APPLICATION NOTE

Determination of Polychlorinated Biphenyls (PCBs) in water using GC-ECD

AN179 v1; SCION Instruments

Introduction

Polychlorinated biphenyls (PCBs) were produced in large quantities between 1930 and 1980, typically used in electrical devices and coolant fluids. They were identified as environmental toxic and were banned in the US in 1979. In 1986, an international agreement banned most uses of PCB's due to environmental concerns. PCBs persist in the environment for long periods of time and can travel through air, water and soil. PCBs are associated with a range of health issues, including skin problems, liver damage and have potential carcinogenic effects on both humans and animals.

PCBs are formed by attaching one or more chlorine atoms to a pair of connected benzene rings, shown in Figure 1[1]. Depending on the number and position of the chlorine atoms attached to the biphenyl structure, 209 different PCBs can be formed.

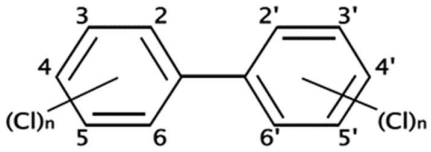

Figure 1 Polychlorinated Biphenyls chemical structure

There are regulations in place across different countries for controlling the usage of PCBs, due to concerns about the environmental and health effects.[2] Regulations aim to prevent and protect people from adverse effects, minimize releases and ensure cleaner ecosystems. Regulations accelerate the removal of PCBs by strict controls on their use, storage and disposal.

It is important to test for PCBs because it ensures compliance with disposal and cleaning requirements. This can contribute in preventing leakages into the environment. Regular testing gives information about the PCB levels in air, water and soil. Monitoring PCBs determines which actions could be taken to control the contamination.

This application can be performed on either the SCION Instruments 8300 GC & 8500 GC (Figure 2) platform with an Electron Capture Detector (ECD) and the SCION 8400PRO Autosampler. A SCION-5MS column is used for obtaining the best separation of the PCB components. Learn more about the MS columns in MS vs Non-MS GC columns technical note.

The ECD with make-up gas Nitrogen is used for the identification and detection of the components. For identification and quantification of the compounds you will require certified analytical standards

Experimental

For this application a PCB standard and an Internal Standard (IS) were purchased for the qualification and the quantification of the unknown samples. The PCB standard contains 15 PCB compounds with a concentration of 10 µg/mL. The internal standard contains 2 components of 1000 µg/mL: Decachlorobiphenyl and Tetrachloro-m-xylene (TCMX).

Table 1: Instrumentation operating conditions GC and SQMS

GC Part	Settings
S/SL Injector	240°C Split program, Initial: 50:1, 0.01 min: off, 0.50 min: 50:1 Pressure pulse: 25 psi, 0.5 min
Injection Volume	1.0 µL
Column	SCION-5MS 30m x 0.25mm x 0.25µm
Carrier Gas	Helium 1 mL/min
Oven Program	80°C (hold 2.0 min), 20°C/min to 325°C (hold 2.75 min)
Detector	ECD 300°C Make-up (N2): 29 mL/min Cell current: CAP
Run Time	17.0 min
Software	CompassCDS

Figure 2 SCION Instruments 8300 & 8500-GC equipped with 8400 PRO Autosampler.

Sample preparation

A calibration set from the PCB standard (Table 2) diluted with iso-octane was prepared at 6 levels ranging from 0.0003 µg/mL up to 0.005 µg/mL for the linearity. For the repeatability calibration standard 3 was used with a concentration of 0.0015 µg/mL. TCMX and Decachlorobiphenyl were used as internal standards (IS) and added to all the standards and samples, this was diluted to get a final concentration of 0.003 µg/mL.

Table 2: Components and CAS numbers of the PCB-standard.

No.	Compound	CAS Number	IS used
1	2,2′,5-Trichlorobiphenyl	37680-65-2	TCMX
2	2,3,3′-Trichlorobiphenyl	38444-84-7	TCMX
3	2,4,4′-Trichlorobiphenyl	7012-37-5	TCMX
4	2,4′,5-Trichlorobiphenyl	16606-02-3	TCMX
5	2,2′,3,5′-Tetrachlorobiphenyl	41464-39-5	TCMX
6	2,2′,5,5′-Tetrachlorobiphenyl	35693-99-3	TCMX
7	2,2′,4,5,5′-Pentachlorobiphenyl	37680-73-2	TCMX
8	2,3,3′,4,4′-Pentachlorobiphenyl	32598-14-4	Decachlorobiphenyl
9	2,3′,4,4′,5-Pentachlorobiphenyl	31508-00-6	Decachlorobiphenyl
10	2,2′,3,4,4′,5′-Hexachlorobiphenyl	35065-28-2	Decachlorobiphenyl
11	2,2′,3,4′,5′,6-Hexachlorobiphenyl	38380-04-0	TCMX
12	2,2′,4,4′,5,5′-Hexachlorobiphenyl	35065-27-1	Decachlorobiphenyl
13	2,2′,3,3′,4,4′,5-Heptachlorobiphenyl	35065-30-6	Decachlorobiphenyl
14	2,2′,3,4,4′,5,5′-Heptachlorobiphenyl	35065-29-3	Decachlorobiphenyl
15	2,2′,3,3′,4,4′,5,5′-Octachlorobiphenyl	35694-08-7	Decachlorobiphenyl

Tap water samples were prepared in triplicate by an extraction of 50 mL tap water with 10 mL iso-octane containing 0.003 µg/mL IS. The solution was stirred for 15 minutes with a magnetic stirrer, two layers appeared. The top (organic) layer was transferred into a sample container then concentrated under N_2-gas to approximately 1 mL. The extract was transferred into an injection vial ready for analysis.

To determine the recovery, six water samples were prepared as the water sample above and spiked with 0.0005 µg/mL PCB standard prior to extraction. These spiked samples were used to determine the LOQ of the method accordance with EPA regulations[2].

Blank injections of iso-octane were performed in between samples, to ensure that the system was not contaminated after sample injections.

Results

Because there are so many components present in this standard and method, not all components will be mentioned in the Results section. If necessary, the full validation report is accessible by request.

The results are shown for the following PCB compounds found in the sample: 2,2′,5-Trichlorobiphenyl (#1), 2,3,3′-Trichlorobiphenyl (#2), 2,2′,4,5,5′-Pentachlorobipheny l(#7), 2,3,3′,4,4′-Pentachlorobiphenyl (#8), 2,2',3,3',4,4',5,5'-Octachlorobiphenyl (#15),

The calibration curves for the PCB standards were prepared at 6 levels from 0.0003 µg/mL up to 0.005 µg/mL.

The system precision of the method was obtained by five consecutive injections of PCB standard (#3) (0.0015 µg/mL).

The system precision results of the selected PCB compounds can be found in Table 3, along with the linearity results (R^2) obtained from the calibration curves.

Table 3: Summary of Results – Linearity and repeatability

No.	Compound	R^2	Area repeatability (%RSD)
1	2,2′,5-Trichlorobiphenyl	0.998	4.41
2	2,3,3′-Trichlorobiphenyl	0.999	3.80
7	2,2′,4,5,5′-Pentachlorobiphenyl	0.997	3.45
8	2,3,3′,4,4′-Pentachlorobiphenyl	0.999	2.71
15	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	0.998	1.19

For all PCB compounds an R^2 of 0.996 or higher was achieved, which is an excellent result, with many regulations requiring an R^2 value of ≥0.98.

Repeatability results show that for most PCB components the relative standard deviations (RSD%) ranged from 1.19% to 4.41%. This is a good precision for the method, since most acceptance criteria for PCB method validation require an RSD ≤15%.[3]

The chromatogram shown in figure 3 shows good separation of the PCBs and IS in CompassCDS. There is an exception for peaks 2,4',5-Trichlorobiphenyl and peak 2,4,4'-Trichlorobiphenyl they are coeluting and are calculated as a sum total. See Integration technical note for integrating peaks in CompassCDS.

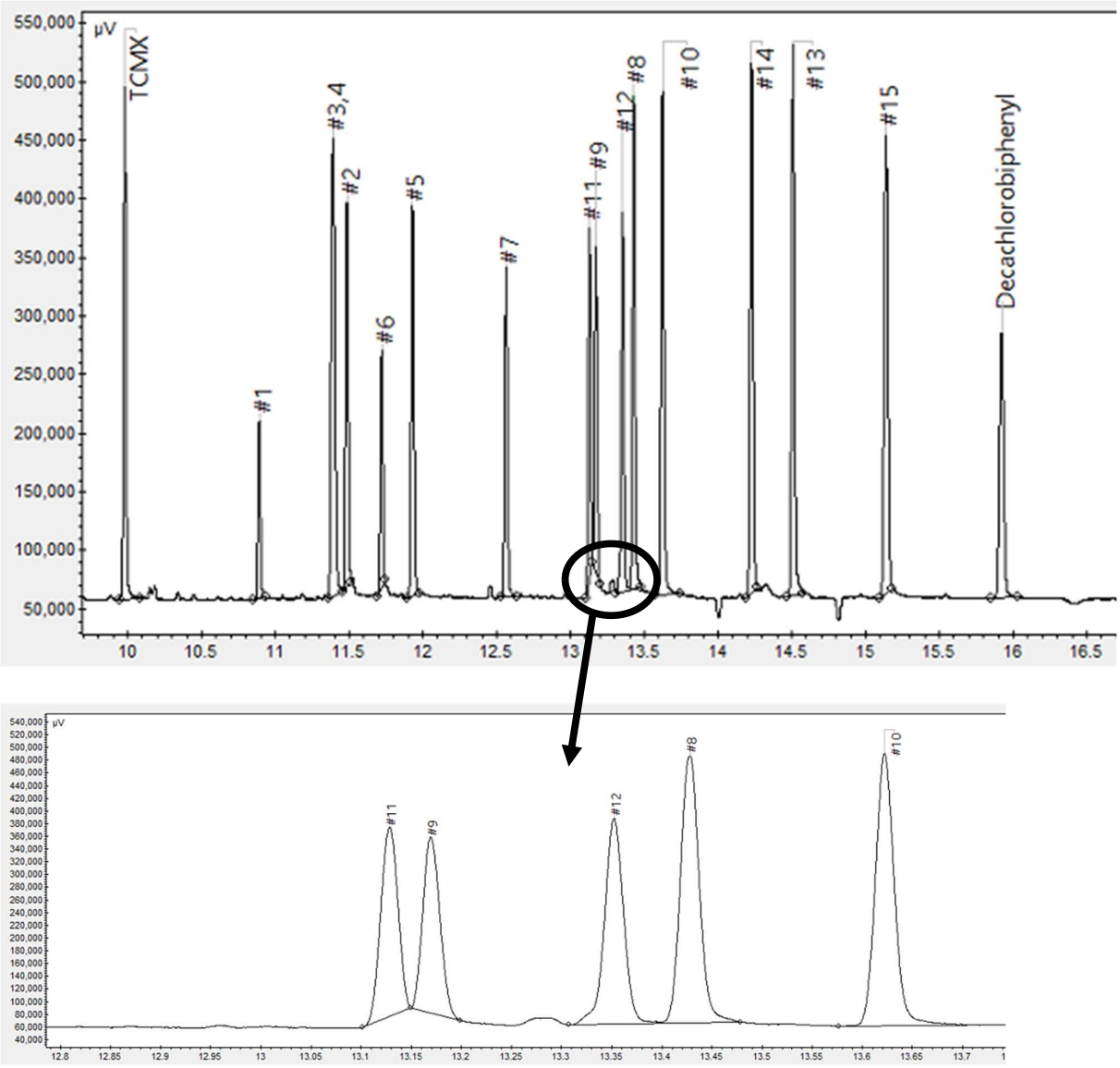

Figure 3: Example chromatogram of PCB standard (1-15) + Internal Standard

Table 4: Summary of Results – Recovery

No.	Compound	Water sample (µg/mL)	Spiked water sample (µg/mL)	Recovery (%)	RSD (%)
1	2,2',5-Trichlorobiphenyl	n.d.	0.00050	82.9	2.09
2	2,3,3'-Trichlorobiphenyl	n.d.	0.00050	82.9	2.18
7	2,2',4,5,5'-Pentachlorobiphenyl	n.d.	0.00050	50.7	2.83
8	2,3,3',4,4'-Pentachlorobiphenyl	n.d.	0.00050	102.6	1.89
15	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	n.d.	0.00050	81.5	0.61

n.d. = not detected

The water sample was prepared with (n=6) and without (n=3) spiked PCB standard to determine the recovery. The results (Table 4) indicate that none of the PCB components were detected in the water sample. The recovery percentages for the spiked samples ranged from 50.7% to 102.6%, with repeatability (RSD) values between 0.61% and 5.26% demonstrating good method efficiency and reliability.

Conclusion

The SCION 8500 GC platform equipped with a split/spitless injector, SCION 5MS column, ECD detector and 8400PRO Autosampler is a perfect solution for analysing PCBs in water for qualitative and quantitative analysis. Good system precision, linearity, results and recovery results are achieved for this application. The LOQ of 0.5 ppb according to EPA regulations[2] was achieved with the spiked recovery sample, confirming good working of the analytical method.[2]

The results were achieved with the SCION Instruments GC-ECD set up and CompassCDS software. The SCION-5MS column shows good separation between the PCBs.

The analysed water sample is, according to multiple results obtained by this application, most likely water that has not been exposed to PCBs.

References

1. Polychlorinated Biphenyls (PCBs) | Biomonitoring California https://biomonitoring.ca.gov/chemicals/polychlorinated-biphenyls-pcbs (Accessed 13-08-2024)

2. National Primary Drinking Water Regulations | US EPA https://www.epa.gov/ground-water-and-drinking-water/national-primary-drinking-water-regulations (Accessed 13-08-2024)

3. HJ 743-2015 English PDF (chinesestandard.net) https://www.chinesestandard.net/PDF/English.aspx/HJ743-2015 (Accessed 16-08-2024)

Order Information

Contact your sales representative for ordering information for the 8500/8300 GC

Suggested Consumables

Part	Part number
15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10	41312148
BTO Septa 9 mm, pk/50	CR298713
10µL fixed needle syringe, 5 cm, 0.47 mm OD, 26 g conical needle	41312133
SCION-5MS column 30m x 0,25mm x 0,25 µm	SC32223
1177 4mm SPLT LINER / FRT-SILTEK PK/5EA	RT210462145

For more information, please contact:

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