

Polychlorinated Biphenyl (PCB) Analysis on Fast and Standard HT8

Polychlorinated biphenyl (PCB) compounds have been found to have serious health implications and be extremely persistent in the environment. As a result, the 209 possible congeners are the subject of strict monitoring procedures within environmental regulating bodies around the world. Monitoring of soil, water and food stuffs for PCBs is commonplace in environmental laboratories.

Figure 1: PCB standard.

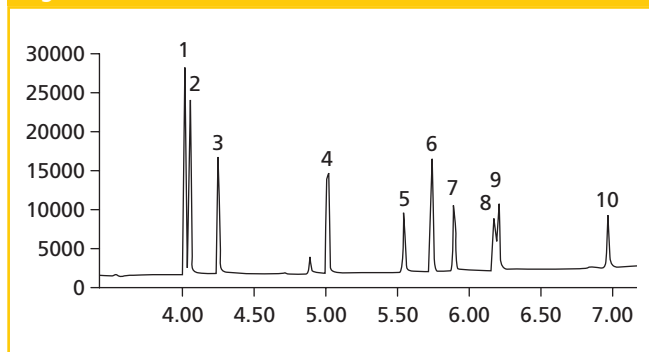
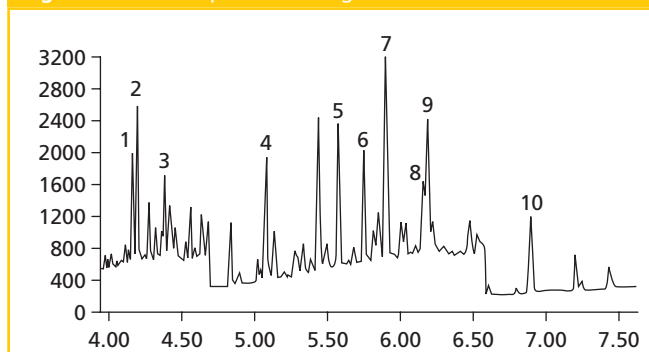


Figure 2: Real sample containing PCBs.



Phase: HT8 0.1 μm Constant flow: On
 Column: 10 m \times 0.1 mm i.d. Average linear velocity: 50 cm/sec
 Initial temp: 90 $^{\circ}\text{C}$, 0.5 min Injection mode: Split
 Rate 1: 35 $^{\circ}\text{C}/\text{min}$ to 210 $^{\circ}\text{C}$ Injection volume: 0.1 μL
 Rate 2: 15 $^{\circ}\text{C}/\text{min}$ to 290 $^{\circ}\text{C}$ Injection temperature: 250 $^{\circ}\text{C}$
 Final temp: 290 $^{\circ}\text{C}$, 5 min Autosampler: Yes
 Detector type: MSD Column part no: 054690
 Carrier gas: He, 54.2 psi
 Carrier gas flow: 0.6 mL/min

The analysis for PCBs in environmental samples is predominately run on a GC-ECD or GC-MS system using a 5% phenyl phase or an SGE HT8 carborane phase. The HT8 phase provides good selectivity for the seven indicator congeners (IUPAC No.'s 28, 52, 101, 118, 153, 138 and 180) and separates them from most of the potential co-eluting congeners. The analysis is usually performed with a 0.25 mm i.d. capillary column and takes between 20 and 60 minutes, depending on the length of the column. The analysis time for PCB analyses can be reduced considerably by using a shorter capillary column with a smaller i.d. These fast columns still maintain excellent separation of the various components while increasing the number of analyses performed; this is beneficial for laboratories with a high sample throughput. SGE produces FAST GC columns (10 m \times 0.1 mm i.d.) with a variety of phases, including HT8. The HT8 FAST PCB column has been successfully used in some German laboratories and is written into an addendum of the relevant DIN Norm (Germany's equivalent to EPA methods) as a good screening capillary column using MS detection.

Recent test results from a Swiss laboratory using the fast HT8 column are shown in Figure 1. The chromatogram of the test mix shows the superb separation of some of the critical pairs, for

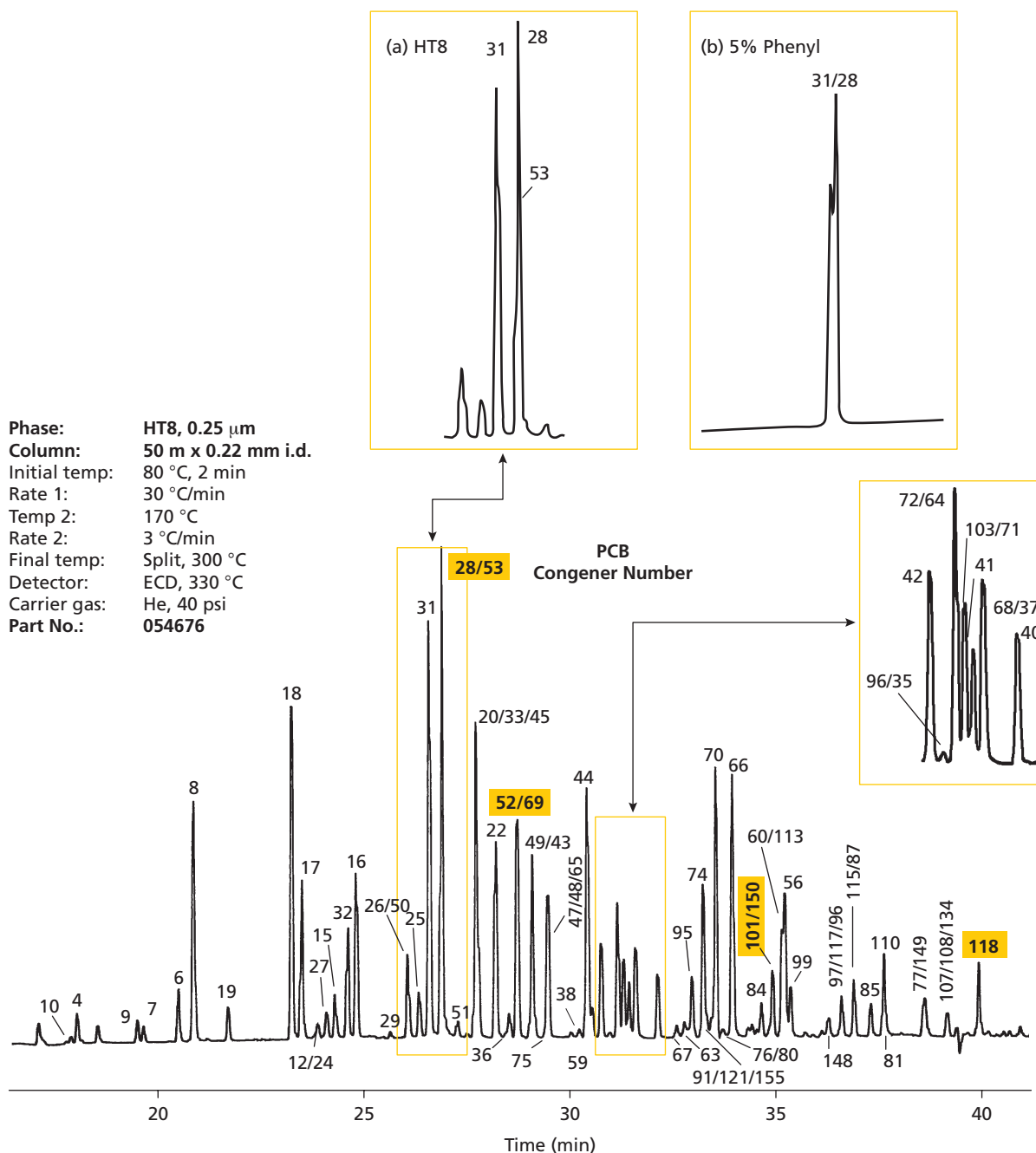
Table 1: Retention times of PCB analytes.

Target Compounds	Retention Time (min)	(QIon)
1) PCB 31	4.12	256
2) PCB 28	4.15	256
3) PCB 52	4.34	292
4) PCB 101	5.06	326
5) PCB 149	5.57	360
6) PCB 118	5.75	326
7) PCB 153	5.90	360
8) PCB 163	6.17	360
9) PCB 138	6.20	360
10) PCB 180	6.93	394

example congeners 31/28 (ca. 80%) and 163/138 (ca. 50%). This is an excellent separation in a total analysis time of less than 10 minutes. Figure 2 shows the chromatogram of an extract, which contains PCBs and again the indicator congeners are visible and identifiable (Table 1). Quantification is also possible if, the matrix background is not too high, as in this example. These encouraging results show that the FAST PCB capillary column is an excellent choice for the screening of samples which may contain PCBs, and if the extract is clean, it can also be used for quantification of the major congeners.

The Aroclor samples are a mixture of specific PCB congeners, which were used for a variety of purposes. Applications such as heat transfer fluids, dielectrics (insulators), in transformer cooling liquids, heat-transfer and hydraulic fluids, vacuum pump fluids, lubricants, plasticizers, fillers in investment casting waxes, surface coatings and sealants, pesticide extenders, and copy paper were commonplace prior to 1970 before their effect on public health came to light. The use of Aroclor mixtures is very restricted and in some countries banned from use.

Figure 3: Analysis of Aroclor 1242 on HT8.



The standard columns used for PCB analysis of Aroclor mixtures are usually a 5% phenyl or the SGE HT8 carborane phase 50 m × 0.22 mm i.d. × 0.25 µm film capillary column shown in Figure 3. PCB congeners are non-polar compounds that separate predominantly on boiling point when analysed on a 5% phenyl column. Because of the similar structures of many of the PCBs, the boiling points are very similar and many of the congeners are unable to be separated on non-polar columns. The analysis of Aroclor mixtures using an SGE HT8 capillary column gives unique separation of several difficult-to-resolve congeners. The unique partitioning properties of the HT8 capillary column come from the Carborane incorporated into the polysiloxane backbone. The Carborane interacts preferentially with ortho-substituted PCBs giving separation of these components to a greater extent than conventional 5% phenyl capillary columns. This can be seen in Figure 3, which is a chromatogram of Aroclor 1242, a mixture of PCB congeners that has a chlorine content of 42%.

Within this chromatogram the unique separation can be shown for congeners 31 and 28 on an HT8 (Figure 3a) compared with a conventional 5% phenyl column where these isomers are unable to be separated (Figure 3b).

The added advantage of the HT8 column is that with the extra polarity and specific interactions with the PCB congeners there is no sacrifice of thermal stability. The HT8 column has a maximum temperature of 360/370 °C, comparable or better than most 5% phenyl columns. Eluting the high boiling PCB congeners on an HT8 column is not a problem.



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