APPLICATION NOTE

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

AN185 v2; SCION Instruments

Introduction

Polychlorinated biphenyls (PCBs) were produced in large quantities between the 1930s and 1980s, typically used in electrical devices and coolant fluids. They were identified as environmentally toxic and were banned in the US in 1979. In 1986, an international agreement banned most uses of PCBs 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.[1]

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.

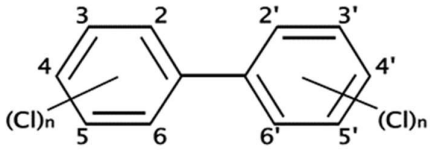

Figure 1 Polychlorinated Biphenyls chemical structure

QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) is a highly efficient sample preparation technique for the analysis of trace contaminants in soil samples. QuEChERS combines a solvent extraction with dispersive solid phase extraction (d-SPE) clean up, and is an ideal choice as a sample preparation method for these pollutants. This method reduces solvent consumption, sample preparation time and costs which makes it an attractive alternative to traditional techniques such as Soxhlet or ultrasonic extraction.

A soil sample must go through an appropriate sample preparation technique so it is suitable for injection onto the gas chromatograph (GC) for analysis. Figure 2 demonstrates how the soil sample looks before any sample preparation and then after using the QuEChERS method.

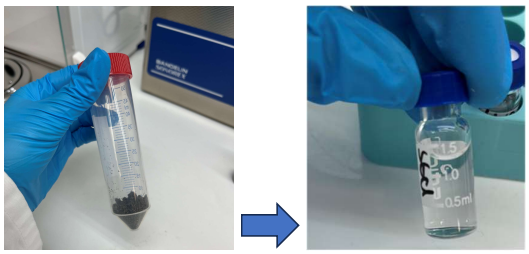

Figure 2 From soil sample to clear sample, suitable for GC analysis

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 to 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 3) 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 and carrier gas of Nitrogen is used for the detection of the compounds. For identification and quantification of the compounds you will require certified analytical standards. Learn more about the importance of using analytical standards in our technical note.

Figure 3 SCION Instruments 8300 & 8500-GC equipped with 8400 PRO Autosampler.

Experimental

For this application a PCB standard and an internal standard (IS) (Table 2) were purchased for the qualification and the quantification of the unknown samples. The PCB standard contained 14 PCB compounds with a concentration of 500 µg/mL. The internal standard contains 2 compounds of 1000 µg/mL: Decachlorobiphenyl (deca-CB) and Tetrachloro-m-xylene (TCMX).

Table 1 Instrumentation operating conditions GC

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, 0.4 min
Injection Volume	1.0 µL
Column	SCION-5MS 30m x 0.25mm x 0.25µm
Carrier Gas	Nitrogen 1 mL/min
Oven Program	50°C (hold 2.0 min), 8°C/min to 325°C (hold 3.63 min)
Detector	ECD 300°C Make-up (N2): 29 mL/min Cell current: CAP
Run Time	40.0 min
Software	CompassCDS

Sample preparation

Linearity samples were prepared in n-Hexane at 6 levels from 2.5 ppb to 100 ppb. The system precision was determined with multiple injections of L3 (20 ppb, n=10). Deca-CB and TCMX (50 ppb) were used as internal standards (IS) and added to all the standards and samples.

Table 2 Compounds of the PCB standard and internal standard.

No.	PCB no.	Compound	IS used
1	PCB-1	2-Chlorobiphenyl	TCMX
2	PCB-3	4-Chlorobiphenyl	TCMX
3	-	TCMX (IS)	-
4	PCB-7	2,4'-Dichlorobiphenyl	TCMX
3	PCB-18	2,2',5-Trichlorobiphenyl	TCMX
6	PCB-28	2,4,4'-Trichlorobiphenyl	TCMX
7	PCB-52	2,2',5,5'-Tetrachlorobiphenyl	TCMX
8	PCB-44	2,2',3,5'-Tetrachlorobiphenyl	TCMX
9	PCB-61	2,3',4',5-Tetrachlorobiphenyl	TCMX
10	PCB-109	2,3,3',4',6-Pentachlorobiphenyl	Deca-CB
11	PCB-142	2,2',3,4',5',6-Hexachlorobiphenyl	Deca-CB
12	PCB-114	2,3',4,4',5-Pentachlorobiphenyl	Deca-CB
13	PCB-153	2,2',4,4',5,5'-Hexachlorobiphenyl	Deca-CB
14	PCB-137	2,2',3,4,4',5'-Hexachlorobiphenyl	Deca-CB
15	PCB-180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	Deca-CB
16	-	Decachlorobiphenyl (IS)	-

The original QuEChERS method (unbuffered) was performed as follows.[3] The samples were prepared by weighing 5 g of soil into the 50 mL tube and adding 15 mL of acetonitrile/water (75%:25%, v/v). The mixture was vortexed for 4 minutes and then sonicated for 20 minutes. The extraction salts were added and vortexed for a further 4 minutes and centrifuged for 10 minutes at 4500 rpm. 6 mL of the supernatant was transferred to a 15 mL tube with clean up salts (900 mg $MgSO_4$, 150 mg Primary Secondary Amine (PSA), 150 mg Octadecylsilane (C18)). The clean-up tube was vortexed for 4 minutes and centrifuged for 10 minutes at 4500 rpm. 1.5 mL of the upper layer was filtered directly into the vial and injected onto the GC.

For determining the recovery, 6 QC samples were spiked with the PCB standard and had internal standard added and 3 QC blank samples were had the internal standard added only prior to the sample preparation with QuEChERS. The results were compiled and the recovery was calculated using the QC spiked and the QC blank samples.

10 soil samples were prepared to determine if there were PCBs present in the soil. The 10 samples had the internal standard added prior to the sample preparation with QuEChERS.

Results

The calibration curves for the PCB standards were prepared at 6 levels from 2.5 ppb up to 100 ppb. The system precision of the method was obtained by 10 consecutive injections of the PCB standard (20 ppb). The system precision results of the PCB compounds can be found in Table 3, along with the linearity results (R^2) obtained from the calibration curves.

For all PCB compounds an R^2 of 0.991 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 compounds the relative standard deviations (RSD%) ranged from 0.12% to 1.82%. This is a good precision for the method, since most acceptance criteria for PCB method validation require an RSD ≤15%.[3]

The recovery results (Table 3) indicate that none of the PCB compounds were detected in the QC blank soil sample. The recovery percentages for the QC spiked samples ranged from 76% to 150%, with repeatability (RSD%) values between 1.08% and 4.13% demonstrating good method efficiency and reliability.

Table 3 Summary of results

No.	PCB no.	Linearity	RSD (%) system precision	RSD (%) QC spiked	QC spiked (ppb)	Recovery (%)	Soil sample (ppb)
1	PCB-1	0.9946	1.09	2.27	64.38	128.76	n.d
2	PCB-3	0.9950	1.32	7.13	38.18	76.36	n.d
4	PCB-7	0.9985	0.12	1.08	58.83	117.66	n.d.
3	PCB-18	0.9915	0.41	1.92	71.56	143.12	n.d.
6	PCB-28	0.9947	0.69	2.66	74.61	149.22	n.d.
7	PCB-52	0.9932	0.33	1.62	67.16	134.32	n.d.
8	PCB-44	0.9945	0.30	1.53	58.32	116.64	n.d.
9	PCB-61	0.9951	0.32	2.58	74.36	148.72	n.d.
10	PCB-109	0.9945	1.82	4.13	59.16	118.32	n.d.
11	PCB-142	0.9936	1.64	4.01	60.61	121.22	n.d.
12	PCB-114	0.9943	1.55	2.91	65.92	131.84	n.d.
13	PCB-153	0.9938	1.59	3.40	61.28	122.56	n.d.
14	PCB-137	0.9948	1.52	2.86	46.76	93.52	n.d.
15	PCB-180	0.9958	1.26	2.42	60.33	120.66	n.d.

n.d. = not detected

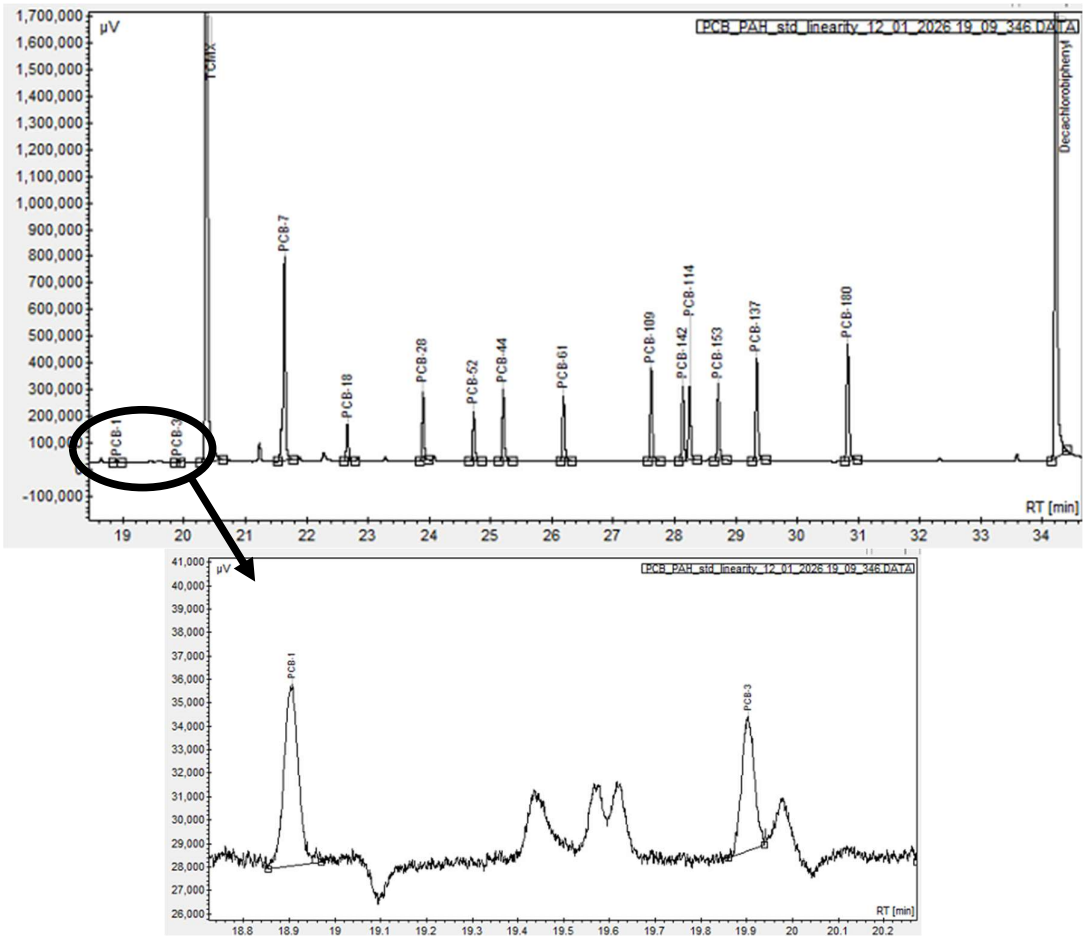

Figure 4 Example chromatogram of 10 ppb PCB standard + Internal Standard

No PCB compounds were found in the soil samples (Table 3).

The chromatogram shown in Figure 4 shows good separation of the PCBs and internal standards in CompassCDS. Validating a method is important because the responses for each compound can be vary. Like in this example the first two peaks (PCB-1 and PCB-3) gave a lower response but can still be positively identified as peaks. See Integration technical note for integrating peaks in CompassCDS.

Method validation

For thorough validation of the method, all samples were analyzed within a single sample sequence. The run began with three solvent blanks to confirm that the system was not contaminated. These were followed by the linearity samples and then the system precision (repeatability) samples.

After this, the 3 QC blank and 6 QC samples were analyzed, followed by the soil samples and bracketed by two additional QC samples. These QC samples ensure consistent system performance and verify the reliability of all previously analysed samples.

The final injection was a solvent blank to confirm that no carry-over occurred. To learn more about QC samples and method validation please visit our website.

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 soil for qualitative and quantitative analysis. Good system precision, linearity, results and recovery results are achieved for this application. The recovery was achieved with the spiked recovery sample, confirming good working of the analytical method.[2]

Samples were prepared using the original QuEChERS method (unbuffered) to prepare soil samples for GC analysis. For more information on using QuEChERS see our technical note on SCION's knowledge centre.

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

This method is also applicable on the SCION Instruments 8300/8500 GC – Mass Spectrometer (GC-MS) which is made for PCBs and PAHs in soil.

References

1. Polychlorinated Biphenyls (PCBs) | Biomonitoring California https://biomonitoring.ca.gov/chemicals/polychlorinated-biphenyls-pcbs (Accessed 22-01-2026)

2. National Primary Drinking Water Regulations | US EPA https://www.epa.gov/ground-water-and-drinking-water/national-primary-drinking-water-regulations (Accessed 22-01-2026)

3. Samia A., T. B. (2023, April 12). Development of the QuEChERS Extraction Method for the determination of polychlorinated biphenyls (aroclor 1254) in soil samples by using GC-MS. p. 11. (Accessed 21-01-2026)

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

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