Bulletin 875C

Supelco Capillary GC Columns

Chemical structures, polarities, operating temperature ranges, and chemical compatibilities of Supelco™ general-purpose capillary GC columns and columns for specific clinical, environmental, food/beverage, and petroleum/chemical applications are summarized in this bulletin. This information should simplify the process of choosing the best column for a particular purpose.

Key Words:

- capillary GC columns capillary GC phases
- column polarity capillary column characteristics

How to Choose a Column

Column selection is based on five primary factors: sample, stationary phase type, column ID and stationary phase film thickness (which are interrelated), and column length. The practical effects of these factors on the performance of a capillary column are discussed briefly on the next few pages. Remember that this information is general; specific situations may be exceptions to these guidelines.

This reference material should make choosing the best capillary column for your purpose an easier process. If you have any questions, our Technical Service chemists can recommend the appropriate capillary column for a given sample type and separation.

Sample and Stationary Phase

The stationary phase is a polymeric film coated on the inner wall of the capillary column. Differences in the chemical and physical properties of the injected organic compounds and their interactions with the stationary phase are the basis for the separation process.

Interactions between the sample components and the stationary phase vary as a function of the properties of the analyzed compounds. When the energy of the analyte-phase interaction differs significantly for two compounds one is retained longer than the other. How long they are retained in the column (retention time) is a measure of the analyte-stationary phase interaction.

Changing the chemical features of the polymeric stationary phase alters its physical properties. Two compounds that do not separate (coelute) on a particular stationary phase might separate on another phase of a different polarity, if the difference in the analyte-phase interactions is significant. This is the basis for providing a variety of capillary column phases – each phase provides a specific combination of interactions for each class of chemical analytes.

The stationary phase also dictates the minimum and maximum temperatures at which a column can be used.

Phase Type

Phase Polarity Choosing the stationary phase is usually the most important choice in selecting a column. The single most important characteristic of the phase is polarity, because it dictates selectivity, or the ability of the column to separate sample components. Phase selection is based on the general chemical principle that "like dissolves like." A nonpolar column is best for analyses of nonpolar compounds. Polar columns most effectively separate polar compounds.

Nonpolar molecules are generally composed only of carbon and hydrogen atoms and contain carbon-carbon single bonds. Normal hydrocarbons (n-alkanes) are the most common nonpolar molecules analyzed by capillary gas chromatography. These compounds are well separated by nonpolar capillary columns (see Table 1). Interactions between nonpolar compounds and a nonpolar phase are dispersive, that is, the compounds enter into and exit from the phase film at random. Thus separation is based exclusively on the boiling points of the molecules.

Table 1. Sample Type/Capillary Phases

Sample Type	Examples	Phases
Nonpolar Molecules C and H atoms only, C – C bonds	normal hydrocarbons (n-alkanes)	SPB-Octyl, SPB-1, SP-2100, SE-30, SPB-5, PTE-5, SE-54
Polar Molecules Primarily C and H atoms, also contain Br, Cl, F, N, O, P, S	alcohols, amines, carboxylic acids, diols, esters, ethers, ketones, polychlorinated biphenyls, thiols	SPB-1301, SPB-20, SPB-35, SPB-50, SPB-1701, SP-2250, PAG, Nukol, SP-1000, SUPELCOWAX 10, CARBOWAX 20M
Polarizable Molecules C and H atoms only, C=C or C=C bonds	alkenes, aromatic hydrocarbons	SP-2330, SP-2331, SP-2380, SP-2340, TCEP

For more information, see column listings (pages 4-5, 10-28).

Polar molecules are composed primarily of carbon and hydrogen atoms, but also contain one or more atoms of bromine, chlorine, fluorine, nitrogen, oxygen, phosphorus, or sulfur. Polar compounds typically analyzed by capillary GC include alcohols, amines, carboxylic acids, diols, esters, ethers, ketones, polychlorinated biphenyls, and thiols. Polarizable molecules are composed of carbon and hydrogen, but contain one or more double or triple carbon-carbon bonds. Polarizable molecules include alkenes and aromatic (benzene ring-containing) hydrocarbons. Polar and polarizable compounds are generally well separated by intermediate polarity-polar capillary columns (see Table 1). In



addition to dispersive interactions, interactions between polar molecules and polar phases include dipole and/or acid-base interactions. Separations are determined by differences in the overall effects of these interactions.

Bonded or Nonbonded Phase Bonded phases are immobilized/ chemically bound (crosslinked) within the tubing, while nonbonded phases are simply coated on the wall. Generally a bonded phase is preferred, because it will bleed less during use, can be used to higher temperatures and, when necessary, can be rinsed with solvents to remove accumulated nonvolatile materials. When a bonded phase is not available, such as for the high polarity phases, look for a stabilized phase. Stabilized phases are not as permanent as bonded phases (they cannot be rinsed), but they have greater thermal stability than nonbonded phases. Supelco offers a stabilized high polarity phase (SP-2380).

Chemical structures, polarities, operating temperature ranges, and chemical compatibilities of Supelco capillary GC phases are described on pages 4 and 5 and 10 through 28.

Column ID

The current range of commercially available capillary column inside diameters enables you to balance two factors: efficiency (number of theoretical plates) and sample capacity (the amount of any one sample component that can be applied to the column without causing the desired sharp peak to overload). Narrower bore columns (0.10mm-0.32mm ID) provide the best resolution (highest efficiency), while wider bore columns (0.53mm and 0.75mm ID) provide the greatest sample capacity (Table 2). The nature of the sample component and phase affect sample capacity: nonpolar phases have higher capacities for nonpolar analytes, polar phases have higher capacities for polar analytes. Temperature programming increases the sample capacity of a column, if you are willing to sacrifice some of the column's resolving ability. You must also consider the characteristics of your detector: special-purpose detectors often enable you to use smaller samples; without a jet separator, a mass spectrometer usually cannot accept the high gas flows from columns larger than 0.20mm-0.25mm ID.

Table 2. Internal Diameter Affects Characteristics of GC Columns[▲]

Internal Diameter	Sample Capacity (ng) (each component)	Efficiency (theoretical plates/meter)	Optimum Flow Rate (cc/min)*
0.10mm	5-10	7000*	0.18 (He)/ 0.37(H ₂)●
0.20mm	5-30	5000	0.4
0.25mm	50-100	4170	0.6
0.32mm	400-500	3330	1.0
0.53mm	1000-2000	1670	2.8
0.75mm	10,000-15,000	1170	5.6
2mm (packed) 20,000	2000	20

60-meter SPB-1 capillary columns, 2-meter SE-30 packed column.

0.25mm & 0.32mm ID columns: 0.25µm film

0.53mm & 0.75mm columns: 1.0µm film

Carrier Gas: helium (except as noted)

Column Temperature: optimized for approx. equal k' values

(145°C-165°C) cample: isothermal test mix

(straight chain hydrocarbons and active compounds)

If a sample contains many components, or if you expect some components to elute closely (e.g., isomers), you may be compelled to use a 0.10mm-0.32mm ID column for maximum resolution. These columns also require the use of specialized capillary equipment, i.e., a sample splitting device and a gas flow controller that reliably controls carrier gas flow at low rates.

If sample components are in high concentrations, or represent a wide range of concentrations, a 0.53mm or 0.75mm ID column will be best. It will provide sufficient sensitivity for you to obtain peaks for the minor sample components, without being overloaded with the major components. In most analyses, the loss of resolution involved in using a wider bore column will not be important. In addition wide bore columns, particularly 0.75mm ID columns, often can be used without sample splitting and with flow control devices typically used with packed GC columns. Thus, with simple modifications to the injection and detector ports, packed column chromatographs are compatible with these capillary columns.

Stationary Phase Film Thickness

Increasing the film thickness generally increases peak width (reduces column efficiency), increases analyte retention times (may also increase resolution) and reduces sample interaction with the tubing wall. Increasing film thickness also increases the maximum sample capacity, and the temperature at which a sample component will elute from the column. Increasing film thickness reduces the upper temperature limit, because column bleed is greater.

In general thin film columns (0.10 μ m-0.25 μ m) are used for analyses of relatively simple samples or for analytes with high boiling points (>300°C). Compounds elute at lower temperatures and shorter retention times from thin film columns. Thicker film columns (1 μ m-5 μ m) are best suited for analytes with low boiling points (e.g., volatile organics and gases). Thick film columns increase retention of highly volatile compounds, thereby eliminating the need for cryogenics. The higher sample capacity of thick film columns reduces sample overloading (peaks that broaden and front) for highly concentrated components.

Phase Ratio (Beta)

Effects of phase film thickness are interdependent with column ID. The phase ratio, beta (β), expresses the ratio of the gas volume and the stationary phase volume in a column:

$$\beta = \frac{\text{column radius (µm)}}{2 \text{ x phase film thickness (µm)}} = \frac{r}{2d_r}$$

Beta values for various combinations of column ID and phase film thickness are listed in Table 3. Consult this table when you wish to change column ID or phase film thickness for a particular analysis, because columns with the same phase ratio will provide very similar retention times and elution order under the same analytical conditions, as shown in Figure A.

In contrast to the relative terms "thick film" and "thin film", β values establish a distinct ranking for columns. The rule to remember for selecting columns by β values is **columns with lower \beta values increase sample capacity and increase component retention.** Some guidelines for selecting columns by β values are given in Table 4.

[◆]For comparative purposes only — all values are approximate.

^{*}Corresponds to 21cm/sec linear velocity (optimum for helium carrier gas).

^{*}Based on k' = 6.0.

^{•15-}meter column, 0.10µm film.

Volatile organic compounds (VOCs) generally have low retention times on general-purpose columns ($\beta=100\text{-}400$). The rule for selecting a column by β indicates that a small β value is needed to increase retention times. VOCOL columns with dimensions of 0.53mm ID x 3.0µm film and 0.25mm ID x 1.5µm film ($\beta=44$) were developed specifically for separating these compounds under ambient temperature conditions.

Table 3. Capillary Column Phase Ratios and Sample Capacities

Column ID (mm)	Film Thickness (µm)	Phase Ratio (β)	Sample Capacity (ng/analyte)
•	0.10	500	10-20
0.20	0.20	250	30-40
	0.80	63	200-300
•	0.10	625	30-40
	0.25	250	100-150
0.25	0.50	125	200-300
	1.0	63	400-500
	2.0	31	700-800
	0.10	800	50-70
	0.25	320	100-200
0.32	0.50	160	200-300
	1.0	80	400-500
	2.0	40	700-900
	4.0	20	1500-2000
	0.10	1325	50-100
	0.25	530	200-300
	0.50	265	500-700
0.53	1.0	133	1000-1500
	1.5	88	1500-2000
	3.0	44	4000-5000
	5.0	27	8000-10,000

Figure A. Columns with Different ID and Phase Film Thickness, But Equal Beta Value, Provide Similar Elution Patterns

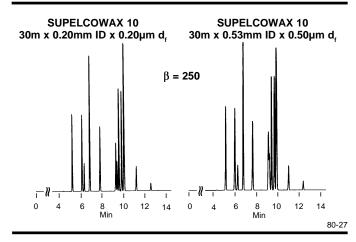


Table 4. Selecting a Column, Using Phase Ratio (β)

β	Sample Components
<100	highly volatile, low molecular weight compounds
100-400	general-purpose analyses, wide range of compounds
>400	high molecular weight compounds

Columns with large β values (β >400) reduce the retention factor and elution temperature for high molecular weight compounds. These columns also generally exhibit lower bleed and have lower sample capacities.

Column Length

A longer column will provide greater resolution than a shorter column. However, there are practical limits to increasing column length: in isothermal analyses a 60-meter column increases resolution by 40%, relative to a 30-meter column (resolution increases according to the square root of the column length), but doubles the analysis time and increases the pressure required to move the sample through the column. A 60-meter column also costs more than a 30-meter column.

Generally a 30-meter column provides the best balance between resolution and analysis time. Use a 15-meter column for screening analyses or for simple samples whose components are dissimilar in chemical nature. Use a 60-meter column when samples are complex or volatile (e.g., gases at room temperature), when you expect some components to elute very closely together (e.g., isomers), or when you can use temperature pro-gramming to minimize the increase in analysis time. For analyzing these difficult samples, however, a 30-meter column with a thicker phase film is often as useful as a 60-meter column. For certain extremely demanding separations, such as analyses of fatty acid methyl esters or petroleum product components, special purpose 100-meter or longer columns are available. Use these columns only when you need extreme resolving ability — longer columns also reduce the optimum linear velocity for an analysis.

Table 5 shows the effects of column length on various performance and operating parameters of 0.25mm ID columns.

Table 5. Performance and Operational Parameters of 0.25mm ID Columns of Different Lengths

Column Length (m)	Plate Number (N)	nC13 Retention Time⁴ (min)	Optimum Gas Velocity (cm/sec, He)	Inlet Pressure (psig)	k ['] (C13)
30	155,000	14.3	23	18	5.8
60	304,000	41.3	19	28	5.5
120	550,000	79.7	17	53	5.6
150	719,000	136.0	14	57	6.3

SPB-5 columns, 0.25mm ID x 1.0 μ m film (β = 62.5), Col. Temp.: 145°C $^{4}k'$ = 6

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Meridian GC/MS Capillary Columns

Meridian columns . . . solving your most difficult separation problems

Supelco has culminated thirty years of experience in gas chromatography with the introduction of this new family of capillary columns. Meridian $^{\text{TM}}$ capillary columns are designed specifically for GC/MS. These columns offer low bleed, inertness and improved thermal stability for all of your capillary calibration challenges.

MDN-1

Nonpolar methylsilicone columns that separate analytes according to boiling point. The bonded polymer matches the polarity of its nonbonded predecessors: SE-30, SP-2100, and SPB-1. Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCl in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed. (Figure B.)

Phase: bonded; poly(dimethylsiloxane)

Temp. Limits: -60°C to 320°C

Similar Phases: SPB-1, DB-1, ULTRA-1, RTx-1, CP-SIL-5CB

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24258
30	1.0	63	24259
0.32mm ID Fuse	ed Silica		
30	0.25	320	24299
30	1.0	80	24300-U

MDN-5

The low phenyl content (5%) improves the thermal stability of the phase, while still providing essentially a boiling point elution order, and a slight increase in selectivity, especially for aromatic compounds. Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCl in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed. (Figure C.)

Phase: bonded; poly(5% diphenyl/95% dimethylsiloxane)

Temp. Limits: -60°C to 320°C

Similar Phases: DB-5MS, HP-5MS, PTE-5, XTI-5

Length (m)	d _, (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24096
0.32mm ID Fuse	ed Silica		
30	0.25	320	24097
30	0.32	250	24203
30	1.0	80	24204-U

MDN-5S

These nonpolar columns feature very low bleed, and excellent inertness for active compounds. Meridian 5S columns feature silphenylene phase technology that provides chromatography similar to 5% phenylsiloxane phases with less bleed, improved thermal stability and inertness. Columns can be rinsed.

Phase: bonded (5% phenyl)methylpolysiloxane

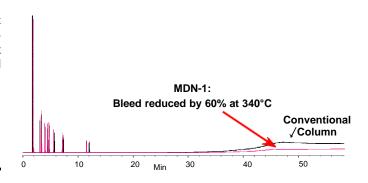
Temp. Limits: 0.25 and 0.32mm ID: -60°C to 325/350°C

0.53mm ID: -60°C to 300/320°C

Similar Phases: DB-5MS, HP-5 trace analysis

Length (m)	d _ε (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.10	625	24378
15	0.25	250	24377-U
30	0.25	250	24384
60	0.25	250	24392
30	0.50	125	24379
30	1.0	250	24385-U
0.32mm ID Fuse	ed Silica		
30	0.25	320	24386
60	0.25	320	24394
30	0.50	160	24393
30	1.0	80	24387-U
0.53mm ID Fuse	ed Silica		
30	1.5	88	25474

Figure B. Bleed Profile for an MDN-1 Column



100% bonded poly(dimethylsiloxane) phases Temp. Program: 110°C (15 min) to 340°C at 10°C/min

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Meridian GC/MS Capillary Columns

MDN-12

Low bleed, low to intermediate polarity, and unique selectivity are features of MDN-12 columns. These Meridian columns are ideal for confirmational analyses and for separating active compounds, pesticides, herbicides, PCBs, and PAHs. They are certified for MS and can be rinsed.

Phase: bonded and crosslinked; proprietary

Temp. Limits: 30°C to 340/360°C

Similar Phase: DB-XLB

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24388
60	0.25	250	24396
30	0.50	125	24395
30	1.0	250	24389
0.32mm ID Fuse	ed Silica		
30	0.25	320	24390-U
60	0.25	320	24398
30	0.50	160	24397
30	1.0	80	24391

MDN-35

These intermediate polarity columns are ideal for confirmational analyses, and have exceptional inertness for active compounds. They are certified for MS. Like other Meridian columns, MDN-35 columns offer very low bleed. Columns can be rinsed.

Phase: bonded (35% phenyl) methylpolysiloxane

Temp. Limits: 50°C to 340/360°C

Similar Phases: DB-35MS

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
30	0.25	250	24382-U
0.32mm ID Fus	ed Silica		
30	0.25	320	24383-U

Figure C. Triazine Pesticides from Apples

Stationary Phases: MDN-5, 30m x 0.25mm ID, 0.25µm phase film

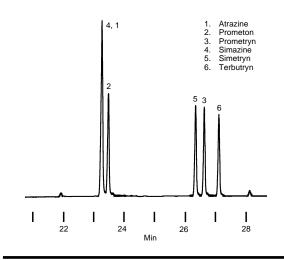
Cat. No.: 24096

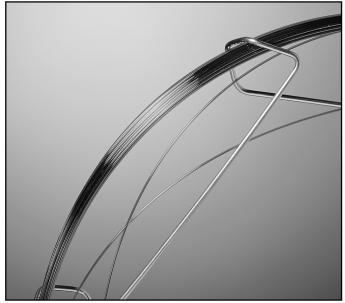
Oven: 80°C (1 min) to 280°C at 6°C/min

Carrier: helium, 40cm/sec. Det.: thermal specific, 250°C

Sample: 1µL of 10µg/mL extract, split/splitless 45 sec, 200°C

795-0842





994-0058

Special Purpose Columns

Stationary Phase	Operating Temperature	Dimensions	Cat. No.	
Food/Beverage				
Omegawax [™] 250 Omegawax 320 Omegawax 530	50° to 280°C 50° to 280°C 50° to 280°C	30m x 0.25mm ID, 0.25µm film 30m x 0.32mm ID, 0.25µm film 30m x 0.53mm ID, 0.5µm film	24136 24152 25374	
SAC™-5	-60°C to 320°C	30m x 0.25mm ID, 0.25µm film	24156	
SP™-2560 SP-2380 SPB™-PUFA	subamb. to 250°C subamb. to 275°C 50°C to 220°C	100m x 0.25mm ID, 0.2µm film 100m x 0.25mm ID, 0.20µm film 30m x 0.25mm ID, 0.2µm film 30m x 0.32mm ID, 0.2µm film	24056 24317 24314 24323	
Environmental				
PTE™-5	-60° to 320°C	30m x 0.25mm ID, 0.25µm film 30m x 0.32mm ID, 0.25µm film 30m x 0.32mm ID, 0.32µm film 30m x 0.32mm ID, 1.0µm film	24135-U 24143 24214 24159	
PTE-5 QTM	subamb. to 320°C	15m x 0.53mm ID, 0.5μm film	25355	
SP-2331 ^▲	subamb. to 275°C	30m x 0.25mm ID, 0.2µm film 60m x 0.25mm ID, 0.2µm film 60m x 0.32mm ID, 0.2µm film	24257 24104-U 24105-U	
SPB-608	subamb. to 300°C	30m x 0.25mm ID, 0.25µm film 15m x 0.53mm ID, 0.5µm film 30m x 0.53mm ID, 0.5µm film	24103-U 25310-U 25312	
SPB-624	subamb.to 250°C (1.4μm film or 1.8μm film) or 230°C (3.0μm film)	30m x 0.25mm ID, 1.4µm film 60m x 0.25mm ID, 1.4µm film 60m x 0.32mm ID, 1.8µm film 30m x 0.53mm ID, 3.0µm film 75m x 0.53mm ID, 3.0µm film	24255 24256 24251 25430 25432	
SPB-Octyl	-60° to 280°C	60m x 0.25mm ID, 0.25μm film	24219-U	
Sup-Herb™	subamb. to 300°C	15m x 0.53mm ID, 0.5µm film	25322	
VOCOL™	subamb. to 250°C (1.5μm film) or 230°C (3μm film)	30m x 0.25mm ID, 1.5µm film 60m x 0.25mm ID, 1.5µm film 60m x 0.32mm ID, 3.0µm film 30m x 0.53mm ID, 3.0µm film 60m x 0.53mm ID, 3.0µm film 105m x 0.53mm ID, 3.0µm film	24205-U 24154 24157 25320-U 25381 25358	
Petrochemical				
Carbowax® Amine PTA-5 Petrocol™ DH	60° to 220°C -60°C to 320°C -60° to 320°C	15m x 0.53mm ID, 1.0µm film 30m x 0.53mm ID, 1.0µm film 60m x 0.53mm ID, 1.0µm film 30m x 0.25mm ID, 0.5µm film 100m x 0.25mm ID, 0.5µm film	25352 25353 25354 24277 24160-U	
Petrocol DH 50.2	-60° to 320°C	50m x 0.20mm ID, 0.5µm film	24160-0 24133-U	
Petrocol DH 150 Petrocol DH Octyl	-60° to 320°C -60°C to 220°C	150m x 0.25mm ID, 1.0μm film 100m x 0.25mm ID, 0.5μm film	24155 24282	

^{▲▲} Stabilized phase

Developed for	Other Applications
C10 - C24+ fatty acid methyl esters by chain length/degree of unsaturation	nitrosamines, flavors & fragrances
sterols, glycerides	
C10 - C24+fatty acid methyl esters positional and geometric fatty acid methyl esters polyunsaturated fatty acid methyl esters	
semivolatile pollutants (US EPA Methods 525, 625.5, 1625)	PCBs, phenols, amines, pesticides, herbicides, anilines, flavorings/foods, solvents in air
semivolatile pollutants at hazardous waste sites ("quick turnaround" methods)	
TCDD isomers	
priority pollutant chlorinated pesticides (US EPA Methods 508, 608, 8081, 8082)	PCBs, herbicides, organophosphorous and pyrethroid pesticides
volatile organic compounds	
polychlorinated biphenyls (PCBs)	detailed hydrocarbon analyses; solvents
nitrogen-containing herbicides	pesticides
volatile priority pollutants (VOCs) (US EPA Methods 502, 602, 8240)	
amines/amino-containing pharmaceuticals and other basic compounds,	volatile basic compounds
amines and other volatile basic analytes complex hydrocarbon mixtures (e.g., gasoline) ASTM D5441 hydrocarbons (gases, complex mixes) ASTM D5134, ASTM D5441 hydrocarbons, gases, complex mixes, ASTM D5441 petroleum products	solvent purity, impurities in monomers and additives

(continued on next page)

Special Purpose Columns

Stationary Phase	Operating Temperature	Dimensions	Cat. No.	
Petrocol 2887 (5" cage)	subamb. to 350°C	5m x 0.53mm ID, 0.5µm film	25323	
Petrocol EX2887	subamb. to 380°C	5m x 0.53mm ID, 0.1µm film	25337	
SPB-1	-60° to 320°C	10m x 0.53mm ID, 0.5μm film	25313	
SPB-1	-60° to 320°C	30m x 0.32mm ID, 2.0µm film	24215-U	
SPB-1 Thin Film	-60° to 350°C	15m x 0.53mm ID, 0.1µm film	25360	
		30m x 0.53mm ID, 0.1µm film	25361	
SPB-1 SULFUR	-60° to 320°C	30m x 0.32mm ID, 4.0µm film	24158	
TCEP	subamb. to 145°C	60m x 0.25mm ID, 0.44µm film	24153	
		60m x 0.32mm ID, 0.51µm film	24161	
Pharmaceutical				
α-DEX™ 120	30° to 230°C	30m x 0.25mm ID, 0.25µm film	24310	
β-DEX 110	30° to 230°C	30m x 0.25mm ID, 0.25µm film	24301	
		60m x 0.25mm ID, 0.25µm film	24302	
β-DEX 120	30° to 230°C	30m x 0.25mm ID, 0.25µm film	24304	
		60m x 0.25mm ID, 0.25µm film	24305-U	
γ-DEX 120	30° to 230°C	30m x 0.25mm ID, 0.25µm film	24307	
α-DEX 225	30° to 230°C	30m x 0.25mm ID, 0.25µm film	24311	
β-DEX 225	30° to 230°C	30m x 0.25mm ID, 0.25µm film	24348	
γ-DEX 225	30° to 230°C	30m x 0.32mm ID, 0.25µm film	24312	
α-DEX 325	30° to 230°C	30m x 0.32mm ID, 0.25µm film	24303	
β-DEX 325	30° to 230°C	30m x 0.25mm ID, 0.25µm film	24308	
γ-DEX 325	30° to 230°C	30m x 0.25mm ID, 0.25μm film	24306	
OVI-G43	-20° to 260°C	30m x 0.53mm ID, 3.0µm film	25396	

Column Dimensions

Actual ID (mm)	OD (mm)	Cage Diameter
0.094-0.106	0.288-0.312	6"/15cm
0.188-0.212	0.335-0.365	6"/15cm
0.238-0.262	0.335-0.365	6"/15cm
0.308-0.332	0.410-0.450	8"/20cm
0.530-0.544	0.640-0.690	8"/20cm
0.670-0.830	0.875-0.925	8"/20cm
	(mm) 0.094-0.106 0.188-0.212 0.238-0.262 0.308-0.332 0.530-0.544	(mm) (mm) 0.094-0.106 0.288-0.312 0.188-0.212 0.335-0.365 0.238-0.262 0.335-0.365 0.308-0.332 0.410-0.450 0.530-0.544 0.640-0.690

[•]Depends on thickness of outside protective coating.

Capillary Starter Kits

Novice capillary chromatographers and experienced chromatographers contending with new samples of unknown nature (e.g., unknown polarity) can experiment with columns of low, intermediate, and high polarity by using these kits. We recommend the 0.25mm ID column kit for experienced capillary chromatographers. The 0.53mm ID kit is ideal for novices and for experienced analysts who suspect analytes in their samples might be present in a very wide range of concentrations.

Ordering Information:

Description	Cat. No.
0.25mm ID Capillary Starter Kit	
30m x 0.25mm ID, 0.25 μ m film fused silica columns, one each: PTE-5, SPB-50, and SUPELCOWAX TM 10	24142
0.53mm ID Capillary Starter Kit	
30m x 0.53mm ID, 0.5µm film fused silica columns,	
one each: SPB-1, SPB-50, and SUPELCOWAX 10	25377

Develope	d for	Other Applications
SIMDIS SIMDIS	(simulated distillation) (ASTM Method D2887) extended ASTM Method D2887) es in gasoline (ASTM D4815)	fingerprinting for forensics; solvents, general purpose nonpolar column general purpose nonpolar column
sulfur co	mixtures, h temperature analyses mpounds s or in complex hydrocarbon mixes)	triglycerides, other high temperature analyses stack gas analysis, light hydrocarbons (gases)
	s in mineral spirits es in gasoline (ASTM D4815)	
chiral co	ral compounds, positional isomers mpounds, positional isomers mpounds, positional isomers	solvents in air flavors & fragrances, aromatics, semivolatiles flavors & fragrances, aromatics, semivolatiles
chiral con chiral con chiral con chiral con chiral con chiral con chiral con	mpounds, positional isomers	flavors & fragrances, aromatics, semivolatiles solvents

Sigma-Aldrich Trademarks

CapSeal Bullet, Carbopack, Carboxen, CLOT, DEX, Fluorcol, GlasSeal, Meridian, Nukol, Omegawax, Petrocol, PTE, SAC, SP, SPB, Sup-Herb, Supelco, Supel, SUPELCOWAX, Supeltex, Thermogreen, VOCARB, VOCOL

Other Trademarks

Apiezon - Biddle Instruments

Balan, Paarlan, Treflan – Eli Lilly & Co.

 $\label{eq:Bentone-National Lead Co., Baroid Sales Div.}$ Bentone – National Lead Co., Baroid Sales Div.

Carbowax – Union Carbide Corp.

Dexsil – Dexsil Chemical Corp.

Dual, Tolban – Ciba-Geigy AG

Eptam, Ordram, Ro-Neet, Sutan, Tillam, Vernam – Stauffer Chemical Co.

GOAL – Rohm and Haas Co.

Hamilton - Hamilton Co.

Mininert - Precision Sampling Corp.

OV – Ohio Valley Specially Chemical Co.

Pluronics - BASF

Porapak - Waters Associates

Prowl - American Cyanamid Co.

Sencor - Bayer AG

Teflon – E.I. du Pont de Nemours & Co., Inc.

Tekmar – Tekmar Co.

UCON - Union Carbide Corp.

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

Special Purpose Columns: Enantiomers/Positional Isomers

α-DEX 120

The chiral stationary phase in α -DEX columns contains permethylated α -cyclodextrin embedded in an intermediate polarity stationary phase. The columns provide unique selectivity for the enantiomeric separation of small molecules; also recommended for separating positional isomers (phenols, xylenes, etc.).

Phase: nonbonded; 20% permethylated $\alpha\text{-cyclodextrin}$ in

SPB-35 poly(35% phenyl/65% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

McReynolds Nos.: x' y' z' u' s' = 102 243 142 221 170

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		_
30	0.25	250	24310

γ-DEX 120

The chiral stationary phase in γ -DEX columns contains 20% permethylated γ -cyclodextrin embedded in an intermediate polarity stationary phase. Because the elution order of the members of a chiral pair frequently reverses (enantioreversal) on a γ -DEX column, compared to the elution order on an α -DEX or β -DEX column, we recommend γ -DEX columns as complements to α -DEX and β -DEX columns.

Phase: nonbonded; 20% permethylated γ -cyclodextrin in SPB-35 poly(35% diphenyl/65% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24307

β-DEX 110, β-DEX 120

The chiral stationary phase in β -DEX columns contains permethylated β -cyclodextrin embedded in an intermediate polarity stationary phase. Recommended for the enantiomeric separation of a wide range of chiral compounds (ketones, esters, alkanes, alkenes, alcohols, acids, ethers, etc.). The 10% (β -DEX 110) and 20% (β -DEX 120) β -cyclodextrin content alters the elution order while maintaining similar enantioselectivity.

Phase: nonbonded; 10% and 20% permethylated β-cyclodextrin in SPB-35 poly(35% diphenyl/

65% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

McReynolds Nos.: x' y' z' u' s' = 112 236 153 130 184

(β-DEX 110)

x' y' z' u' s' = 119 264 154 134 187(\(\beta\)-DEX 120)

Length (m)	d _f (µm)	Beta	Cat. No.
β-DEX 110, 0.25	mm ID Fused Si	ilica	
30	0.25	250	24301
60	0.25	250	24302
β-DEX 120, 0.25	mm ID Fused Si	ilica	
30	0.25	250	24304
60	0.25	250	24305-U

Cyclodextrin Column Selection Kit I

This kit provides you with the tools you need to perform most chiral separations. Identities of enantiomers can be confirmed by monitoring changes in their elution order (enantioreversal) from an α -DEX column to a β -DEX column, a β -DEX column to a γ -DEX column.

Kit includes one 30m x 0.25mm ID, 0.25 μ m film column of each type: α -DEX 120, β -DEX 120, γ -DEX 120.

Description	Cat. No.
Cyclodextrin Column Selection Kit I	24340

Cyclodextrin Column Selection Kit II

In combination with Kit I, this kit provides you with a library of columns that spans the full range of DEX column enantioselectivity at substantial savings, relative to purchasing individual columns.

Kit includes one 30m x 0.25mm ID, 0.25μm film column of each type: β -DEX 120, β -DEX 225, γ -DEX 225, β -DEX 325.

Description	Cat. No.
Cyclodextrin Column Selection Kit II	24328-U

Recommended Reading

See our complete line of reference books on gas chromatography on our website: www.sigma-aldrich.com.

Special Purpose Columns: Enantiomers/Positional Isomers

DEX-225 and DEX-325

Because it is difficult to predict the best phase for a given chiral or positional isomer separation requirement, we have extended our selection of cyclodextrin-based materials, to broaden the range of selectivity possibilities. We prepare the columns on this page using dimethyl- and diacetyl-derivatized cyclodextrins. These columns will separate volatile chiral molecules, including alcohols, aldehydes, carboxylic acids, epoxides, esters, and halogenated compounds. We are continually developing specific applications on our cyclodextrin columns, and suggest that you regularly consult our Web site for the most current chiral applications.

α-DEX 225

The chiral stationary phase in α -DEX 225 columns contains 2,3-di-O-acetyl-6-O-TBDMS- α -cyclodextrin embedded in an intermediate polarity phase.

Phase: nonbonded; 25% 2,3-di-O-acetyl-6-O-TBDMS-

α-cyclodextrin embedded in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
30	0.25	250	24311

β-DEX 225

The chiral stationary phase in β -DEX 225 columns contains 2,3-di-O-acetyl-6-O-TBDMS- β -cyclodextrin embedded in an intermediate polarity phase. These columns provide unique selectivity for enantiomeric separations of small molecules: alcohols, aldehydes (e.g., 2-phenylpropionaldehyde), esters (e.g., methyl malate, methyl lactate), flavor compounds, and ketones.

Phase: nonbonded; 25% 2,3-di-O-acetyl-6-O-TBDMS-

β-cyclodextrin embedded in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24348

γ-DEX 225

The chiral stationary phase in γ -DEX 225 columns contains 2,3-di-O-acetyl-6-O-TBDMS- γ -cyclodextrin embedded in an intermediate polarity phase.

Phase: nonbonded; 25% 2,3-di-O-acetyl-6-O-TBDMS-

γ-cyclodextrin embedded in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
30	0.25	250	24312

α-DEX 325

The chiral stationary phase in α -DEX 325 columns contains 2,3-di-O-methyl-6-O-TBDMS- α -cyclodextrin embedded in an intermediate polarity phase.

Phase: nonbonded; 25% 2,3-di-O-methyl-6-O-TBDMS-

α-cyclodextrin embedded in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
30	0.25	250	24303

β-DEX 325

The chiral stationary phase in β -DEX 325 columns contains 2,3-di-O-methyl-6-O-TBDMS- β -cyclodextrin embedded in an intermediate polarity phase.

Phase: nonbonded; 25% 2,3-di-O-methyl-6-O-TBDMS-

β-cyclodextrin embedded in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
30	0.25	250	24308

γ-DEX 325

The chiral stationary phase in γ -DEX 325 columns contains 2,3-di-O-methyl-6-O-TBDMS- γ -cyclodextrin embedded in an intermediate polarity phase.

Phase: nonbonded; 25% 2,3-di-O-methyl-6-O-TBDMS-

γ-cyclodextrin embedded in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

Temp. Limits: 30°C to 230°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fus	sed Silica		
30	0.25	250	24306

Special Purpose Columns: Environmental

SPB-608

Specially tested with 18 chlorinated pesticides at low concentration with an electron capture detector (ECD). These columns meet the criteria for minimum breakdown of 4,4'-DDT and endrin for EPA Methods 508, 608, 8080, 8081, and SW-Pesticides. Proprietary bonded phase.

Temp. Limits: subambient to 300°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24103-U
0.25mm ID Fuse	ed Silica		
15	0.50	265	25310-U
30	0.50	265	25312

SP-2331

A highly polar cyanosilicone stationary phase, specially tested for analyses of TCDD (dioxin) isomers. The phase is stabilized, providing a maximum temperature slightly higher than nonbonded cyanosilicone phases, such as SP-2330.

Temp. Limits: subambient to 275°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
30	0.20	313	24257
60	0.20	313	24104-U

Sup-Herb

Specially tested intermediate polarity column for analyses of herbicides, per US EPA Method 507. Proprietary bonded phase.

Temp. Limits: subambient to 300°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.53mm ID Fus	ed Silica		
15	0.50	265	25322

SPB-624

Specially tested for separation, efficiency, and baseline bleed; designed for purge-and-trap analyses of volatile halogenated, nonhalogenated, and aromatic contaminants from air, drinking water, wastewater, and soil. SPB-624 columns meet the requirements of various US EPA methods: CLP-VOA, 502.2, 524.2, 601, 602, 624, 1624, TO-1, TO-2, TO-3, TO-14, 5041, 8010, 8015, 8020, and 8260. Proprietary bonded phase.

Temp. Limits: subambient to 250°C (1.4μm or 1.8μm film) or 230°C (3.0μm film)

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	1.4	44	24255
60	1.4	44	24256
0.32mm ID Fuse	ed Silica		
60	1.8	44	24251
0.53mm ID Fuse	ed Silica		
30	3.0	44	25430
75	3.0	44	25432

VOCOL

These intermediate polarity columns, designed for volatile organic compounds (VOCs) analysis, ensure greater retention and resolution of the more volatile compounds. Use in direct injection ports or coupled to purge-and-trap systems, for US EPA volatiles methods, including 502.2, 524.2, 624, 8240, 8260, and 8021. Proprietary bonded phase.

Temp. Limits: subambient to 250°C (1.5μm film) or 230°C (3μm film)

Length (m)	d _f (µm)	Beta	Cat. No.
0.20mm ID Fus	ed Silica		
10	1.2	42	24129-U
0.25mm ID Fuse	ed Silica		
30	1.5	42	24205-U
60	1.5	42	24154
0.32mm ID Fuse	ed Silica		
60	1.8	44	24217-U
60	3.0	27	24157
0.53mm ID Fuse	ed Silica		
30	3.0	44	25320-U
60	3.0	44	25381
105	3.0	44	25358

PTE-5

Tested environmental column with an SE-54-type phase, for analyses of semivolatile priority pollutants. Tested to meet or exceed all column performance specifications of US EPA Methods 625, 1625 and 8270. Low bleed columns recommended for use with GC/MS systems.

Phase: bonded; poly(5% diphenyl/95% dimethylsiloxane)

Temp. Limits: -60°C to 320°C

McReynolds Nos.: x' y' z' u' s' = 1974649362

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24135-U
0.32mm ID Fuse	ed Silica		
30	0.25	320	24143
30	0.32	250	24214
30	1.0	80	24159

PTE-5 OTM

Tested environmental column with an SE-54-type phase, developed for rapid screening of semivolatile priority pollutant samples according to the US EPA Quick Turnaround Methods protocol.

Phase: bonded; poly(5% diphenyl/95% dimethylsiloxane)

Temp. Limits: -60°C to 320°C

McReynolds Nos.: x' y' z' u' s' = 1974649362

Length (m)	d _, (μm)	Beta	Cat. No.
0.53mm ID Fus	ed Silica		
15	0.50	265	25355



Special Purpose Columns: FAMEs/Sterols

Omegawax

These columns were developed to provide highly reproducible analyses of fatty acid methyl esters, specifically the omega 3 and 6 fatty acids. The columns are checked for reproducibility of FAME equivalent chain length (ECL) values and resolution of key components.

Phase: bonded; poly(ethylene glycol)

Temp. Limits: 50°C to 280°C

Length (m)	d _f (μm)	Beta	Cat. No.		
Omegawax 250	, 0.25mm ID Fus	ed Silica			
30	0.25	250	24136		
Omegawax 320	, 0.32mm ID Fus	ed Silica			
30	0.25	320	24152		
Omegawax 530, 0.53mm ID Fused Silica					
30	0.50	265	25374		

SP-2560

Specially prepared and tested columns, designed to separate geometric-positional (cis/trans) isomers of fatty acid methyl esters. Recommended for separating FAMEs in hydrogenated vegetable oil samples.

Phase: nonbonded; biscyanopropyl polysiloxane

Temp. Limits: subambient to 250°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
100	0.20	313	24056

SP-2380

This column was developed for high resolution and efficiency, and fast analyses of positional and geometric isomers of fatty acid methyl esters.

Phase: stabilized poly(90% biscyanopropyl/10%

cyanopropylphenyl siloxane)

Temp. Limits: subambient to 275°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		<u>.</u>
100	0.20	313	24317

SPB-PUFA

These columns provide highly reproducible analyses of polyunsaturated fatty acid methyl esters. The lower polarity poly (alkylene glycol) phase features improved "carbon number" separations over the poly(ethylene glycol) Omegawax columns.

Phase: bonded; poly(alkylene glycol)

Temp. Limits: 50°C to 220°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
30	0.20	313	24314
0.32mm ID Fus	ed Silica		
30	0.20	400	24323

SAC-5

An SE-54 type phase, developed and tested for reproducible analyses of plant sterols, cholesterol, and other animal sterols.

Phase: bonded; poly(5% diphenyl/95% dimethylsiloxane)

Temp. Limits: -60°C to 320°C

McReynolds Nos.: x' y' z' u' s' = 1974649362

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
15	0.25	250	24151
30	0.25	250	24156

Special Purpose Columns: Hydrocarbons

Petrocol DH 50.2

Narrow bore column for detailed hydrocarbon analyses of naphthas, gasolines and similar samples according to ASTM Test Method D5134 (Figure D).

Phase: bonded; poly(dimethylsiloxane)

Temp. Limits: -60°C to 320°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.20mm ID Fus	ed Silica		
50	0.50	100	24133-U

Petrocol DH

Highly reproducible column displaying more than 400,000 theoretical plates, designed for detailed analyses of petroleum products; used for PNA, PONA and PIANO-type analyses. Includes an extensive retention index data sheet (400+ analytes).

Phase: bonded; poly(dimethylsiloxane)

Temp. Limits: -60°C to 320°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fus	sed Silica		
100	0.50	125	24160-U

Petrocol DH 150

The longest capillary column commercially available as a stock item. Columns typically display more than 600,000 theoretical plates. For detailed purity analyses of light hydrocarbon gases and petroleum products (oxygenates, solvents, naphthas, gasolines, etc.).

Length (m)	d _r (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
150	1.0	63	24155

Petrocol DH Octyl

These highly reproducible columns are designed for detailed analyses of petroleum products. They offer unique selectivity not obtainable with poly(dimethylsiloxane) columns, such as baseline separations of benzene/1-methylcyclopentene and toluene/2,3,3-trimethylpentane (Figure D).

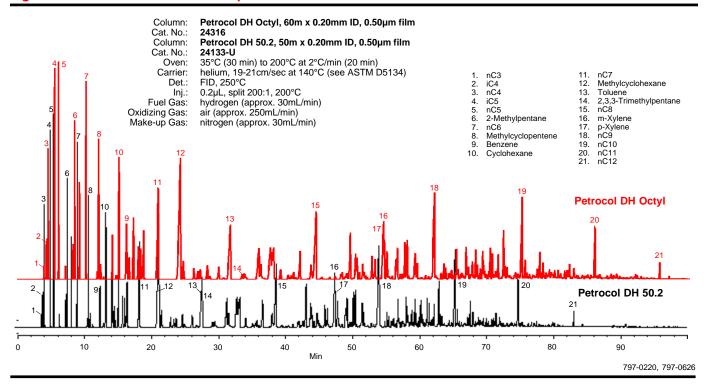
Phase: bonded; poly(50% n-octyl/50% methylsiloxane)

Temp. Limits: -60°C to 220°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
100	0.50	125	24282

Refer to SPB-Octyl, SPB-1, and SUPELCOWAX 10 listings for additional 100-meter columns.

Figure D. Qualitative Reference Naphtha Standard on Two Petrocol Columns





Special Purpose Columns: Hydrocarbons/Sulfur/Amines

Petrocol 2887

Developed and tested to meet or exceed column performance requirements for simulated distillation of petroleum fractions having boiling points up to 1000°F, according to ASTM Test Method D2887.

Phase: bonded; poly(dimethylsiloxane) Temp. Limits: subambient to 350°C

Length (m)	d _r (µm)	Beta	Cat. No.
0.53mm ID Fus	ed Silica (5" cage	e)	
5	0.50	265	25323

Petrocol EX2887

A thin film version of the Petrocol 2887 column, developed for extended D2887 SIMDIS analysis of samples having final boiling points greater than 1000°F.

Phase: bonded; poly(dimethylsiloxane) Temp. Limits: subambient to 380°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.53mm ID Fuse	ed Silica		
5	0.10	1325	25337

HT-5

SGE aluminum-clad columns coated with a carborane phase, offering the highest maximum temperature of any commercially available column. They display low bleed for GC/MS and simulated distillation analyses.

Phase: bonded; siloxane-carborane (5% phenyl equivalent)

Temp. Limits: 10°C to 460°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.32mm ID Fuse	ed Silica		
12	0.10	800	25002
25	0.10	800	25003
0.53mm ID Fuse	ed Silica		
6	0.10	1325	25004
12	0.15	883	25005-U

SPB-1 SULFUR

A very thick film version of our SPB-1 columns, specially developed for analyses of sulfur gases and other volatile sulfur compounds. The column displays relatively low column bleed even for the exceptionally thick film (4µm) of stationary phase, which makes it compatible for use with the Sievers Sulfur Chemiluminescence Detector (SCD) and other sulfur-specific detectors (Figure E).

Phase: bonded; poly(dimethylpolysiloxane)

Temp. Limits: -60°C to 300°C

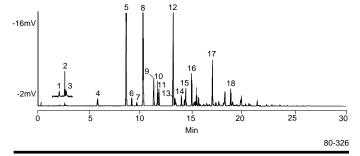
Length (m)	d _f (µm)	Beta	Cat. No.
0.32mm ID Fuse	ed Silica		
30	4.0	20	24158

Figure E. Natural Gas Condensate on an SPB-1 SULFUR Column

SPB-1 SULFUR, 30m x 0.32mm ID, 4.0µm film Column: Cat. No.: 24158 Oven: -10°C (3 min) to 300°C at 10°C/min, hold 2 min sulfur chemiluminescence detector 0.5mL natural gas condensate, split 10:1

- Hydrogen sulfide 47pg S Carbonyl sulfide 116pg S Sulfur dioxide 66pg S Methanethiol 523pg S Ethanethiol 5924pg S Dimethylsulfide 414pg S Carbon disulfide 192pg S i-Propanethiol 11,206pg S t-Butanethiol 1814pg S
- n-Propanethiol 1363pg S 13.
- n-Propanetniol 1363pg S Methylethylsulfide 1048pg S s-Butanethiol 5594pg S i-Butanethiol 423pg S Diethylsulfide 515pg S n-Butanethiol 1183pg S Dimethyldisulfide 1567pg S

2-Ethylthiophene 2324pg S Diethyldisulfide 929pg S



Chromatogram provided by Sievers Research Inc., Boulder, Colorado, USA.

Carbowax Amine

Carbowax Amine columns are specially prepared, base-deactivated polyethylene glycol columns designed for the analysis of primary, secondary, and tertiary amines and other volatile basic analytes.

Phase: nonbonded; base-modified poly(ethylene glycol)

Temp. Limits: 60°C to 200°C

Length (m)	d _f (µm)	Beta	Cat. No.
0.53mm ID Fuse	ed Silica		
15	1.0	133	25352
30	1.0	133	25353
60	1.0	133	25354

Special Purpose Columns: Volatiles/Amines

SPB-HAP

This column was developed to provide the best resolution of very volatile, regulated hazardous air pollutants. The thick film focuses analytes on the inlet of the column, without cryogenic focusing.

Phase: bonded; poly(dimethylsiloxane)

Temp.: -60°C to 300°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.32mm ID Fuse	ed Silica		
60	4.0	20	25020-U

See the Chemical Standards section of our catalog for hazardous air pollutants standards.

PTA-5

Specially prepared, base-deactivated poly (5% diphenyl/95% dimethylsiloxane) columns designed for analyses of amines and other basic analytes.

Phase: bonded; base-modified poly(5% diphenyl/95% dimethylsiloxane)

Temp. Limits: -60°C to 320°C

Length (m)	d _r (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.50	125	24277
30	1.0	625	24330
0.32mm ID Fuse	ed Silica		
30	0.5	160	24331
30	1.0	80	24332
30	1.5	53	24333
0.53mm ID Fuse	ed Silica		
30	0.5	265	25437
30	1.5	88	25438
30	3.0	44	25439

See our catalog for applications.

OVI-G43

This column is specially prepared and tested to meet the requirements of United States Pharmacopoeia (USP) Method 467 and the European Pharmacopoeia general method for determining residual organic solvents in pharmaceutical preparations. Use this column to separate organic volatile impurities (OVIs) for research purposes or qualitative analysis (Figure F). The USP and European Pharmacopoeia methods also specify using a deactivated 5-meter guard column.

Phase: bonded; poly(6% cyanopropylphenyl/

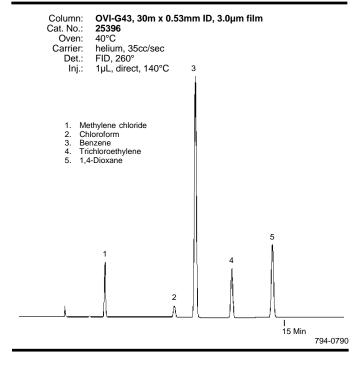
94% dimethylsiloxane)

Temp. Limits: -20°C to 260°C

Length (m)	d _f (μm)	Beta	Cat. No.
0.53mm ID Fuse	ed Silica		
30	3.0	44	25396

Description	Cat. No.
Deactivated Guard Column for OVI-G43 5m x 0.53mm ID	25339
Other Columns for Residual Solvents Analysis G27 (SPB-5) 30m x 0.53mm ID, 5.0µm	25347
G16 (SUPELCOWAX 10) 30m x 0.53mm ID, 1.0μm	25301-U

Figure F. USP <467> Organic Volatile Impurities on an OVI-G43 Column



Special Purpose Columns: PLOT/SCOT

Mol Sieve 5A PLOT

Like their packed column counterparts, Mol Sieve 5A PLOT columns separate permanent gases. Oxygen, nitrogen, carbon monoxide and methane can be separated in less than 5 minutes. More difficult separations, such as argon from oxygen, can be achieved by using subambient temperatures. A proprietary procedure fixes the molecular sieve to the fused silica tubing and ensures the particles will not be dislodged from the tubing in normal use. The 0.53mm ID column can be used in packed column chromatographs.

Dimensions (fused silica)	Max. Temp. (°C)	Cat. No.
30m x 0.32mm ID	300	24243
30m x 0.53mm ID	300	25463

Carboxen-1006 PLOT

Carboxen[™]-1006 porous layer open tubular (PLOT) columns separate permanent gases and C1, C2, and C3 light hydrocarbons, using above-ambient initial temperatures. They also are ideal for resolving formaldehyde/water/methanol (formalin) mixtures and monitoring impurities in ethylene. A proprietary procedure fixes a porous carbon molecular sieve (surface area ≥ $750\text{m}^2/\text{gram}$) to fused silica tubing and ensures the carbon layer will not be dislodged from the tubing in normal use. Use Carboxen-1006 columns with high flow rates and rapid temperature programs, up to 250°C, to ensure excellent, fast separations. The 0.53mm ID column can be used in packed column chromatographs.

Dimensions (fused silica)	Max. Temp. (°C)	Cat. No.
30m x 0.32mm ID	250	24241-U
30m x 0.53mm ID	250	25461

SCOT

Support-coated open tubular (SCOT) columns are prepared by depositing a layer of liquid phase-coated support particles on the inner wall of the tubing. This technology, developed by Perkin-Elmer, makes available many phases that cannot be obtained on conventional wall-coated open tubular capillary columns. SCOT columns combine the sensitivity and excellent sample resolution of capillary GC with the extensive stationary phase library of packed column GC.

Stainless Steel SCOT Columns

50' x 1/32" OD x 0.02" ID with 1/16" connections

Liquid Phase	Max. Temp. (°C)	Beta	Cat. No.
Bentone 34/DNDP■	150	45	25521
Squalane	120	50	25535
TĊEP	150	40	25536
BMEA	100	40	25538

[■]Di-n-decylphthalate

Supel-Q PLOT

SupelTM-Q PLOT columns contain a porous divinylbenzene polymer that effectively resolves carbon dioxide and C1-C4 hydrocarbons at above ambient temperatures. It also is suitable for analyses of other gases, such as sulfur gases, and alcohols, ketones, aldehydes, and many polar compounds. Gasoline and other petroleum fractions can be analyzed as well. These columns exhibit very little bleed, even at the maximum temperature. Relative to packed columns (e.g., Porapak®-Q), Supel-Q PLOT columns offer better resolution in less time. A proprietary procedure itizes the polymer to the fused silica tubing and ensures the particles will not be dislodged from the tubing in normal use. The 0.53mm ID column can be used in packed column chromatographs.

Dimensions (fused silica)	Max. Temp. (°C)	Cat. No.
30m x 0.32mm ID	250	24242
30m x 0.53mm ID	250	25462

Carboxen-1010 PLOT

A carbon molecular sieve column for separating the permanent gases: hydrogen, nitrogen, carbon monoxide, methane, carbon dioxide, C2 and C3 hydrocarbons. Oxygen is separated from nitrogen. The 0.53mm ID columns can be used in packed column chromatographs. Manufactured under US patents 5,599,445 and 5,607,580.

Dimensions (fused silica)	Max. Temp. (°C)	Cat. No.
30m x 0.32mm ID	250	24246
30m x 0.53mm ID	250	25467

[◆]Patent pending.

General Purpose Columns: Metallon

Metallon Stainless Steel Capillary Columns

- Inert
- Unbreakable
- Wide variety of stationary phases

Easy-to-handle metal columns, deactivated to provide inertness equivalent to fused silica. Metallon columns have the same outer diameter as standard 0.53mm ID columns, so no special ferrules are required. We also can prepare columns having other dimensions and phases. If you are looking for a metal column that is not listed, just call us with your requirements.

MET-1

Phase: polydimethylsiloxane **Temp. Limits:** -60°C to 320°C

McReynolds Nos.: x' y' z' u' s' = 458435638

Similar Phases: DB-1, OV-1, OV-101, SPB-1, BP-1, HP-1,

ULTRA-1, CPSIL-5CB, RTx-1, 007-1

Length (m)	d _f (μm)	Beta	Cat. No.			
0.53mm ID Fused Silica						
15	0.5	265	25487			
30	0.5	265	25477			
15	1.5	88	25473			
30	1.5	88	25488			
60	1.5	88	25479			
15	3.0	44	25476			
30	3.0	44	25478			
15	5.0	27	25475			
30	5.0	27	25489			

MET-5

Phase: 5% phenyl/95% methylpolysiloxane

Temp. Limits: -60°C to 320°C

McReynolds Nos.: x' y' z' u' s' = 19 74 64 93 72 Similar Phases: DB-5, SPB-5, CPSIL-8CB, HP-5, ULTRA-2, RTx-5, 007-2

Length (m)	d _f (µm)	Beta	Cat. No.
0.53mm ID Fuse	ed Silica		
15	0.5	265	25480-U
30	0.5	265	25481
15	1.5	88	25490
30	1.5	88	25482
60	1.5	88	25483

MET-WAX

Phase: polyethylene glycol **Temp. Limits:** 35°C to 280°C

McReynolds Nos.: x' y' z' u' s' = 305 551 360 562 484

Similar Phases: DB-WAX, CP-WAX-51, CP-WAX-57CB, BP-20,

CW20MS, HP-20M, SUPELCOWAX 10,

Stabilwax, 007-CW

Length (m)	d _, (μm)	Beta	Cat. No.
0.53mm ID Fus	ed Silica		
15	1.0	133	25484
30	1.0	133	25485
60	1.0	133	25486

General Purpose Columns: Nonpolar

SPB-Octyl

poly(50% n-octyl/50% methylsiloxane)

The polarity of SPB-Octyl columns approaches that of squalane, and is substantially less than that of the widely used "nonpolar" methyl silicone phase. Because they offer unique selectivity compared to the commonly used low and intermediate polarity columns, we recommend SPB-Octyl columns for multidimensional or confirmational analyses of PCB-containing samples.

Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCI in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed.

Figure H shows PCB congeners on an SPB-Octyl column.

Phase: bonded; poly(50% n-octyl/50% methylsiloxane)

Temp. Limits: -60°C to 280°C (isothermal)

McReynolds Nos.: x' y' z' u' s' = 3 14 11 12 11

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24218-U
60	0.25	250	24219-U
30	1.0	63	24232
60	1.0	63	24233-U
0.53mm ID Fuse	ed Silica		
60	3.0	44	25398

Figure H. PCB Congeners in Stream Sediment[®]

Column: SPB-Octyl, 30m x 0.25mm ID, 0.25µm film

24218-U Cat. No.:

Oven:

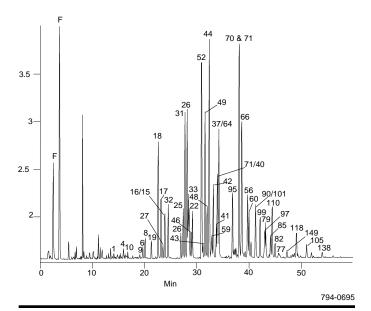
50°C (1 min) to 150°C at 10°C/min 5g stream sediment, downstream from industrial sites Sample:

SPME Fiber: PDMS, 100µm film

57300-U Cat. No.:

794-0200

headspace, 90°C (30 min) 280°C (2 min), splitless Extraction: Desorption:



Peak IDs are PCB congener numbers from Ballschmiter and Zell, Fresenius' Z. Anal. Chem., 302: 20-31 (1980). For additional information, request free Bulletin

General Purpose Columns: Nonpolar

SPB-1

Poly(dimethylsiloxane)

713-0338

Nonpolar methylsilicone columns that separate sample components according to boiling point. This bonded polymer matches the polarity of its nonbonded predecessors, SE-30 and SP-2100. The SPB-1 phase is used in many of our Petrocol specialty columns.

Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCI in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed. (Figure I.)

Phase: bonded; poly(dimethylsiloxane)

Temp. Limits: -60°C to 320°C

McReynolds Nos.: x' y' z' u' s' = 4 58 43 56 38

Figure I. Bacterial Acid Methyl Esters on an SPB-1 Column

Column: SPB-1, 15m x 0.10mm ID, 0.10µm film

Cat. No.: 24338

150°C (2 min) to 250°C at 50°C/min, hold 5 min Oven:

hydrogen, 60cm/sec (set at 150°C) FID, 280°C Carrier:

Det.:

1µL Bacterial Acid Methyl Esters CP Mix

(Cat. No. 47080-U) containing 10µg total analytes,

split 200:1, 250°C

1.	Me undecanoate
2.	Me 2-hydroxydecanoate
3.	Me dodecanoate
4.	Me tridecanoate
5.	Me 2-hydroxydodecanoate
6.	Me 3-hydroxydodecanoate
7.	Me tetradecanoate
8.	Me 13-methyltetradecanoate
9.	Me 12-methyltetradecanoate
10.	Me pentadecanoate
11	Me 2-hydroxytetradecanoate

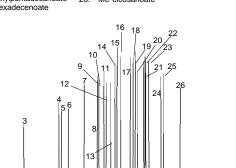
- 15. Me hexadecanoate Me 15-methylhexadecanoate
 Me cis-9,10-methylenehexadecanoate Me heptadecanoate
 Me 2-hydroxyhexadecanoate
 Me cis-9,12-octadecadienoate 19. 20.
- Me cis-9-octadecenoate Me trans-9-octadecanoate & Me cis-11-octadecenoate Me octadecanoate
- Me cis-9,10-methyleneoctadecanoate Me 2-hydroxytetradecanoate Me 3-hydroxytetradecanoate Me nonadecanoate Me 14-methylpentadecanoate

3

Min

Me cis-9-hexadecenoate

1



4

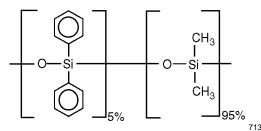
797-0400

Length (m)	d _f (µm)	Beta	Cat. No.			
0.10mm ID Fus	0.10mm ID Fused Silica					
15	0.10	250	24338			
0.20mm ID Fus	ed Silica					
15	0.20	250	24162			
30	0.20	250	24163			
12	0.33	152	24229-U			
25 10	0.33 1.20	152 42	24230-U 24134-U			
		42	24134-0			
0.25mm ID Fus 30	ed Silica 0.10	625	24261			
15	0.10	250	24026			
30	0.25	250	24028			
60	0.25	250	24030-U			
100	0.25	250	24198			
15	1.0	63	24027			
30	1.0	63	24029			
60	1.0	63	24031			
100	1.0	63	24220-U			
0.32mm ID Fus		222	0.4000			
30	0.10	800	24290			
15 30	0.25 0.25	320 320	24099 24044 <i>*</i>			
60	0.25 0.25	320 320	24044			
100	0.25	320	24228-U			
15	1.0	80	24098-U			
30	1.0	80	24045-U			
60	1.0	80	24047			
100	1.0	80	24213-U			
30	2.0	40	24215-U			
60	2.0	40	24216-U			
30	5.0	16	24296			
60	5.0	16	24297			
0.53mm ID Fus		1225	25260			
15 30	0.10 0.10	1325 1325	25360 25361			
15	0.50	265	25314			
30	0.50	265	25315			
60	0.50	265	25382			
15	1.0	133	25416			
30	1.0	133	25417			
15	1.5	88	25302-U			
30	1.5	88 88	25303 25388			
60 15	1.5 3.0	88 44	25388 25340			
30	3.0	44	25340 25341-U			
60	3.0	44	25348			
15	5.0	27	25344			
30	5.0	27	25345-U			
60	5.0	27	25349			

^{*}Recommended for most samples.

General Purpose Columns: Low Polarity

SPB-5
Poly(5% diphenyl/95% dimethylsiloxane)



The low phenyl content, 5%, provides improved thermal stability to the phase, while still providing essentially a boiling point elution order, and a slight increase in selectivity, relative to SPB-1, especially for aromatic compounds.

Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCI in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed.

Phase: bonded; poly(5% diphenyl/95% dimethylsiloxane)

Temp. Limits: -60°C to 320°C

McReynolds Nos.: x' y' z' u' s' = 1974649362

Figure J. Rancid Corn Oil (SPME/GC)

Sample: 3.0g corn oil SPME Fiber: polydimethylsiloxane, 100µm film Cat. No.: 57300-U Sampling: 40°C, 45 min (headspace) 250°C, 1.5 min SPB-5, 30m x 0.53mm ID, 5.0µm film Desorption: Column: Cat. No.: 25347 Oven: 40°C (5 min) to 220°C at 4°C/min helium, 5mL/min Carrier: Det.: FID, 300°C splitless (1 min), 250°C Propane Pentane Pentanol Hexanal 2-Hexenal 2-Heptanone 2-Heptanone 2-Heptenal 1-Octen-3-ol 2-Pentylfuran 3-Octen-2-one 2-Octenal 2-Nonenal 13. 14. 15. 2-Decenal trans,cis-2,4-Decadienal trans.trans-2.4 Decadienal 2-Undecenal BHT * Indicators of rancidity 30 20 Min

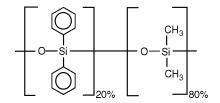
Length (m)	d _f (μm)	Beta	Cat. No.
0.10mm ID Fus	ed Silica		
15	0.10	250	24341
0.20mm ID Fus	ed Silica		
15	0.20	250	24165-U
30	0.20	250	24166
60	0.20	250	24167
12	0.33	152	24234-U
0.25mm ID Fus	ed Silica		
15	0.25	250	24032
30	0.25	250	24034
60	0.25	250	24036
15	1.0	63	24033
30	1.0	63	24035
60	1.0	63	24037
0.32mm ID Fus	ed Silica		
15	0.25	320	24101-U
30	0.25	320	24048*
60	0.25	320	24050
30	0.50	160	24360
25	0.52	154	24359
15	1.0	80	24100-U
30	1.0	80	24049
60	1.0	80	24051
0.53mm ID Fus	ed Silica		
15	0.50	265	25316
30	0.50	265	25317
60	0.50	265	25383
30	1.0	133	25420-U
15	1.5	88	25304
30	1.5	88	25305-U
60	1.5	88	25389
15	3.0	44	25342
30	3.0	44	25343
60	3.0	44	25350
15	5.0	27	25346
30	5.0	27	25347
60	5.0	27	25351

^{*}Recommended for most samples.

General Purpose Columns: Intermediate Polarity

SPB-20

Poly(20% diphenyl/80% dimethylsiloxane)



713-0340

SPB-20 columns have intermediate polarity as a result of the higher (20%) phenyl content of the stationary phase. The higher polarity produces different elution order for polar compounds, thereby providing confirmational information.

Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCI in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed.

Phase: bonded; poly(20% diphenyl/80% dimethylsiloxane)

Temp. Limits: -25°C to 300°C

McReynolds Nos.: $x' \ y' \ z' \ u' \ s' = 67 \ 116 \ 117 \ 174 \ 131$

Length (m)	d _ε (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24086
60	0.25	250	24087-U
30	1.0	63	24196
0.32mm ID Fuse	ed Silica		
30	0.25	320	24088*
60	1.0	80	24194
0.53mm ID Fuse	ed Silica		
30	0.50	265	25329-U
30	1.0	133	25333

^{*}Recommended for most samples.

See our catalog for applications.

SPB-35

Poly(35% diphenyl/65% dimethylsiloxane)

713-0341

SPB-35 columns have higher polarity than SPB-20 columns as a result of a greater phenyl content (35%). These columns are useful for analyses of polar compounds, because these compounds are retained longer, relative to nonpolar compounds.

Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCI in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed.

Phase: bonded; poly(35% diphenyl/65% dimethylsiloxane)

Temp. Limits: 0°C to 300°C

McReynolds Nos.: x' y' z' u' s' = 101 146 151 219 202

Length (m)	d _r (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24092
0.32mm ID Fuse	ed Silica		
30	0.25	320	24094 *
0.53mm ID Fuse	ed Silica		
30	0.50	265	25331
30	1.0	133	25335

^{*}Recommended for most samples.

General Purpose Columns: Intermediate Polarity

SPB-1701

Poly(14% cyanopropylphenyl/86% dimethylsiloxane)

Intermediate polarity SPB-1701 columns have a mixed functionality which provides unique elution order characteristics, relative to the phenyl-containing silicone phases.

Cyano functionality renders this phase more susceptible to damage by oxygen, moisture, and HCI than other silicone phases. Columns can be rinsed.

Phase: bonded; poly(14% cyanopropylphenyl/

86% dimethylsiloxane)

Temp. Limits: subambient to 280°C

McReynolds Nos.: x' y' z' u' s' = 67 170 153 228 171

Length (m)	d _ε (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
15	0.25	250	24112
30	0.25	250	24113
60	0.25	250	24114
0.32mm ID Fuse	ed Silica		
15	0.25	320	24183
30	0.25	320	24184
60	0.25	320	24185
0.53mm ID Fuse	ed Silica		
15	0.50	265	25368
30	0.50	265	25369
15	1.0	133	25366
30	1.0	133	25367

Recommended Reading

To see our complete line of reference books on gas chromatography, visit our website: www.sigma-aldrich.com.

SPB-50

713-0342

Poly (50% diphenyl/50% dimethylsiloxane)

The highest phenyl content of the common phenyl-containing phases, and hence the highest polarizability. Useful for analyses of polar materials and to provide confirmational information.

Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases, but stable to low levels of HCI in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed.

Phase: bonded; poly(50% diphenyl/50% dimethylsiloxane)

Temp. Limits: 30°C to 310°C

McReynolds Nos.: x' y' z' u' s' = 125 175 183 268 220

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24181
0.32mm ID Fuse	ed Silica		
30	0.25	320	24187
0.53mm ID Fuse	ed Silica		
30	0.50	265	25363

SP-2250

A nonbonded 50% phenyl polymer that is matched in polarity by the bonded version, SPB-50.

Phase: nonbonded; poly(50% phenyl/50% methylsiloxane)

Temp. Limits: 0°C to 250°C

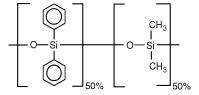
McReynolds Nos.: x' y' z' u' s' = 119 158 162 243 202

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
15	0.2	313	24009
30	0.2	313	24010
60	0.2	313	24011-U
0.32mm ID Fuse	ed Silica		
15	0.2	400	24147
30	0.2	400	24148

General Purpose Columns: Intermediate Polarity

SPB-17

Poly (50% diphenyl/50% dimethylsiloxane)



713-0343

This bonded, crosslinked (50% diphenyl) dimethylpolysiloxane phase is excellent for confirmational analyses. SPB-17 columns have intermediate polarity. Columns can be rinsed.

Phase: bonded; poly(50% diphenyl/50% dimethylsiloxane)

Temp. Limits: 0.25mm and 0.32mm ID: 40°C to 280/300°C

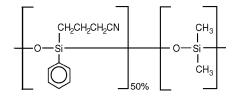
0.53mm ID: 40°C to 260/280°C

McReynolds Nos.: x' y' z' u' s' = 125 169 174 253 207

d _f (µm)	Beta	Cat. No
ed Silica		
0.25	250	24374-U
0.25	250	24380-U
ed Silica		
0.25	320	24381
0.50	160	24376
ed Silica	100	24
	ed Silica 0.25 0.25 ed Silica 0.25 0.50	ed Silica 0.25 250 0.25 250 ed Silica 0.25 320 0.50 160

SPB-225

Poly(50% cyanopropylphenyl/50% dimethylsiloxane)



797-0678

This bonded, crosslinked poly(50% cyanopropylphenyl 50% dimethylsiloxane phase is excellent for separating cis and trans FAMEs. SPB-225 columns have intermediate to high polarity. Columns can be rinsed.

Phase: bonded; poly(50% cyanopropylphenyl/

50% dimethylsiloxane)

Temp. Limits: 45°C to 220/240°C

Length (m)	d _r (µm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.15	417	24334
30	0.25	250	24335
0.32mm ID Fuse	ed Silica		
30	0.15	533	24336
30	0.25	320	24337

General Purpose Columns: Polar

Nukol

Poly(ethylene glycol) modified with nitroterephthalic acid

$$\begin{array}{c|c} \mathsf{HO_2C} & & & \\ \hline \mathsf{C} & & \\ \mathsf{NO_2} & & \\ \end{array} \\ \begin{array}{c} \mathsf{OCH_2\,CH_2} \\ \mathsf{DCH_2\,CH_2} \\$$

This bonded PEG-type phase, incorporating acidic functional groups, displays an acidic character and is useful for analyses of volatile acidic compounds. Even free carboxylic acids can be analyzed with excellent peak shape and minimal adsorption.

Chemically compatible with water and other injection solvents, but solvents such as water and methanol must be vaporized before reaching the column inlet. Avoid these solvents when using on-column injection techniques. Sensitive to strong inorganic acids. Columns can be rinsed.

Phase: bonded; modified poly(ethylene glycol)

Temp. Limits: 60°C to 200°C

McReynolds Nos.: x' y' z' u' s' = 314 569 372 578 504

Length (m)	d _ε (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
15	0.25	250	24106-U
30	0.25	250	24107
0.32mm ID Fuse	ed Silica		
15	0.25	320	24130
30	0.25	320	24131
60	0.25	320	24132
15	1.0	80	24206-U
30	1.0	80	24207
60	1.0	80	24208
0.53mm ID Fuse	ed Silica		
15	0.50	265	25326 *
30	0.50	265	25327
60	0.50	265	25386
30	1.0	133	25357

^{*}Recommended for most samples.

See our catalog for applications.

SPB-1000

Poly(ethylene glycol) modified with nitroterephthalic acid

SPB-1000 is an improved version of our Nukol™ phase. It is a bonded, PEG-type phase incorporating acidic functional groups and displaying a polarity closer to the SP-1000 phase than does Nukol. This new phase displays the acidic character necessary for analyses of volatile acidic compounds. It also offers improved performance for analyses of glycols, compared to Nukol.

Phase: bonded; modified poly(ethylene glycol)

Temp. Limits: 60°C to 200°C

McReynolds Nos.: x' y' z' u' s' = 308 565 368 567 511

Length (m)	d _f (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
30	0.25	250	24313
0.32mm ID Fuse	ed Silica		
30	0.25	320	24315
0.53mm ID Fuse	ed Silica		
30	0.50	265	25445

PAG

Poly(alkylene glycol)

CH₃

CH₂ - CH₂ - O

CH₂ - CH - O

H

Tale

The polyalkylene glycol stationary phase is less polar than polyethylene glycol phases, due to the incorporation of propylene oxide into the polymer backbone. This provides a phase that fills the polarity gap between 50% phenyl columns and Carbowax®-type columns (polarity similar to UCON® and Pluronics® phases).

Chemically compatible with water and other injection solvents, but solvents such as water and methanol must be vaporized before reaching the column inlet. Avoid these solvents when using on-column injection techniques. Sensitive to strong inorganic acids. Columns can be rinsed.

Phase: bonded; poly(alkylene glycol)

Temp. Limits: 30°C to 220°C

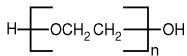
McReynolds Nos.: x' y' z' u' s' = 252 499 310 489 416

Length (m)	d _f (µm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
15	0.25	250	24222-U
30	0.25	250	24223
0.32mm ID Fus	ed Silica		
15	0.25	320	24225-U
30	0.25	320	24226
60	0.25	320	24227
0.53mm ID Fus	ed Silica		
15	0.50	265	25422-U
60	0.50	265	25424

General Purpose Columns: Polar

SUPELCOWAX 10

Poly(ethylene glycol)



713-0344

This polar PEG-type phase is the bonded equivalent to the CARBOWAX 20M phase, with much higher thermal stability. Because this phase offers higher polarity than any of the phenylsilicone phases, it is widely used for separation and purity analyses of many polar compounds, including alcohols, aromatics, and other solvents, flavors, fragrances, and FAMEs.

Chemically compatible with water and other injection solvents, but solvents such as water and methanol must be vaporized before reaching the column inlet. Avoid these solvents when using on-column injection techniques. Sensitive to strong inorganic acids. Columns can be rinsed.

Phase: bonded; CARBOWAX 20M poly(ethylene glycol)

Temp. Limits: 35°C to 280°C

McReynolds Nos.: x' y' z' u' s' = 305 551 360 562 484

Length (m)	d _f (µm)	Beta	Cat. No.		
0.10mm ID Fuse	ed Silica				
5	0.10	250	25025-U		
10	0.10	250	25026-U		
15	0.10	250	24343		
0.25mm ID Fuse	ed Silica				
15	0.25	250	24077		
30	0.25	250	24079 *		
60	0.25	250	24081		
30	0.50	125	24284		
60	0.50	125	24285-U		
0.32mm ID Fuse	ed Silica				
15	0.25	320	24078		
30	0.25	320	24080-U *		
60	0.25	320	24082		
15	0.50	160	24083		
30	0.50	160	24084		
60	0.50	160	24085-U		
30	1.0	80	24211		
60	1.0	80	24212		
0.53mm ID Fuse	ed Silica				
15	0.50	265	25324		
30	0.50	265	25325		
60	0.50	265	25385		
15	1.0	133	25300-U		
30	1.0	133	25301-U		
60	1.0	133	25391		
30	2.0	66	25375-U		
60	2.0	66	25376		

^{*}Recommended for most samples.

See our catalog for applications.

SP-2330

Poly(80% biscyanopropyl/20% cyanopropylphenylsiloxane)

Substitution of the bis-cyanopropyl and phenyl groups on the polymer backbone provides the phase with both polar and polarizable characteristics. These columns (and all high cyanopropyl-substituted polymers) are useful for both high and low temperature separations of samples such as geometric isomers of fatty acid methyl esters, dioxins, and aromatic compounds.

Cyano functionally renders this phase more susceptible to damage by oxygen, moisture, and HCI than other silicone phases. Avoid solvents such as water and methanol when using oncolumn injection techniques. Columns should not be rinsed.

Phase: nonbonded; poly (80% biscyanopropyl/

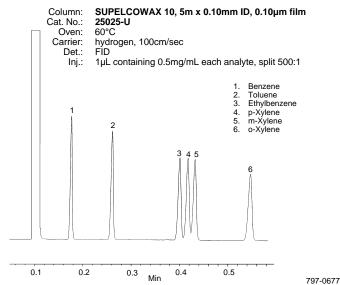
20% cyanopropylphenyl siloxane)

Temp. Limits: subambient to 250°C

McReynolds Nos.: x' y' z' u' s' = 382 610 506 710 591

Length (m)	d _f (µm)	Beta	Cat. No.	
0.25mm ID Fuse	ed Silica			
15	0.20	313	24018	
30	0.20	313	24019	
60	0.20	313	24020-U	
0.32mm ID Fus	ed Silica			
15	0.20	400	24102-U	
30	0.20	400	24073	
60	0.20	400	24074	

Figure K. Ultra-Fast BTEX Separation, Using 0.10mm ID SUPELCOWAX 10 Column



SUPELCO

General Purpose Columns: Polar

SP-2380

poly(90% biscyanopropyl/ 10% cyanopropylphenyl siloxane)

713-0348

Between the SP-2330 and SP-2340 phases in polarity. The high polarity of this phase allows the separation of geometric (cis/ trans) fatty acid methyl ester isomers as a group. Stabilized phase with a maximum temperature slightly higher than the traditional nonbonded cyanosilicone phases (SP-2330 and SP-2340). Significantly more stable than SP-2330.

Cyano functionally renders this phase more susceptible to damage by oxygen, moisture, and HCI than other silicone phases. Avoid solvents such as water and methanol when using oncolumn injection techniques. Columns should not be rinsed.

Phase: stabilized poly(90% biscyanopropyl/ 10% cyanopropylphenyl siloxane)

Temp. Limits: subambient to 275°C

McReynolds Nos.: x' y' z' u' s' = 402 629 520 744 623

Length (m)	ngth (m) d _f (µm) Beta		Cat. No.	
0.25mm ID Fuse	ed Silica			
15	0.20	313	24109	
30	0.20	313	24110-U	
60	0.20	313	24111	
100	0.20	313	24317	
0.32mm ID Fuse	ed Silica			
30	0.20	400	24116	
60	0.20	400	24117	
0.53mm ID Fuse	ed Silica			
30	0.20	663	25319	

See our catalog for applications.

SP-2340

Poly(biscyanopropylsiloxane)

713-0347

The highest polarity of any of the general purpose cyanosilicone phases. As with all cyano phase columns, these columns are useful for both high and low temperature separations of samples such as geometric isomers of fatty acid methyl esters, dioxins, and aromatic compounds.

Cyano functionally renders this phase more susceptible to damage by oxygen, moisture, and HCI than other silicone phases. Avoid solvents such as water and methanol when using oncolumn injection techniques. Columns should not be rinsed.

Phase: nonbonded; poly(biscyanopropyl siloxane)

Temp. Limits: subambient to 250°C

McReynolds Nos.: x' y' z' u' s' = 419 654 541 758 637

Length (m)	d _ε (μm)	Beta	Cat. No.
0.25mm ID Fuse	ed Silica		
15	0.20	313	24021
30	0.20	313	24022
60	0.20	313	24023
0.32mm ID Fuse	ed Silica		
15	0.20	400	24138
30	0.20	400	24075 *
60	0.20	400	24076

^{*}Recommended for most samples.

TCEP

This highly polar phase offers unique polarity for certain separations, despite its relatively low temperature limit and the fact that it is not a bonded phase. Because many aromatic compounds have retention indices greater than 1100 on TCEP, it is used for analyses of aromatics in mineral spirits and impurities in individual aromatics and oxygenates. Columns should not be rinsed.

Phase: nonbonded; 1,2,3-tris-2-cyano-ethoxypropane

Temp. Limits: subambient to 145°C

McReynolds Nos.: x' y' z' u' s' = 594 857 759 1031 917

Length (m)	d _, (μm)	Beta	Cat. No.
0.25mm ID Fus	ed Silica		
60	0.44	142	24153
0.32mm ID Fus	ed Silica		
60	0.51	157	24161

Custom Columns

Specify What You Need . . .

- A phase not commonly available on capillary columns (Table 6)
- A column with a Supelco stationary phase in an unusual length, ID, or film thickness (Table 7)
- A mixed phase for optimum peak separations and analysis speed
- A 0.10mm ID column for fast screening or special applications
- Testing with a sample appropriate to your analysis

We usually can provide you with the product you need, with the consistency and reproducibility you have come to expect from Supelco's ISO 9001-registered manufacturing, processes. All columns are prepared using our standard production and testing processes, and are priced at or below prices for our stock capillary columns.

To order your custom column, call Order Processing and provide us with your required phase, film thickness, ID, length, and other necessary details.

Table 6. Typical Nonstandard Stationary Phases

Other phases are available; please inquire.

Polarity	Phase
Nonpolar ¹	Apiezon L, Apiezon L/KOH, Dexsil 300, OV-1, OV-3, OV-61, OV-101, Squalane Squalene
Intermediate Polarity ²	Dexsil 400, OV-17, OV-22, OV-25, OV-215, PPE-20, PPE-21, SP-2401
Polar ³	BDS, Dexsil 410, OV-225, Silar 5 CP, Silar 7 CP, SP-2300, SP-2310, UCON LB-550X, UCON 50HB-280X,
	UCON 50HB-2000, UCON 50HB-51000, XE-60
Very Polar⁴	BC-150, OV-275, Silar 9 CP, Silar 10 CP

¹Polarity similar to SPB-1, SPB-5.

Table 7. Standard Stationary Phases and Film Thicknesses

For column lengths up to 60m. Please inquire about other lengths and film thicknesses.

	Fused Silica (mm ID)					
Stationary Phase	0.10	0.20	0.25	0.32	0.53	0.75
SPB-Octyl, SPB-1, SPB-5	0.05-0.5	0.10-1.0	0.10-3.0	0.10-5.0	0.10-7.0	0.10-7.0
SE-30	0.05-0.5	0.10-1.0	0.10-1.5	0.10-2.0	0.10-7.0	0.10-7.0
SP-2100	0.05-0.5	0.10-1.0	0.10-1.5	0.10-2.0	0.10-2.0	0.10-2.0
SE-54	0.05-0.5	0.10-1.0	0.10-1.5	0.10-2.0	0.10-7.0	0.10-7.0
SPB-20, SPB-35, SPB-50	0.05-0.5	0.10-1.0	0.10-1.5	0.10-2.0	0.10-3.0	0.10-3.0
SP-2250	0.05-0.1	0.10-0.20	0.10-0.25	0.10-0.30	0.10-0.50	0.10-0.50
PAG, SUPELCOWAX 10	0.05-0.2	0.10-0.40	0.10-1.0	0.10-2.0	0.10-3.0	0.10-1.0
CARBOWAX 20M	0.05-0.2	0.10-0.40	0.10-0.50	0.10-1.0	0.10-1.5	0.10-1
Nukol	0.05-0.2	0.10-0.40	0.10-0.50	0.10-1.0	0.10-1.0	0.10-1.0
SP-1000	0.05-0.2	0.10-0.40	0.10-0.50	0.10-1.0	0.10-1-0	0.10-1.0
SP-2330, SP-2340	0.05-0.1	0.10-0.20	0.10-0.20	0.10-0.20	0.10-0.20	0.10-0.20
SP-2380	0.05-0.1	0.10-0.20	0.10-0.25	0.10-0.30	0.10-0.50	0.10-0.75
SPB-17	0.05-0.5	0.10-1.0	0.10-1.50	0.10-2.0	0.10-3.0	0.10-3.0
SPB-1701	0.05-0.5	0.10-1.0	0.10-1.50	0.10-2.0	0.10-3.0	0.10-3.0
SPB-225	0.05-0.5	0.10-1.0	0.10-1.50	0.10-2.0	0.10-3.0	0.10-3.0
SPB-1301	0.05-0.5	0.10-1.0	0.10-1.50	0.10-2.0	0.10-3.0	0.10-3.0
Carbowax Amine	0.05-0.2	0.10-0.40	0.10-0.50	0.10-1.0	0.10-1.5	0.10-1.0
OVI-G43	0.05-0.5	0.10-1.0	0.10-1.50	0.10-2.0	0.10-3.0	0.10-3.0
DEX α, β, γ	0.25	0.25	0.25	0.25	0.25	0.25

Some thicker films are not available in 60m lengths.

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

PLOT*	Diameters
Alumina, Alumina KCI	0.25-0.75
Mol Sieve 5A	0.25-0.75
Carboxen 1006, 1010	0.25-0.75
Supel-Q	0.25-0.75

^{*}Column lengths up to 60 meters.



²Polarity similar to SPB-20, SPB-35, SPB-50.

³Polarity similar to SUPELCOWAX 10.

⁴Polarity similar to SP-2330, SP-2340, SP-2380.

Fused Silica Tubing

Use as transfer lines, guard columns, or retention gaps, or to make your own columns.

Tubing can be coupled through GlasSeal™ fused silica or glass connectors. If necessary, use polyimide glue to provide a permanent seal.

Tubing Treatment	Application
Untreated	General purposes, where high inertness is not necessary
Nonpolar (methyl)	Low polarity solvents (e.g., alkanes, carbon disulfide, ethers)
Intermediate Polarity (phenyl/methyl)	Intermediate polarity solvents (e.g., acetone, methylene chloride, toluene)
Polar (PEG)	Polar solvents (e.g., acetonitrile, methanol, water)

Fused Silica Tubing

		Deactivated,	Deactivated,	Deactivated,
ID ()	Untreated	Nonpolar	Intermediate Polarity	Polar
ID (mm)	Cat. No.	Cat. No.	Cat. No.	Cat. No.
3 x 1-meter lengths				
0.10	25700-U	25704	25705	25710
0.20	_	24057	25706	25711
0.25	24024	24025	25707	25712
0.32	25702	24058	25708	25713
0.53	25703	25307	25709	25714
3-meter length				
0.10	25715	25720-U	_	25730-U
0.20	_	25721	25726	25731
0.25	25717	25722	25727	_
0.32	25718	25723	25728	_
0.53	25719	25724	25729	25734
5-meter length				
0.10	25735	25740-U	25745-U	_
0.20	_	25741	25746	_
0.25	25737	25742	25747	_
0.32	25738	25743	25748-U	25752-U
0.53	25739	25744	25339 ●	25753
15-meter length				
0.20	_	25755	_	25763
0.25	24059	25756	25760-U	_
0.32	24062	25757	25761	25765
0.53	25306	25758	25762	25766
30-meter length				
0.20	25767	25768-U	25772	25776
0.25	_	-	_	25777
0.32	24063	25770-U	25774	25778
0.53	25308	25771	25775-U	25779
60-meter length				
0.20	25780-U	25782	25786	_
0.25	24061	25783	25787	25791-U
0.32	24064	25784	25788-U	25792
0.53	25781	25785	25789	-

[•]Deactivated according to USP 467.

Column Test Mixes

After you install a column in your system, use a test mix to make sure you haven't also installed some surprises (such as ferrule or tubing fragments in the column, or small leaks). Weekly tests thereafter will keep little problems from growing into big problems. Test mixes are an inexpensive aid to obtaining high quality chromatograms.

Acidity Column Test Mix

Even a highly efficient column can adsorb acidic or basic compounds. To determine the acid/base affinity of your column, simply inject this mix and compare peak heights (Grob & Grob, *Chromatographia*, **4**:421, 1971). Instructions included.

0.05% each component in methylene chloride.

2,6-Dimethylaniline 2,6-Dimethylphenol

Description	Cat. No.
2mL	48255-U

Hydrocarbon Test Mix

This easy-to-use mix is ideal for checking column installation when you use a capillary column in a modified packed column system. You also can use it to determine theoretical plates.

C12-C17 hydrocarbons, 500-2000µg/mL in chloroform.

Description	Cat. No.
2mL	48244

Isothermal Test Mixes

Use isothermal test mixes to indicate column efficiency, leaks, dead volume, and sample adsorption. Each mix includes simple, detailed instructions.

Isothermal Test Mix Kit

2mL each of the isothermal test mixes listed below.

Polar Column Test Mix (47302) Intermediate Polarity Column Test Mix (47301) Nonpolar Column Test Mix (47300-U)

Description	Cat. No.
Kit	47303

Polar Column Test Mix

For SUPELCOWAX 10, SP-1000, and other polar phases.

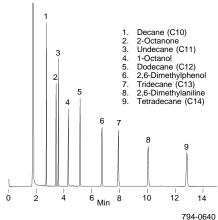
Description	Cat. No.
2mL	47302

Intermediate Polarity Column Test Mix

For SPB-20, SPB-35, and other intermediate polarity phases.

Description	Cat. No.
2mL	47301

Column: SPB-35, 30m x 0.25mm ID, 0.25µm film
Oven: 114°C
Carrier: helium, 30cm/sec
Det.: FID, 220°C
Inj.: 1µL Cat. No. 47301, 220°C, split
100:1



Nonpolar Column Test Mix

For SPB-1, SPB-5, and other nonpolar phases.

Description	Cat. No.
2mL	47300-U

Methane Standard

Use 40µL injections of this dilute methane standard (100ppm in helium) for more accurate flow measurements than with smaller quantities of more concentrated methane. Use with the methane syringe, syringe adapter, and pressure regulator listed below. Disposable cylinder.

100ppm in helium.

Description	Cat. No.
Methane Standard, 14L	307200
Accessories Hamilton® 1725N Syringe Syringe Adapter Pressure Regulator	20705 609010 513010

Programmed Test Mix

This mix is for a sensitive, temperature programmed analysis (Grob, et al., *J. Chromatogr.* **156**:1, 1978) that tests a column's affinity for many compounds. Prepared at concentrations convenient for setting split ratios and sample sizes. In use, on-column quantities are those recommended by Grob, et al.

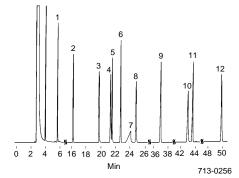
Each component at quantity indicated in the figure, in methylene chloride.

Description	Cat. No.
2mL	47304

Column: SPB-1, 30m x 0.25mm ID
Oven: 50°C to 200°C at 2°C/min
helium, 20cm/sec

Det.: FID, 250°C Inj.: 1µL Cat. No. **47304**, 250°C, split





Column Test Mixes

Specific Phase Test Mixes

For popular Supelco capillary columns. Active components and inactive hydrocarbons.

Carbowax Amine Column Test Mix

Description	Cat. No.
1mL	48278

α-DEX 120 Column Test Mix

Description	Cat. No.
1mL	48013

β-DEX 120 Column Test Mix

Description	Cat. No.
1mL	48028

Omegawax Test Mixes

Use these mixes to assess the performance of an Omegawax capillary column in analyses of fatty acid methyl esters. The Omegawax Column Test Mix and the Menhaden Oil standard are based on natural mixtures of fatty acids. Relative peak sizes may vary from lot to lot.

Omegawax Column Test Mix

Approximately 50mg FAMEs/mL in hexane.

Description	Cat. No.
1mL	48476

Menhaden Oil

Approximately 100mg FAMEs/mL in hexane.

Description	Cat. No.
1mL	48473

Methyl Nervonate

1000µg/mL in hexane.

Description	Cat. No.
1mL	48262

Petrocol DH Column Test Mix

Use this mix to assess performance of a Petrocol DH 100-meter capillary column.

Description	Cat. No.
1mL	48872

Petrocol D2887 Column Test Mix

Description	Cat. No.
6 x 1mL	48889

SPB-1 Thin Film Column Test Mix

For 0.10µm film SPB-1 columns.

Description	Cat. No.
1mL	48273

SPB-1 Thick Film Column Test Mix

For 3µm and 5µm film SPB-1 columns.

Description	Cat. No.
1mL	48275-U

SPB-50 Column Test Mix

Description	Cat. No.
1mL	48280-U

Sup-Herb Test Mixes

For Sup-Herb columns or herbicide columns of comparable selectivity.

Herbicides Mix 1

 $100\mu g/mL$ each component in ethyl acetate.

Eptam®	Terbacil
Sutan®	Sencor®
Tillam® (Pebulate)	Bromacil
Ordram® (Molinate)	Paarlan® (Isopropalin)
Ro-Neet® (Cycloate)	GOAL® (Oxyfluorfen)
Treflan® (Trifluralin)	Velpar (Hexazinone)

Description	Cat. No.
1mL	49136

Herbicides Mix 2

 $100\mu g/mL$ each component in ethyl acetate.

Vernam [®]	Tolban® (Profluralin)
Propachlor	Dual®
Balan [®]	Prowl [®]
Simazine	Oxadiazon
Propazine	

Description	Cat. No.
1mL	49138-U

Chromatography Tips

Evaluating Column Performance

Retention factor (k') is a measure of how much time a solute spends in the stationary phase, and therefore is a measure of film thickness. It can be calculated by using the following equation::

$$k' = \frac{t_R - t_M}{t_M} = \frac{t_R'}{t_M}$$

where k' = retention factor

 t_{R} = retention time of solute

 $t_{_{M}}$ = retention time of unretained peak

 t_{R}' = adjusted retention time of solute

Effective plate number (N_{eff}) is a measure of a column's resolving power. The more plates, the greater the resolving power. Effective plate number can be calculated by using the following equation:

$$N_{eff} = 5.545 \left(\frac{t_{R}'}{W_{h}} \right)^{2}$$

where N_{eff} = effective plate number

 t_{R}' = adjusted retention time of solute

W_b = peak width at one-half peak height

An alternative to $N_{\rm eff}$ is the **effective plate height (H_{\rm eff}).** The smaller the height equivalent to a plate, the higher the resolving power of the column. Effective plate height can be calculated by using the following equation:

$$H_{eff} = \frac{L}{N_{eff}}$$

where H_{eff} = effective plate height

L = column length

 N_{eff} = effective plate number

Coating efficiency is the ratio between the calculated number of theoretical plates for a column and the potential number of theoretical plates the column could have. A perfect column would have a coating efficiency of 100% (although measurement variation can lead to calculated values greater than 100%). Columns can have coating efficiencies lower than 100% without being poor columns. This stems from the way the maximum potential number of theoretical plates is calculated. The complete equation takes into account the film thickness of the stationary phase and the linear velocity of the mobile phase. If the film thickness is small, it is negligible and can be ignored. Likewise, if the linear velocity is at optimum, this term can be ignored. When these terms have values such that they are no longer negligible, but are still ignored (such as with very thick film columns, or when columns are tested at linear velocities far from optimum), calculated coating efficiencies will appear small even though the column is of good quality.

Operating Temperature Limits

Lower Limit The minimum operating temperature for a stationary phase is the lowest temperature that a column having that phase can be subjected to before realizing a loss of 50% in column efficiency. This lower limit approximately coincides with the melting temperature for the stationary phase.

Using the column at temperatures below the lower operating limit of the stationary phase yields atypically broad peaks.

Upper Limit The upper temperature limit for a stationary phase generally depends on the type of polymer backbone, functional group type, percent substitution of the functional group to the polymer backbone, and the column operating conditions. The upper temperature limit of phenyl-containing polysiloxanes generally increases with increasing phenyl substitution. Backbones of polysiloxane are more thermally stable than polyethylene glycol. Polysiloxane phases containing high percentages of cyanopropyl are less thermally stable than polysiloxanes with phenyl groups.

The upper temperature limit is determined by the column bleed level, column life-time, and reproducibility of data for columns operated at high temperatures. The recommended upper temperature limits listed for Supelco columns are for temperature-programmed analyses (generally held for less than 10 minutes). The maximum isothermal temperature for a column is 10-20°C below the listed limit. Columns operated isothermally near their upper temperature limits will gradually show signs of deterioration (e.g., tailing peaks, shorter retention times, and loss of resolution). The upper temperature limit is attainable only when the carrier gas is purified of moisture, oxygen, and particles.

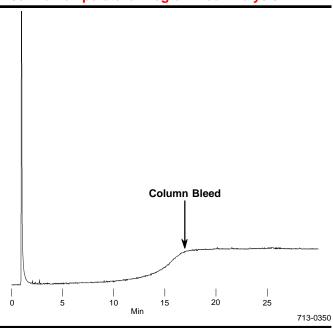
Column Bleed

Column bleed is the chromatographic background signal which results from the elution of stationary phase degradation or depolymerization products. Even low levels of volatile degradation products can produce relatively strong signals by the highly sensitive detectors commonly used in capillary gas chromatography. These materials are always present in at least trace amounts and are not necessarily an indication of a damaged column.

During a temperature programmed analysis, bleed is detected as a smooth baseline rise with no discrete peaks (Figure L). The baseline remains relatively level before the rise begins and after the maximum temperature is reached. The smooth nature of the baseline rise is a consequence of the continuous generation of stationary phase degradation products. As the oven temperature is increased, the rate at which these products are generated and eluted also increases.

Columns containing larger quantities of stationary phase generate correspondingly larger quantities of degradation products. Thus, column bleed increases with increasing film thickness and column length. Polar phases generally are less thermally stable than less polar phases, and exhibit higher bleed levels. The extent to which bleed is detected depends on the sensitivity and selectivity of the detector. For example, a nitrogen-phosphorus detector (NPD) will be much more sensitive to degradation products derived from a cyanopropyl phase than those from a dimethylsilicone phase, due to the presence of nitrogen in the stationary phase.

Figure L. Column Bleed Creates the Baseline Rise in a Temperature Programmed Analysis



Degradation of silicone stationary phases follows two reaction pathways. One involves depolymerization of the polymeric stationary phase, forming volatile cyclic silicone oligomers. This reaction is catalyzed by inorganic acids and bases. Therefore, samples contaminated with these materials should not be injected onto the column. Alternatively, organic side groups can be cleaved from the polymer backbone, generating volatile fragments. Because this process is promoted by oxygen, contaminated carrier gas and leaks in the gas lines or injector increase column bleed.

Discrete peaks in a blank analysis are not an indication of column bleed, but rather are the result of column contamination. Contamination generally arises from septum bleed, carrier gas impurities, or residual material from a previous analysis.

Capillary Injection Techniques

Four primary injection techniques are used in capillary gas chromatography: split, splitless, direct, and on-column injections. Each of these injection types has a specific purpose. A common denominator in all of these techniques is the importance of proper installation of the column in the injection port. It is critical to follow the instrument manufacturer's recommended insertion distances for installing the column, as the distance will differ from manufacturer to manufacturer, as well as among the various injection techniques.

Split injection is a vaporizing-type injection and is probably the most commonly used injection technique. The technique is designed to reduce the amount of sample reaching the column. It is primarily used with highly concentrated samples, with per component quantities ranging from 0.1-20µg/µL. This technique is used because capillary columns have a very small sample capacity, relative to packed columns. Split injection provides the highest efficiency and resolution of any of the injection techniques used in capillary GC, because high carrier gas velocities are used to transfer the sample to the column. Both split and splitless injection require specialized pneumatics systems designed for use with capillary columns, i.e., the so-called split/splitless injector.

In split injection, the sample is injected into a heated injection port and is vaporized in an area of very high carrier gas flow. As the vaporized sample flows through a tortuous path provided by the design of the inlet liner (splitter liner), it is mixed with carrier gas. Because of differences in the carrier gas flow rate to the column and through the split vent, a small portion of the sample is transferred to the capillary column and the bulk of the sample exits through the split vent port. The difference in flows establishes the split ratio. The typical calculation for determining the split ratio and setting a split ratio of 100:1 is:

Split Ratio =
$$\frac{\text{column} + \text{split vent} + \text{septum purge flows}}{\text{column flow}}$$
=
$$\frac{1\text{cc/min} + 97\text{cc/min} + 2\text{cc/min}}{1\text{cc/min}}$$
=
$$100:1$$

Because of the very high injector carrier gas flow velocities and rapid transfer of the sample to the column, which is important in providing the high efficiencies for split injection, discrimination can occur in split injection. This occurs when a sample contains components with a very broad molecular weight distribution. Due to slight differences in vaporization rates, the higher molecular weight components require slightly more time to vaporize and thus may not be thoroughly vaporized prior to the split. Another key point of split injection is that since it is a vaporizing-type injection, thermally labile components can break down. It is also important to make the injection as rapidly as possible. If injection is slow, band broadening will occur, reducing some of the inherent efficiency of this injection technique.

Splitless injection is a vaporizing injection technique based on the use of a split injection system in the non-splitting mode. It is primarily used for trace level analysis of sample components.

In splitless injection, a large amount of dilute sample is injected into a heated injection port, where it is vaporized, and a low flow of carrier gas sweeps the majority of the vaporized sample into the inlet of the column. During injection in classical solvent effect splitless injection, the temperature of the column is kept 10°-20°C below the boiling point of the sample solvent matrix, so that the vaporized sample entering the column recondenses or "focuses" in a tight band at the column inlet. Focusing is critical to the injection process. If the sample does not recondense in a tight band at the column inlet, the resulting peak widths will reflect the volume of the injection port rather than the efficiency of the column.

After approximately 1.5-2 volumes of carrier gas have passed through the injection port and into the column, the split vent valve is opened and any residual sample remaining in the injection port is vented through the split vent port. After a predetermined period of time, the oven temperature is programmed and the increasing temperature initiates sample component elution through the column

Sample introduction in splitless injection is typically slow, in comparison to the rapid injections used in split injection. A slow injection is required because volume is limited in the splitless injector liner – a typical 10cm x 2mm ID splitless injector liner has an internal volume of 0.31cc. Depending on the amount of sample introduced and the expansion coefficient of the solvent matrix, this internal volume potentially can be overloaded, causing sample to be forced back into the carrier gas lines.

Since the injected vaporized sample will be recondensed at the inlet of the column, the analyst should not see any decrease in column efficiency due to the slower injection process. Once the sample has been injected and allowed to recondense at the column inlet, the solenoid valve on the split vent is opened to vent residual sample from the injection port. Timing of switching the solenoid valve is important as this, in conjunction with the injector liner internal diameter, carrier gas volumetric flow rate, and sample volume are key variables in determining the time to open the solenoid valve. Once the valve is opened the chromatographic process continues as a typical temperature programmed analysis.

Direct injection is a vaporizing injection technique typically used with wide bore capillary columns (ID \geq 0.53mm) in a packed column gas chromatograph which has been converted for use with these columns.

In this technique, the sample is injected slowly into the heated injector liner, vaporized, and transported in its entirety directly to the column in the carrier gas flow. No splitting or specialized pneumatics are required, although a low flow mass flow controller might be needed to allow proper control of the low volumetric flow rates typically used with these columns. Since all of the sample is transferred to the column, sample discrimination does not occur. Thermally labile samples still can be decomposed in this process, however, because the process is a vaporization-type procedure.

It is important to use a slightly reduced injection speed. The injector liner has a limited volume and, if exceeded, the vaporizing sample could backflash onto the cooler bottom face of the septum or into the carrier gas inlet lines, and recondense. This will produce broad, tailing peaks, especially for the solvent. Since all of the sample is transferred to the column, direct injection is ideal for quantitative analysis.

Cold on-column injection is a nonvaporizing injection technique in which the liquid sample is directly deposited at the inlet of the capillary column. The oven temperature program is then used to vaporize the sample components and begin the elution process. With narrow bore capillary columns (ID \leq 0.32mm), a specialized injection system and syringes with narrow OD needles are required to properly insert the needle into the bore of the column. With 0.53mm ID columns, a specialized liner is required to properly guide the needle into the column, but standard 26 gauge needles can be used.

Because this is a nonvaporizing injection technique, and all sample components are quantitatively deposited directly into the column, it is ideal for use with thermally labile analytes and provides very good results for quantitative analyses. It is important to inject the sample slowly, to eliminate the potential for aerosol formation, which would broaden peaks and lose some of the efficiency of the column.

Secondary cooling of the entire column, or of a short section of the inlet end of the column, also can be used to aid in condensing the injected sample in a tight band at the column inlet.

Injection Port Liners

The injector liners used in conjunction with the various capillary injection techniques play a vital role in the quantitative transfer of the sample to the capillary column. Both the design and the inertness of the injector liners impact the overall system performance.

In general, inlet liners should have a proper expansion volume, to allow proper vaporization of the injected liquid sample according to the chosen injection technique. The liners should be very well deactivated, to minimize adsorption of active sample components. We use state of the art silylation techniques to deactivate all of our injector liners.

Split Injection Liners Examples of typical split injector liners are shown in our general catalog. The cup liner is one of the most commonly used liners for split injections.

Key design features of all split injection liners are a large expansion volume followed by a constricted area. The large expansion volume is where the sample vaporizes and begins to mix with carrier gas. The constricted area, the cup, is a critical part of the design. This is where turbulent flow is established, to aid in proper mixing of the vaporized sample before it reaches the split point and column inlet. Proper mixing ensures that a representative part of the sample enters the column. If the sample is inadequately mixed, discrimination can occur, leading to poor quantitative results.

Deactivation of the liner is also important, because a poorly deactivated liner will adsorb active sample components.

Splitless injector liners are typically straight tubes with none of the constrictions in split injector liners.

The critical factor in the design of the splitless liner is the internal diameter and the corresponding internal volume. Typical internal diameters for splitless injector liners are 2mm and 4mm. A limited internal volume is important, because the objective of the technique is to transfer as much of the sample to the column as possible before opening the solenoid valve for the split vent. If a liner with a large internal volume is used with the low carrier gas volumetric flow rates in capillary GC, excessive purge times will be needed to ensure that much of the sample has been transferred to the column prior to venting the remaining sample from the injection port.

Also critical is the inertness of the liner. Because the vaporized sample has a significantly longer residence time in this liner, compared to the time in a split injection liner, there is increased opportunity for adsorption of active sample components.

Carrier Gas Purity

Gas purifiers and traps are absolutely necessary in a high performance capillary chromatography system. Clean, contaminant-free gases are essential for optimized performance from the capillary column, universal detectors, and specific detectors. Even trace amounts of contaminants in your carrier and makeup gas delivery system can cause column damage, detector noise, baseline irregularities and, in general, poor analytical performance.

Use of high capacity moisture and oxygen traps, combined with an oxygen/moisture indicating trap, will allow you to maintain or improve the overall performance of the chromatographic system. Routine monitoring and maintenance of your gas delivery system will help ensure good analyses and minimize expensive down time and troubleshooting.

SUPELCO

Septa

When performing temperature-programmed analyses, you might observe peaks or a baseline rise you cannot trace to the sample, or to column bleed. These problems are often caused by septum bleed. Volatile materials from the septum accumulate at the column inlet during the cooldown portion of a temperature program. When the column is reheated for the next sample, these volatiles also are eluted, producing extra peaks, a general baseline rise, or both artifacts. You can minimize these problems by understanding the factors that create septum bleed.

Septum Quality The quality traits to look for in a septum are low bleed, resistance to leaks, and easy penetration. If you are using a sensitive detector, such as an ECD, NPD, or MSD, poor quality septa can make the baseline extremely unstable.

Septum Conditioning Septum bleed can be greatly reduced by conditioning the septum overnight. Changing the septum at the end of the day allows sufficient conditioning, and is recommended by most septum manufacturers. Our Thermogreen™ LB-2 septa are conditioned as part of the manufacturing process, saving you the time, effort, and expense of solvent extraction, heating, or other treatments.

Septum Durability A septum should allow easy needle penetration, but resist fragmentation. Septum fragments in the injector liner bleed and adsorb active sample components. Thermogreen LB-2 septa offer easy penetration and excellent resistance to fragmentation.

Septum Tips

- Keep Thermogreen septa in the original metal container until you use them. Storing them in plastics can contaminate them.
- Use forceps to remove septa from the container.
 Finger oils can create bleed.

For more information about septa, request Application Note 82.

Rinsing Capillary Columns

If the performance of your column is deteriorating (as revealed by using a test mix, for example), and you know the injection liner is clean and properly deactivated, the problem could be adsorptive sample residue or septum fragments contaminating the inlet of the column. You can cure this problem by cutting two loops from the column inlet. Alternatively, you can rinse Supelco bonded phase capillary columns (with phase films of less than 1.5µm) with pentane, methylene chloride, or acetone, using our solvent rinse kit (Catalog No. 23626).

For more information about rinsing capillary columns, and other tips, request Bulletin 853, *Troubleshooting Guide for Capillary Gas Chromatography*.

Guard Columns and Retention Gaps

A one-meter piece of fused silica tubing attached to the capillary column inlet protects the column from nonvolatile sample components and "dirty" samples. When the guard column becomes contaminated (e.g., performance with a test mix begins to deteriorate), simply replace it with a new one.

Fused silica tubing attached to the column inlet also can serve as a retention gap, allowing large samples to be injected without solute band broadening.

A short length of flexible fused silica tubing also makes an ideal GC/MS or GC/FTIR transfer line and, in a purge and trap system, is excellent for connecting the concentrator or trap to the column inlet.

Capillary Column Connectors

The Supelco™ Butt Connector consists of a double-tapered ferrule and a stainless steel compression housing with a threaded cap (Catalog No. 23804). It provides a gas-tight seal within the ferrule, with no change in column efficiency or inertness. Light in weight (<4.5 grams), the butt connector can be suspended without putting undue stress on the column. It is a valuable tool for mending a broken fused silica column, attaching a guard column to an analytical column, connecting two columns of different polarity in series (for tuned separations), eliminating end straightening for glass columns, and routing a permanent fused silica mass spectrometer manifold line to the source, for easy column changes.

Ferrules for the butt connector are available with internal diameters suitable for all Supelco capillary columns.

GlasSeal™ Connectors (Catalog Nos. 20479 and 20480) enable you to connect polyimide-coated fused silica tubing ends, for a leak-tight connection without the use of any tools. They are useful for repairing broken columns, adding a retention gap or guard column, or connecting columns in series. The connection is made by simply pressing two tubing ends of any diameter (same or different, 0.20mm to 0.53mm ID) into the connector. The connectors are light in weight, inert, and have low dead volume.

*Supelco US patent No. 4,529,230.

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For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

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