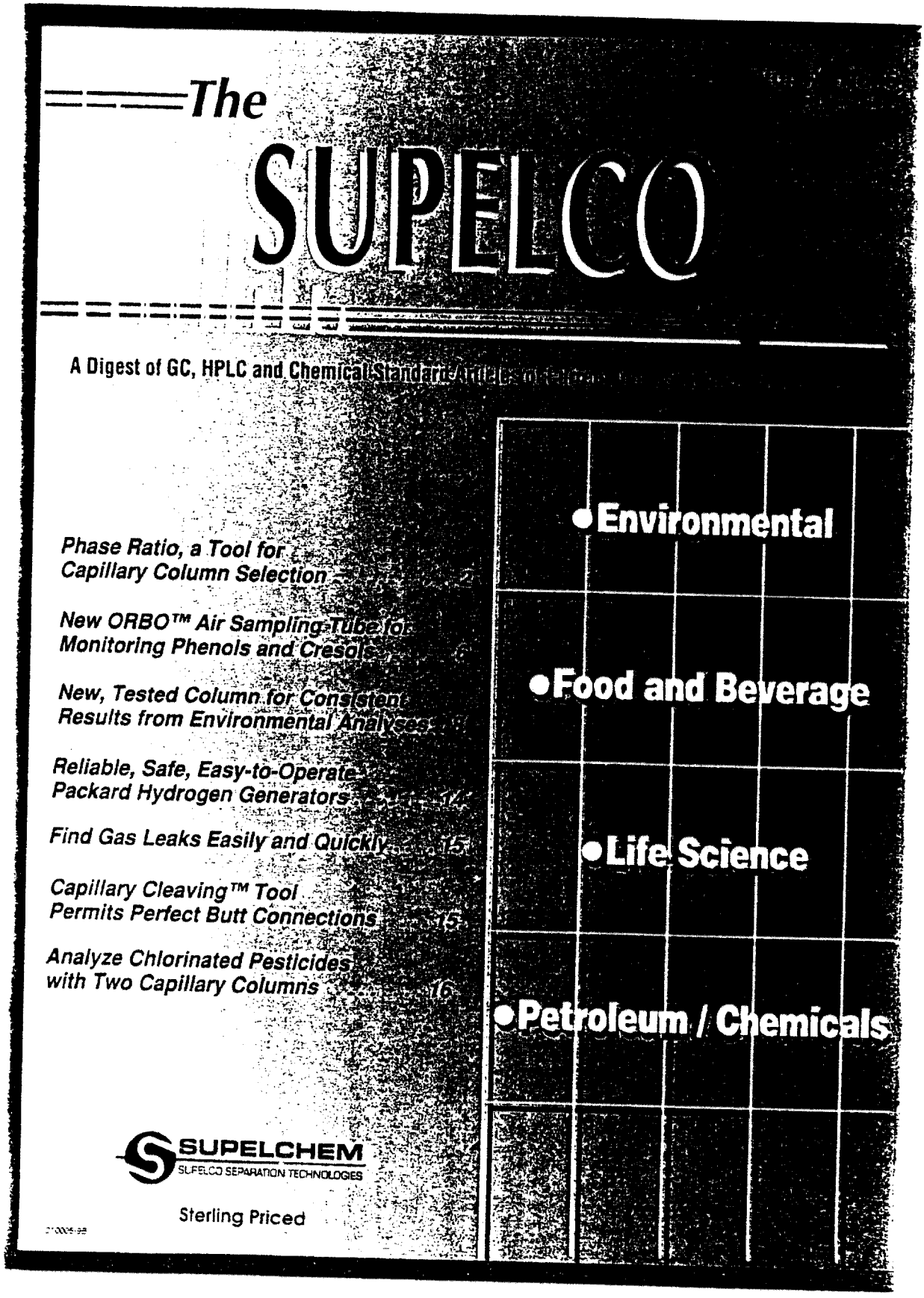
The
SUPELCO
A Digest of GC, HPLC and Chemical Standard Articles
Phase Ratio, a Tool for
Capillary Column Selection
New ORBO™ Air Sampling Tube for
Monitoring Phenols and Cresols
New, Tested Column for Consistent
Results from Environmental Analyses
Reliable, Safe, Easy-to-Operate
Packard Hydrogen Generators
Find Gas Leaks Easily and Quickly
Capillary Cleaving™ Tool
Permits Perfect Butt Connections
Analyze Chlorinated Pesticides
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Phase Ratio, a Tool for Capillary Column Selection

Use phase ratio (β) to select the optimum column for your petroleum application.

Analysts using capillary gas chromatography have a wide variety of columns to choose from. The many possible combinations of internal diameter and stationary phase film thickness make the choice of column dimensions particularly difficult. Phase ratio, or β, can be used as a guide in determining the optimum combination of column dimensions for an analysis.

Phase ratio is a dimensionless number that expresses the ratio of the internal diameter of a capillary column to the thickness of the stationary phase film (Figure A). In other words, β represents the ratio between the gas volume and the stationary phase volume in a column.

β is a useful tool for column selection because columns with the same β value, given the same analytical conditions and stationary phase, produce similar relative retention times and indices. This means that columns of the same β value can be interchanged even though they have different film thicknesses and column IDs (Table 1).

Column dimensions can be selected for the particular application at hand, while taking into account the resulting tradeoffs in sample capacity and efficiency. For example, to increase sample capacity, a wider bore column with the same β value can be used; or to obtain greater efficiency a narrower bore column with the same β value can be used.

Figure B illustrates the use of β value to correlate columns of different dimensions. The same solvent mixture, containing butanol, ethylbenzene, chloroform, toluene, para and meta xylene — and various acetates — was analyzed on two columns with similar β values: a *SUPELCOWAX™ 10 30m x 0.20mm x 0.20µm column* (β =250) and a *SUPELCOWAX 10 30m x 0.53mm x 0.50µm column* (β =265). The very close β values result in identical elution patterns with no

Figure A — β Defined

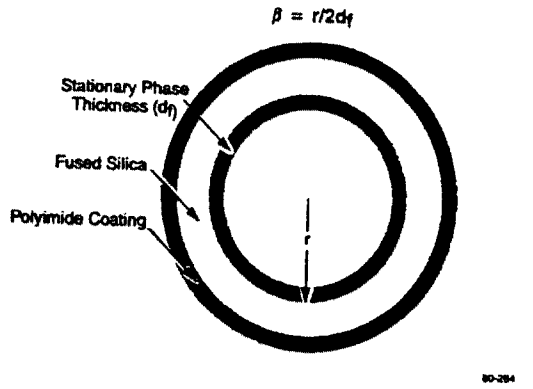


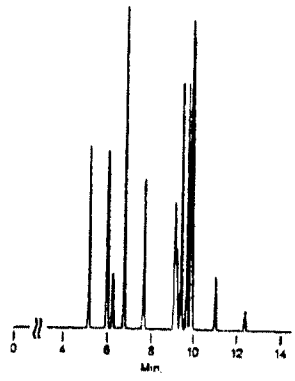
Table 1 — Examples of Columns with Equivalent β Values

β	Column ID mm	Film Thickness µm
63	0.20	0.80
	0.25	1.00
	0.32	1.28
	0.53	2.12
	0.75	3.00
250	0.20	0.20
	0.25	0.25
	0.32	0.32
	0.53	0.53
	0.75	0.75
400	0.20	0.13
	0.25	0.16
	0.32	0.20
	0.53	0.33
	0.75	0.47

Figure B — Similar Elution Patterns for a Solvent Mixture on SUPELCOWAX 10 Capillary Columns

SUPELCOWAX 10 capillary column, 30m x 0.20mm x 0.20µm (β=250)

SUPELCOWAX 10 capillary column, 30m x 0.53mm x 0.50µm (β=265)



80-27

Col. Temp.: 50°C for 4 min., to 175°C at 4°C/min., Inj. & Det. Temp.: 220°C. Linear Velocity: 20cm/sec. at 75°C, He. Det.: FID (4 x 10^{11} AFS. Sample: 1µl. Split: 100:1.

Table 2 — β Guidelines

Range	β	Application
Low	<100	low capacity (k') solutes low M.W. compounds
Medium	100 - 400	wide range k' solutes general purpose analyses wide range of compounds
High	>400	high k' solutes high M.W. compounds

peak shifting. The larger ID column is less efficient and therefore exhibits a slight loss of resolution.

The analyses presented in Figure B could occur in a situation where a methods development lab uses a narrow bore column for most of their work and then transfers the method to a pilot plant. The pilot plant analysts sample their product stream and inject larger samples on their column, overloading the narrow bore column, which has less on-column sample capacity, and masking some peaks. By changing to a wider bore column with the same β value, the analyst trades greater sample capacity for slightly lower resolution.

In addition to correlating columns of different dimensions, β can be used as a guide for column selection. Columns having a β value of less than 100 are considered low-β columns and are used for analysis of low molecular weight compounds. Columns with β values between 100 and 400 are considered standard-β columns and are used for most analyses. They are well suited for the analysis of a wide range of compounds and can be used for unique applications such as simulated distillation (SIMDIS). High molecular weight compound analyses generally require high-β columns. Table 2 summarizes guidelines for selecting a capillary column.

Although the following examples for each range of β values come from petroleum applications, the concept can be applied to other disciplines where analysis of very complex samples is required. For example, a related article in this publication, *New, Tested Column for Consistent Results from Environmental Analyses,* discusses bonded-phase, SE-54-type capillary columns with β values of 250, 265, and 320.

Figure C illustrates the use of a very low-β (26.5) column for analysis of low boiling point hydrocarbons. These separations were performed on a *60m x 0.53mm x 5.0µm, SPB™-1 fused silica capillary column* using ambient column temperature (30°C). Ethylene and ethane (Figure C1) were baseline separated. Propylene and propane were partially separated. As the inset shows, isobutane and ethanol, which co-eluted at ambient temperature, were baseline separated at 0°C. The same column was used to detect impurities in propylene (Figure C2), again resulting in baseline separation of ethylene and ethane. However propane, if present, would be masked by the large propylene peak.

If the corresponding olefins are not present, a slightly higher β column can be used. Figure D shows analysis of natural gas on a shorter column of the same type with a thinner film. A *30m x 0.53mm x 3.0µm SPB-1 fused silica capillary column*

(cont'd on page 4

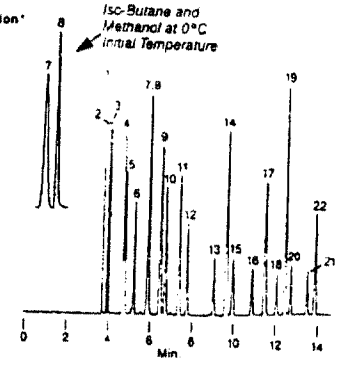
(contd. from page 3)

Figure C — Low Boiling Point Hydrocarbons on a Very Low β (26.5) Column

C1 — C1-C6 Hydrocarbons and C1-C4 Alcohols

Component	Concentration*
1. Methane	0.3
2. Ethylene	0.2
3. Ethane	0.2
4. Propylene	0.3
5. Propane	0.2
6. Propadiene	0.2
7. Iso-Butane	0.2
8. Methanol	0.2
9. Butene-1	0.3
10. n-Butane	0.2
11. cis-2-Butene	0.3
12. Ethanol	0.2
13. Iso-Propanol	0.1
14. n-Pentane	0.4
15. tert-Butanol	0.1
16. n-Propanol	0.1
17. Methyl-tert-butyl ether	0.3
18. sec-Butanol	0.1
19. n-Hexane	0.4
20. Iso-Butanol	0.1
21. n-Butanol	0.1
22. Benzene	0.2

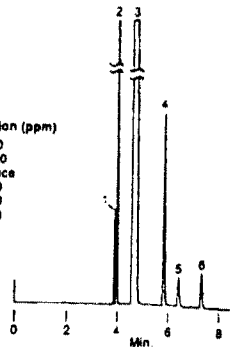
*(Vol. %)



80-265

C2 — Impurities in Propylene

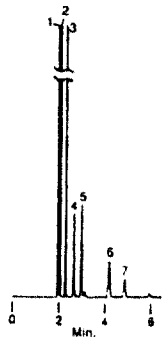
Component	Concentration (ppm)
1. Ethylene	100
2. Ethane	1000
3. Propylene	balance
4. Methanol	200
5. n-Butane	100
6. Ethanol	100



80-267

SPB-1 fused silica capillary column, 60m x 0.53mm x 5.0µm film, Col. Temp.: 30°C for 5 min., to 200°C at 20°C, Inj. Temp.: 200°C, Det. Temp: 200°C, Linear Velocity: 19-21cm sec., He, Det.: FID (8 x 10^{-11} AFS), Sample: 250µl, Split: 100:1.

Figure D — Natural Gas on a 44.2 β Column

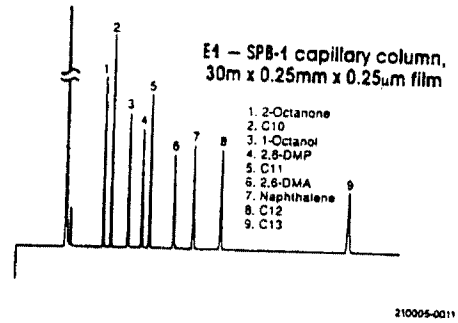


Component	Concentration (Vol. %)
1. Methane	70.5
2. Ethane	9.0
3. Propane	6.0
4. Iso-Butane	3.0
5. n-Butane	3.0
6. Iso-Pentane	1.0
7. n-Pentane	1.0
Helium, Nitrogen	balance

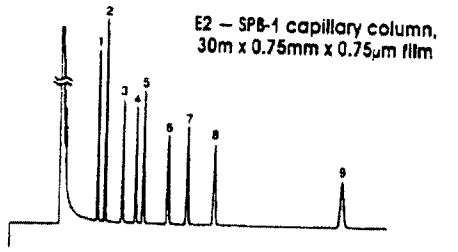
80-256

Column and conditions: see Figure C.

Figure E — Nonpolar Test Mix on Columns With Same β (β = 250)



210005-0011



210005-0010

Col. Temp.: 110°C, Inj. & Det. Temp.: 220°C, Linear Velocity: 19-21cm/sec., He, Det.: FID (4 x 10^{-11} AFS), Sample: 1µl (Cat. No. 4-7300), Split: 100:1.

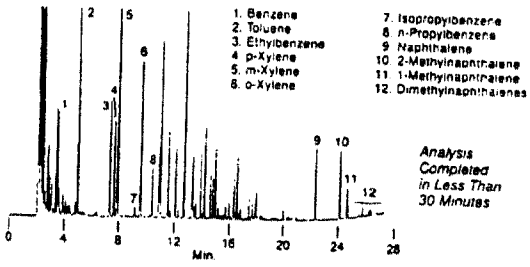
with a β value of 44.2 was used to perform the separation at ambient column temperature (30°C), requiring 6 minutes for complete separation of all compounds.

The chromatograms presented in Figures E and F were generated by columns with a β value of 250. This value is in the center of the 100 to 400 range and is probably the most commonly used β value column. (If a column's ID in millimeters is the same as its film thickness in microns, the column is, by definition, a 250 β column.)

The same nonpolar test mix was separated on a 0.25mm ID and a 0.75mm ID SPB-1 capillary column, both with a β of 250 (Figure E). Even though the internal diameters of these columns are widely different, the analyses are virtually identical. The calculated retention indices for each of the active probes in the test sample are also identical, indicating no difference in polarity.

Figure F shows the analysis of aromatics in gasoline on a *30m x 0.20mm x 0.20µm SUPELCOWAX capillary column*. As shown previously, the standard β value (250) of this column allows the analysis of a wide range of compounds.

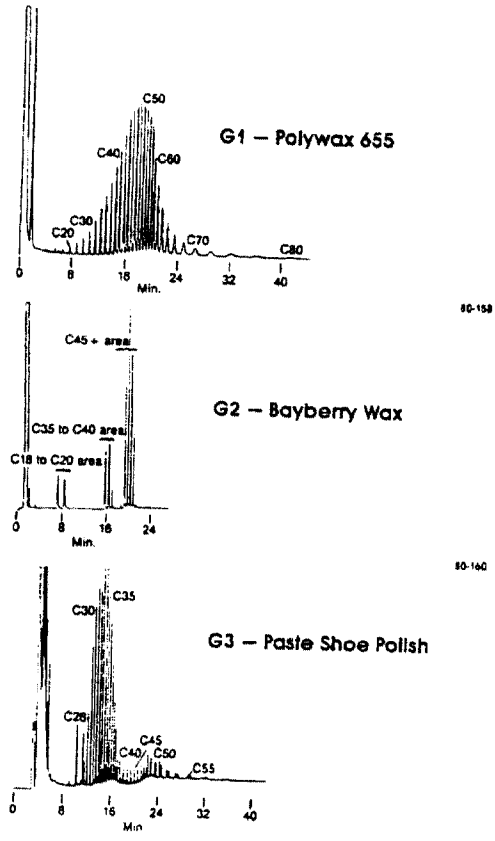
Figure F — Aromatics in Gasoline



80-103

SUPELCOWAX 10 capillary column, 30m x 0.20mm x 0.20µm film, Col. Temp.: 60°C for 8 min., to 210°C at 8°C/min., hold 5 min., Inj. & Det. Temp.: 220°C, Linear Velocity: 25cm/sec., He, Det.: FID (8 x 10^{-11} AFS), Sample: 0.1µl gasoline, Split: 50:1.

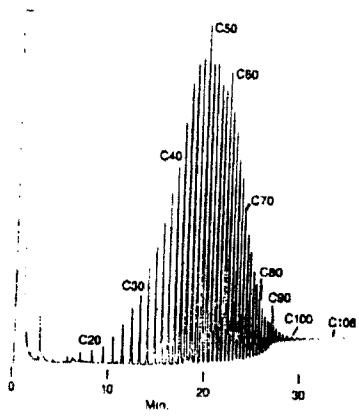
Figure G — Waxes on a High β (1325) Column



80-162

SPB-1 fused silica capillary column, 30m x 0.53mm x 0.10µm film, Col. Temp.: 50°C to 350°C at 15°C/min., Inj. Temp.: cool on-column, Det Temp.: 390°C, Flow Rate: 5ml/min., He, Det.: FID (8 x 10^{-11} AFS), Sample: 1µl, Split: 100:1.

Figure H — Polywax 655 on a Very High β (1875) Column



80-145

SPB-1 glass capillary column, 7m x 0.75mm x 0.10µm film, Col. Temp.: 40°C for 1 min., to 430°C at 15°C/min., hold 15 min., Inj. Temp.: cool on-column, Det. Temp.: 450°C, Flow Rate: 10ml/min., He, Det.: FID (128 x 10^{-10} AFS), Sample: 0.1µl (Cat. No. 4-8482), Split: 100:1.

Figure G illustrates the use of a high-β column for the separation of high boiling point materials. A *30m x 0.53mm x 0.10µm SPB-1 fused silica capillary column*, with a β of 1325, was used to analyze several waxes — including *Polywax™ 655*, Bayberry wax, and paste shoe polish, a finished wax product. Carbon numbers up to C80 were eluted on this column.

Increasing the β value extends the upper range of compounds that can be analyzed. Figure H shows the separation of Polywax 655 using a very high β. 7m x 0.75mm x 0.10µm SPB-1 column. In this case, the very high β value (1875) permits the analysis of compounds with carbon numbers as great as C108.

The capillary columns discussed here represent a small sample of the available range. Determining the proper β value is only one step in optimizing your capillary column application. Film thickness and column ID — the two factors that make up β — as well as factors not related to β, such as column length and stationary phase, can be optimized. Supelco offers a complete selection of capillary columns. And, if you can't find the column you need in our stock selection, we can make a custom column to your specifications. Call us.

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

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4004039644

New ORBO™ Air Sampling Tube for Monitoring Phenols and Cresols

As an improvement over previous methods for monitoring phenols and cresols, the Occupational Health and Safety Administration (OSHA) developed a simplified sampling procedure for analyzing both analytes. Breakthrough volumes, detection limits, and stability studies performed by OSHA indicate that a single sampling tube containing a 100mg front section and 50mg back section of Amberlite® XAD®-7■ resin effectively adsorbs and desorbs a combined atmosphere of phenol and the ortho-, para-, and meta-isomers of cresol (1).

Our new *ORBO-47 adsorbent tubes* — prepared according to OSHA Method 32 — contain Supelpak™-70 resin, a highly purified grade of Amberlite XAD-7 resin. ORBO-47 tubes will help ensure minimal background for accurate monitoring of airborne phenol and cresols.

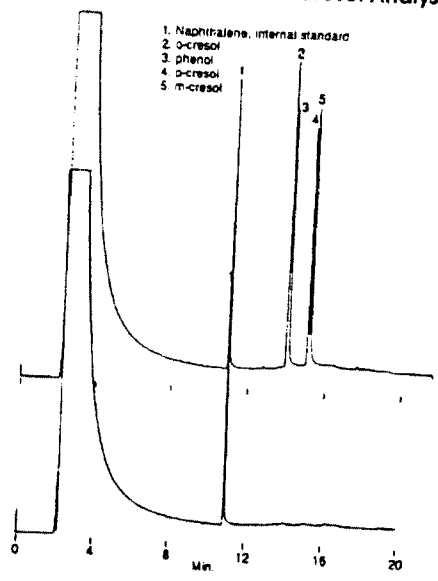
An ORBO-47 adsorbent tube effectively adsorbs and desorbs phenol and the cresol isomers — singly or in combinations

Results of OSHA investigations determined the detection limits of the analytical procedure to be 12ng and 14ng for phenol and cresol, respectively. We established our quality assurance test limits for these tubes to approximate the analytical limit of the method, or 11.4ng on-column for each analyte. These test levels ensure that Supelpak-70 resin purification procedures are functional, reproducible, and meet the requirements of OSHA Method 32.

Studies at Supelco, using ORBO-47 tubes, provided additional information about the reliability of the adsorbent and the sampling procedure. Adsorbent purity, bed weight reproducibility, and phenol and cresol desorption efficiency were determined.

For purity determination, three unused tubes from each of two ORBO-47 tube lots were extracted with solvent according to OSHA Method 32. Chromatograms obtained from these samples were compared to chromatograms for an external calibration standard approximately equal to the 9.5mg/m³ action limit of o-cresol, and the 11mg/m³ action limit of phenol, p-cresol, and m-cresol in air. The tubes contained no contaminants that could interfere with the phenol and cresol analysis at 1/10 of these on-tube concentrations (Figure A).

Figure A — ORBO-47 Tubes Ensure Minimal Background for Phenol and Cresol Analysis



P10100-0040-41

SUPELCOWAX-10 borosilicate glass capillary column, 60m x 0.75mm ID, 1.0µm film, Col. Temp.: 100°C (no hold) to 210°C at 8°C/min, Flow Rate: 10ml/min., He, Det.: FID, Sample: 1µl methanol containing 120ng each phenol, o-cresol, m-cresol, and p-cresol and 50ng int. std. or 1µl of 2ml methanol extract from blank ORBO-47 tube (int. std. added at 50ng/1µl).

Consistent bed weights also help ensure accurate results. The bed weight specified for the front section of ORBO-47 tubes is 100mg. Front bed weights for six ORBO-47 tubes averaged 100.8mg ±1.7mg. The small standard deviation signifies highly consistent bed weights.

Consistent adsorbent bed weights in ORBO-47 tubes help ensure accurate sampling

We measured desorption efficiency of phenol and cresol from Supelpak-70 using two methods. For the first method, 4 ORBO-47 tubes were spiked with 240µg/ml each of phenol and the cresol isomers. Air was drawn across the tube at 0.1 liters/min., the OSHA Method 32 recommended flow rate, for 2 hours. Front and back beds were desorbed separately in 2ml of methanol. Final concentration of the standards in the extract was 120µg/ml. An internal standard, naphthalene was added to the methanol at 50µg/ml to eliminate injection volume variances.

Using the second method, three front beds of ORBO-47 tubes, containing Supelpak-70, were placed in sample vials. We spiked the vials with 240µg each standard compound to approximate the action limit of each compound. After equilibration, Supelpak-70 was desorbed with 2ml of methanol as in the previous method.

Table 1 — ORBO-47 Desorption Efficiency

	% Recovery			
	Phenol	o-Cresol	p-Cresol	m-Cresol
Method 1	99	94	93	92
	104	105	104	102
	101	103	101	100
	111	106	104	105
Method 2	102	105	104	102
	106	108	106	103
	103	106	111	114
x-bar	103.9	103.7	103.3	102.6
%RSD	3.8	4.4	5.3	6.4

For each procedure, control vials were made by spiking 2ml of methanol with 240µg of each standard. Table I shows that desorption efficiency is excellent for both methods.

Our results — combined with those obtained from the OSHA group — show that the ORBO-47 tube is an effective tool for monitoring airborne phenols and cresols according to OSHA Method 32.

You will also receive your ORBO-47 tubes very quickly, since they are in stock and ready for delivery.

ORBO-47 Adsorbent Tubes, pk. of 50
2-0349T **£95**

SUPELCOWAX™-10 Wide Bore Capillary Column
60m x 0.75mm ID Borosilicate Glass, 1.0µm film
2-3723T **£525**

For other custom or capillary columns, refer to our catalogue or call our Order Processing Department.

■ This product contains media manufactured under one or more of US Pat. Nos. 4,224,415; 4,256,840; 4,382,124; and 4,501,826. The purchaser is entitled to utilize this product under US Pat. No. 4,297,220.

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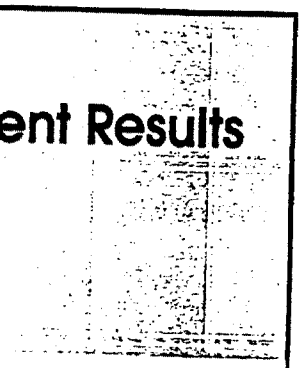
Reference

1. Method 32 (Organics Methods Evaluation Branch, OSHA Analytical Laboratory, Salt Lake City, Utah, USA (November 1981).

Reference not available from Supelchem.

New, Tested Column for Consistent Results from Environmental Analyses



Since we introduced our 30m x 0.25mm ID PTE™-5 capillary column last year, many analysts have inquired about the availability of an equivalent 0.32mm ID column. In response to these requests, we now offer a *30m x 0.32mm ID (0.25µm phase film) PTE-5 column*. Like their 0.25mm ID counterparts, every 0.32mm ID column is tested and unconditionally guaranteed to ensure consistent, highly reliable characteristics for analyses of semivolatile compounds, according to many of the US Environmental Protection Agency's 500, 600, SW-846, and Contract Laboratory Program methods. We test every PTE-5 column, to ensure reproducible relative retention times, consistent high inertness, and low column bleed.

PTE-5 columns are unconditionally guaranteed to ensure consistent characteristics for analysis of semivolatile compounds by US EPA methods

Bonded phase, SE-54-type capillary columns (5% phenyl/95% dimethyl silicone) — such as PTE-5 columns — are among the most widely used columns for environmental analyses. These columns are cited in Environmental Protection Agency (EPA) methods for many semivolatile organic compounds, including compounds that are acidic, basic, or neutral.

To provide *consistent* results for these widely diverse analyses, a column must be manufactured and tested under rigid specifications. We evaluate the new 0.32mm ID PTE-5 columns by using the same tests and the same 15-component mixture we use to test our 0.25mm ID columns (Table 1). As a result, we unconditionally guarantee *all* PTE-5 columns for three important criteria: consistent polarity (measured by reproducibility of relative retention times), inertness (measured by reproducibility of relative response factors), and consistently low column bleed.

Reproducible relative retention times (RRT) are a critical requirement of a column used for environmental analyses, because many analysts establish and rely on retention time windows for preliminary identification of sample components. Even small column-to-column differences in polarity can alter relative retention times and put identification of sample components in jeopardy. We monitor the RRT for three components of the 15-component test mixture — one eluting early, one at the middle, and one near the high point of the column temperature profile. These three components are sensitive indicators of shifts in column polarity that could adversely affect the consistency of column-to-column performance.

Table 1 — Test Mixture for Evaluating PTE-5 Columns

Component	Concentration (ng/µl)
N-Nitrosodimethylamine	10
Aniline	10
Benzoic acid	10
2,2'-Difluorobiphenyl (int. std.)	5
3-Nitroaniline	10
2,4-Dinitrophenol	10
4-Nitrophenol	10
4-Nitroaniline	10
Pentachlorophenol	10
Phenanthrene	5
Anthracene	5
Benzidine	10
3,3'-Dichlorobenzidine	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(g,h,i)perylene	10

Table 2 shows the mean RRT for these compounds obtained from 27 of our new 0.32mm ID PTE-5 columns. Standard deviations and coefficients of variation are minimal, demonstrating very good column-to-column reproducibility.

Linear relative response factors, critical in a column used for environmental analyses, are a reflection of column inertness. For accurate quantification of sample components, many US EPA methods require that linear calibration curves be established over the concentration range of interest. We have determined that 0.32mm ID PTE-5 columns pro-

Linear relative response factors show that PTE-5 columns are inert

vide a linear calibration curve (six data points) with on-column quantities of 5 to 200 nanograms for each of the 15 components in our test mixture. This includes the five known to exhibit erratic chromatographic behavior: benzoic acid, 2,4-dinitrophenol, 3-nitroaniline, 4-nitroaniline, and pentachlorophenol. Figure A shows typical calibration curves for an acidic compound and a basic compound from one PTE-5 column. These linear data plots confirm the highly inert nature of PTE-5 columns.

From these response data, we have selected five components of the 15-component test mixture to routinely monitor on every PTE-5 column as indicators of column inertness. These compounds — benzidine, benzoic acid, 2,4-dinitrophenol, 4-nitroaniline, and pentachlorophenol — exhibit the greatest variability in linearity of response evaluations, and therefore are the most stringent test of PTE-5 column performance. As a consequence of this stringent testing, you can be sure PTE-5 columns are consistently inert.

In the proposed US EPA Contract Laboratory Program (CLP) semivolatiles procedure (1), relative standard deviations for response factors (relative to deuterated internal standards) for initial calibrations of 54 target compounds must fall below specified values ranging from 15 to 30%. Eleven compounds, including 4-nitrophenol, 2,4-dinitrophenol, and 4-nitroaniline, have no maximum %RSD criteria because of their "erratic and poor linearity and sensitivity". For a 0.32mm ID PTE-5 column, randomly selected from inventory, %RSD values for these problem compounds are comparable to — or lower than — values for better behaved compounds (Table 3).

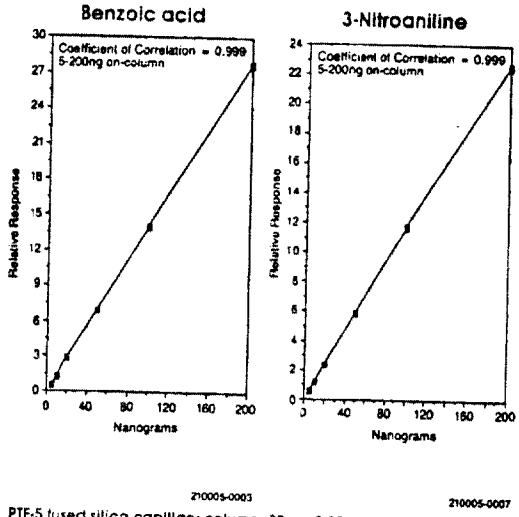
Furthermore, linear responses are reproducible

Table 2 — Highly Consistent Relative Retention Times for PTE-5 Columns

	Relative Retention Time*		
	N-Nitroso-dimethylamine	4-Nitrophenol	Benzo(g,h,i)-perylene
Mean**	0.184	1.162	2.273
Standard Deviation	0.009	0.003	0.005
% Relative Standard Deviation	4.79	0.26	0.22

*Retention of 2,2'-difluorobiphenyl = 1.
**N = 27 0.32mm ID columns.

Figure A — Obtain Linear Calibration Curves for Acidic and Basic Compounds from the same PTE-5 Column



PTE-5 fused silica capillary column, 30m x 0.32mm ID, 0.25um film.

Table 3 — Highly Consistent Relative Responses for Problem Compounds — Using a 0.32mm ID PTE-5 Column

Compound	Overall Relative Response Factor*		
	Mean	Standard Deviation	%Relative Standard Deviation
Benzidine	0.831	0.073	8.81
2,4-Dinitrophenol	0.431	0.066	15.35
4-Nitroaniline	0.694	0.019	2.79
4-Nitrophenol	0.836	0.028	3.39
Pentachlorophenol	0.894	0.029	3.21

*3 analyses at each of 5 concentrations: 20, 50, 80, 120, 160ng/µl.
Fused silica columns manufactured under HP US Pat. No. 4,293,415

(cont'd. on page 10)

(cont'd from page 9)

from one PTE-5 column to the next. Table 4 shows average coefficient of correlation values for six-point calibration curves for 12 randomly selected columns. The mean values are all near 1.000, and standard deviations are minimal.

Column bleed is the third column performance criterion we evaluate. Low bleed is necessary to meet sensitivity requirements of most US EPA methods, to shorten stabilization times, and to minimize the need for cleaning mass spectrometer ion sources. We evaluate PTE-5 column bleed at 300°C, using an FID. Bleed must be below 7.5 picoamps, but these columns typically exhibit less than 5 picoamps of bleed under these test conditions.

Several applications demonstrate the inertness and versatility of new 0.32mm ID PTE-5 columns. Phenols, as analyzed according to US EPA Method 8040 and Method 604, are among the most difficult environmental analytes to analyze. Because these compounds interact with poorly deactivated columns and injector liners, the ability to resolve underivatized phenols, while maintaining peak sharpness and symmetry, is a severe test for a capillary column. Figure B shows that 18 phenols mentioned in EPA Method 8040 are well resolved — with excellent peak shape — in less than 23 minutes.

PTE-5 columns typically exhibit less than 5 picoamps of bleed at 300°C

Polynuclear aromatic hydrocarbons (PAHs), analyzed according to EPA Method 8100 and Method 610 and the CLP semivolatiles protocol, also are demanding of column inertness. Figure C shows that a PTE-5 column will resolve the 16 PAHs listed in Methods 8100 and 610, plus dibenzofuran and carbazole. Note, in particular, the separation of benzo(b)fluoranthene and benzo(k)fluoranthene, and indeno (1,2,3-cd)pyrene and dibenzo(a,h)anthracene).

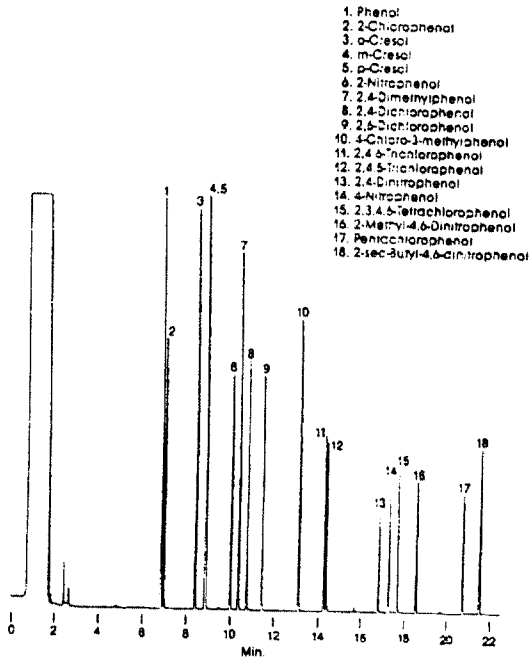
Analysis of chlorinated herbicides in drinking water (EPA Method 515.1) calls for converting the chlorinated acids and phenols to methyl derivatives, then analyzing them with electron capture detection. Figure D shows the analysis of 10 of these

Table 4 — Relative Responses are Consistently Linear for 0.32mm ID PTE-5 Columns

Compound	Coefficient of Correlation		
	Mean*	Standard Deviation	Coefficient of Variation (%)
N-Nitrosodimethyl-amine	1.000	0.000	0.028
Aniline	1.000	0.001	0.055
Pentachlorophenol	0.999	0.001	0.055
Benzoic acid	0.999	0.001	0.104
2,4-Dinitrophenol	0.997	0.002	0.198
3-Nitroaniline	0.999	0.002	0.156
4-Nitroaniline	0.997	0.002	0.155
4-Nitrophenol	0.998	0.002	0.196
Benzidine	0.993	0.007	0.697

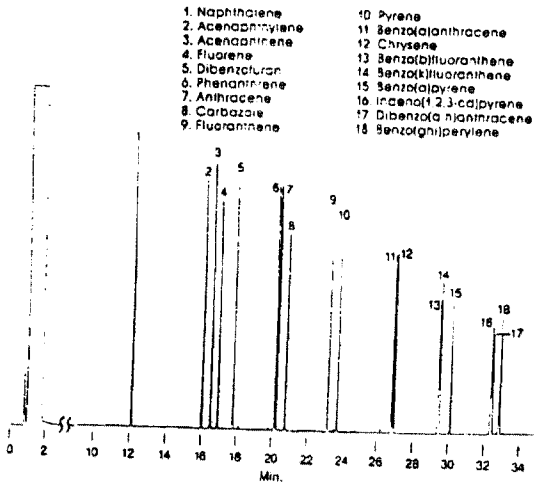
*N = 12 randomly selected columns.

Figure B — Phenols by EPA Method 8040 on a 0.32mm ID PTE-5 Column



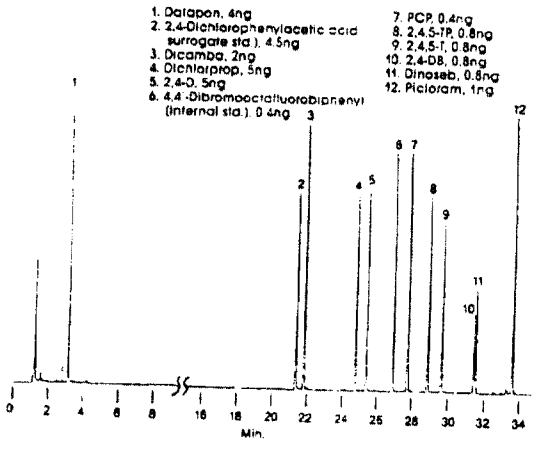
PTE-5 fused silica capillary column, 30m x 0.32mm ID, 0.25µm film. Col. Temp.: 50°C for 3 min., then to 200°C at 8°C/min., Linear Velocity: 30cm/sec, He, Det.: FID (16 x 10^{-11} AFS), Sample: 1µl 2-propanol containing 50ng each phenol.

Figure C — Polynuclear Aromatic Hydrocarbons by EPA Method 8100



PTE-5 fused silica capillary column, 30m x 0.32mm ID, 0.25µm film, Col. Temp.: 35°C for 4 min., then to 320°C at 10°C/min., Linear Velocity: 40cm/sec, He, Det.: FID (16 x 10^{-12} AFS), Sample: 1µl methylene chloride containing 25ng each PAH.

Figure D — Chlorinated Pesticides as Methyl Derivatives, by EPA Method 515.1



PTE-5 fused silica capillary column, 30m x 0.32mm ID, 0.25µm film, Col. Temp.: 60°C to 300°C at 4°C/min., Linear Velocity: 30cm/sec, He, Det.: ECD (256 x 10^{-13} AFS), Sample: 1µl hexane, on-column quantities of herbicides listed on figure.

herbicide derivatives on a 0.32mm ID PTE-5 column, at concentrations of 0.4 to 5.0ng/µl. All compounds are separated.

If you are performing analyses of semivolatiles in environmental samples on a bonded SE-54-type capillary column, and highly consistent column-to-column performance is important to you, we highly recommend our 0.25mm ID and new 0.32mm ID PTE-5 columns. We test every column to ensure highly consistent performance. And we guarantee you will be consistently satisfied with PTE-5 column performance — or you can return the column for a new one or for a refund.

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Reference

1. Table 2 (Relative Response Factor Criteria for Initial and Continuing Calibration of Semivolatile Organic Compounds) in section *Method for the Determination of Extractable Semivolatile Organic Compounds* (Oct. 18, 1989) of draft *Statement of Work for Organic Analysis, Multimedia, Multiconcentration*, US EPA Contract Laboratory Program (Oct. 1989).

Reference not available from Supelchem.

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4004039 70

(contd. from back page) Analyze Chlorinated Pesticides with Two Capillary Columns

ensures excellent column inertness. In addition, the SPB-608 column offers exceptionally low ECD bleed levels.

When analyzing for Method 608 pesticides in samples of unknown origin, compound identification should be supported by at least one confirmational analysis. This confirmation is frequently performed on a second column, containing a stationary phase with a different polarity than the primary analytical column. Our PTE-5 column is a logical choice for the confirmational analysis.

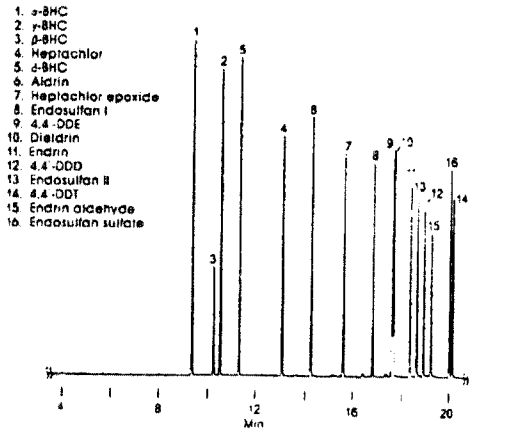
A PTE-5 capillary column separates the organochlorine pesticides listed in EPA Method 608 in less than 30 minutes (Figure B). It also shifts the *elution order* — relative to that provided by the SPB-608 column — of nine of these 16 pesticides (Table 1). And, compared to the same analysis on the SPB-608 column, it shifts the *relative retention times* (RRT) of all the pesticides. These RRT shifts — caused by differences in column polarity — provide greater assurance that the compounds have been correctly identified. Elution orders of the pesticides listed in Table 1 were confirmed by GC/MS.

When we developed our PTE-5 column for the demanding analyses of the semivolatile pollutants listed in US EPA Methods 625, 1625, and 8270 (1), we significantly improved the inertness of the bonded SE-54 type stationary phase. We were able to do this by improving manufacturing procedures for this column. We check each column's inertness with priority pollutant compounds notorious for poor chromatographic performance. Among these compounds are benzoic acid, 2,4-dinitrophenol, pentachlorophenol and benzidine.

Our priority pollutant mix is also used to check relative response factors. For this evaluation, we use on-column quantities of 5-10 nanograms for each compound. These levels are at, or below, the minimum detection level (MDL) of EPA Method 625. In addition, we check each PTE-5 column's polarity to ensure that relative retention times of the priority pollutants fall within the retention time windows specified in EPA Method 625.

Each PTE-5 column will provide linear calibration curves for the analytes listed in EPA Method

Figure B — Chlorinated Pesticides Using PTE-5 Capillary Column for Confirmation in Dual Column Procedure



909-100-0058

PTE-5 fused silica capillary column, 30m x 0.25mm ID (0.25µm film thickness), Linear Velocity: 25cm/sec, He at 290°C, Col. Temp.: 150°C (hold for 4 minutes), 6°C/min. to 290°C (hold for 5 minutes or until all 16 pesticides elute), Det.: ECD, (256 x 10⁻¹³ AFS) at 310°C, Split Inj.: Vent Flow 65-70cc/min., Temp. 250°C, Vol.: 0.5µl, Sample: 16 Chlorinated Pesticides Mixture (100pg each compound).

Table 1 — Different Elution Patterns of Chlorinated Pesticides■

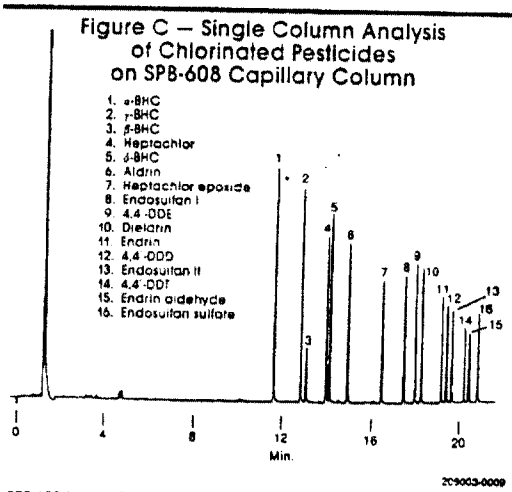
Component	SPB-608 Column RT (min.)	RRT**
1. α-BHC	13.52	0.758
2. γ-BHC	15.09	0.846
3. β-BHC	15.46	0.867
4. Heptachlor	16.58	0.929
5. δ-BHC	16.83	0.943
6. Aldrin	17.84	1.000
7. Heptachlor epoxide	19.9	1.115
8. Endosulfan I	21.2	1.188
9. 4,4'-DDE	21.98	1.232
10. Dieldrin	22.28	1.249
11. Endrin	23.48	1.316
12. 4,4'-DDD	23.91	1.340
13. Endosulfan II	24.13	1.353
14. 4,4'-DDT	24.96	1.399
15. Endrin aldehyde	25.18	1.411
16. Endosulfan sulfate	25.72	1.442

Component	PTE-5 Column RT (min.)	RRT**
1. α-BHC	9.54	0.654
* 2. β-BHC	10.72	0.735
* 3. γ-BHC	10.44	0.716
* 4. δ-BHC	13.36	0.916
* 5. Heptachlor	11.52	0.790
6. Aldrin	14.59	1.000
7. Heptachlor epoxide	15.96	1.094
8. Endosulfan I	17.2	1.179
9. 4,4'-DDE	18.08	1.239
10. Dieldrin	18.02	1.235
11. Endrin	18.8	1.289
* 12. Endosulfan II	19.38	1.328
* 13. 4,4'-DDD	19.1	1.309
* 14. Endrin aldehyde	20.64	1.415
* 15. Endosulfan sulfate	19.7	1.350
* 16. 4,4'-DDT	20.5	1.405

■Chromatographic conditions shown in Figures A and B.
*Indicates elution order shift compared to the SPB-608 column.
**Relative to Aldrin.

625, within the specifications of the method. And, like our SPB-608 column, our PTE-5 column resolves endrin and 4,4'-DDT from direct injection samples at on-column quantities of 1 picogram. FID and ECD bleed levels for the PTE-5 column are very low (less than 5 picoamps FID bleed at 300°C).

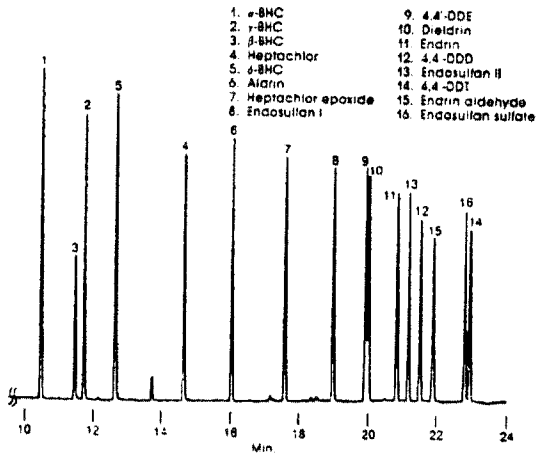
Figure C — Single Column Analysis of Chlorinated Pesticides on SPB-608 Capillary Column



209003-0009

SPB-608 fused silica capillary column, 30m x 0.25mm ID (0.25µm film thickness). Linear Velocity: 25cm/sec., He. Col. Temp.: 150°C (hold for 4 min.), 8°C/min. to 290°C (hold for 5 minutes), Det.: ECD (32 x 10⁻¹³ AFS), Inj. Vol.: 0.5µl. Sample: 16 Chlorinated Pesticides Mixture (200pg each compound).

Figure D — Single Column Analysis of Chlorinated Pesticides on PTE-5 Capillary Column



209003-0001

PTE-5 fused silica capillary column, 30m x 0.25mm (0.25µm film thickness). Linear Velocity: 25cm/sec., He., Col. Temp.: 150°C (hold for 3 min.), 5°C/min. to 290°C, Det.: ECD (2 x 10⁻¹³ AFS), Inj. Vol.: 0.5µm. Sample: 16 Chlorinated Pesticides Mixture (200pg each compound).

After selecting the columns for the primary and confirmational pesticides analyses, we saved time by performing both analyses simultaneously. To obtain effective dual column confirmational analyses — without loss of column performance — you must have similar linear velocities through the two columns. This is best accomplished with columns of equal length and diameter. We used 30-meter columns with internal diameters of 0.25mm.

Also, different temperature programs cause a shift in the retention order of at least one pair of closely eluting peaks. We achieved optimum separation of all listed pesticides, on *both* columns, with a temperature program rate of 6°C/minute. A comparison of the dual column analyses in Figures A and B with the single column analyses in Figures C and D shows no obvious deterioration in chromatographic performance.

Under the conditions we established for this dual column, single injection procedure, our SPB-608 and PTE-5 capillary columns make excellent choices for the analysis of the organochlorine pesticides listed in EPA Method 608. Different stationary phase polarities result in different elution patterns for these pesticides, ensuring a more conclusive confirmational analysis. And by performing simultaneous analyses from a single sample injection, you save precious analytical time.

PTE-5 Fused Silica Capillary Column 30m x 0.25mm ID (0.25µm film thickness)	**2-4135T**	**£359**
SPB-608 Fused Silica Capillary Column 30m x 0.25mm ID (0.25µm film thickness)	**2-4103T**	**£379**
Pesticide Mix (for Method 608 analyses)	**4-8858T**	**£38**
Supelpreme™-HC Pesticides Mix (does not contain 4,4'-DDE for Method 608)	**4-8903T**	**£81**
Supeltex™ M-2A Ferrule (two-hole, 0.4mm ID)	**2-2467T**	**£30/5**

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

Reference

1. Quality Control Protocol for the Fused Silica Capillary Column Gas Chromatography/Mass Spectrometry Determination of Semivolatile Priority Pollutants. Revision 3.1. Sept. 1981. US EPA. Las Vegas. Nevada. USA

Reference not available from Supelchem.

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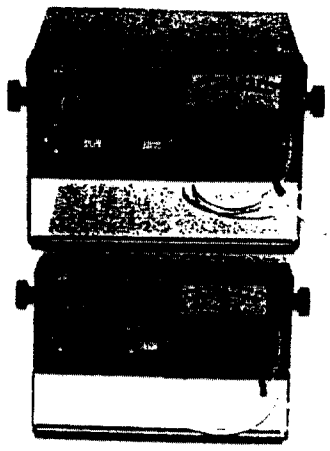

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Stop worrying about annoying, costly, or potentially dangerous gas leaks. This easy-to-use *GOW-MAC Leak Detector* will locate even minute leaks. Sensitive to helium, hydrogen, Freon®, carbon monoxide, carbon dioxide — or any gas with a thermal conductivity sufficiently different from air — the GOW-MAC Leak Detector works equally well at ambient or elevated temperatures. And it is more sensitive than liquid leak detectors.

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Capillary Cleaving Tool **2-3740T** **£42**

Analyze Chlorinated Pesticides with Two Capillary Columns

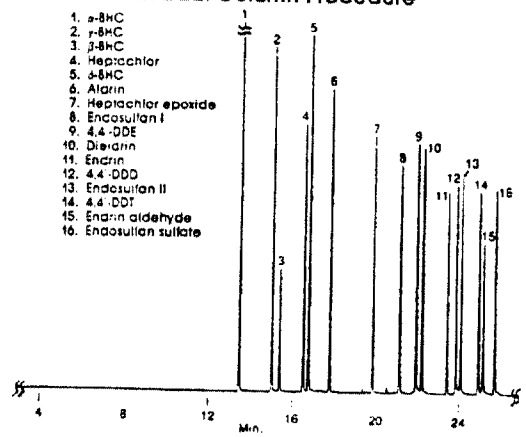
To save time when performing dual column confirmational analyses of US EPA Method 608 organochlorine pesticides, use our *30m x 0.25mm ID SPB™-608* and *PTE™-5 capillary columns*. Simply connect both columns to the same injection port with a two-hole ferrule, and use two electron capture detectors (ECDs). Using a single injection, the SPB-608 column serves as the primary analytical column, while the PTE-5 column provides the confirmational analysis.

Our SPB-608 capillary column was developed specifically for the separation of the organochlorine pesticides and PCBs listed in EPA Method 608. Under the conditions developed for our confirmational procedure, the SPB-608 column separates Method 608 pesticides in less than 30 minutes (Figure A).

Each SPB-608 column is tested with a mixture of 16 chlorinated pesticides, selected from the compounds listed in Method 608. The relative ECD response for endrin and 4,4'-DDT is also measured. Minimal breakdown of these unstable components — at an on-column level of 160 picograms —

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

Figure A — Chlorinated Pesticides Using SPB-608 Capillary Column for Primary Analysis in Dual Column Procedure



SPB-608 fused silica capillary column, 30m x 0.25mm ID (0.25μm film thickness). Linear Velocity: 25cm/sec. He at 290°C. Col. Temp.: 150° (hold 4 minutes), 6°C/min. to 290°C (hold for 5 minutes or until all 16 pesticides elute), Det.: ECD (128 x 10^{-13} AFS) at 310°C, Split Inj.: Vent Flow 65-70cc/min., Temp. 250°C, Vol. 0.5μl. Sample: 16 Chlorinated Pesticides Mixture (100pg each compound).

Iconic on page 12

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