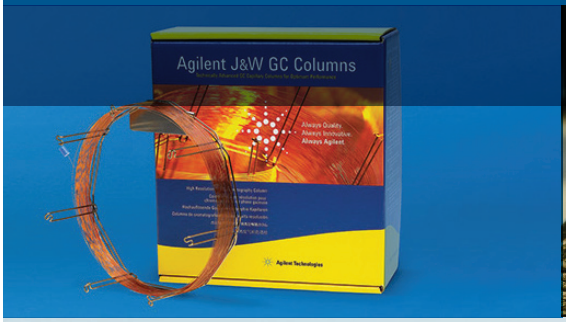
Polychlorinated Biphenyls (PCB) Analysis in Environmental Samples by GC/MS

Consumables Workflow Ordering Guide



Agilent

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For many years, PCBs were widely used as dielectrics in electrical apparatus and coolant fluids. However, when they were identified as environmentally toxic, PCBs were classified as persistent organic pollutants and their production has been banned worldwide since early 1970s. Despite being banned for decades, they are still found in soils, surface waters and sediments, due to their ability to accumulate and persist in aqueous environmental matrices for many years.

Several regulations have been put in place to monitor PCBs in environmental matrices.

US Regulations

In the US, EPA 525.1 (drinking and raw source water), EPA 8082A (solid, tissue, and aqueous matrix), EPA 1628 (Water, Soil, Sediment, Biosolids, and Tissue).

EPA 1628 and EPA 525.1 recommend a 30 m x 0.25 mm id fused silica capillary column coated with a 0.25 µm bonded film of polyphenylmethylsilicone (J&W DB-5 or equivalent).

EPA 8082A method describes procedures for both single-column and dual-column analyses using a DB-5 or equivalent) or fused-silica capillary column chemically bonded with 14% cyanopropylmethylpoly-siloxane (DB-1701, or equivalent). The dual-column approach generally employs a single injection that is split between two columns that are mounted in a single gas chromatograph.

EU regulations

In the European Union (EU), PCB measurement in sediments and biota is recognized as a cost-effective water quality monitoring approach. It describes the general contamination status, supplies reference values for local and regional monitoring, and identifies areas of concern where extra monitoring effort is needed.[1-3] The analysis of PCBs in soils generally follows liquid extraction and clean-up procedures. While the methodology and analytical set up may vary from country to country, the central role of the seven EU PCBs (PCB28, PCB52, PCB101, PCB118, PCB138, PCB153, and PCB180) in monitoring is clear and unequivocal. These are not selected because of their specific toxicity but are to provide an indication of the distribution of the congeners per degree of chlorine substitution. Interfering PCBs that elute close or co-elute may hamper quantification. This is particularly the case for the pair 28/31, for which a resolution minimum of 0.5 is set in methods such as EN 17322. This method describes procedures for single column GC/MS analysis using a 5% phenyl-methyl liquid phase (DB-5ms) or equivalent. The dual column approach for GC/ECD advises a 14% cyanopropylmethylpoly-siloxane phase (DB-1701, VF-1701ms) for the confirmatory column. In some cases, the DB-XLB is used for its 28/31 separation potential.

China regulations

In China, HJ 743-2015 (Soil and sediment), HJ 715-2014 (Water quality), HJ 891-2017 Solid Waste). China HJ method analyzes for 18 critical PCBs in the environment. Among the 18 targeted PCB congeners, six congeners are indicator PCBs, and 12 are coplanar PCBs. Coplanar PCBs are dioxin-like PCBs with high environmental toxicity.

While both GC electron capture detector (ECD) and GC mass selective detector (MSD) have been used for PCB analysis, the GC/MSD based method enables the use of retention time (RT) and characteristic ions of each PCB for congener identification. The selective monitoring of MSD on targeted ions ensures fewer false positive identifications compared to the GC ECD analysis approach, especially under the interference of heavy matrix.

Agilent 8890 GC/5977B GC/MSD/7650A.

HJ 743-2015 recommends a 5% phenyl 95% dimethylpolysiloxane type stationary phase for the 18 PCB analysis. The Agilent HP-5ms column is coated with this type of polymer. Meanwhile, the Agilent DB-5ms column is coated with 5% phenyl 95% dimethyl arylene siloxane and has similar selectivity as the HP-5ms column. Both HP-5ms and DB-5ms columns were tested for their resolution of the targeted analytes (see Table 1b for Method parameters). The chromatograms of the 18 PCB calibration standards on DB-5ms column showed better resolution on PCB123 and PCB118 (Figure 1).[4]

The Agilent J&W DB-XLB has exceptionally low bleed and low polarity and provides good separation EU PCB pair 28/31. Pressed for time and productivity, labs might opt for a DB-5ms analysis first with subsequent confirmation using the DB-XLB when the levels are close to regulation limits or exceed them.

Table 1a. Instrument consumables.

Consumables used for application development	
Column	Agilent DB-5ms, 30 m x 0.25 mm, 0.25 µm (p/n 122-5532)
Liner	Agilent Ultra Inert, Splitless liner with glass wool (5190-2293)
Septum	Agilent bleed optimized, nonstick 11 mm septa (p/n 5183-4757)
Syringe	Agilent ALS syringe, 0 µL tapered, fixed needle (p/n 5181-3354)
Vials	Agilent A-line certificated 2 mL amber screw top vials (p/n 5182-0716)
Caps	Caps Agilent screw cap, blue, certified, PTFE/silicone/PTFE septa (p/n 5182-0723)

Table 1b. Instrument configuration and analytical parameters.

Agilent 8890 GC/5977B GC/MSD instrument parameters	
Autosampler	Agilent 7650A automatic liquid sampler
Split/Splitless Inlet Mode	Splitless
Inlet Temperature	280 °C
Purge Flow	60 mL/min
Purge Time	0.75 min
Carrier Gas	Helium
Column Flow Rate	1.2 mL/min, constant flow
Oven Ramp Program	40 °C, hold 0 minutes 20 °C/min to 230 °C, hold 0 minutes 5 °C/min to 260 °C, hold 1.5 minutes 20 °C/min to 310 °C, hold 2 minutes
Transfer Line Temperature	300 °C
Extraction Ion Source Temperature	250 °C
MS Quad Temperature	150 °C
Acquisition Type	SIM
EMV Mode	Gain factor
Gain Factor	0.5

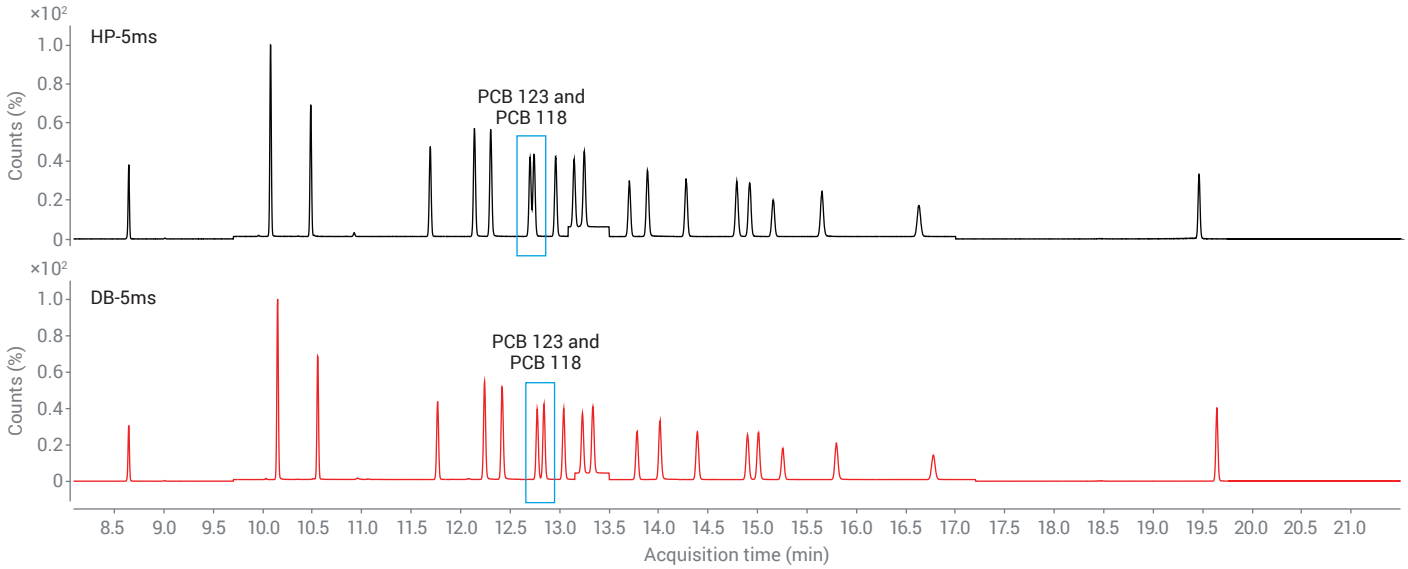

Figure 1. Chromatograms of 18 PCBs analyzed on Agilent HP-5ms and DB-5ms columns showed better resolution of PCB123 and PCB118. See Table 1 for Instrument configuration, consumables and analytical parameters.

Sample Preparation of Soil Samples for PCB analysis

Accelerated solvent extraction and solid phase extraction are generally required for extraction and cleanup of PCBs in soil. These are tedious and time-consuming procedures. An Agilent optimized extraction and cleanup procedure uses the dispersive QuEChERS kits for analyzing PCBs in soil.[5]

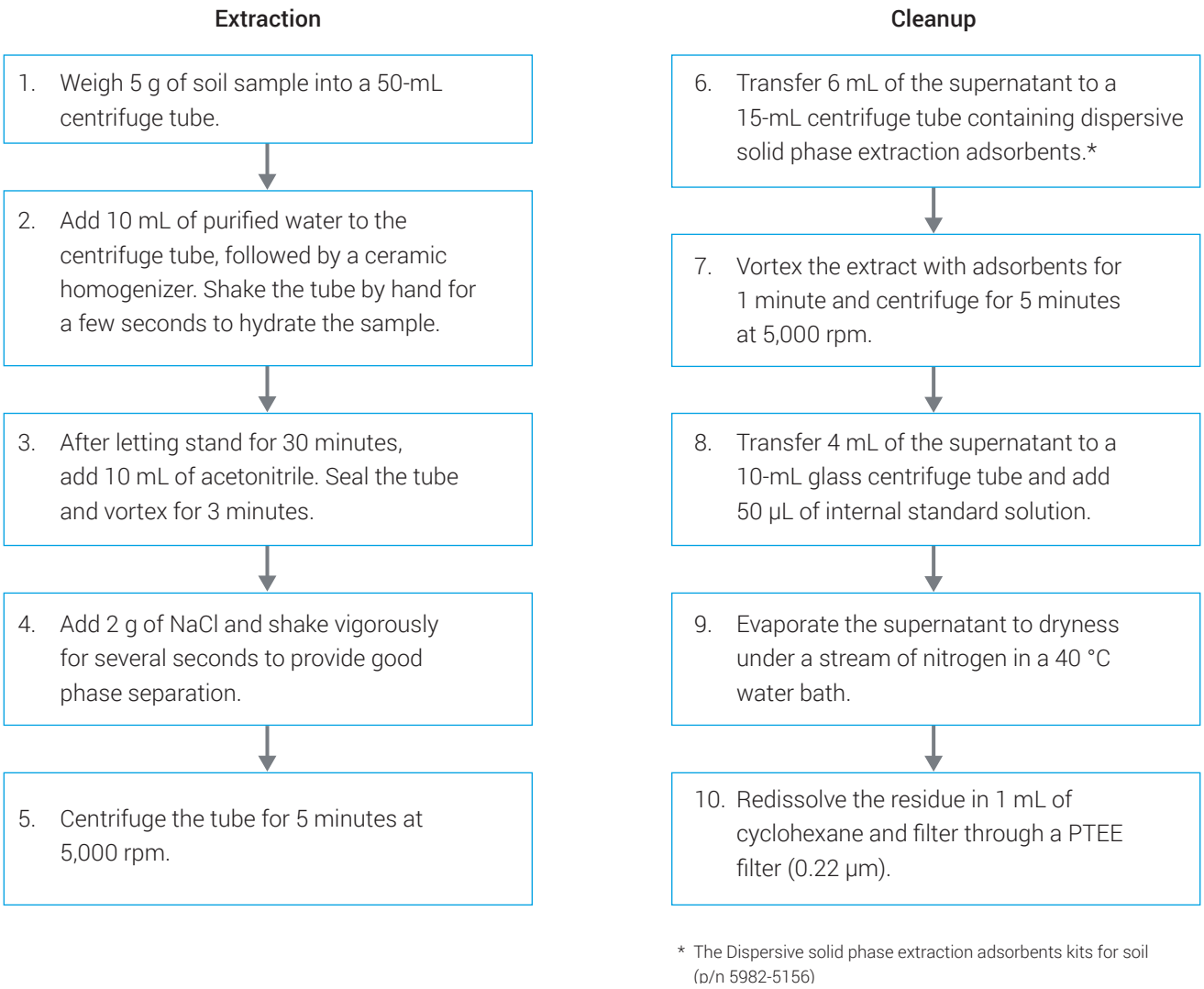

Figure 2. Optimized extraction and cleanup procedure.

References

1. Guidance for sediment and biota monitoring under the common implementation strategy for the water framework directive. Trends in Analytical Chemistry, Vol 36, 2012.
2. ISO 13876:2013; Soil quality - Determination of polychlorinated biphenyls (PCB) by gas chromatography with mass selective detection (GC/MS) and gas chromatography with electron-capture detection (GC-ECD)
3. EN17322:2020; Environmental Solid Matrices - Determination of polychlorinated biphenyls (PCB) by gas chromatography - mass selective detection (GC/MS) or electron-capture detection (GC-ECD)
4. Analysis of Polychlorinated Biphenyls on the Agilent 8890 GC/5977B GC/MSD by Following the China HJ 743-2015 Method 5994-1464EN
5. Determination of Selected Polychlorinated Biphenyls in Soil Using QuEChERS-based Method and Gas Chromatography Tandem Mass Spectrometry 5991-6980EN

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Description	Part No.
MyList 1: GC columns for PCB analysis	
Agilent J&W DB-5ms Ultra Inert, 30 m x 0.25 mm id x 0.25 µm (recommended)*	122-5532UI
Agilent J&W DB-1701, 30 m x 0.25 mm id x 0.25 µm (for dual column setup EPA 8082A)	122-0731
Agilent J&W VF-1701 ms, 30 m, 0.25 mm id, 0.25 µm (Confirmatory columns for EN17322 and ISO 13876)	CP9151
Agilent J&W DB-XLB GC Column, 30 m, 0.25 mm, 0.50 µm, 7-inch cage (confirmatory column for 28/31 pair)	122-1236
MyList 2: GC Supplies	
Agilent Ultra Inert, splitless liner with glass wool, 1/pk	5190-2293
Inlet septa, Advanced Green, nonstick 1 mm, 50/pk	5183-4759
Agilent bleed temperature optimized, nonstick 11 mm septa, 50/pk	5183-4757
15%Graphite/85% Vespel Ferrules, 0.4 mm id, 10/pk	5181-3323
Ultra Inert gold seal, with washer, 1/pk	5190-6144
Ultra Inert gold seal, with washer, 10/pk	5190-6145
Self-Tightening column nut, collared, inlet	G3440-81011
Replacement collar for self-tightening nut	G3440-81012
Agilent ALS syringe, 10 µL tapered, fixed needle	5181-3354
MyList 3: Vials and Caps	
Agilent A-Line certificated 2 mL amber screw top vials, 100/pk	5182-0716
2 mL screw top amber, write-on spot, deactivated, certified, 100/pk	5183-2072
Screw Caps, blue, certified, PTFE/silicone/PTFE septa, 100/pk	5182-0723

Description	Part No.
MyList 4: Sample Preparation (Soil Samples)	
QuEChERS Dispersive Solid Phase Extraction Kit, 50/pk	5982-5156
QuEChERS Dispersive Solid Phase Extraction Kit, 50/pk	5982-5156CH
MyList 5: Standards	
7 analytes, PCB calibration standard, 10 µg/mL, isooctane, 1 mL	RPCM-200-1
14 analytes PCB calibration standard, 10 µg/mL, isooctane, 1 mL	RPCM-210-1
12 analytes, WHO standard, 10 µg/mL, isooctane, 1 mL	RPCM-220-1
18 analytes, ISS PCB standard, 10 µg/mL, isooctane, 1 mL	RPCM-230-1
32 analytes WHO/ISS PCB calibration mixture, 10 µg/mL, isooctane, 1 mL	RPCM-240-1
19 analytes, PCB congener standards for EPA method 8082, 100 µg/mL, isooctane	RPCM-8082-1
20 analytes, PCB congener calibration check at 0.2 µg/mL respectively, 0.2 µg/mL, isooctane	RPC-EPA-1
20 analytes, PCB congener calibration checks at 100 µg/mL respectively, 100 µg/mL, acetone	RPC-EPA2-1
41 analyte, PCB congener mix, various concentrations, isooctane	RPCM-245-1
Aroclor 1260, 100 µg/mL, hexane	PP-361-1
Aroclor 1254, 100 µg/mL, hexane	PP-351-1
Aroclor 1242, 100 µg/mL, hexane	PP-311-1
Aroclor 1248, 100 µg/mL, hexane	PP-341-1
Aroclor 1016, 100 µg/mL, hexane	PP-281-1
Aroclor 1232, 100 µg/mL, hexane	PP-301-1
Aroclor 1221, 100 µg/mL, hexane	PP-291-1
Aroclor 1262, 100 µg/mL, hexane	PP-371-1
Aroclor 1268, 100 µg/mL, hexane	PP-381-1
9 analyte, concentration calibration, various concentrations, hexane	CB-681MN-1
2 analytes, Method 8082A calibration mix, 1000 µg/mL, isooctane	PPM-8082-1
8 analytes, Method 525.1 PCB mixture, 500 µg/mL, acetone	RPCM-525A-1
8 analyte, Method 525.1 PCB mixture, 100 µg/mL, acetone	RPCM-525-1
Singles, 100 µg/mL, isooctane	RPC-(prefix)**
Internal & Surrogates	
Decachlorobiphenyl, 1,000 µg/mL, toluene	PPS-150-1
2,4,5,6-tetrachloro-m-xylene, 2,000 µg/mL, acetone	IST-440-1

* *The Ultra Inert column is recommended over DB-5 (122-5032) or DB-5ms (122-5532) for best analytical results. Surface inertness prevents analyte breakdown, response loss and peak shape distortion delivering excellent consistency, stability, and durability over multiple sample injections.*

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