Determination of PCBs and PAHs in soil using GC-MS

AN184 v1; SCION Instruments

Introduction

Polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) are among the most persistent and hazardous organic pollutants found in contaminated soils. PCBs were produced in large quantities between the 1930s and 1980s, typically used in electrical devices and coolant fluids. In 1986, an international agreement banned the use of most PCBs due to environmental concerns.[1] PAHs are primarily formed and released into the environment through the incomplete combustion of organic materials. This can occur from natural forest and grassland fires, volcanic activities and food preparation.

PCBs are formed by attaching one or more chlorine atoms to a pair of connected benzene rings, shown in Figure 1.[2] Depending on the number and position of the chlorine atoms attached to the biphenyl structure, 209 different PCBs can be formed.

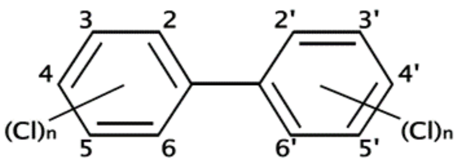

Figure 1 Polychlorinated Biphenyls chemical structure

PAHs consist of two or more fused aromatic benzene rings (Figure 2). They are non-polar, hydrophobic and chemically stable. The aromatic structure of PAHs makes them significantly stable. The toxicity of PAHs is due to their physical and chemical characteristics, such as high melting and boiling points and low vapor pressure.[2]

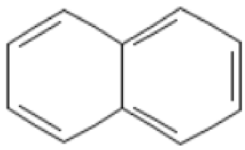

Figure 2 Naphthalene chemical structure

PCBs and PAHs persist in the environment for long periods of time and can travel through air, water and soil. They are associated with a range of health issues, liver damage, skin problems and have potential carcinogenic effects on both humans and animals.[1]

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 technique 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 to be suitable for injection onto the gas chromatograph (GC) for analysis. Figure 3 conveys how the soil sample looks before sample preparation and then after using QuEChERS method.

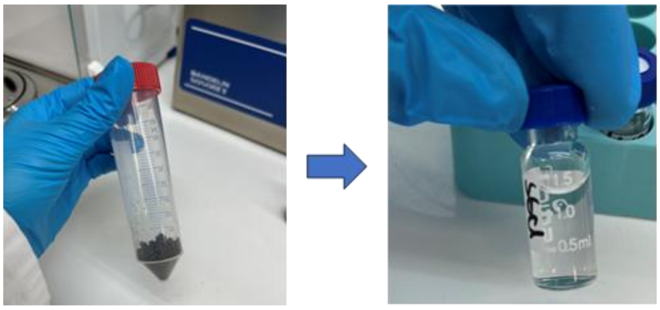

Figure 3 From soil sample to clear sample, suitable for GC analysis

Different countries have varying regulations for controlling the usage of PCBs and PAHs, due to concerns about the environment and health effects. Regulations aim to prevent and protect people from adverse effects, minimize releases and ensuring cleaner ecosystems. Regulations are put into place to accelerate the removal of PCBs and PAHs by implementing strict controls on their use, storage and disposal.

Testing for PCBs and PAHs is very important because it ensures compliance with disposal and cleaning regulations and to prevent leakages into the environment. Regular testing gives critical information about PCB and PAH levels in air, water and soil. Monitoring PCBs and PAHs 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 4) platform with an 8700 MS (SQ) and the SCION 8400PRO Autosampler. A SCION-5MS column is used for obtaining the best separation of the PCB and PAH compounds. Learn more about MS columns in MS vs Non-MS GC columns technical note.

MS is used for the identification of the target compounds, using the integrated NIST library search tool (Libraries are available for purchase separately).

Experimental

For this application a PCB/PAH standard and an internal standard (Table 2) were purchased for the qualification and the quantification of the unknown samples. The standard contains 29 different PCB and PAH 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 and MS (SQ)

GC Part	Settings
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	Helium 1 mL/min
Oven Program	50°C (hold 2.0 min), 8°C/min to 325°C (hold 3.63 min)
Run Time	40 min
Software	MSWS
MS Part	**Settings**
MS transfer line temp	275°C
Ion source temp	300°C
MS mode	Electron Ionization
Delay collection time	8.00 min
Scan mode	SIM mode

Figure 4 SCION Instruments 8300 & 8500-GC and 8700 MS and 8400PRO Autosampler

Sample preparation

Linearity samples were prepared in n-Hexane at 9 levels with concentrations ranging from 10 ppb to 400 ppb. The system precision was determined with multiple injections of L4 (60 ppb, n=9). Deca-CB and TCMX (50 ppb) were used as internal standards (IS) and added to all the standards and samples.

Table 2 Compounds and CAS numbers of the PCB/PAH standard and internal standard.

No.	PCB no.	Compound	CAS Number
1	-	Isophorone	78-59-1
2	-	Acenaphthylene	38444-84-7
3	PCB-1	2-Chlorobiphenyl	2051-60-7
4	PCB-3	4-Chlorobiphenyl	2051-62-9
5	-	Fluorene	86-73-7
6	-	TCMX (IS)	877-09-8
7	PCB-7	2,4'-Dichlorobiphenyl	34883-43-7
8	-	Hexachlorobenzene	118-74-1
9	PCB-18	2,2',5-Trichlorobiphenyl	37680-65-2
10	-	Phenanthrene	85-01-8
11	-	Anthracene	120-12-7
12	PCB-28	2,4,4'-Trichlorobiphenyl	7012-37-5
13	PCB-52	2,2',5,5'-Tetrachlorobiphenyl	35693-99-3
14	PCB-44	2,2',3,5'-Tetrachlorobiphenyl	41464-39-5
15	PCB-61	2,3',4',5-Tetrachlorobiphenyl	32598-11-1
16	-	Pyrene	129-00-0
17	PCB-109	2,3,3',4',6-Pentachlorobiphenyl	38380-03-9
18	PCB-142	2,2',3,4',5',6-Hexachlorobiphenyl	38380-04-0
19	PCB-114	2,3',4,4',5-Pentachlorobiphenyl	31508-00-6
20	PCB-153	2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1
21	PCB-137	2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2
22	-	Benz(a)anthracene	56-55-3
23	-	Chrysene	218-01-9
24	PCB-180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3
25	-	Benzo(b)fluoranthene	205-99-2
26	-	Benzo(k)fluoranthene	207-08-9
27	-	Decachlorobiphenyl (IS)	2051-24-3
28	-	Benzo[a]pyrene	50-32-8
29	-	Indeno(1,2,3-cd)pyrene	193-39-5
30	-	Dibenz(a,h)anthracene	53-70-3
31	-	Benzo(g,h,i)perylene	191-24-2

The original QuEChERS (unbuffered) method 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 then centrifuged for 10 minutes at 400 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 then centrifuged for 10 minutes at 4500 rpm. 1.5 mL of the upper layer was filtered directly into the vial and injected into the GC.

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

9 soil samples were prepared to determine if PCBs and PAHs were present in the soil. The 9 samples were spiked with internal standard prior to the sample preparation with QuEChERS.

There were two compounds present in the internal standard. Table 3 shows which IS was used for each compound as well as the quantifier and qualifier ions used.

Results

Due to the number of compounds present in this standard, not all compounds will be mentioned in the Results section. If necessary, the full validation report is accessible by request.

The calibration curves for the PCB/PAH standards were prepared at 9 levels from 10 ppb to 400 ppb. The system precision of the method was obtained by 9 consecutive injections of PCB/PAH standard (#4) (60 ppb).

The precision results of the selected PCB/PAH compounds can be found in Table 4, along with the linearity results (R^2) obtained from the calibration curves. For all PCB/PAH components an R^2 of 0.993 or higher was achieved, which is an excellent result, with many regulations requiring an R^2 value of ≥0.99.

Repeatability results show that the PCB/PAH compounds relative standard deviations (RSD%) are below 6%. This is a good precision for the method, since most acceptance criteria for PCB/PAH method validation require an RSD ≤15%.[3]

Table 3 Quantifier and qualifier ions used, IS used and scan time

No.	Quantifier Ion and Qualifier ions	IS used
1	54/82/138	TCMX
2	151/152/153	TCMX
3	152/153/188	TCMX
4	152/188/190	TCMX
5	165/166/167	TCMX
6	207/209/244	-
7	142/284/286	TCMX
8	152/222/224	TCMX
9	186/256/258	TCMX
10	152/176/178	TCMX
11	176/178/179	TCMX
12	186/256/258	TCMX
13	220/290/292	TCMX
14	220/290/292	TCMX
15	220/290/292	TCMX
16	200/202/203	Decachlorobiphenyl
17	324/326/328	Decachlorobiphenyl
18	290/360/362	Decachlorobiphenyl
19	324/326/328	Decachlorobiphenyl
20	145/360/362	Decachlorobiphenyl
21	290/360/362	Decachlorobiphenyl
22	226/228/229	Decachlorobiphenyl
23	226/228/229	Decachlorobiphenyl
24	324/394/396	Decachlorobiphenyl
25	250/252/253	Decachlorobiphenyl
26	250/252/253	Decachlorobiphenyl
27	178/214/498	-
28	250/252/253	Decachlorobiphenyl
29	138/276/274	Decachlorobiphenyl
30	139/276/278	Decachlorobiphenyl
31	138/274/276	Decachlorobiphenyl

Table 4 Summary of results – linearity and repeatability

No.	Compound	R^2	Repeatability (%RSD)
3	2-Chlorobiphenyl	0.9974	4.39
5	Fluorene	0.9976	4.93
16	Pyrene	0.9993	2.38
21	2,2',3,4,4',5'-Hexachlorobiphenyl	0.9984	1.86

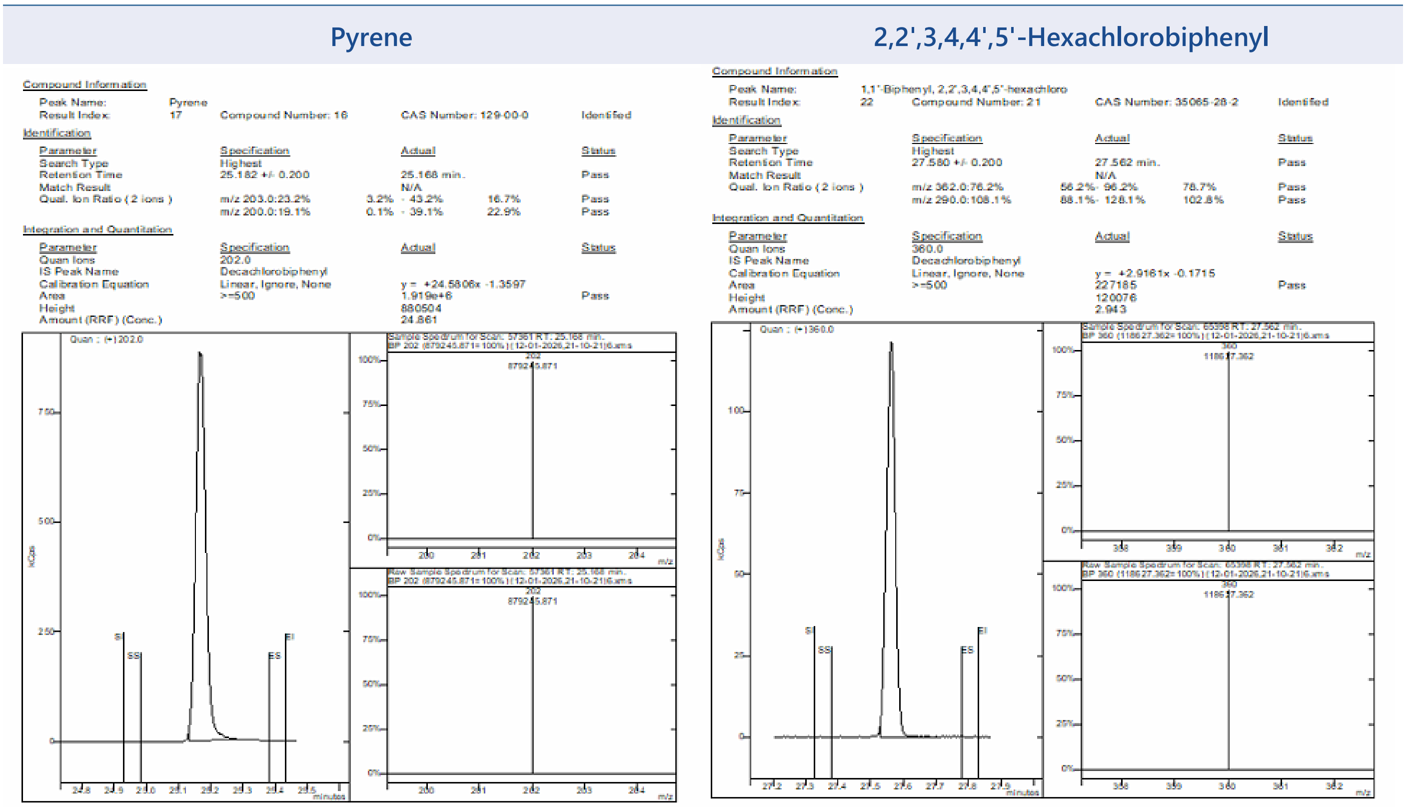

Figure 5 Example compounds chromatograms with corresponding spectra

Figure 5 shows an example chromatogram from MSWS of the peaks Pyrene and 2,2',3,4,4',5'-Hexachlorobiphenyl and their corresponding mass spectra.

The TIC of a SIM run shown in Figure 6 shows good separation of the PCB/PAH and internal standard compounds.

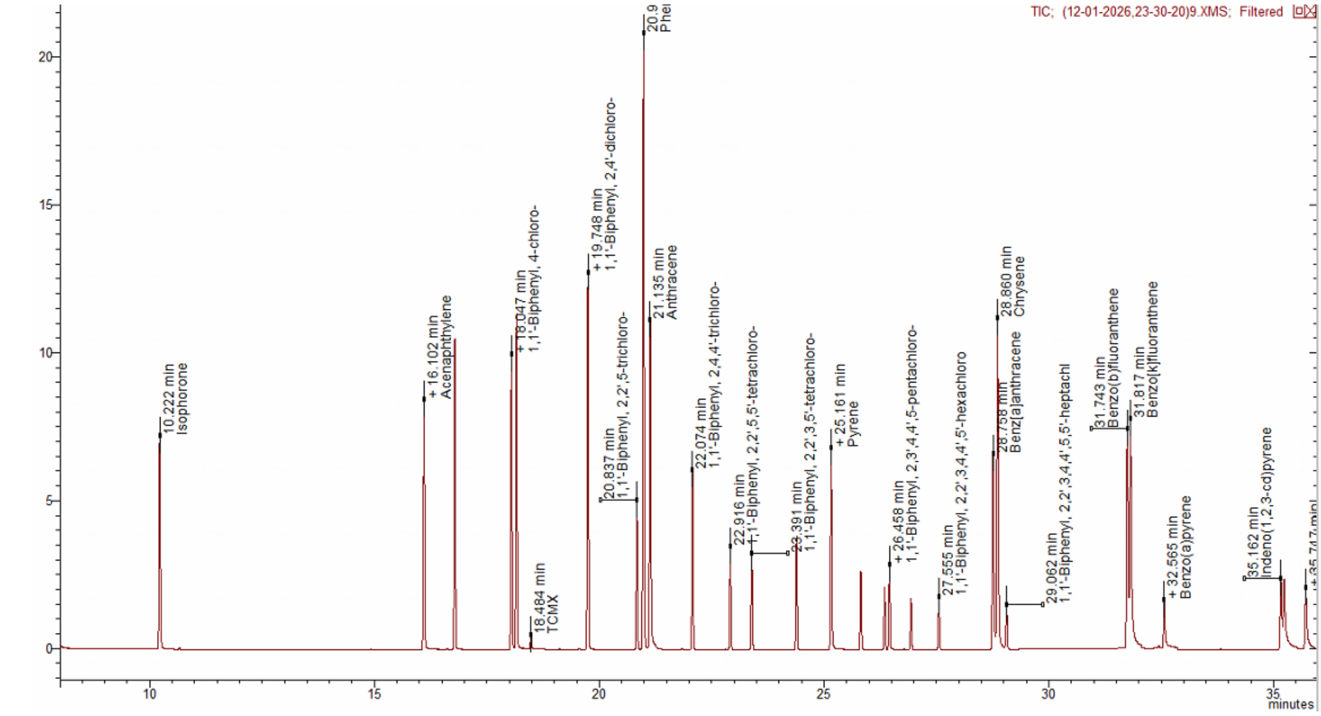

Figure 6 Total Ion Chromatogram (TIC)

Table 5 Summary of results – recovery

No.	Compound	Recovery (%)	RSD (%)
5	Fluorene	109.79	4.93
9	2,2',5-Trichlorobiphenyl	132.78	5.68
16	Pyrene	122.60	2.38
19	2,3',4,4',5-Pentachlorobiphenyl	97.74	1.90
31	Benzo(g,h,i)perylene	130.84	3.16

The recovery percentages (shown in Table 5) for the spiked QC samples ranged from 58% to 150%, with repeatability (RSD) values between 1.00% and 9.72% demonstrating good method efficiency and reliability.

No PCB compounds were found in the soil samples but several PAH compounds were detected throughout the samples. Table 6 lists all compounds detected and their relevant concentrations which were between 6 ppb and 202 ppb. Repeatability (RSD) was determined from the samples (which originated from the same soil source). These were found to be between 1.97% and 13.63%. Further proving the robustness of the method.

Table 6 Summary of Results – PAHs found

No.	Compound	Soil sample (ppb)	RSD (%)
2	Acenaphthylene	6.12	13.63
5	Fluorene	10.49	7.97
10	Phenanthrene	63.86	3.13
11	Anthracene	24.44	2.87
16	Pyrene	127.07	2.30
22	Benz(a)anthracene	144.59	1.94
23	Chrysene	133.16	3.77
25	Benzo(b)fluoranthene	201.78	2.00
26	Benzo(k)fluoranthene	87.71	3.04
28	Benzo[a]pyrene	173.96	1.97
29	Indeno(1,2,3-cd)pyrene	134.28	8.64
30	Dibenz(a,h)anthracene	43.60	3.00
31	Benzo(g,h,i)perylene	99.10	3.63

Figure 7 shown below shows a comparison from the 100 ppb standard (red) compared to the soil sample (green). A clear peak for pyrene was found in the soil sample. The two peaks beside pyrene are PCBs which were not found in the soil sample.

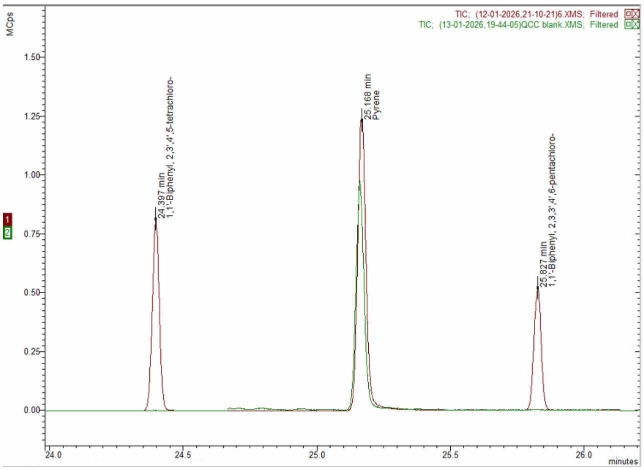

Figure 7 100 ppb std (red) vs soil sample (green)

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 analyzed 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 and 8700 MS and 8400PRO sampler is a perfect solution for analyzing PCBs and PAHs in soil for qualitative and quantitative analysis. Good system precision, linearity, results and recovery results were achieved for this application. A LOQ of 10 ppb was achieved and 50 ppb spiked sample was recovered, confirming good working of the method.

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 results were achieved with the SCION Instruments GC-MS set up and MSWS software. The SCION-5MS column shows good separation between the PAH and PCB compounds.

The analyzed soil samples is, according to multiple results obtained by this application, most likely from soil that has not been exposed to PCBs but it has been exposed to some PAHs.

This method is also applicable on the SCION Instruments 8300/8500 GC- platform with Electron Capture Detector (GC-ECD) which is made for PCBs in soil only.

References

1. Environment.ec.europa. (n.d.). Retrieved from Europian Commission: https://environment.ec.europa.eu/topics/waste-and-recycling/pcbspcts_en (Accessed 21-01-2026)

2. MDPI. (2025). Retrieved from Toxics: https://www.mdpi.com/2305-6304/13/3/151 (Accessed 21-01-2026)

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

Ordering Information for the 8300 GC	
Part	**Part Number**
8300 GC with 8700-MS-SQ EI Select, with S/SL inlet (120V)	SCIONSQ83SEL311
8300 GC with 8700-MS-SQ EI Select, with S/SL inlet (230V)	SCIONSQ83SEL312
8400 PRO Autosampler for 8300 GC and 8500 GC	840000001
MS WorkStation Software	394195791
NIST 20 MS Library and Search Program for MSWS	4121057
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

SCION offers other MS options such as the 8700 SQ Premium and 8900 TQ, as well as additional spectral libraries such as Designer Drugs and Wiley, please contact your local SCION sales representative to discuss your needs.

For more information, please contact:

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