

## SPB-Octyl Capillary Column Resolves Coplanar PCBs

Traditional gas chromatography separation of PCB congeners does not offer the selectivity necessary for separation of highly toxic coplanar PCBs. SPB-Octyl capillary columns contain a unique, nonpolar bonded phase that provides well resolved, efficient separation of coplanar PCBs. (ChromFax: 394048)

### Key Words:

- PCBs • coplanar PCBs • SPB-Octyl capillary column
- toxicity

The widespread use of polychlorinated biphenyls (PCBs) as dielectric and heat transferring fluids, fluids in hydraulic and vacuum systems, plasticizers, and flame retardants, combined with their stable molecular structure, has created a persistent environmental problem. Of the 209 possible PCB congeners, approximately 150 have been reported in the environment.

Coplanar, or non-*ortho*-substituted PCBs, particularly those that have two *para*-chlorine atoms and at least one *meta*-chlorine atom (IUPAC nos. 81, 77, 126, and 169), are of increasing concern because they exhibit toxicity similar to 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), i.e., high “dioxin-like” toxicity (1).

Gas chromatography separation of PCB congeners has been based on low and intermediate-polarity phases, such as SE-54 and OV®-1701. SE-54 capillary columns are well documented, with retention times of all 209 congeners determined for this phase (2,3). However, problems with coelutions, poor detection at low levels, and the need for better separation of the highly toxic coplanar PCBs demand columns offering differing selectivities.

The SPB™-Octyl capillary column offers unique selectivity characteristics that are ideal for multidimensional or confirmational separations of PCB-containing samples. The SPB-Octyl column contains a bonded, highly dispersive, 50% octyl/50% methyl polysiloxane phase. The polarity of the octyl-methyl silicone phase approaches that of squalane, and is substantially less than that of the widely used “nonpolar” methyl silicone (SPB-1) phase (Table 1).

**Table 1. McReynolds Constants for Nonpolar Phases**

Phase	x'	y'	z'	u'	s'
Squalane	0	0	0	0	0
SPB-Octyl*	3	14	11	12	11
SPB-1	4	58	43	56	38
SPB-5	19	74	64	93	62

\*Mean of five columns.

x'	benzene
y'	1-butanol
z'	2-pentanone
u'	1-nitropropane
s'	pyridine

A well resolved, efficient separation of a mixture of 68 PCB congeners was obtained, using a 60m x 0.25mm ID x 0.25µm film SPB-Octyl column (Figure A). The 26 compounds that eluted between 24 and 29 minutes included several non-*ortho*-substituted PCBs (IUPAC nos. 35-39 and 77-81), that were well separated from the other PCBs with few coelutions. A similar separation shows excellent resolution of some other non-*ortho*-substituted PCBs (Figure B).

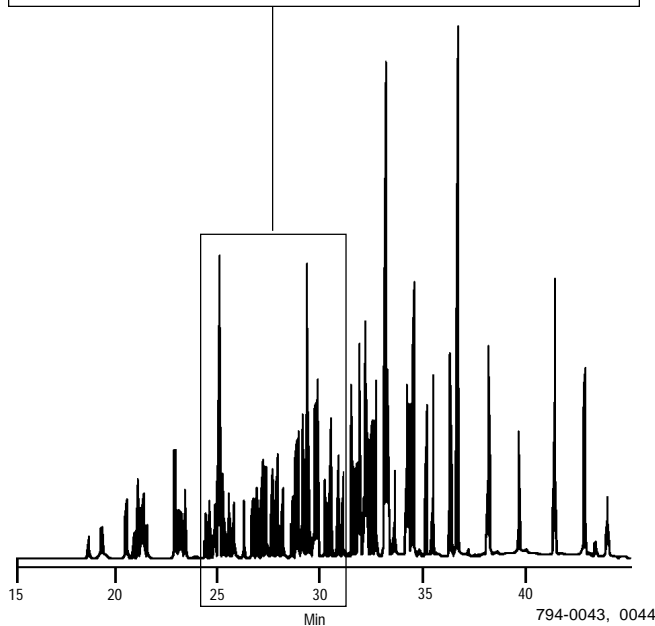
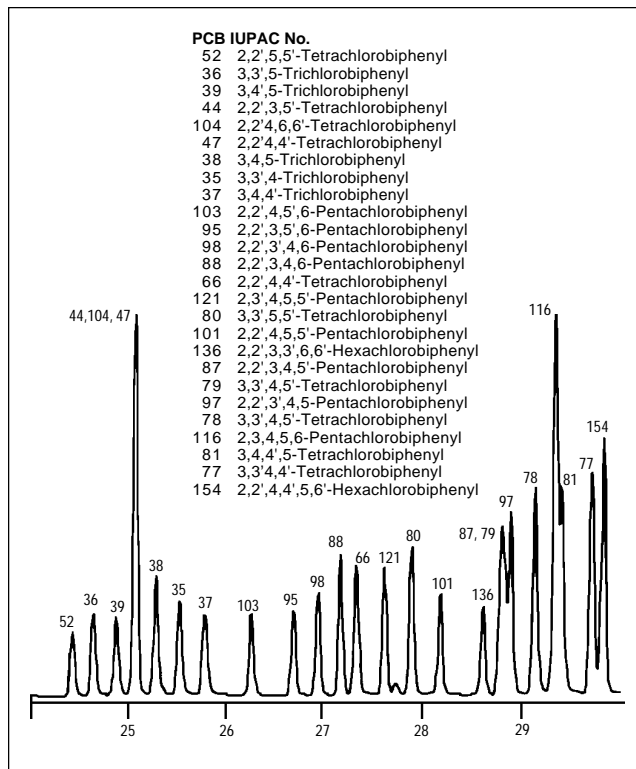
A final column temperature of 320°C was used for this application in order to elute the penta- and hexachlorobiphenyl congeners. Although the recommended operating temperature range for the SPB-Octyl column is -60°C to 280°C (isothermal), with additional conditioning, columns can be used to temperatures up to 320°C in a temperature-programmed run. However, expect increased bleed and a shorter lifetime due to loss of phase.

Even after sustaining a 320°C temperature for 16 minutes, column efficiency, activity, and selectivity remained virtually unchanged with only a modest decrease in absolute retention times. Many analysts would find this sacrifice acceptable in exchange for the analytical information obtained for the more highly substituted congeners.

The SPB-Octyl column provides excellent resolution and efficiency for separations of PCB mixtures. We recommend this column for multidimensional or confirmational analyses of environmental samples containing PCBs. We anticipate that the unique nonpolar functionality of this bonded phase will also prove useful for a wide range of foods, fragrances, pharmaceutical, and petrochemical applications.

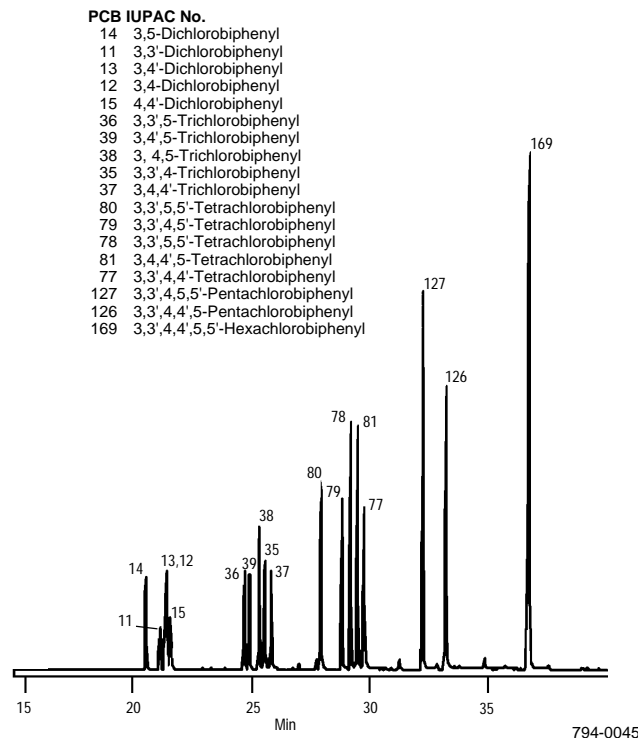
**Figure A. 68 PCB Congeners Resolved**

Column: **SPB-Octyl, 60m x 0.25mm ID, 0.25µm film**  
 Cat. No.: **24219-U**  
 Oven: 150°C (4 min) — 6°C/min — 320°C (16 min)  
 Carrier: helium, 20cm/sec (set at 200°C)  
 Det.: ECD (340°C)  
 Inj.: 1µL, 68-component PCB mixture in isooctane (50-200ng/mL each component), splitless injection, split vent off 45 sec (250°C)



**Figure B. Selected Non-ortho-Substituted (Coplanar) PCBs**

Column: **SPB-Octyl, 60m x 0.25mm ID, 0.25µm film**  
 Cat. No.: **24219-U**  
 Oven: 150°C (4 min) — 6°C/min — 320°C (16 min)  
 Carrier: helium, 20cm/sec (set at 200°C)  
 Det.: ECD (340°C)  
 Inj.: 1µL, 68-component PCB mixture in isooctane (50-200ng/mL each component), splitless injection, split vent off 45 sec (250°C)



**Ordering Information:**

**SPB-Octyl Fused Silica Capillary Column**  
 60m x 0.25mm ID, 0.25µm film **24219-U**

**References**

- Pastor, M.D., J. Sanchez, D. Barceló, and J. Albaigés, J. Chromatogr., 629:329-337 (1993).
- Ballschmitter, K., R. Bacher, and A. Mennel, J. High Res. Chromatogr., 15:260-270 (1992).
- de Boer, J., Q. Dao, and R. van Dortmond, J. High Res. Chromatogr., 15:249-255 (1992).

References not available from Supelco.

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OV — Ohio Valley Specialty Chemical Co.  
 SPB — Supelco, Inc.  
 Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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