

Application

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SPB-50 Capillary Column for Environmental Applications

US EPA methods for monitoring phenols, chlorinated pesticides, base-neutrals and phthalates specify packed gas chromatography columns using a 50% phenyl polysiloxane phase (SP-2250 or OV-17). Supelco's SPB-50 capillary column provides the polarity of the 50% phenyl polysiloxane phase with the advantages of a capillary column. (ChromFax: 394049)

Key Words:

- packed gas chromatography • phenyl polysiloxane phase

The SPB™-50 column expands Supelco's line of phenyl-containing capillary columns (Table 1). The bonded 50% phenyl polysiloxane phase offers better selectivity for compounds of similar structure and boiling points than the 5% phenyl polysiloxane phase of the widely-used PTE™-5 column. The SPB-50 phase has the highest phenyl content, and therefore the highest polarizability of the bonded, phenyl-containing series of capillary phases. SPB-50 columns exhibit excellent selectivity for the analysis of polar semivolatile pollutants. In addition to its high polarizability, the very low bleed of the SPB-50 column under GC/MS conditions (typically only 2pA at 290°C and 9pA at 320°C) makes it an excellent choice for many EPA methods involving semivolatile pollutants. (We recommend operating the SPB-50 column within temperature limits of 30°C to 310°C for longer column life.)

Table 1. McReynolds Numbers

	x'	y'	z'	u'	s'
SPB-5 (PTE-5)	19	74	64	93	62
SPB-20	67	116	117	174	131
SPB-35	101	146	151	219	202
SPB-50	125	175	183	268	220

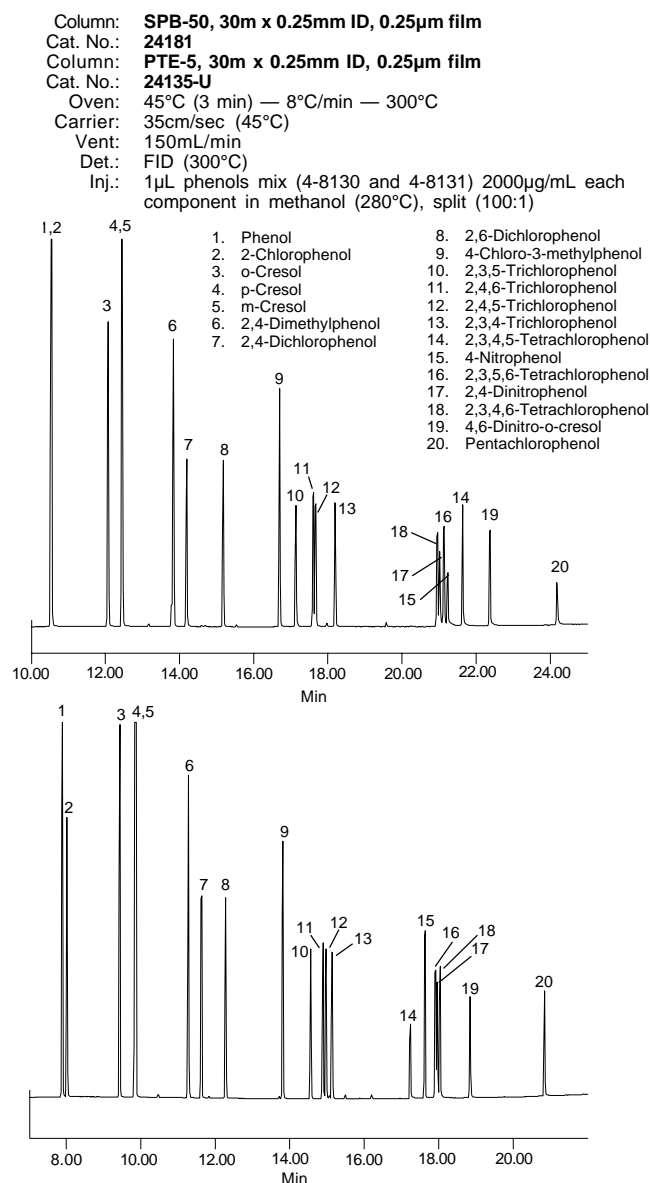
x' benzene y' 1-butanol z' 2-pentanone u' 1-nitropropane s' pyridine

We compared the performance of PTE-5 and SPB-50 columns for separating phenols, base-neutral/phthalates, and chlorinated pesticides. In every case, the SPB-50 column yielded slightly longer retention times (due to increased analyte interaction with the phase), but provided unique elution orders, and often higher resolution compared to the popular PTE-5 column.

Because of its unique selectivity, the SPB-50 is an excellent column for confirmational analysis of phenols. The elution order of 2,3,4,5-tetrachlorophenol, 4-nitrophenol, 2,3,5,6-tetrachlorophenol, 2,4-dinitrophenol, and 2,3,4,6-tetrachlorophenol (peaks 14-18) on the SPB-50 column is reversed compared to the PTE-5 column (Figure A).

When a mixture of base-neutrals and phthalates was separated using SPB-50 and PTE-5 columns, the cluster of peaks 6-12 was resolved better on the SPB-50 column, while peaks 13-18 were resolved better on the PTE-5 column. The combination of the two capillary columns clearly assists in identifying and quantifying these pollutants.

Figure A. Phenols on PTE-5 and SPB-50 Columns

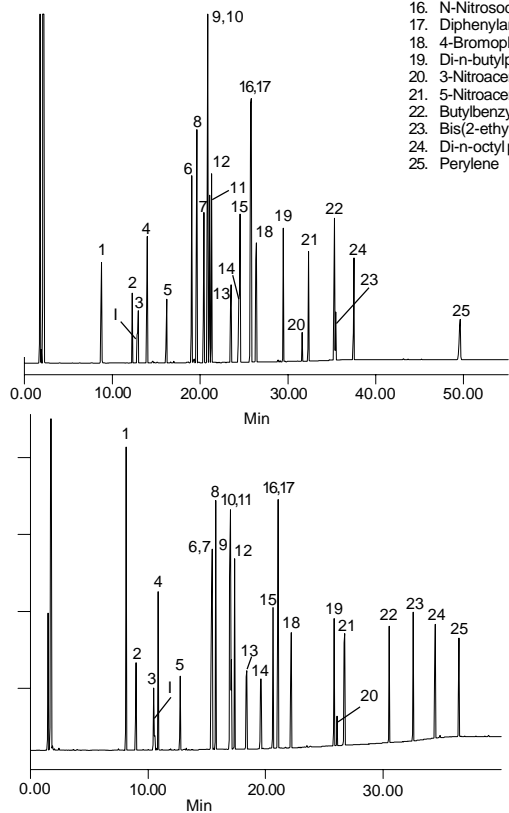


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Figure B. Base Neutrals and Phthalates on PTE-5 and SPB-50 Columns

Column: **SPB-50, 30m x 0.25mm ID, 0.25µm film**
 Cat. No.: **24181**
 Column: **PTE-5, 30m x 0.25mm ID, 0.25µm film**
 Cat. No.: **24135-U**
 Oven: 45°C (3 min) — 8°C/min — 300°C
 Carrier: 35cm/sec (45°C)
 Vent: 150mL/min
 Det.: FID (300°C)
 Inj.: 1µL base neutrals and phthalates mix (4-8134 and 4-8135), 2000µg/mL each component in methylene chloride (280°C), split 100:1

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|---|---------------------------------|
| 1. Camphene | 8. Methylnaphthalene |
| 2. Bis(2-chloroethyl) ether | 9. 2-Chloronaphthalene |
| 3. Bis(2-chloroisopropyl) ether | 10. Biphenyl |
| 4. Impurity of Bis(2-chloroisopropyl) ether | 11. Chloronaphthalene |
| 5. N-Nitrosodi-n-propylamine | 12. Diphenyl ether |
| 6. Bis(2-Chloroethoxy) methane | 13. 2,6-Dinitrotoluene |
| 7. 2-Methylnaphthalene | 14. 2,4-Dinitrotoluene |
| | 15. 4-Chlorophenyl phenyl ether |
| | 16. N-Nitrosodiphenylamine |
| | 17. Diphenylamine |
| | 18. 4-Bromophenyl phenyl ether |
| | 19. Di-n-butylphthalate |
| | 20. 3-Nitroacnaphthalene |
| | 21. 5-Nitroacnaphthalene |
| | 22. Butylbenzylphthalate |
| | 23. Bis(2-ethylhexyl) phthalate |
| | 24. Di-n-octyl phthalate |
| | 25. Perylene |

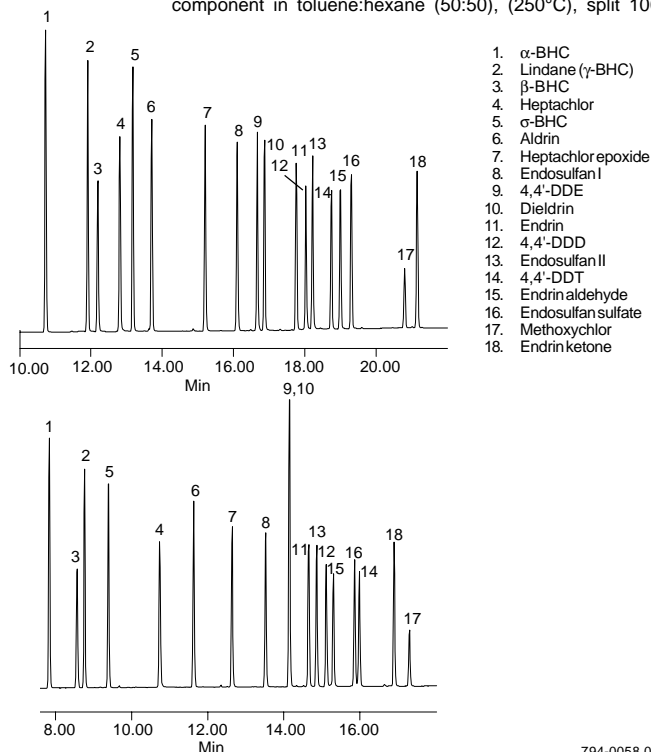


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The SPB-50 column resolved a mixture of 18 chlorinated pesticides with no coelutions (Figure C). The combined breakdown of endrin and 4,4'-DDT on the SPB-50 column was less than 6% with splitless injections of 20pg per component. The elution order of these pesticides and their breakdown levels are nearly the same as those of the specially-tested SPB-608 capillary column. The elution order of the PTE-5 column is quite different, making it a good choice for a confirmational column. 4,4'-DDE and dieldrin (peaks 9 and 10), which coeluted on the PTE-5 column, were well resolved on the SPB-50 column.

Figure C. Chlorinated Pesticides on PTE-5 and SPB-50 Columns

Column: **SPB-50, 30m x 0.25mm ID, 0.25µm film**
 Cat. No.: **24181**
 Column: **PTE-5, 30m x 0.25mm ID, 0.25µm film**
 Cat. No.: **24135-U**
 Oven: 150°C (4 min) — 8°C/min — 290°C
 Carrier: 30cm/sec (150°C)
 Vent: 150mL/min
 Det.: ECD (310°C)
 Inj.: 1µL chlorinated pesticides mix (4-8913), 2000ng/mL each component in toluene:hexane (50:50), (250°C), split 100:1



794-0058.0059

The SPB-50 column offers a useful alternative to SPT™-2250 and OV®-17 packed columns and was designed to meet or exceed the quality characteristics of other available bonded 50% phenyl capillary columns. With low bleed, high efficiency, long-term stability, and high polarizability, the SPB-50 column is an ideal column for monitoring polar semivolatiles pollutants in environmental samples.

Ordering Information:

Fused Silica Capillary GC Columns

SPB-50, 30m x 0.25mm ID, 0.25µm film	24181
SPB-50, 30m x 0.32mm ID, 0.25µm film	24187
PTE-5, 30m x 0.25mm ID, 0.25µm film	24135-U

Trademarks

OV — Ohio Valley Specialty Chemical Co.
 PTE, SP, SPB — Supelco, Inc.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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