

GC/ECD Analysis of Organochlorine Pesticides or Polychlorinated Biphenyls

Using a Low-Bleed Rtx®-XLB Column and Restek Reference Materials

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- ✓ Rtx®-XLB column shows extremely low bleed and excellent inertness, improving sensitivity for active compounds.
- ✓ 20 common organochlorine pesticides in 3 convenient reference concentrations.
- ✓ 19 US EPA Method 8082A PCB congeners in one solution.

Various methods have provided guidelines for GC/electron capture detection (GC/ECD) analysis of organochlorine pesticides and PCBs in aqueous and soil matrices. Pesticides and PCB congeners now are analyzed by separate methods, to ensure more accurate PCB data and eliminate complications that arise in combined analysis. Analyses of individual PCB congeners greatly simplify quantitative studies, and improve data, relative to the difficult quantitative studies of PCBs as mixtures (e.g., Aroclor® mixtures)—especially with mixtures weathered by long exposure in the environment.

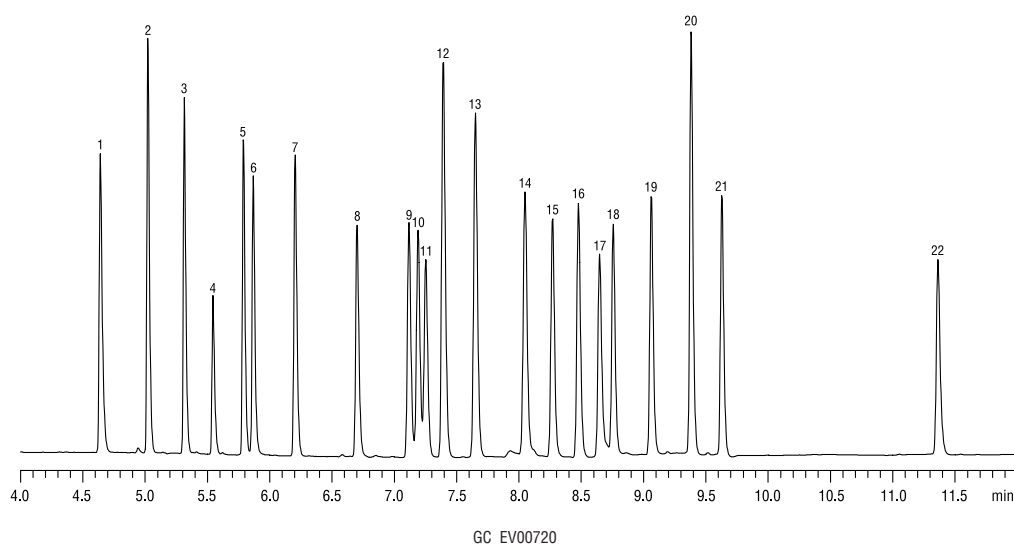
GC analysis of organochlorine pesticides and PCBs can be very challenging because of lengthy calibrations, linearity problems, and potential breakdown of some of the pesticides. In addition to adequate resolution of the target analytes, the column for this analysis must exhibit low bleed. A wide-bore (0.53mm ID) column is listed in US EPA Methods 8081A and 8082A for organochlorine pesticides,

but a narrow-bore column may be used in single-column analyses. Our new 0.32mm ID, 0.5µm phase Rtx®-XLB column is ideal for analyses of active compounds, due to improvements in polymer synthesis and tubing deactivation. Figure 1, an analyses of 20 organochlorine pesticides (Organochlorine Pesticide Mix AB #2, cat.# 32292), demonstrates the superior efficiency and low bleed characteristic of the new column, even at 330°C. The column, in combination with a high initial temperature, 120°C, reduced analysis time to 11.5 minutes, with excellent separation. Very low bleed and high thermal stability ensure reliable detection at the 80/160/800 ppb level. The very low bleed also minimizes detector contamination, prolonging intervals between cleanings and thus increasing throughput over time. Note that to minimize breakdown of labile pesticides we minimized sample contact with metal surfaces by using a Drilled Uniliner® inlet liner to convey the sample directly onto the column.

Restek chemists carefully reviewed EPA Methods 8080 and 8081A, then developed three calibration mixes that include 20 most often monitored organochlorine pesticides. The mix used to obtain Figure 1 has varied concentrations of the target analytes, from 8 to 80µg/mL, because these pesticides exhibit significantly differing responses.* The other two mixes include the 20 analytes at a single concentration, 200µg/mL or 2000µg/mL. The 2000µg/mL concentration often is more practical than lower concentrations, especially if several mixes must be combined. We also offer all surrogates and internal standards currently required for these analyses.

PCBs are persistent in the environment, and accurately determining their presence and concentrations is very important. A common question is whether such analyses should be focused on mixtures of PCBs (e.g., Aroclor® mixes) or on individual congeners. Congener-specific analyses have important advantages over analyses of mixtures: generally, congener analyses offer lower detection limits and greater information content. In addition, compositions of weathered, degraded, and metabolized PCB mixtures can be measured and interpreted more easily. Also, it is easier to detect interferences caused by other chemicals, and quantification of individual congeners is more accurate. However, coelution of analytes is a problem in a PCB congener analysis, so a strong quality assurance program and reliable reference materials are needed by the analyst. To facilitate congener-specific analyses, we now make a reference mix of 19 PCB congeners at 100µg/mL each in isoctane, suitable for EPA Method 8082A. Depending on regulatory and project requirements,

Figure 1 Organochlorine pesticides separated in less than 12 minutes, using an Rtx®-XLB column.

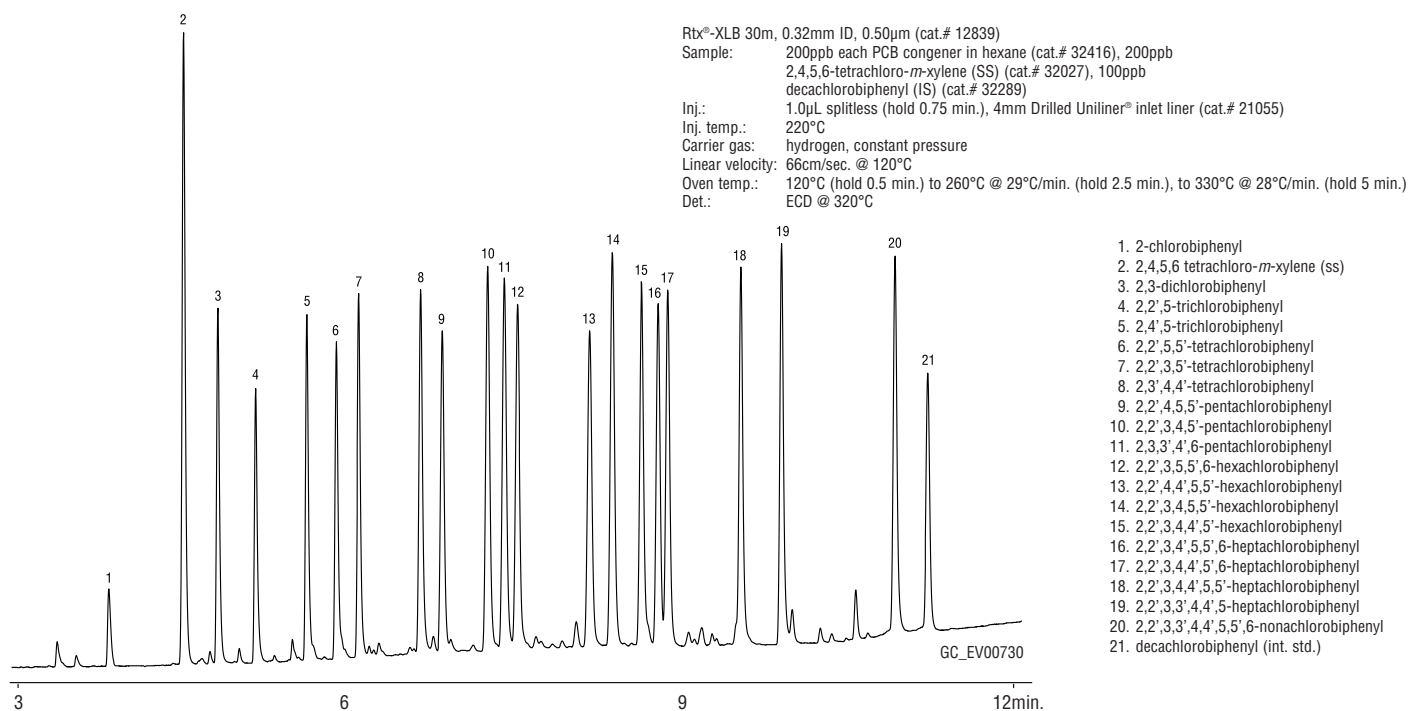


1. 2,4,5,6-tetrachloro-*m*-xylene (ss)
2. α -BHC
3. γ -BHC
4. β -BHC
5. δ -BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ -chlordane
10. α -chlordane
11. endosulfan I
12. 4,4'-DDE
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. endrin aldehyde
18. 4,4'-DDT
19. endosulfan sulfate
20. methoxychlor
21. endrin ketone
22. decachlorobiphenyl (ss)

GC_EV00720
Rtx®-XLB 30m, 0.32 ID, 0.5µm (cat.# 12839)
Sample: Organochlorine Pesticide Mix AB (cat.# 32292) 80/160/800ppb in hexane
2,4,5,6-tetrachloro-*m*-xylene (cat.# 32027) surrogate, 80ppb
decachlorobiphenyl (cat.# 32029) surrogate, 160ppb
Inj.: 1.0µL splitless (0.75 min. hold), 4mm Drilled Uniliner® inlet liner (cat.# 21055)
Inj. temp.: 220°C
Carrier gas: hydrogen, constant pressure
Linear velocity: 60cm/sec. @ 120°C
Oven temp.: 120°C (hold 0.5 min.) to 260°C @ 29°C/min. (hold 2.5 min.), to 330°C @ 28°C/min. (hold 3 min.)
Det.: ECD @ 320°C

*For mix composition, see page 8 of this Advantage.

Figure 2 19 PCB congeners separated in less than 12 minutes, using an Rtx®-XLB column.



the mix can be used for reporting either PCB congener results or total PCBs. Decachlorobiphenyl and tetrachloro-*m*-xylene are appropriate as internal standard and surrogate standard, respectively. The PCB congener standard is a very useful addition to our group of Aroclor® reference mixes.

Figure 2 is a GC/ECD analysis of the 19 PCB congeners, with the internal and surrogate standards. To simplify the work of analysts who monitor both pesticides and PCBs, we used the same 30m, 0.32mm

ID, 0.5µm Rtx®-XLB column and the same conditions in both analyses: the conditions used to obtain Figure 2 are optimal for very rapid analysis (11.5 min.) of the Method 8082A PCB congeners, as was true for the pesticides.

If you are performing analyses of organochlorine pesticides and/or PCBs, an Rtx-XLB® column and Restek reference materials will save time, help simplify your analysis, improve the quality of your data, and increase your productivity.

Rtx®-XLB Columns (fused silica)

(proprietary low-polarity phase)

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.25mm	0.10	30 to 340/360°C		12808	
	0.25	30 to 340/360°C	12820	12823	12826
	0.50	30 to 340/360°C		12838	
0.32mm	1.00	30 to 340/360°C	12850	12853	
	0.10	30 to 340/360°C		12809	
	0.25	30 to 340/360°C	12821	12824	12827
0.53mm	0.50	30 to 340/360°C		12839	
	1.00	30 to 340/360°C		12854	
	1.50	30 to 340/360°C	12867	12870	

ID	df (µm)	temp. limits	12-Meter	20-Meter	25-Meter
0.18mm	0.18	30 to 340/360°C		42802	
0.20mm	0.33	30 to 340/360°C	42815		42820

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Additional reference mixes listed on page 8.

For more **info** on the Rtx®-XLB column, request lit. cat.# 59957.

Suitable for **European Methods or ASTM D-4059-96**

PCB Congener Mix, Method 8082A

- 2-chlorobiphenyl (BZ #1)
- 2,3-dichlorobiphenyl (BZ #5)
- 2,2',5-trichlorobiphenyl (BZ #18)
- 2,4',5'-trichlorobiphenyl (BZ #31)
- 2,2',3,5'-tetrachlorobiphenyl (BZ #44)
- 2,2',5,5'-tetrachlorobiphenyl (BZ #52)
- 2,3',4,4'-tetrachlorobiphenyl (BZ #66)
- 2,2',3,4,5'-pentachlorobiphenyl (BZ #87)
- 2,2',4,5,5'-pentachlorobiphenyl (BZ #101)
- 2,3,3',4',6-pentachlorobiphenyl (BZ #110)
- 2,2',3,4,4',5'-hexachlorobiphenyl (BZ #138)
- 2,2',3,4,5,5'-hexachlorobiphenyl (BZ #141)
- 2,2',3,5,5',6-hexachlorobiphenyl (BZ #151)
- 2,2',4,4',5,5'-hexachlorobiphenyl (BZ #153)
- 2,2',3,3',4,4',5'-heptachlorobiphenyl (BZ #170)
- 2,2',3,4,4',5,5'-heptachlorobiphenyl (BZ #180)
- 2,2',3,4,4',5',6-heptachlorobiphenyl (BZ #183)
- 2,2',3,4',5,5',6-heptachlorobiphenyl (BZ #187)
- 2,2',3,3',4,4',5,5',6-nonachlorobiphenyl (BZ #206)

Each	5-pk.	10-pk.
32416	32416-510	—
w/data pack		
32416-500	32416-520	32516

PCB Congener Standard #1

- 2,4,4'-trichlorobiphenyl (BZ #28)
- 2,2',5,5'-tetrachlorobiphenyl (BZ #52)
- 2,2',4,5,5'-pentachlorobiphenyl (BZ #101)
- 2,2',3,4,4',5'-hexachlorobiphenyl (BZ #138)
- 2,2',4,4',5,5'-hexachlorobiphenyl (BZ #153)
- 2,2',3,4,4',5,5'-heptachlorobiphenyl (BZ #180)

Each	5-pk.	10-pk.
32290	32290-510	—
w/data pack		
32290-500	32290-520	32390

PCB Congener Standard #2

- BZ #28, BZ #52, BZ #101, BZ #138, BZ #153, BZ #180, plus 2,3',4,4',5-pentachlorobiphenyl (BZ #118)

Each	5-pk.	10-pk.
32294	32294-510	—
w/data pack		
32294-500	32294-520	32394

GC/ECD Analysis of Organochlorine Pesticides or Polychlorinated Biphenyls

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8140/8141 Internal & Surrogate Standards

1,000µg/mL in acetone, 1mL/ampul

Each	5-pk.	10-pk.
Internal Standard: 1-bromo-2-nitrobenzene		
32279	32279-510	—
w/ data pack		
32279-500	32279-520	32379
Surrogate: 4-chloro-3-nitrobenzotrifluoride		
32282	32282-510	—
w/ data pack		
32282-500	32282-520	32382

2,4,5,6-Tetrachloro-*m*-xylene

Each	5-pk.	10-pk.
200µg/mL in acetone, 1mL/ampul		
32027	32027-510	—
w/ data pack		
32027-500	32027-520	32127
200µg/mL in acetone, 5mL/ampul		
32028	32028-510	—
w/ data pack		
32028-500	32028-520	32128

Decachlorobiphenyl (BZ #209)

Each	5-pk.	10-pk.
10µg/mL in isooctane, 1mL/ampul		
32289	32289-510	—
w/ data pack		
32289-500	32289-520	32389
200µg/mL in acetone, 1mL/ampul		
32029	32029-510	—
w/ data pack		
32029-500	32029-520	32129
200µg/mL in acetone, 5mL/ampul		
32030	32030-510	—
w/ data pack		
32030-500	32030-520	32130

508.1 Internal Standard

pentachloronitrobenzene

100µg/mL in ethyl acetate, 1mL/ampul

Each	5-pk.	10-pk.
32091	32091-510	—
w/ data pack		
32091-500	32091-520	32191

Organochlorine Pesticide Mix AB #2

aldrin	8µg/mL	dieldrin	16
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (isomer B)	8
4,4'-DDT	16	methoxychlor	80

In hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32292	32292-510	—
w/ data pack		
32292-500	32292-520	32392

Organochlorine Pesticide Mix AB #1

20 compounds listed for cat.# 32292 above

200µg/mL each in hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32291	32291-510	—
w/ data pack		
32291-500	32291-520	32391

Organochlorine Pesticide Mix AB #3

20 compounds listed for cat.# 32292 above

2,000µg/mL each in hexane:toluene (1:1), 1mL/ampul

Each	5-pk.	10-pk.
32415	32415-510	—
w/ data pack		
32415-500	32415-520	32515