which results from a 5-hour sampling period (recommended sampling period plus one hour). Then, conditioned air from a dynamic test gas facility was drawn through the setup (1 l/min). Subsequently, both tubes were prepared and analysed separately (see [Sections 5](#) and [6](#)). No breakthrough was observed.

10.5 Storage stability

10.5.1 Storage stability before sampling

The sample carriers have a limited storage stability of only 14 days, as they must be spiked with the internal standard solution (pentachlorobenzene) before sampling. This was verified as part of the validation of the method.

10.5.2 Storage stability of the loaded sample carriers

The storage stability of the loaded Florisil tubes was determined by spiking nine sample carriers each with both concentrations of $0.16 \mu\text{g}/\text{m}^3$ and $0.62 \mu\text{g}/\text{m}^3$ as described in [Section 10.1](#). The sample carriers were conditioned for four hours with air of high humidity (approx. 80%) at a volumetric flow rate of 1 l/min. The tubes were then sealed with caps and stored at room temperature. Three tubes in each case were then processed and analysed as described in [Sections 5](#), [6](#) and [7](#) after one day, one week and two weeks.

No loss of the analytes could be detected during this period.

10.6 Selectivity

This method was developed for the sampling of PCBs that may be generated in the production of silicone products involving peroxidic crosslinking with bis(2,4-dichlorobenzoyl) peroxide. A chromatogram with the internal standard and the three investigated PCBs is shown in [Figure 2](#).

Products containing PCBs from other sources such as transformer oils are complex PCB mixtures. In comparative tests with Aroclor mixtures, it was shown that interferences in the determination of PCB 47, PCB 51 and PCB 68 cannot be ruled out.

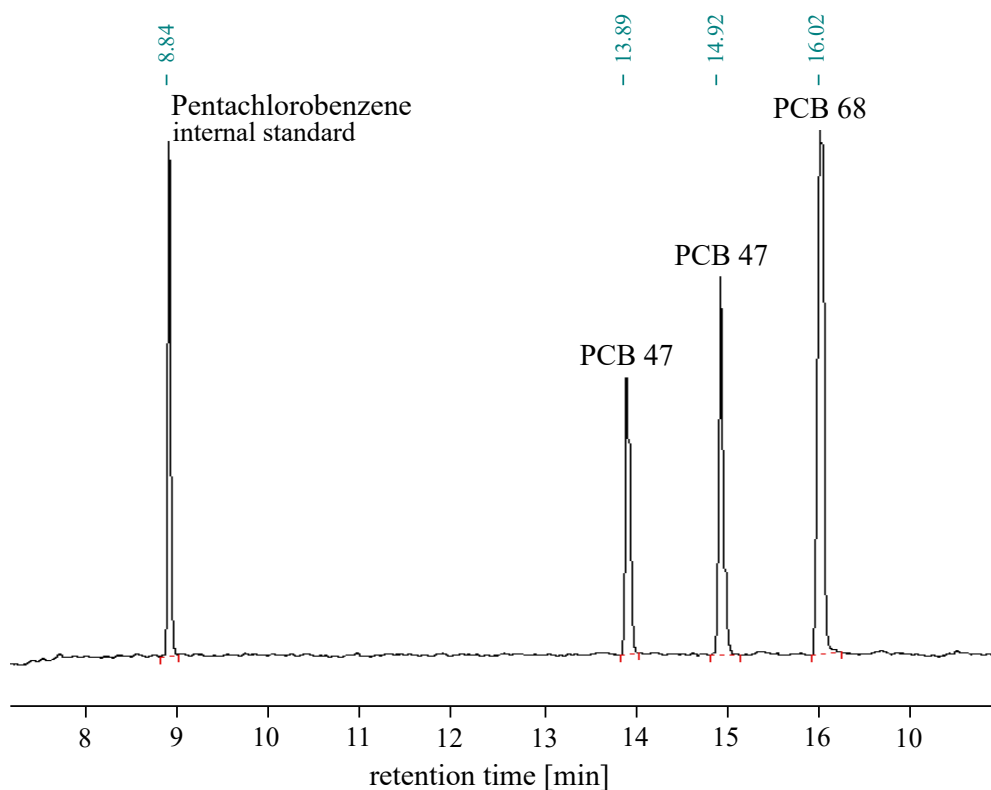


Fig. 2 Example of a chromatogram for the gas chromatographic separation of the three polychlorinated biphenyls PCB 47, PCB 51, PCB 68 and the internal standard pentachlorobenzene

11 Discussion

The analytical method described here permits the determination of PCB 47, PCB 51 and PCB 68 in the workplace air in a concentration range from 0.11 to 0.62 $\mu\text{g}/\text{m}^3$. No negative influence from the humidity (of up to 80% rel.) on the measurement results and the storage stability could be detected.

In principle, for PCB 47, PCB 51 and PCB 68 a sampling system must be used that is suitable for particle and vapour mixtures as stipulated in DIN EN ISO 23861 (DIN 2023). The suitability of the method was tested by performing workplace measurements during the manufacturing of silicone tubes and sealants. In this case, PCB 47 was only detected on the Florisil tube. If the presence of a particulate phase cannot be excluded, then the loaded glass fibre filter must also be analysed.

Notes

Competing interests

The established rules and measures of the Commission to avoid conflicts of interest (www.dfg.de/mak/conflicts_interest) ensure that the content and conclusions of the publication are strictly science-based.

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