SUPELCO[®]

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Product Information

Special Purpose SLB[™]-5ms Capillary GC Columns

A Non-Polar Column for Trace Analyses

Low levels of detection are a requirement for chromatographers in many different fields. Environmental chemists must meet stringent reporting requirements. Analysts in the pharmaceutical, food/ beverage, flavor/fragrance, and personal product industries must insure that harmful compounds are not present in consumer goods. Material scientists must fully characterize raw materials. Forensic chemists must identify accelerants in fire debris and drugs in body fluids. Analysts rely on GC-MS and other GC methods to enable highly sensitive, low level detection. When measurement is required at the ppb or even ppt level, extreme care must be taken to ensure that nothing interferes with the analysis.

When capillary GC was in its infancy, the column was not the limiting factor in determining the limit of detection. However, the other components (autosamplers, injection ports, detectors, data systems) that make up a GC system have become more sophisticated, allowing analysts to achieve lower and lower limits of detection. Using modern instrumentation, the column may in fact play a prime role in determining the lowest possible limit of detection. For this reason, today's chemists require capillary columns specifically designed to allow analysts to achieve the low detection limits specified by their applications. The Supelco Low Bleed SLB-5ms is such a column.

Low Bleed Columns, Consistently

SLB-5ms represents the 4th generation 5-type capillary GC column from Supelco. The primary design input was to consistently achieve low bleed characteristics while maintaining a selectivity close to that of the classical 5% phenyl polymethylsiloxane.

Why use a 5-type column? 1) Many existing methods for the analysis of environmental samples specify that a 5-type column be used. 2) The best approach to a new separation is to run it on a non-polar column that separates primarily by boiling point. 3) While there are other columns in the 'non-polar' range, the 5-type is considered by many as the 'best' non-polar column.

Why is low bleed important? 1) Simply stated, lower bleed results in lower detection limits. The greater the area of the peak that is above the baseline, the more counts the software observes. This is known as the signal-to-noise ratio. 2) Extraneous m/z from excessive column bleed interferes with the ability of mass spectral software to properly identify analytes and unknowns.

Why is consistency important? In today's competitive business environment, you simply cannot afford to perform method development when switching columns. Improvements result in SLB columns being highly reproducible, column-to-column.



For the user, SLB provides the values of lower detection limits, easier mass spectral identification, less instrument downtime, shorter analysis times, shorter installation times, and confidence in column choice.

SLB Improvements

Unique Advances in Polymer Synthesis: Our polymer chemists have developed a new polymer that provides unsurpassed ruggedness. Traditional methyl silicone based polymers are structured such that small volatile cyclic siloxane molecules can be released from the polymer chain and seen by the detector as column bleed. As an alternative, the SLB-5ms polymer is designed to significantly reduce column bleed through increased stability and extensive crosslinking. This crosslinking between polymer chains yields polymers that are among the most rugged we have ever seen.

Proprietary Surface Deactivation Chemistry: In order to make a good column, one must start with a good surface, so deactivation of the fused silica tubing is a very important part of the manufacturing process. Our R&D chemists have developed a proprietary formulation and procedure for superior deactivation of the fused silica tubing that we use in the manufacturing of our SLB columns. This is done to insure that the fused silica tubing is highly inert prior to bonding the most rugged polymers our polymer chemists have ever made. In addition to eliminating surface activity and improving inertness, deactivation also improves the wettability of the surface. Wettability ensures that the stationary phase film will spread evenly.

Innovative Manufacturing Processes: Our manufacturing group applied nearly three decades worth of learning into the development of this column. We have painstakingly investigated every variable in the column-making process to be able to offer our customers the best product we can. Many months were spent and many experiments were performed to refine these processes. Additionally, a very durable technique to chemically bond the phase to the fused silica surface was developed. Simply stated, we add phase to the fused silica tubing and it stays there.



SLB-5ms for Environmental Applications

Numerous environmental methods specify the use of a 5-type column for analysis. Example methodologies include semivolatiles using GC-MS, organochlorine pesticides and PCBs using GC-ECD, herbicides using GC-ECD, organophosphorus pesticides using GC-NPD, polynuclear aromatic hydrocarbons using GC-FID, and phenols using GC-FID or GC-ECD, among others.

Of these, US EPA Method 8270 for semivolatiles using GC-MS is a formidable challenge for a column because of the low detection limits required by the method and also the diverse functionality of the analytes. The ideal column should be inert enough to provide excellent peak shape and also exhibit very low bleed. The SLB-5ms capillary column was evaluated for use in performing this method. The equivalent of a daily continuing calibration standard (a mixture of 72 analytes and eight surrogates each at 50 ng on-column plus six internal standards each at 40 ng on-column) was analyzed. The resulting chromatogram is presented in Figure 1. Analytes that are not completely resolved or that co-elute are mass resolved by the MSD based on their quant ions. As evident, SLB-5ms is highly suited to perform this and other environmental applications.

SLB-5ms for Other Applications

The best approach to a new separation is to first run it on a nonpolar column that separates primarily by boiling point. This is useful since elution order can be predicted. Columns with higher polarity can then be used to assist in confirming identity. While there are other columns in the 'non-polar' range, the 5-type is considered by many as the 'best' non-polar column. The slight phenyl content adds desirable stability and selectivity compared to the more non-polar 100% methyl column.

Trace Analyses: For existing applications where a non-polar column is currently being used, switching to SLB-5ms will allow low limits of detection to consistently be achieved. This is especially true if the method being followed was developed at a time when column technology was ahead of other system components. If instrumentation has been upgraded, the column may be the deciding factor on how low the limit of detection. To get the most out of your system, consider upgrading the column.

Figure 1. Analysis of US EPA Method 8270D Semivolatiles Using SLB-5ms

column:	SLB-5ms, 30 m x 0.25 mm I.D., 0.25 μm (28471-U)
oven:	40 °C (2 min.), 22 °C/min. to 240 °C, 10 °C/min. to 330 °C, (1 min.)
inj.:	250 ℃
MSD interface:	330 ℃
scan range:	40-450 m/z
carrier gas:	helium, 1.0 mL/min. (11 min.), 10 mL/min ² to 1.5 mL/min. (hold remainder of run)
injection:	0.5 μL, splitless (0.50 min.)
liner:	2 mm I.D., straight
sample:	50 ng on-column of a 72 component semivolatile standard and 8 surrogate compounds, plus 6 internal standards (at 40 ng on-column)



Extend Column Life With Guard Columns

A decrease in peak shape quality in a capillary GC system can typically be traced to the inlet end of the column. Over time, the inlet end of the column becomes contaminated from an accumulation of nonvolatile material. The phase can also be damaged from the continuous condensation and vaporization of solvent and analytes. Inevitably, active analytes will adsorb to the contaminated / damaged section, leading to peak tailing, loss in resolution, and reduced response. When chromatography degrades to an unacceptable level, performance is restored by clipping the contaminated / damaged section off the inlet end of the column.

To extend the lifetime of capillary GC columns, Supelco recommends using a 3-5 m long guard column. A guard column is a short piece of uncoated deactivated fused silica tubing which is placed in-line between the GC injection port and the analytical column. The guard column will take the brunt of the contamination / damage. By clipping the guard column periodically to restore performance instead of the analytical column, the analytical column remains unaltered. Therefore, chromatography (retention times and resolution) is not affected.

Confirmatory Analyses With "Y" Connectors

The use of MS detection provides valuable structural data, thereby allowing the user to confirm identity with a high degree of certainty. To obtain the same confidence level in identity with non-MS applications, it is common practice to analyze the sample on two columns, each of different polarity. This can most easily be accomplished by splitting the sample downstream of the injection port into two columns, each of which is connected to a separate detector. One injection results in two analyses.

To do this, a short 3-5 m length of uncoated deactivated guard column is placed in-line between the injection port and the "Y" connector. Each column is attached to a free leg of the "Y" connector. For performing analyses per US EPA Methodologies for pesticides, PCBs, and herbicides, a possible arrangement would be a 5-type column and a 1701-type column, each connected to a separate ECD. For research needs, it is possible for the two detectors to be of different types. That is, one column going to a FID and one column going to an ECD. In this case, the columns can be of the same or different polarity.

- N-nitrosodimethylamine 1.
- 2. Pyridine
- 3. 2-fluorophenol (surr.)
- 4. Phenol-d₆ (surr.)
- 5. Phenol
- 6. Aniline
- Bis(2-chloroethyl)ether 7. 8.
- 2-chlorophenol-d₄ (surr.) 2-chlorophenol 9.
- 1.3-dichlorobenzene 10.
- 1.4-dichlorobenzene-d₄ (I.S.) 11
- 1.4-dichlorobenzene 12.
- 13. Benzyl alcohol
- 14
- 1,2-dichlorobenzene-d₄ (surr.)
- 15. 1.2-dichlorobenzene 16. 2-methylphenol
- 17 Bis(2-chloroisopropyl)ether
- 18. N-nitroso-di-n-propylamine
- 19. 4-methylphenol
- 20 Hexachloroethane
- 21. Nitrobenzene-d₅ (surr.)

72,73

- 22. Nitrobenzene
- 23. Isophorone
- 24. 2-nitrophenol
- 2.4-dimethylphenol 25.
- 26 Bis(2-chloroethoxy)methane
- Benzoic acid 27. 2.4-dichlorophenol
- 28. 1,2,4-trichlorobenzene 29.
- 30. Naphthalene-d₈ (I.S.)
- Naphthalene 31.
- 32 4-chloroaniline
- 33. Hexachlorobutadiene
- 34. 4-chloro-3-methylphenol
- 35
- 2-methylnaphthalene Hexachlorocyclopentadiene 36.
- 37. 2,4,6-trichlorophenol
- 38 2,4,5-trichlorophenol
- 39 2-fluorobiphenyl (surr.)
- 40. 2-chloronaphthalene
- 41 2-nitroaniline
- 42.
- Dimethyl phthalate

43. 2,6-dinitrotoluene

44. Acenaphthylene

- 45. 3-nitroaniline
- Acenaphthene-d₁₀ (I.S.) 46.
- 47 Acenaphthene
- 48. 2,4-dinitrophenol
- 4-nitrophenol 49.
- Dibenzofuran 50.
- 51. 2,4-dinitrotoluene
- Diethyl phthalate 52.
- 53 4-chlorophenyl phenyl ether
- 54. Fluorene
- 55. 4-nitroaniline
- 56
- 2-methyl-4,6-dinitrophenol N-nitrosodiphenylamine 57.
- 58 Azobenzene
- 59
- 2,4,6-tribromophenol (surr.) 60
- 4-bromophenyl phenyl ether
- 61. Hexachlorobenzene
- 62 Pentachlorophenol
- 63. Phenanthrene-d₁₀ (I.S.)

- 64. Phenanthrene
- 65. Anthracene
- 66 Carbazole
- Di-n-butyl phthalate 67.
- 68. Fluoranthene
- Benzidine 69.
- 70. Pvrene
- 71. Terphenyl-d₁₄ (surr.)
- 3.3'-dimethylbenzidine 72.
- Butylbenzyl phthalate 73.
- 74. 3.3'-dichlorobenzidine
- Benzo(a)anthracene 75.
- 76. Bis(2-ethylhexyl)phthalate
- Chrysene-d₁₂ (I.S.) 77.
- Chrysene 78.
- Di-n-octyl phthalate 79.
- Benzo(b)fluoranthene 80.
- Benzo(k)fluoranthene 81.
- 82. Benzo(a)pyrene
- 83. Perylene-d₁₂ (I.S.)
- 84 Indeno(1,2,3-cd)pyrene
- 85. Dibenzo(a,h)anthracene
- 86. Benzo(g,h,i)perylene



Ordering Information:

SLB-5ms

Phase: bonded and highly crosslinked; silphenylene polymer virtually equivalent in polarity to 5% phenyl polymethylsiloxane

Temp. Limits: 0.10 to	o 0.32 mm I.D.: -60 °C	to 340 °C	(isothermal)
	-60 °C	to 360 °C	(programmable)
	0.53 mm I.D.: -60 °C	to 330 °C	(isothermal)

.53 mm I.I	⊃.: -60 °C	to 330 °	C (isothermal)
	-60 °C	to 340 °	C (programm	able)

Length (m)	d _f (µm)	Beta	Cat. No.	
0.10 mm I.D. Fused Silic	0.10 mm I.D. Fused Silica			
10	0.10	250	28465-U	
15	0.10	250	28466-U	
0.18 mm I.D. Fused Silic	a			
20	0.18	250	28564-U	
12	0.30	150	28566-U	
30	0.30	150	28575-U	
20	0.36	125	28576-U	
0.20 mm I.D. Fused Silic	a			
30	0.20	250	28513-U	
0.25 mm I.D. Fused Silic	a			
30	0.10	625	28467-U	
15	0.25	250	28469-U	
30	0.25	250	28471-U	
60	0.25	250	28472-U	
15	0.50	125	28577-U	
30	0.50	125	28473-U	
60	0.50	125	28474-U	
30	1.0	63	28476-U	
0.32 mm I.D. Fused Silica				
15	0.25	320	28557-0	
30	0.25	320	28482-U	
30	0.32	250	28532-0	
15	0.50	160	28597-U	
30	0.50	160	28484-0	
30	1.0	80	28487-0	
0.53 mm I.D. Fused Silic	a	265	205 42 11	
15	0.50	265	28542-0	
30	0.50	265	28541-0	
30	1.0	132	28559-0	

GlasSeal[™] Capillary Column "Y" Connectors

Use a GlasSeal "Y" connector to split a sample to two columns for confirmatory analysis. Silanized for an inert inside surface, these can be used with our 0.10-0.53 mm I.D. tubing. To make this an extremely durable connection, use a small drop of polyimide sealing resin (cure at 200 °C, maximum temperature 350 °C).

Description	Cat. No.
Borosilicate Glass, each	20480
Fused Silica, each	23631
Fused Silica, pack of 3	23632
Polyimide Sealing Resin, 5 g	23817

Fused Silica Guard Columns

For use as a guard column to protect your analytical column from damaging sample components. Match the deactivation of the tubing with the polarity of the injection solvent.

Deactivation Non-Polar Intermediate Polarity Polar	Injection Solvents Alkanes, carbon disulfide, ethers Acetone, methylene chloride, toluene Acetonitrile, methanol, water		Max. Temp 360 °C 360 °C 260 °C	
Length (m)	I.D.	Cat. No.		
Non-Polar Deactivation				
3	0.25	25722		
5	0.25	25742		
3	0.32	25723		
5	0.32	25743		
Intermediate Polarity Deactivation				
3	0.25	25727		
5	0.25	25747		
3	0.32	25728		
5	0.32	25748-U		
Polar Deactivation				
5	0.32	25752-U		

Capillary Column Butt Connector



This device consists of a double-tapered ferrule and a stainless steel compression housing with a threaded cap. Small and light (2.3 cm x 0.6 cm, 4.4 g with ferrule), it provides a gas tight seal. This unit maintains inertness with no change in column efficiency.

Description	Cat. No.
Capillary Column Butt Connector, body only	23804
Supeltex [™] M-2B Ferrules, pack of 2	
To connect 0.10/0.25 mm I.D. to 0.10/0.25 mm I.D.	22453
To connect 0.32 mm I.D. to 0.32 mm I.D.	22454
To connect 0.53 mm l.D. to 0.53 mm l.D.	22591
To connect 0.10/0.25 mm I.D. to 0.53 mm I.D.	22455-U
To connect 0.32 mm l.D. to 0.53 mm l.D.	22586

Trademarks

GlasSeal, SLB, Supelco, Supeltex — Sigma-Aldrich Co.

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