msp

Gas Chromatography Troubleshooting and Reference Guide

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msp

GC Troubleshooting and Reference Guide

Troubleshooting

Reference

The gas chromatograph and capillary column function as a complete system and not as two individual parts. A problem or deficiency in any part of the system usually will result in some type of chromatographic difficulty. The same problem can be caused by a number of different system deficiencies. A logical and controlled roubleshooting procedure will quickly and accurately identify the source of the problem. This will result in the fastest, easiest and most complete solution to the problem.

Troubleshooting is a skill that becomes easier with practice. Someone equipped with the right tools and a rudimentary understanding of capillary column gas chromatography, can identify, locate and correct problems with minimal amount of effort.

Troubleshooting Tools

Flowmeter

A digital or manual model with a range of 10 to 500 mL / min is suitable.

New Syringe

A working syringe that has not been used for samples should be available. Some problems may actually be syringe or autosampler related.

Methane or Another Nonretained Compound

A non-retained compound is used to set and verify carrier gas flow and to check out injector operation and setup.

New Septa, Ferrules and Injector Liners

These are used to replace parts that eventually become defective, worn out or dirty.

Leak Detector

Electronic models are recommended. Liquid leak detection fluids are satisfactory, but care has to be exercised to avoid possible contamination problems.

Column Test Mixture or Reference Sample

These are used to diagnose select system and column problems. They are useful to compare current system performance to past performance.

Checkout Column

This is a column that is not used for samples. The performance and quality is known so that evaluation of the system can be made. It helps to verify or eliminate the previous column as the source of a problem.

Instrument Manuals

These are not a last resort. The manuals are a good source of troubleshooting information special to a particular model of gas chromatograph. Performance specifications are often contained in the manuals.

Eight Problem Categories

Most performance problems can be placed within one of eight areas. These are baseline disturbances, irregular peak shapes or sizes, retention time shifts, loss of separation or resolution,quantitation difficulties, rapid column deterioration, ghost peaks and broad solvent fronts. It is not uncommon to have more than one of these problems occurring at the same time. Sometimes, it is difficult to determine the actual nature of the problem. This makes a logical and systematic approach to problem solving very important.

It is important to realize that the following comments and recommendations are generalizations and simplifications. Every possible problem or correction cannot be covered, nor can every detail be mentioned. The page where additional information can be found is shown in parentheses following each solution.

Baseline Disturbances

(Figure 1) see page 2 for figure

Spiking:

1. Particulate matter passing through the detector.
   Solution: Clean the detector per the instruction manual.
2. Loose connections on cables or circuit boards (usually random spiking).
   Solution: Clean and repair the electrical connections as needed.

Noise:

1. Contaminated injector and / or column.
   Solution: Clean the injector. Solvent rinse the column (pg 26).
2. The column is inserted into the flame of an FID, NPD or FPD.
   Solution: Reinstall the column.
3. Air leak when using an ECD or TCD.
   Solution: Find and repair the leak.
4. Incorrect combustion gases or flow rates when using an FID, NPD or FPD.
   Solution: Check and reset the gases at their proper values.
5. Physical defect in the detector.
   Solution: Clean or replace parts as necessary.
6. Defective detector board.
   Solution: Consult the instruction manual or contact the GC manufacturer.

Baseline Disturbances *(Continued)*

Wander:

1. Contaminated carrier gas if using isothermal conditions.
   Solution: Change the carrier gas or use (change) carrier gas impurity traps (pg 25).
2. Contaminated gas chromatograph.
   Solution: Clean the injector and / or gas lines. Solvent rinse the column (pg 26).
3. Poor control of the carrier gas or detector gas flows.
   Solution: Clean, repair or change the flow controller.
4. Poor thermal control of the detector.
   Solution: Consult the instruction manual or contact the GC manufacturer.

Drift (Upward):

1. GC or column contamination.
   Solution: Clean the injector. Solvent rinse the column (pg 26).
2. Damaged stationary phase.
   Solution: Replace the column. Determine the cause of the damage (oxygen, thermal or chemical) to prevent future problems (pg 15).

Drift (Downward):

1. Incomplete conditioning of the column.
   Solution: Condition the column until a stable baseline is obtained (pg 15).
2. Unequilibrated detector.
   Solution: Allow the detector enough time to equilibrate.

Baseline Disturbances

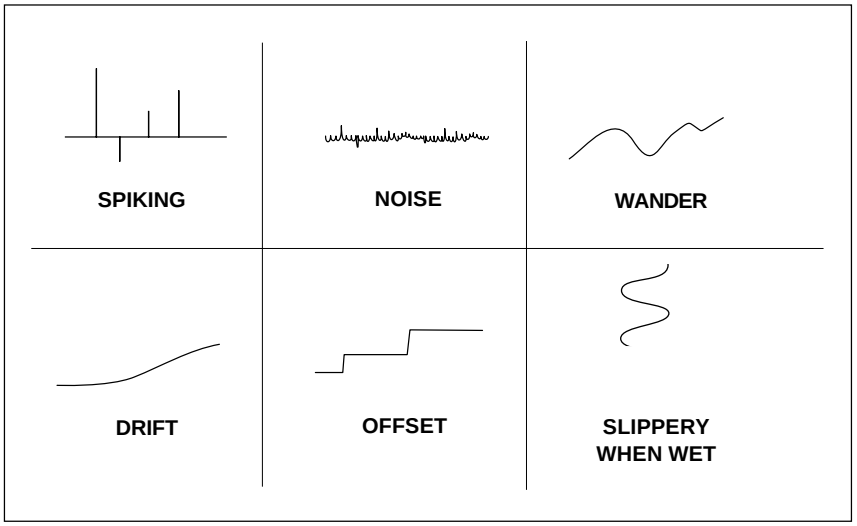


Figure 1

Offset:

1. Injector or column contamination.
   Solution: Clean the injector. Solvent rinse the column (pg 26).
2. Column is inserted into the flame of an FID, NPD or FPD.
   Solution: Reinstall the column.
3. Contaminated carrier or detector gases.
   Solution: Change the gases or install (change) impurity traps (pg 25).
4. Contaminated detector.
   Solution: Clean the detector.
5. Malfunctioning or improperly set recording device.
   Solution: Check the recorder settings. Consult the instruction manual, or contact the manufacturer.

Irregular Peak Shapes or Sizes

(Figure 2) See page 3 for figure

No Peaks:

1. Plugged syringe.
   Solution: Clean the syringe or use a new syringe.
2. Broken column.
   Solution: Replace or reinstall the column.
3. Injecting the sample into the wrong injector.
   Solution: Use the correct injector or move the column to the correct injector.
4. Column installed into the wrong detector.
   Solution: Reinstall the column into the correct detector.
5. Integrator or recording device is connected to the wrong detector or not connected at all.
   Solution: Connect the integrator to the correct detector.
6. Detector gases improperly set or not on.
   Solution: Check and reset the detector gases.

Irregular Peak Shapes or Sizes (Continued)

7. Very low or no carrier gas flow.
Solution: Immediately lower the column temperature to 35-40C. Measure and verify the carrier gas flow rate (pg 17). Check for leaks.

All Peaks Reduced in Size:

1. Partially plugged syringe.
Solution: Clean the syringe or use a new syringe.

2. Change in the injection technique.
Solution: Check the injection technique and verify that it is the same as before.

3. Large leak in the injector (usually accompanied by poor peak shapes).
Solution: Find and repair the leak.

4. Split ratio is too high.
Solution: Lower the split ratio (pg 19).

5. Too short of a purge activation time for splitless injections.
Solution: Increase the purge activation time (pg 20).

6. Very high septum purge flow.
Solution: Decrease the septum purge flow (pg 18).

7. Too low of an injector temperature (especially for high molecular weight or low volatility compounds).
Solution: Increase the injector temperature (pg 18).

8. Column temperature is not hot enough.
Solution: Increase the column temperature or the upper temperature value of the column temperature program (pg 12).

9. Initial temperature of the column is too high for splitless or on-column injections.
Solution: Decrease the initial column temperature or use a higher boiling solvent (pg 20).

10. High background signal caused by contamination, excessive column bleed (damage) or autozero problem.
Solution: Clean the GC. Solvent rinse the column (pg 26). Replace the bleeding column (pg 12-13). Check the autozero function and setting.

11. Improperly operated detectors.
Solution: Consult the instruction manual for the proper gas flows and type and operating guidelines.

12. Impurities in the detector gas.
Solution: Use impurity traps and / or replace the contaminated gas (pg 25).

13. Detector-compound mismatch.
Solution: Make sure that the detector will respond to the compounds being analyzed.

14. Excessive attenuated integrator signal.
Solution: Check and verify the attenuation settings.

15. Sample concentration or integrity problems.
Solution: Check the sampleís concentration or stability.

Irregular Peak Shapes and Sizes

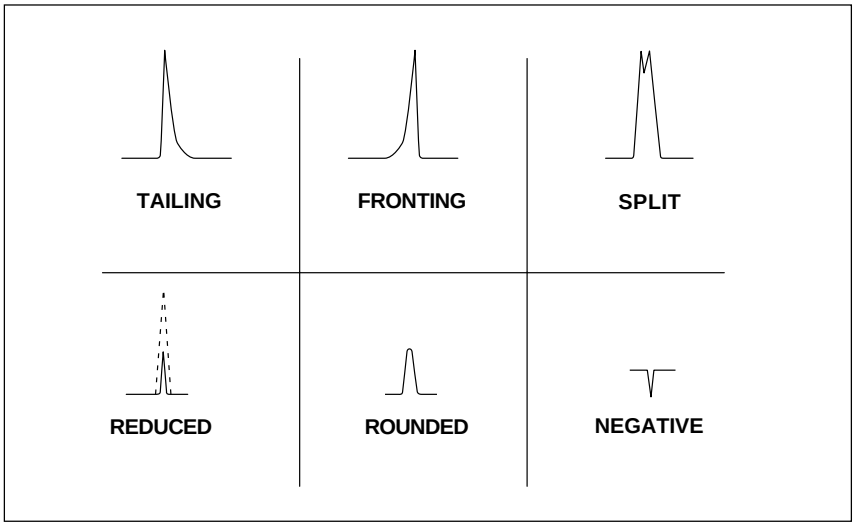


Figure 2

Select Peaks Reduced in Size:

1. Column and / or liner activity or contamination, if the reduction or loss is for active compounds (e.g., amines, carboxylic acids, alcohols, diols).
Solution: Clean or replace the injector liner (pg 22-23). Solvent rinse or replace the column (pg 26).

2. Leak in the injector, if the reduction or loss is the most volatile compounds.
Solution: Find and repair the leak.

3. Too high of an initial column temperature for splitless or on-column injections.
Solution: Decrease the initial column temperature or use a higher boiling solvent (pg 20).

4. Mixed sample solvents for splitless or on-column injections.
Solution: Use a single solvent for sample injection (pg 20).

5. Decomposition or error in the sample.
Solution: Check and verify the sample integrity and concentration.

Irregular Peak Shapes or Sizes *(Continued)*

Tailing Peaks:

1. Active injector liner or column.
   Solution: Clean or replace liner (pg 22-23). Replace the column if it is damaged.
2. Contaminated injector liner or column.
   Solution: Clean or replace the injector liner (pg 22-23). Solvent rinse the column (pg 26).
3. Dead volume caused by a poorly installed column, liner or union.
   Solution: Check and verify the installation of each fitting. Re-install the column, if necessary.
4. Poorly cut column end.
   Solution: Recut and reinstall the column (pg 15).
5. Polarity mismatch of the stationary phase, solute or solvent.
   Solution: Change to a solvent or phase that have a better polarity match (pg 8).
6. Cold spot in the flow path.
   Solution: Check the flow path of the sample for possible cold spots or zones.
7. Solid debris in the liner or column.
   Solution: Clean or replace the liner (pg 22-23). Cut the ends of the column until the debris is removed (pg 15).
8. Poor injection technique (usually too slow of an injection).
   Solution: Change injection technique.
9. Too low of a split ratio.
   Solution: Increase the split ratio (pg 19).
10. Overloading on a PLOT column.
    Solution: Decrease the amount of sample reaching the column.

    Some compounds such as alcoholic amines, primary and secondary amines, and carboxylic acids tail on most columns.
11. ***Solution:*** Use a pH-modified stationary phase. Derivatize the compounds. Some peaks will always exhibit some tailing.

Rounded or Flat-Topped Peaks:

1. Overloaded detector.
   Solution: Decrease the amount of sample reaching the detector.
2. Exceeding the range of the integrator or recording device (especially for computer systems).
   Solution: Reset the range or attenuation levels on the recorder.

Split Peaks:

1. Poor injection technique (jerky or erratic).
   Solution: Change injection technique (smooth and steady plunger depression).
2. Poorly installed column in the injector.
   Solution: Recut the column end (pg 15) and reinstall in the injector.
3. Column temperature fluctuations.
   Solution: Check the oven temperature or contact the GC manufacturer.
4. Coelution of two or more compounds.
   Solution: Check for any changes in the operational parameters. Contamination or a change in the sample will introduce additional compounds to the injected sample. Check for these possibilities.
5. Mixed sample solvent for splitless or on-column injections.
   Solution: Use a single solvent for sample injections (pg 20).

Negative Peaks:

1. All peaks are negative.
   Solution: Check the polarity of the recorder connections.
2. Select peaks on a TCD.
   Solution: Compound has greater thermal conductivity than the carrier gas; a negative peak is expected in this case.
3. After a positive peak on an ECD.
   Solution: Dirty or old ECD cell. Clean or replace the ECD.

Retention Time Shifts

1. Different column temperature.
   Solution: Check and verify the column temperature or temperature program.
2. Different carrier gas flow rate or linear velocity.
   Solution: Check and verify the carrier gas flow rate or linear velocity (pg 16-17).
3. Leak in the injector, especially the septum.
   Solution: Find and repair the leak. Change the septum.
4. Contaminated column.
   Solution: Solvent rinse the column (pg 26).
5. Change in the sample solvent.
   Solution: Use the same solvent for all samples and standards.

Loss of Separation or Resolution

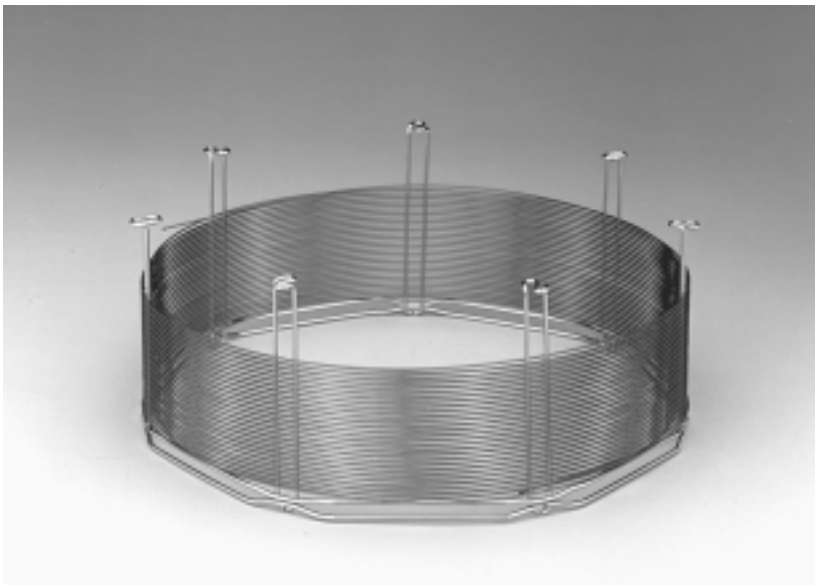
1. Contaminated column.
   Solution: Solvent rinse the column (pg 26).
2. Damaged stationary phase.
   Solution: Replace the column. Excessive bleed should be evident also (pg 12-13).
3. Different column temperature, carrier flow rate or column.
   Solution: Check and verify temperature programs, flow rates and column identity.
4. Large changes in the sample concentration.
   Solution: Adjust or compensate for the concentration change.
5. Improper injector operation.
   Solution: Check the temperature, split ratio, purge time and type of liner (pg 18-23). Also check for leaks.

Quantitation Difficulties

1. Injection technique.
   Solution: Use a consistent injection technique.
2. Split discrimination.
   Solution: Use a consistent injection technique (volume, injector temperature and split ratio) (pg 18-19).
3. Using a different purge activation time for splitless injection.
   Solution: Use a consistent purge activation time (pg 20).
4. Baseline disturbances.
   Solution: See the section on baseline disturbances (pg 1-2).
5. Improper integrator or recorder settings.
   Solution: Check and verify the integrator and recorder settings.
6. Inconsistent detector gas flows or temperatures.
   Solution: Check and verify detector operation.
7. Column or liner activity (adsorption).
   Solution: Clean or replace the injector liner (pg 22-23). Solvent rinse or replace the column.

Rapid Column Deterioration

1. Exposure of the column to air (oxygen) at elevated temperatures.
   Solution: Find and repair any leaks (pg 1). Check the quality of the impurity traps and carrier gas (pg 25).
2. Exceeding the upper temperature limit of the column for prolonged periods.
   Solution: Replace the column. Do not exceed the upper temperature limits (pg 12).
3. Chemical damage.
   Solution: Do not inject inorganic acids or bases (pg 13).
4. Contamination of the column with high molecular weight materials.
   Solution: Use a sample preparation technique to remove the problem contaminants. Use a guard column (pg 24, 26).
5. Column breakage.
   Solution: Avoid abrading or scratching the column. Avoid sharp turns or bends in the tubing (pg 14-15).



Ghost Peaks

1. Contamination of the injector or column.
   Solution: Clean the injector and liner (pg 22-23). Solvent rinse the column (pg 26).
2. Septum bleed.
   Solution: Use a higher temperature septum. Lower the injector temperature. Condition septum before use (pg 18).
3. Previous run terminated too soon.
   Solution: Use a higher temperature to elute all of the sample components. Prolong the run time to allow the complete elution of the sample.

Broad Solvent Front

1. Poorly installed column.
   Solution: Recut (pg 15-16) and reinstall the column.
2. Leak in the injector.
   Solution: Find and repair the leak.
3. Too low of a split ratio.
   Solution: Use a higher split ratio (pg 19).
4. Too low of an injector temperature.
   Solution: Use a higher injector temperature (pg 18).
5. Too long of a purge activation time for splitless injections.
   Solution: Use a shorter purge activation time (pg 20).
6. Large injection volume.
   Solution: Decrease the injection size.
7. Low column temperatures and high boiling solvent.
   Solution: Use a higher initial column temperature or a lower boiling solvent (pg 20).
8. High column temperatures and low boiling solvent.
   Solution: Use a lower initial column temperature or a higher boiling solvent (pg 20).

MSP offers a variety of products that assist with troubleshooting. Please contact us to get the current cataolog.

ADM1000 Model Flowmeter

Flow rates are critical to efficient GC operation. Make sure flow rates are correct by using the J&W model ADM series of flowmeters. They are based on ìacoustic displacementî technology. No bubbles, messy liquids or breaking glassware to deal with. Ideal for field or laboratory use. These flowmeters are compatible with all noncorrosive gases. A computer-optimized calibration incorporating a NIST calibrated flow standard ensures the highest available accuracy, making ISO9000 and GLP compliance that much easier.

ADM1000 Model Flowmeter

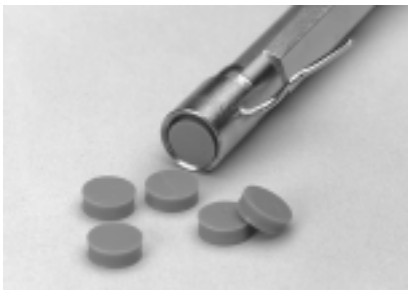
Hamilton Cemented Needle

Be sure to have a clean, working syringe. Problems can sometimes be traced to the autosamplers. J&W offers a complete line of Hamilton Syringes.

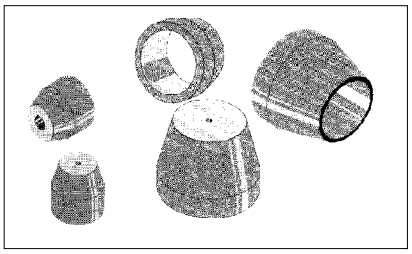
Hamilton Cemented Needle

Septa and Ferrules

MSP offers a complete line of silicone septa and ferrules. Over-used septa and ferrules are prone to leaks, which can cause column bleed due by allowing oxygen to be introduced. Particulates from the overused septa and ferrules can also cause problems when they contaminate the liner.



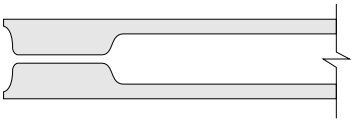
Septa



Ferrules

4 mm Splitless Liner

Pyrolyzed compounds can build up on liner walls. This buildup causes clogging and sample adsorption, which can result in a nonrepresentative chromatogram.



4 mm Splitless Liner

Technical Support

MSP employs skilled scientists whose first priority is to answer your technical questions. These scientists offer analytical consulting and assist you in selecting columns and accessories. No matter which produts you are using, theyëre here to help you with your chromatography questions.

msp
MSP KOFEL
Industriestrasse 26
CH-3052 Zollikofen
mass spectrometer & signal processing products

Telefon: 031 972 31 52
Fax: (+41) 31 971 46 43
info@msp.ch

Capillary Column

Upon first inspection, fused silica capillary columns appear to be quite simple. Further investigation reveals that capillary columns are actually complex, highly sophisticated devices. Considerable technological knowledge, attention to detail and refined techniques are required to produce capillary columns of the highest quality. Capillary columns are much more than just tubes containing a polymer.

What Is a Capillary Column?

A capillary column is composed of three parts (Figure 1):

1. Fused silica tubing
2. Polyimide coating
3. Stationary phase

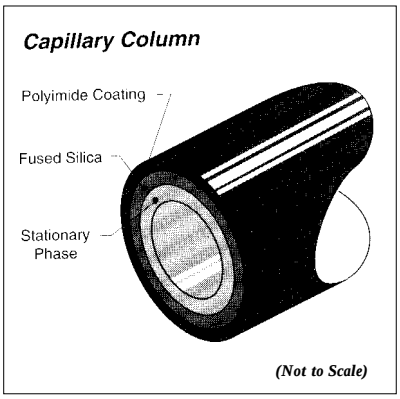


Figure 1

Fused Silica Tubing

The fused silica used to manufacture capillary columns is synthetic quartz typically containing less than 1 ppm metallic impurities. Blanks (preforms) of fused silica are drawn through a furnace at a carefully metered rate. Laser micrometers are used to ensure a constant tube diameter. As part of the column manufacturing process, the inner surface of the tubing is purified and deactivated. This process is used to minimize chemical activity (unwanted interactions between the tubing and the injected sample) and to create a chemically uniform surface for the stationary phase.

Polyimide Coating

Immediately after the drawing process, the outer surface of the tubing is coated with polyimide. This polyimide coating serves two functions. First, it fills any flaws in the tubing. Second, it provides a strong, waterproof barrier. Both functions add to the strength and durability of the tubing. Any damage to the polyimide coating will result in a weak point and is a potential for tubing breakage. The color of the polyimide often varies between columns. ***Color differences will have no effect on column performance or durability because the polyimide coating is on the outer surface of the column.*** Column performance is strictly a function of the deactivation of the fused silica tubing and the quality of the stationary phase coated onto its inner walls.

Stationary Phase

The stationary phase is a polymer that is coated onto the inner wall of the fused silica tubing. The thickness, uniformity and chemical nature of the stationary phase are extremely important. It is the stationary phase that has the greatest influence on the separations obtained.

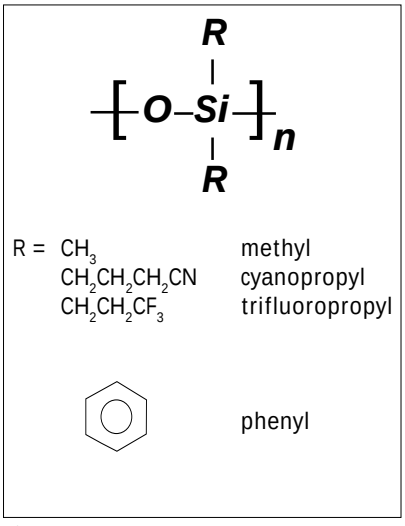


Figure 2

The most common capillary stationary phases are silicone polymers (Figure 2). The type and amount of substitution on the polysiloxane backbone distinguishes each phase and its properties. The phase description refers to the amount and type of substitution on the polysiloxane backbone. For example, a (5%-phenyl)-methyl phase has two phenyl groups bonded to 2.5%, by number, of the silicon atoms; the remaining 97.5% of the silicon atoms have methyl groups bonded to them.

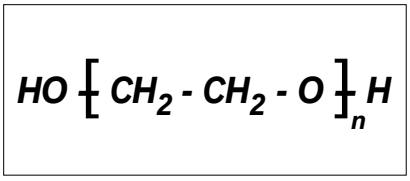


Figure 3

Another widely used stationary phase is polyethylene glycol (Figure 3). Carbowax® 20M is one of the most widely used polyethylene glycols to be used as a gas chromatographic phase. The major disadvantage to polyethylene glycol phases is their high susceptibility to structural damage by oxygen at elevated temperatures. Damage occurs at lower temperatures and lower oxygen levels than most polysiloxane stationary phases. The high polarity and unique separation characteristics of polyethylene glycol stationary phases are useful; thus, the liabilities are tolerated.

A newer class of capillary column contains a gas-solid adsorption type of stationary phase. These columns are often called porous layer open tubular or PLOT columns. PLOT columns contain a layer of solid particles coated onto the inner walls of the fused silica tubing. Instead of a gas-liquid partitioning process between the injected sample and stationary phase, a gas-solid adsorption process occurs. Examples of PLOT stationary phases include polystyrene, aluminium oxide and molecular sieve.

Stationary Phase Considerations

Within a constant set of operating conditions, it is the structure of the stationary phase that determines the ***relative*** retention (elution order) of the compounds. Focusing only on the column, the stationary phase determines the relative amount of time required for two compounds to travel through the column. The stationary phase ìretardsî the progress of the compounds moving through the column. If any two compounds take the same amount of time to migrate through the column, these two compounds will not be separated (i.e., they co-elute). If any two compounds take a different amount of time, these two compounds will be separated. In other words, the stationary phase retains one compound to a greater extent than the other.

Stationary Phase Polarity

Columns are often selected on the basis of their polarity. Polarity is a bulk property of the stationary phase and is determined by the structure of the polymer. Stationary phase polarity does not have a direct influence on the separations obtained. Polarity will have an effect on a variety of column characteristics. Some of the most important characteristics are column lifetime, temperature limits, bleed levels and sample capacity. It is the selectivity of the stationary phase that directly influences the separations. Synonymous use of polarity and selectivity is not accurate but is very common.

Stationary Phase Selectivity

As for polarity, stationary phase selectivity is determined by its structure. Stationary phase selectivity is not completely understood, nor can it be easily explained or characterized. ***Using a severe simplification and condensation,*** selectivity can be thought of as the ability of the stationary phase to differentiate between two compounds by virtue of a difference in their chemical and / or physical properties. From the perspective of a stationary phase, if there is a discernible difference in the properties of two compounds, the amount of interaction between the compounds and the phase will be different. If there is a significant difference in the interactions, one compound will be retained to a greater extent and separation will occur. If there are no discernible differences, coelution will occur. The compounds may have different structures or properties, but if a particular stationary phase cannot distinguish between the compound differences, coelution will occur.

Stationary phase and solute factors such as polarizability, solubility, magnitude of dipoles and hydrogen bonding behavior will influence selectivity. In many cases, more than one factor will be significant, thus there will be multiple selectivity influences. Unfortunately, most compound characteristics, such as the strength of hydrogen bonding or dipoles, are not readily available or easily determined. This makes it very difficult to accurately predict and explain the separations obtained for a column and set of compounds. However, some generalizations can be made. All stationary phases will have polarizability related interactions. Increased retention occurs for solutes that are more polarizable. For methyl- and phenyl-substituted polysiloxanes, it will be the only significant interaction. Solubility of the solute in the stationary phase will affect retention. The more soluble a solute is in the stationary phase, the greater its retention. Polyethylene glycols and cyano- propyl-substituted polysiloxanes have strong dipole and hydrogen bonding characteristics. Trifluoropropyl-substituted polysiloxanes will have a moderate dipole characteristic. As previously stated, because of the inexactness of these characteristics, predictions and precise explanations of solute separations are very difficult.

Bonded and Cross-Linked Stationary Phases

The first capillary columns had stationary phase coated onto the inner tubing walls without any type of chemical attachment. The stationary phase was easy to disrupt or damage with solvents, heat or contaminants. Removal of a short piece of tubing at the front of the column was often necessary to return column performance after phase disruption had occurred. The advent of bonded and crosslinked phases substantially increased the stability and lifetime of capillary columns. The stationary phase is bonded to the inner surface of the fused silica tubing by means of covalent bonds. Crosslinking is the joining of the individual strands of the polymer. Unlike nonbonded phases, bonded and crosslinked phases can be solvent rinsed if they become contaminated, and they also exhibit better thermal and solvent stability.

Column Length

The effects of column length on a separation become less important as column length increases. Resolution is a function of the square root of column length. This means that doubling the resolution between two peaks without changing any other column dimension or operational parameter, requires a fourfold increase in column length (e.g., 30 meters increased to 120 meters). To halve the resolution via length alone will require a reduction in length of 75% (e.g., 30 meters reduced to 7.5 meters). A large portion of the column length can be lost before resolution (separation) is reduced significantly. In a practical sense, removing 1 meter from a 30 meter column will decrease the resolution by only 1.7%.

Shorter column lengths are intended for samples containing a relatively small number of compounds, especially if they are not very similar in structure, polarity or volatility. Shorter columns are also useful for screening analyses. Most analyses are performed with intermediate column lengths (20-30 meters). Usually, 60 meter or longer columns are necessary only for extremely complex samples and special applications. Longer columns will exhibit higher bleed than a corresponding shorter column because of the proportionally greater amount of stationary phase.

Increased retention will be obtained with longer columns. Longer analysis times will result, especially for isothermal temperature conditions. For a temperature program situation, the extra analysis time can be somewhat reduced by using a faster ramp rate.

Column Diameter

The internal diameter will have a direct impact on the efficiency, retention characteristics and sample capacity of a column. Smaller diameter columns are more efficient than larger diameter columns. For two columns of equivalent phase, film thickness, length and quality, the smaller diameter column will provide better resolution of the peaks. Increased resolution is especially beneficial when there are closely eluting sample components.

As column diameter decreases, the retention of a given solute will increase providing no other changes to the chromatographic system have been made. This inverse relationship is approximately linear in nature. Figure 4 illustrates the difference in retention attributable to column diameter. Bleed increases slightly as column diameter increases. Larger diameter columns have greater sample capacities.

Effect of Column Diameter on Retention

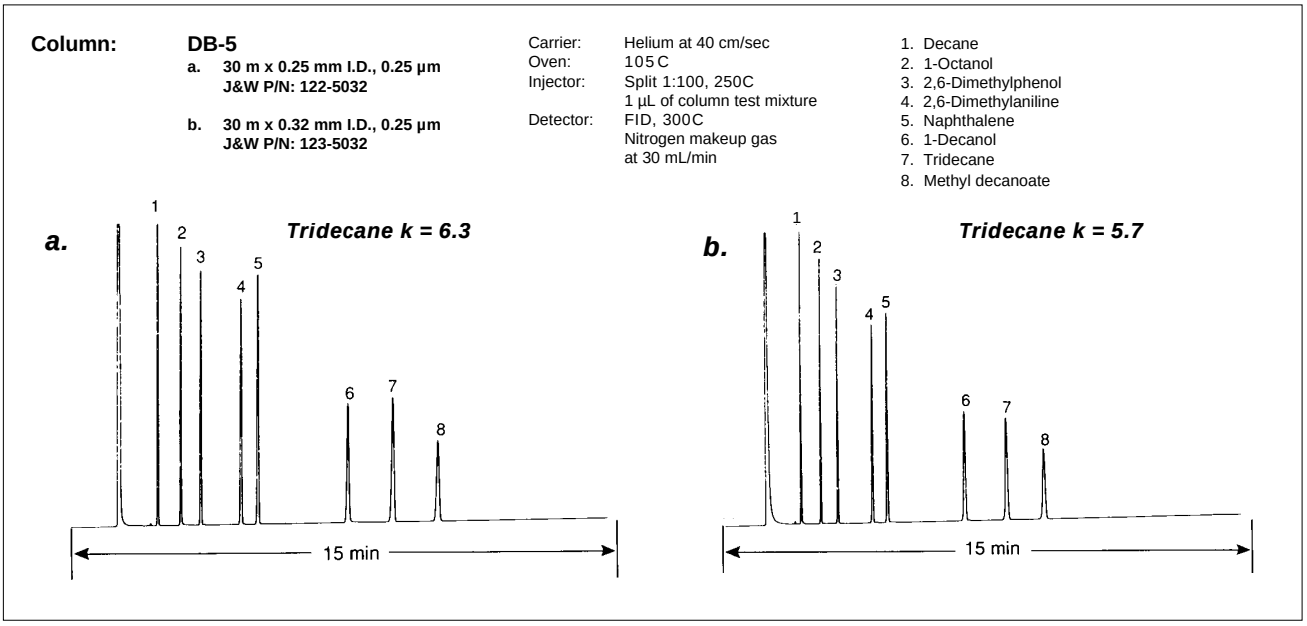


Figure 4

Film Thickness

Film thickness will primarily affect the retentive character and capacity of a column. Increasing film thickness will cause a substantial increase in the retention of a solute. Thick film columns are used primarily for the separation of extremely volatile solutes without the use of cryogenic cooling. For fast eluting solutes, the increased retention results in improved resolution. For medium to slow eluting solutes, the increased retention results in no resolution improvement, and a loss in resolution may actually occur. Increasing the film thickness to improve separation will be effective only for poorly retained solutes.

Thin film columns are useful for the analysis of low volatility or high boiling samples. Thick film columns will excessively retain the sample. Unnecessarily long analysis times or high column temperatures will result when too thick of a stationary phase is used. Figure 5 illustrates the effect of film thickness on solute retention. Sample capacity increases dramatically with increasing film thickness. Column bleed is much higher for thick film columns.

Effect of Film Thickness on Retention

Column:	**DB-5**
a. J&W P/N:	**123-5032**
	30 m x 0.32 mm I.D., 0.25 µm
b. J&W P/N:	**123-5033**
	30 m x 0.32 mm I.D.,1.0 µm
Carrier:	Helium at 40 cm/sec
Oven:	105 C
Injector:	Split 1:100, 250C
	1 µL of column test mixture
Detector:	FID; 300C
	Nitrogen makeup gas at 30 mL/min

1. Decane
2. 1-Octanol
3. 2,6-Dimethylphenol
4. 2,6-Dimethylaniline
5. Naphthalene
6. 1-Decanol
7. Tridecane
8. Methyl decanoate

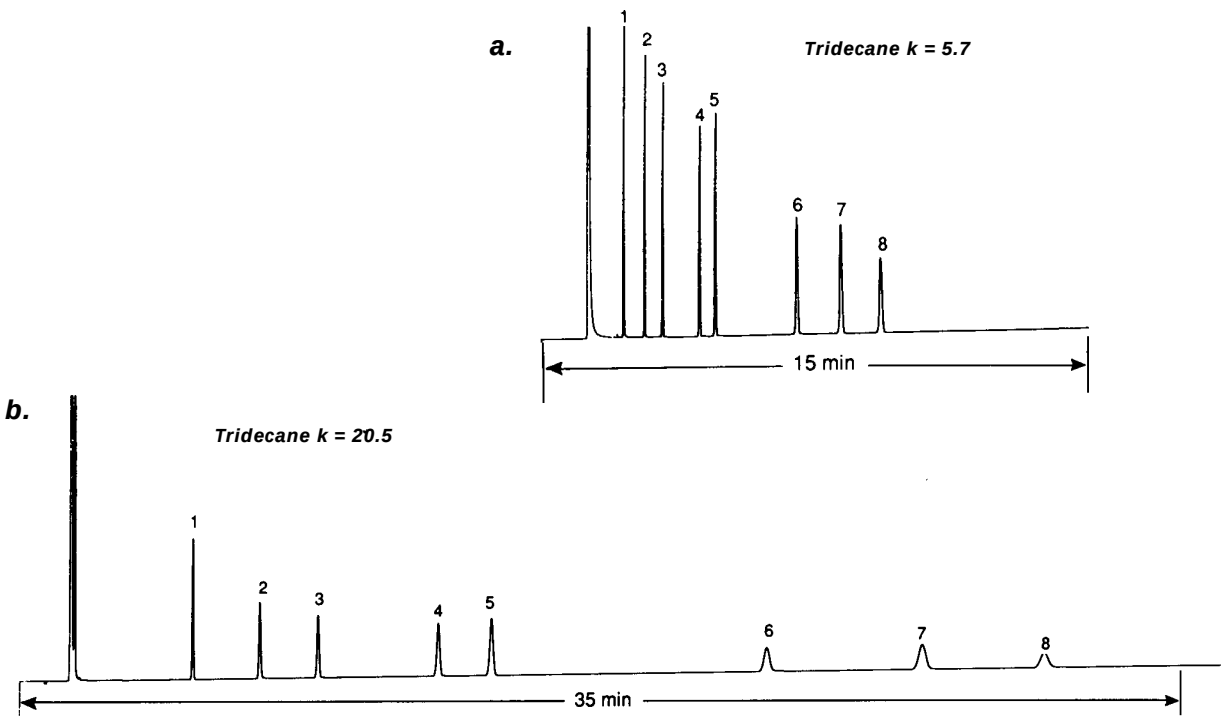


Figure 5

Phase Ratio

The combined effect of column diameter and film thickness on retention can be understood by comparing the phase ratio ( ) of different columns with the same stationary phase.

Equation 1:

$$K_C = k\beta = k \frac{r}{2d_f}$$

where
r = column radius (µm)
d_f = film thickness (µm)
k = partition ratio
K_C = distribution constant

The distribution constant (K_C) is the ratio of the concentration of the solute in the stationary phase and mobile phase (c_s / c_m). The distribution constant remains the same for column temperature, stationary phase and solute.

Under these conditions, solute retention (k) is inversely proportional with column diameter (radius, r) and proportional to film thickness (d_f). Solute retention increases with decreasing column diameter; solute retention decreases with decreasing film thickness. For the same solute and the same column temperature, the relative magnitudes of the phase ratio for two columns will allow the prediction of which column will be more retentive for a given solute. The column with the smaller phase ratio will be more retentive for a given solute than a column with a larger phase ratio. Table 1 lists the phase ratios for a variety of column diameters and film thicknesses.

Diameter (mm)	***Film*** (µm)	***Phase Ratio*** (ß) component)	***Capacity**** (ng per
0.10	**0.18	250	40-50
	0.20	225	55-70
	0.30	150	80-100
	0.40	113	125-150
0.25	0.10	625	40-50
	0.15	417	60-70
	**0.25	250	100-150
	0.50	125	200-250
	1.0	63	350-400
0.32	0.10	800	60-70
	0.15	533	70-80
	**0.25	320	150-200
	0.50	160	250-300
	1.0	80	400-450
	3.0	27	1200-1500
	5.0	16	2000-2500
0.53	0.15	883	80-90
	**1.0	133	1000-1200
	1.5	88	1400-1600
	3.0	44	3000-3500
	5.0	27	5000-6000

Table 1

** Capacity for solutes that are well matched with the stationary phase. Amounts are for each component (single peak) in the sample and not the amount of the entire sample. Capacity is defined as the amount where peak broadening by 10% at half-height occurs. If the inner diameter you are using is not listed, please call MSP for additional information.*

*** Standard.*

Capacity

The capacity of a column is defined as the maximum amount of sample that can be injected into a column before significant peak distortion occurs. Capacity is directly related to film thickness, column diameter and stationary phase polarity. Increased capacity results as film thickness and column diameter increase. Table 1 lists the capacity ranges for a variety of column sizes. Notice the increase in sample capacity with a decrease in the phase ratio.

The more soluble a solute is in the stationary phase, the greater the column capacity for the solute. For example, a polar stationary phase (e.g., DB-WAX) will have a higher capacity for a polar solute (e.g., methanol) than a nonpolar solute (e.g., hexane).

Exceeding the column capacity or "overloading" is indicated by peak broadening or asymmetry. Usually, overloading is evident as a peak with a leading edge (fronting or sharkfin shaped). For gas-solid phases (e.g., GS-Q and GS-Alumina), an overloaded peak will appear to be tailing. Injector problems can also give peak shapes similar to those of overloaded peaks.

Temperature Limits

All stationary phases have upper and lower temperature limits which define the temperature range over which the column can be safely used. The lower temperature limit is the point where the properties of the stationary phase are not conducive to chromatography. At or below the lower temperature limit, the column will exhibit poor separation and peak shape problems. Permanent damage will not result if the column is exposed to temperatures below the lower limit. Heating the column above the lower temperature limit will restore its performance.

Exceeding the upper temperature limit will result in accelerated degradation of the stationary phase. Upper temperature limits are usually given as two numbers. The first or lower temperature is called the isothermal limit. This is the temperature at which the column can be maintained for indefinite periods of time. The second or higher number is called the temperature program limit. The column should not be used at this temperature for prolonged periods (>10 min). The upper temperature limits are not precise thresholds, but temperatures where column lifetime is substantially decreased. Exceeding the upper temperature for short periods of time will not result in instant phase damage; however, the rate of column degradation increases with increasing temperatures.

Slight thermal damage is normally evident as decreased column lifetime. As thermal damage increases, peak tailing and excessive column bleed may be seen. Very rapid and substantial thermal damage occurs whenever the column is exposed to elevated temperatures without carrier gas flow. Polar phases are much more susceptible to thermal damage than nonpolar phases. To prolong lifetime, use any column at the lowest reasonable temperature. There is a direct relationship between increased column lifetime and lower operating temperatures. Leaving the column oven at lower temperatures when the column is not in use will extend its lifetime.

Column Bleed

Column bleed is defined as the normal background signal caused by the elution of stationary phase (polymer) degradation products. These degradation products are always present and are not necessarily a sign of a damaged column. Every column, regardless of the source or quality, will exhibit some column bleed. ***The amount of normal column bleed will increase with increasing film thickness, column diameter and length.*** Polar phases will usually exhibit slightly higher bleed levels than nonpolar phases. A bleed trace (normal background) is shown in Figure 6. Approximately 10 ng of tetradecane was injected into the column to use as a scale.

The bleed trace is a record of the baseline rise during a temperature program. The rise is caused by the increased normal degradation of the stationary phase at higher column temperatures. For a blank run (no injection), there are several important characteristics for the bleed trace:

1. The baseline rise starts about 30-40°C below the isothermal upper temperature limit of the column.
2. The baseline remains relatively level before the rise begins and after the maximum temperature is reached and held.
3. There are no discrete peaks present.

The presence of discrete peaks in a blank run indicates that the inlet or front portion of the column is contaminated. Stationary phase degradation is a continuous process and not an isolated, one-time event. A single point introduction of compound(s) into the column is required to obtain peaks. Since stationary phase degradation is a continuous process and peak gener-

Normal Bleed Profile

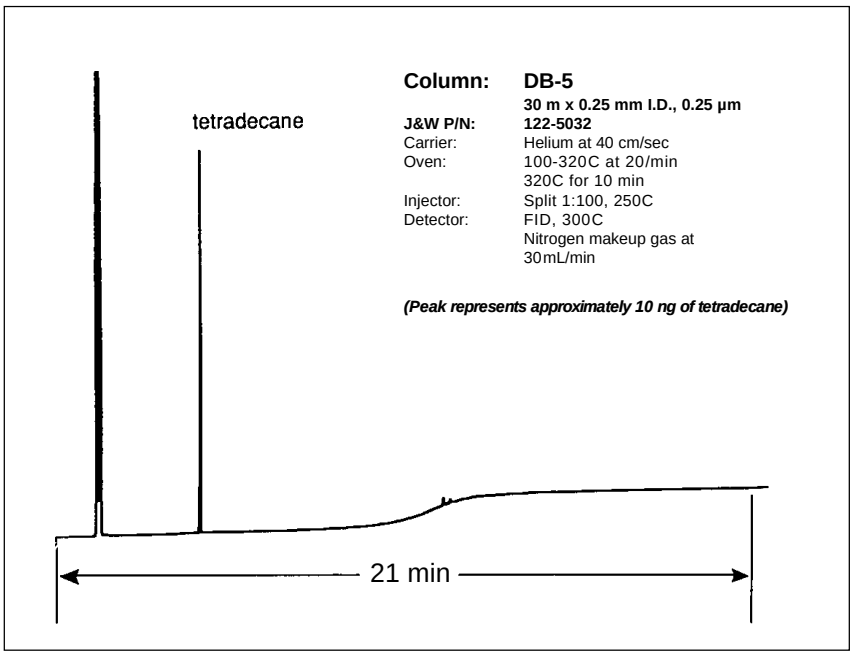


Figure 6

ation is the result of an isolated event (momentary introduction of sample into the column), ***discrete peaks in a blank run cannot be due to a degrading stationary phase.*** The presence of peaks in a blank run is a sure sign of contamination.

The actual magnitude of the baseline offset at higher temperatures will be dependent upon the sensitivity levels used. Column bleed becomes significant for trace level analysis requiring high sensitivity or for very sensitive detectors. Selective detectors may exhibit higher bleed levels for certain phases. For example, the baseline offset for a cyanopropyl-phenyl containing phase will be greater with a nitrogen - phosphorus detector (NPD) than a flame ionization detector (FID) due to the presence of nitrogen in the stationary phase degradation products. The NPD is several orders of magnitude more sensitive to nitrogen than a FID.

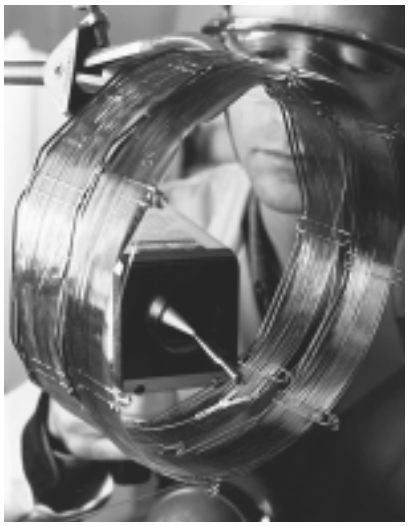
There are two reasons for excessive column bleed or behavior that appears to be column bleed: damage to the stationary phase or contamination. Exposure of the stationary phase to oxygen at elevated temperatures will result in phase damage. The damage is magnified and accelerated at higher temperatures. Oxygen exposure usually occurs when a leak is present in the gas lines or injector. Thermal damage from overheating the column or heating the column without carrier gas flow will also result in excessive bleed due to phase damage. Chemical damage to the phase from the injection of inorganic acids or bases will cause a column to bleed excessively. Contamination of the injector and/or column by semivolatile residues introduced with the injected samples is another common source of excessive baseline rise. The elution of these residues during later GC runs, normally during the higher temperature portion of the program, creates the appearances of column bleed. The presence of discrete peaks or humps in the blank run indicates that there are residues contaminating the GC system. Contaminated columns can be cleaned by solvent rinsing.

Chemical Compatibilities

Bonded and cross-linked phases are not damaged by injecting water or organic solvents. Poor results may be evident with certain solvents depending upon phase/solvent polarity mismatches; however, no damage will occur. The only substances that will damage a stationary phase are strong, inorganic acids (HCl, H_2SO_4, H_3PO_4, HNO_3, etc.) and bases (KOH, NaOH, NH_4OH, etc.). Organic acids and bases will not damage the stationary phase unless they are allowed to reside in the column for prolonged periods. At low ppm concentrations, HCl and NH_4OH in the presence of water will do minimal or no damage to the phase, providing the column temperature exceeds 100°C during the analysis. Non-aqueous samples containing low levels of HCl or NH_4OH will not cause significant phase damage.

Column Storage

Capillary columns should be stored so that abrasion of the tubing is avoided. The ends should be sealed with a GC septa and the column returned to its original box. Upon re-installation, the column ends need to be trimmed to ensure that a small piece of septa has not become lodged in the column.



Dual Columns

GC Column Tubing Diameters

Column I.D. (mm)	*J&W Nomenclature*	*Column O.D.* (mm)*	*Ferrule I.D. (mm)*
0.05	Microbore	0.363	0.4
0.10	Microbore	0.363	0.4
0.18	Minibore	0.340	0.4
0.20	Narrowbore	0.340	0.4
0.25	Narrowbore	0.350	0.4
0.32	Widebore	0.430	0.5
0.45	Hi Speed Megabore	0.673	0.8
0.53	Megabore	0.673	0.8

**** Nominal outer diameter specifications***

Selecting Capillary Columns

The following guidelines and generalizations may be helpful when attempting to select columns.

Stationary Phase

1. Use a stationary phase with polarity closely matching that of the solutes (e.g., nonpolar phase for nonpolar solutes).
2. Use the least polar phase that will provide satisfactory separation; nonpolar phases exhibit superior lifetimes over polar phases.
3. For general purpose use, the best column is a DB-1 or DB-5 .
4. For solutes with dipoles or hydrogen bonding capabilities, use a cyanopropyl (DB-1301, DB-1701, DB-225 and DB-23) or Carbowax (DB-WAX and DB-FFAP) stationary phase. Do not forget to consider polarity and temperature characteristics.
5. For light hydrocarbons or inert gases, use a PLOT column (GS-Q, GS-Alumina, or GS-Molesieve).
6. If possible, avoid using phases containing the specific element corresponding with element specific detectors (e.g., cyanopropyl phases with NPDs and trifluoropropyl phases with ECDs).
7. The widest range of selectivities using the smallest number of columns will be DB-1 or DB-5, DB-1701, DB-17 and DB-WAX. More than 90% of all analyses can be adequately performed with these five columns.

Diameter

1. Use a 0.25 mm I.D. column for split and splitless injections when sample overloading is not a problem. High column efficiencies are realized with these small diameter columns.
2. Use 0.32 mm I.D. columns for splitless and on-column injections, especially when injecting large amounts of sample.
3. Use 0.53 mm I.D. (Megabore) columns as replacements for packed columns or for many purge and trap applications.
4. Use 0.45 mm I.D. columns when ease of use like Megabore is desired but greater column efficiency is needed.
5. Use 0.18 mm I.D. columns for GC/MS systems with low pumping capacities or when very high column efficiencies are needed.

Film Thickness

1. Use a standard film thickness column for most applications.
2. Use thin film columns for high boiling solutes such as petroleum waxes, triglycerides, steroids, etc.
3. Use thick film columns for very volatile solutes such as gases, low boiling solvents and purgeables.

Length

1. A 30 meter column is suitable for most applications.
2. Use a 15 meter column for simple samples (less than 10 components) or for sample screening purposes.
3. Use a 60 meter or longer column for very complex samples or for situations requiring the highest possible number of theoretical plates. These long columns are usually limited to use for the analysis of complex samples such as petroleum products, PCB congeners, dioxins, etc.

Other sources of column selection information:

1. ***Chromatograms in the catalog.***
2. ***Literature references.***
3. ***MSP Support: 031 972 31 52***

Column Installation Tips

Cutting the Column

The importance of a properly cut column is often under appreciated. A poorly cut column will have chips of polyimide or fused silica exposed to the solutes and the flow path of the carrier gas. This may result in tailing, split or broadened peaks due to poor sample introduction into the column. Active compounds may also experience some adsorptive losses.

To properly cut a column, a carbide or diamond tipped pencil or column cleaving tool is needed. Lightly scribe the outer surface of the column at the desired location. Very little force is necessary since only the thin coating of polyimide needs to be cut. Attempting to cut through the fused silica will result in an uneven and unsatisfactory cut. After scribing the polyimide, grasp the column on each side of the scribe. By pulling along the tubing length and bending away from the scribe point, a clean cut will be obtained. This may seem awkward at first, but after several attempts, the process will become quite routine. The end of the column should be examined with a 10-20X magnifier to verify that a clean and straight cut has been obtained.

It is important to cut the column ends after the column nut and ferrule have been placed onto the column. Very small pieces of ferrule material may enter the column upon placement of the ferrule over the column end. If present, these ferrule scrapings will cause peak broadening or tailing, loss of resolution or solute losses due to adsorption. In severe cases, blockage of the column by the solid ferrule particles may occur.

Column Placement

The column should be placed in the GC oven so that the tubing is not subjected to tight bends or turns. Bending places a high amount of stress on the tubing. These areas of stress are more likely to break especially if the polyimide coating has been abraded or scratched in these areas. The avoidance of unnecessary bending stress is critical with larger diameter columns. Large diameter tubing cannot tolerate sharp turns or bends as well as small diameter tubing. Megabore (0.53 mm I.D.) columns should not be wound on cages less than seven inches in diameter, or premature breakage may occur. It is important that any points of possible contact of the tubing with sharp edges such as column identification tags, oven walls, column hangers, etc. are eliminated. The forced air currents inside the GC oven will cause movement of the column, and these contact points will abrade the column. This will eventually lead to column breakage at these abrasion points.

Column Conditioning

After a column has been properly installed, it will need to be conditioned. ***Heating a column without carrier gas flow will result in serious and permanent damage of the stationary phase.***

Before the oven is heated for column conditioning, it is absolutely necessary to verify that there is carrier gas flow through the column and there are no leaks. The most common sites for leaks are the septum and injector fittings. These leaks can often be found with an electronic leak detector. Caution must be exercised when using a liquid leak detector such as Snoop®. If the leak is large, small amounts of the Snoop® may aspirate into the leak and contaminate the injector, detector or column. Using a nonretained compound to verify the injector

Column Installation Tools.

Column Installation Tips *(Continued)*

setup and operation is recommended.

After verifying a leak-free system, the column can be safely conditioned. Heat the column to its isothermal upper temperature limit or a temperature at least 20-30°C above the highest oven temperature that will be used. ***Do not exceed the upper temperature limit of the column.***

After 30-60 minutes at the conditioning temperature, a steady (flat) baseline should be obtained. If the baseline is unstable or excessively high after 60-90 minutes of conditioning, do not continue to condition the column. There is either a leak in the system or the GC is contaminated. If there is a leak, prolonged heating of the column will result in serious damage to the stationary phase. The injector may have residues from previously injected samples. In many cases, the installation of a new column into a contaminated injector will result in a contaminated column.

Saturated gas traps, poor quality carrier gas or a dirty detector will contribute to an elevated or erratic background signal. Be sure that the integrator or recording device is not set at one of its most sensitive levels. Normal, but small, deviations in the detector signal will appear to be very large at these sensitive settings.

Detector	Volatile Compounds
FID/TCD	***methane, butane***
ECD	***methylene chloride*, other halogenated methanes****
PID	***ethylene, acetylene***
NPD	***acetonitrile****
MS	***butane, air, halogenated methanes****

Table 2

** Do not inject a liquid (neat) sample directly into the column, or overloading will occur. Make a headspace injection by placing a small volume of the appropriate solvent in a septum capped vial. Shake the vial, then insert the syringe needle into the headspace above the liquid. Pull up 1-3 μL of the headspace vapors and inject.*

Carrier Gas

After conditioning the column, the carrier gas has to be accurately set. The carrier gas flow rate is dependent on column temperature. It is important to set the carrier gas at the same column temperature for a given analysis. Significant changes in the resolution can occur with small changes in the carrier gas flow rate. For convenience, the carrier gas is often set at the initial temperature of the temperature program.

For capillary columns, carrier gas flow is best expressed as an average linear velocity, ($\bar{\mu}$, cm / sec) instead of a volumetric flow rate (F, mL / min). The average linear velocity can be thought of as the average rate at which a nonretained compound travels through the column or the "speed" of the carrier gas.

Due to the low flow rates used with capillary columns, a bubble flowmeter will not provide an accurate and reproducible measure of the carrier gas flow rate nor can the linear velocity be directly determined. The linear velocity is calculated by injecting a highly volatile compound that is not retained by the column. The recommended compounds for various detectors are listed in Table 2. From the retention time of the nonretained peak, the average linear velocity can be calculated using Equation 2. Using Equation 3, the average volumetric flow rate can also be calculated.

Equation 2: Average linear velocity

$$\bar{\mu}\,(\text{cm/sec}) = \frac{L}{t_M}$$

where

L = ***column length (cm)***

t_M = ***retention time of a nonretained peak (sec)***

Equation 3: Average volumetric flow rate

$$F\,(\text{mL/min}) = \frac{\pi r^2 L}{t_M}$$

where

r = ***column radius (cm)***

L = ***column length (cm)***

t_M = ***retention time of a nonretained peak (min)***

The nonretained peak should be very sharp and symmetrical. Any peak broadening or tailing is unacceptable. Leaks in the injector, a poorly installed column or a lack of sufficient makeup gas flow will all cause tailing or broad, nonretained peaks. ***Any problems will have to be corrected before proceeding.***

The effect of the carrier gas linear velocity on column efficiency is best described using van Deemter curves (Figure 7). The curves illustrate that there is an optimal linear velocity (μ_{opt}) that provides the highest efficiency. This point is where the curve reaches the small-

Carrier Gas *(Continued)*

est value of H_{min} (the point of greatest column efficiency). The best resolution will be obtained when using a linear velocity that generates the highest efficiency for a column. The curves also show that using a linear velocity that is too low or high will result in a rapid loss of column efficiency.

Usually a linear velocity that is greater than the value corresponding to the minimum in the van Deemter curve is used. A value 1.5-2 times the μ_{opt} (called the OPGV or optimal practical gas velocity) is the point where maximum column efficiency per unit time is obtained. Setting the linear velocity at a higher value also compensates for the decrease in linear velocity with increasing column temperature as encountered when using a temperature program.

With helium and hydrogen as carrier gases, the minimum in the van Deemter curves occurs over a much broader range and at higher linear velocities than with nitrogen. Using nitrogen provides the

van Deemter Curves

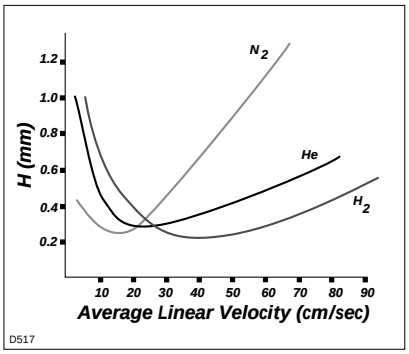


Figure 7

greatest column efficiency, but the minimum in the van Deemter curves occurs over a very narrow range and at a low linear velocity. Substantial analysis speed must be sacrificed for optimal resolution when using nitrogen. For helium and hydrogen, high linear velocities can be used to reduce analysis times without sacrificing a large amount of efficiency. The faster flow rates also sweep the injector faster, which improves the sample introduction process. Helium, and especially hydrogen, provide the best resolution when the analytes elute over a wide temperature range. Nitrogen is not recommended as a carrier gas for capillary columns even though it provides the highest efficiency. Recommended linear velocities for helium and hydrogen are listed in Table 3.

Use the highest purity carrier gas for maximum column life. The use of impurity traps (water and oxygen) on the gas lines is highly recommended to extend column lifetime and to improve detector sensitivities. The slightly higher cost of high purity gases will be offset by longer column and trap life. Table 4 lists recommended minimum purities for carrier and detector gases.

Makeup Gas

Most commercially available GC detectors require 30-40 mL / min total gas flows for best sensitivity and peak shape. Carrier flows for capillary columns range from less than 1 mL / min to over 10 mL / min. These flow rates are well below the range where most detectors will exhibit optimal performance. To supplement the carrier gas flows, makeup gas is added at the column exit to obtain a total gas flow of 30-40 mL / min into the detector. The makeup gas can be the same as the carrier gas or a different gas depending on the type of detector being used. The makeup gas is independent of the flame gases in combustion type detectors (e.g., FID, NPD and FPD). For some detectors, such as the ECD, the moderating or auxiliary gas may act as the makeup gas. Exact recommendations for makeup gas flows and type can be found in the instruction manual for the GC being used.

Recommended Linear Velocities for 30 Meter Columns

Column Diameter (mm)	Linear Velocity (cm/sec)		Flow Rate (mL/min)	
	H_e	H_2	H_e	H_2
0.18	30-45	45-60	0.5-0.7	0.7-0.9
0.25	30-45	45-60	0.9-1.3	1.3-1.8
0.32	30-45	45-60	1.4-2.2	2.2-2.9
0.45	30-45	45-60	2.9-4.3	4.3-5.7
0.53	30-45	45-60	4.0-6.0	6.0-7.9

Table 3

Recommended Minimum Gas Purities

hydrogen	99.99%	carrier
	99.95%	FID
helium	99.995%	carrier
nitrogen	99.999%	carrier and ECD moderating gas
	99.95%	makeup

Table 4

Capillary GC Injectors

There are two goals of sample introduction. One is to introduce the sample into the column such that it

will occupy the shortest length of column. The shorter the sample band is at the beginning of the process, the sharper and more narrow the peaks will be on the chromatogram. The end result is more sensitivity and better resolution. The second goal is to have the composition of the sample introduced into the column be as close to the composition of the sample before the injection. There should be no sample degradation or adsorptive losses occuring or caused by the injector.

Backflash

With the exception of on-column injection, all injectors utilize vaporization to introduce the sample into the capillary column. The injected sample is rapidly vaporized in the heated injector, and a gas (the carrier gas) flowing through the injector carries the sample into the column. One problem that vaporization injection techniques have is backflash. Upon sample vaporization, the gaseous sample will expand to fill the injector liner volume. Backflash is when the vaporized sample expands beyond the capacity of the liner volume and into the injector body. Since the sample now occupies a larger volume, it takes longer for the sample to be carried into the column. A large and tailing solvent front is obtained. If the vaporized sample comes in contact with cold spots like the septum and gas inlets of the injector, small amounts of the sample may condense. This condensation can result in carry over problems (ghost peaks) on subsequent injections. The metal injector is not inert and loss of active compounds may occur when they come in contact with the metal surfaces.

Backflash problems can be minimized by:

1. Using a septum purge with split/splitless injectors.
2. Using small injection volumes.
3. Using large volume injector liners.
4. Using the optimal injector temperature.

Injector Temperatures

The injector temperature should be just hot enough to insure "instant" vaporization of the entire sample without degrading any of the sample components. If the injector temperature is too low, carry over problems, incomplete sample vaporization or broad peaks (especially the solvent front) will be obtained. If the injector temperature is too high, excessive backflash or sample degradation may occur. Using an injector temperature above the upper temperature limit of the column stationary phase will not damage the column. For most samples, 250°C is a good injector temperature. Some experimentation may be warranted to obtain the smallest amount of backflash and the maximum amount of sample vaporization.

Inlet Discrimination

Upon injection, the less volatile sample components will not vaporize as rapidly as the more volatile sample components. Immediately following injection, the vaporized sample has a greater proportion of the more volatile compounds than the less volatile compounds. Thus, more of the volatile compounds are introduced into the column. This effect is called discrimination. The peaks for the less volatile compounds will be smaller than the more volatile compounds. The longer the sample spends in the heated injector, the less severe the discrimination.

Septum Purge

Most split/splitless capillary injectors have a septum purge function. The septum purge minimizes the amount of septum bleed materials that may contaminate the GC system. The septum purge gas sweeps the bottom face of the septum and carries the contaminants out through the septum purge vent. The septum purge flow is usually between 0.5 and 5 mL/min. High or higher than optimum septum purge flows may result in the loss of some of the more volatile sample components. The septum purge function is not essential for good chromatographic results; however, any septum bleed problems may be minimized.

Injection Techniques

There are four major capillary injection techniques - split, splitless, on-column and Megabore direct.

Nearly every standard capillary injector is capable of split and splitless injections. On-column injections require a dedicated capillary on-column injector. Megabore injections utilize packed column injectors that have been converted to a Megabore injector.

Split Injection

Split injection is very simple and the most common of the capillary injection techniques. The highest resolution and system efficiencies are obtained with split injections. Split injections are used for highly concentrated samples with typical per component concentrations of 0.1-10 µg / µL. Injection volumes of 1-2 µL are normally used, but volumes up to 5 µL can be used without significant problems.

Split injection is a vaporization technique (see Figure 8). The sample is vaporized upon injection and rapidly mixed with carrier gas. A small amount of the carrier gas enters the column, and a much larger amount leaves the injector via the split vent. Since the vaporized sample is mixed with the carrier gas, only a small amount of the injected sample actually enters the column. The total gas flow through the injector at the moment of injection is quite high (the sum of the column and split vent flows). The sample is rapidly swept into the column which accounts for the high efficiency of split injections. This also accounts for the severe discrimination obtained with split injections. The less volatile compounds do not have sufficient time to fully vaporize before they are discarded via the split vent.

Split Ratio: The amount of sample entering the column will be dependent on the carrier gas flows into the column and out of the split vent. By measuring the column flow and the split vent flow, the amount of sample "splitting" that occurs can be calculated. This value is called the split ratio. The split ratio is normally reported with the column flow rate normalized to 1. The split ratio is determined using Equation 4. The split ratio can be used to estimate the amount of sample entering the column. A split ratio of 1:50 would indicate that one part of the sample enters into the column and 50 parts are discarded out of the split vent. Therefore, 1 / 51 of the total sample theoretically makes it into the column. Typical split ratios range from 1:10 to 1:100.

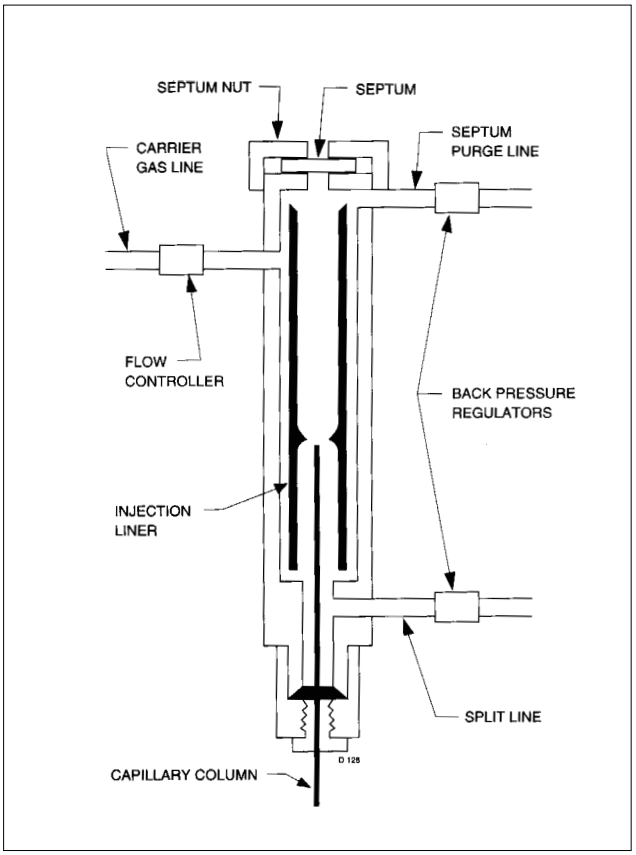


Figure 8

Applications involving highly concentrated samples or very small diameter columns may require the use of higher split ratios.

Note: Split ratio can be directly measured using ADM flowmeters.

Equation 4: Split ratio

$$\frac{\text{split vent flow}}{\text{column flow}} = \text{split ratio}$$

Example:
Column flow = 2 mL/min
Split vent flow = 100 mL/min

$$\text{Split ratio} = \frac{100}{2} = 50$$

Therefore, the split ratio is 1:50.

Injection Techniques

Splitless Injection

Splitless injections are used for trace level analyses or when the per component amounts are no more than approximately 200 ng. Splitless injections are slightly more complex than split injections and are subject to several restrictions and conditions.

The injected sample is vaporized and carried into the column by the carrier gas. At the moment of injection, the flow through the injector is the same (1-2 mL / min) as the column flow (see Figure 9, purge off). Fifteen to ninety (15-90) seconds after the moment of injection, additional carrier gas flow is introduced into the injector (see Figure 9, purge on). This extra gas purges the injector of any remaining sample that has not entered the column. The time at which the extra gas flow is introduced is called the purge activation time (or purge on).

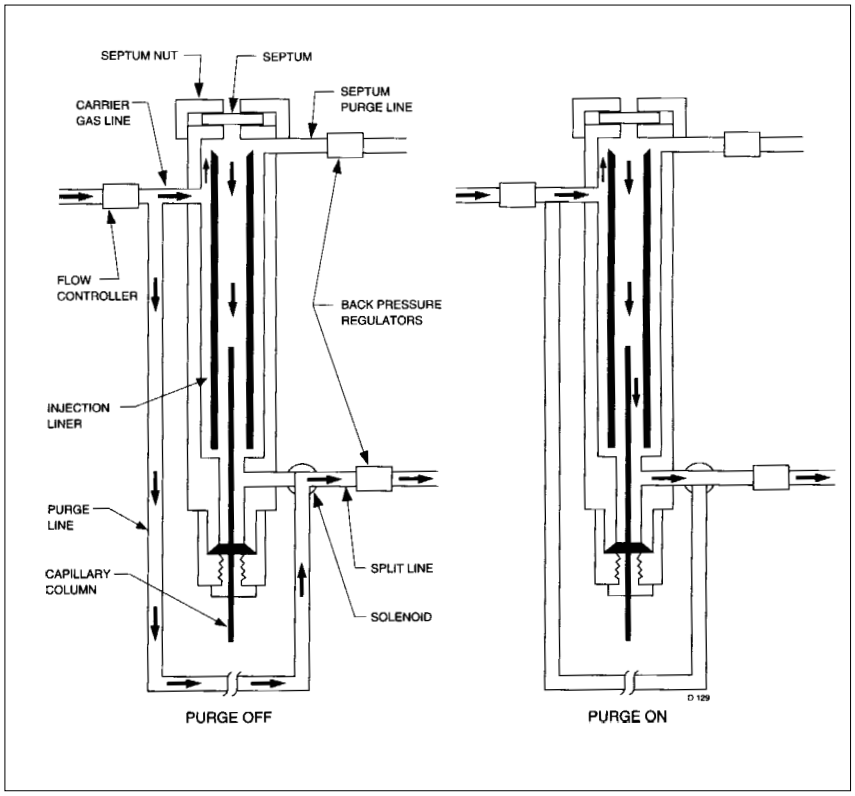


Figure 9

Solvent Effect and Cold Trapping: With splitless injections, the sample is introduced into the column at a much slower rate than for split injections. To avoid extremely broad peaks, the sample needs to be refocused before starting the chromatographic process. ***One requirement of most splitless injections is that the initial temperature of the column is at least 10°C below the boiling point of the sample solvent.*** When the vaporized solvent leaves the injector and enters the cooler column, the solvent rapidly condenses at the front of the column. A solvent film forms, and this film will trap and refocus the sample. This is called the solvent effect. Starting at too high of a column temperature can result in broad and malformed peaks. The solvent film cannot form, and the sample is introduced into the column over a long time (equal to the purge activation time). The sample is not refocused as required for good peak shapes. The earlier eluting peaks will suffer greater peak shape degradation than the later eluting peaks. If this occurs, either a lower initial column temperature or a higher boiling solvent will have to be used. If the sample components boil at 150°C or above the initial column temperature, the solvent effect does not have to occur for good peak shapes. These high boiling compounds will cold trap in the column and refocus into a short sample band without the aid of the solvent effect.

Other Considerations for Splitless Injections: Injection sizes are usually limited to 2 µL or less for splitless injectors. Large injection volumes will normally result in broader peaks. Another limitation of splitless injections is peaks that elute before the solvent front will be malformed. Changing to a lower boiling solvent may remedy this situation.

Inlet discrimination is less severe for this technique than for split injections. If the sample solvent and the column stationary phase polarities are very different, poor peak shapes may occur. The polarity mismatch between the phase and solvent causes this problem. The peak shapes can be improved by installing a retention gap (guard column).

Injection Techniques
(Continued)

On-column Injection

On-column injection is not a vaporization technique. The sample is deposited directly into the column with a syringe. On-column injection provides the optimum in capillary column performance by eliminating discrimination and degradation effects that can result from using a vaporization technique. On-column injections are particularly well suited for high boiling compounds like petroleum waxes, triglycerides and thermally labile compounds.

On-column injection requires the solvent effect or cold trapping to obtain acceptable peak shapes. Some on-column injectors use a secondary cooling function to eliminate the need to cool the entire column down to the appropriate temperature for the solvent effect. Only a small portion of the front of the column is cooled at the moment of injection.

One on-column injector design forces air around a short length of the front of the column, which recondenses the sample in this region. After the injection, the air is turned off and this region of column will rapidly equilibrate to the higher oven temperature. The sample will now begin the chromatographic process. Usually a savings in time will result since the column oven does not have to be cooled to low temperatures before each analysis.

Another injection design uses a telescoping injector that allows the front of the column to be pulled out of the heated GC oven and into the ambient air. The surrounding air cools this portion of the column so that the injection can be made. After injection, the unit is collapsed and the column is returned to the heated oven to start the chromatographic process. This injector can be added to a GC without disabling existing injectors.

Retention gaps (guard columns) are almost always required for on-column injections. Substantially improved peak shapes will result when a retention gap is used. The retention gap will also protect the analytical column. Since the entire sample is deposited directly into the column, potentially large amounts of contaminants or other column damaging materials enter the column. These materials will deposit in the retention gap instead of the column and longer column life and performance will result.

Megabore Direct Injection

Packed column injectors that have been converted to accept Megabore capillary columns (0.45 and 0.53 mm I.D.) are very simple and relatively trouble free. Megabore columns can tolerate large injection volumes (5-6 µL) and high sample concentrations (1-10 µg). Megabore systems are used for converted packed column instruments or for situations where large sample capacities are needed. Megabore injections are well suited for trace level analyses also. Highly concentrated samples may have to be diluted to avoid overloading the column. Small diameter capillary columns (0.32 mm I.D. or less) are not compatible with Megabore injectors.

A Megabore injector consists of a glass injector liner that is held in place with a metal injector fitting. The ferrule in the fitting seals and holds the liner in place. The liner reduces the volume of the packed column injector and provides an inert environment for the sample to vaporize (see the section on Megabore Direct Injector Liners, page 250). The sample is injected and rapidly vaporized in the liner (see Figure 10). The carrier gas sweeps all of the vaporized sample into
the Megabore column. The gas flow through the injector is high (4-10 mL / min), thus the sample is rapidly introduced into the column. There is no splitting of the sample or solvent / temperature requirements.

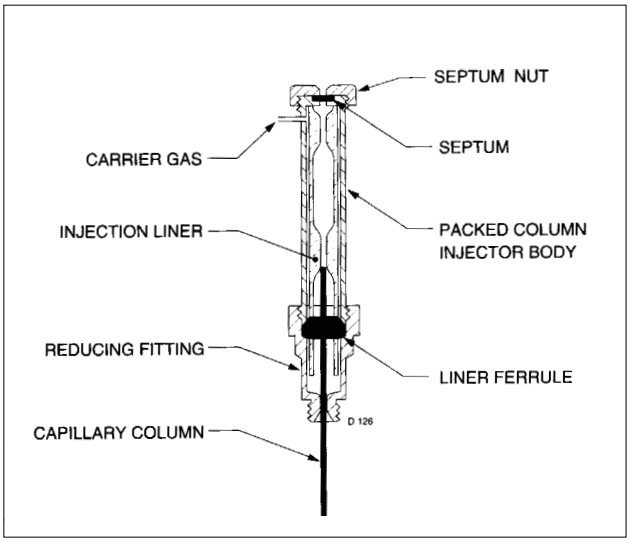


Figure 10

Injector Port Liners

Injector liners provide an inert environment where the sample can vaporize and be properly introduced into the column. Liner design will vary depending on the type of injection technique being used. Liners that are dirty, broken, poorly installed or incorrectly selected will often contribute to inferior chromatographic results.

Split Injector Liners

The split injector liner must have a large enough volume to accommodate the expansion of the vaporized sample. However, the volume must be small enough for the gas flow to quickly sweep the vaporized sample into the column. Liners with too small of a volume will be subject to severe backflash problems. Larger liner volumes become less important at high split ratios since the high carrier gas flows rapidly sweep the injector. Flow disruption within the liner will insure thorough mixing of the sample, thus minimizing discrimination problems. Various types of flow disruption liners are available. The greatest discrimination is obtained with straight bore liners while the inverted cup liners discriminate the least. The hourglass shaped liners provide good sample mixing, and they are easier to clean than the inverted cup liners.

Packing a split injection liner with silanized glass wool is another way to insure flow disruption. The packed liner has a higher thermal mass which will aid in the rapid volatilization of the less volatile components. Additionally, the glass wool acts as a filter to help to trap some of the nonvolatile materials in the injected sample. The glass wool should be lightly packed so unnecessary peak broadening will not occur. Do not use regular laboratory grade glass wool.

Splitless Injector Liners

Splitless injector liners are normally straight tubes without any flow disruption devices. Any flow disruption in the injector will usually cause peak broadening. For this reason, it is not recommended to pack a splitless liner with silylated glass wool. Sometimes there is a restriction at the bottom of the liner. This is to keep the column centered in the liner and away from the liner walls. Small volume liners will result in greater inlet efficiency since the sample is transferred into the column over a shorter period of time. The smaller volume liners are more subject to backflash problems. For small injection volumes (<1 µL), the 2 mm I.D. splitless liner is recommended; for larger injection volumes, the 4 mm liner is recommended to minimize backflash problems.

Megabore Injector Liners

The type of direct injection liner used will greatly impact the quality of Megabore chromatography. There are three general types of direct injection liners: the straight tube, the direct flash vaporization liner and the hot on-column.

Straight Tube Liners: Using a straight tube type of Megabore liner is not recommended. It will typically give a very broad and tailing solvent front which may interfere with some of the early eluting peaks of interest. Due to the lack of any type of restrictions, the vaporized sample can readily backflash out of the liner. The severity of solvent front tailing will be more pronounced for large injection volumes, volatile sample solvents and solutes, low carrier gas flows and excessively hot injectors.

Direct Flash Vaporization Liners:
The direct flash vaporization liner has a restriction at the top of the liner and another restriction several centimeters below the upper restriction. The sample is injected into the chamber formed by the two restrictions. When the syringe needle is inserted into the liner for injection, the needle blocks most of the upper restriction. This prevents the vaporized sample from escaping from the top of the liner. The lower restriction is tapered so that the Megabore column will become lightly wedged in the taper. The polyimide column coating will compress and cause a leak-free seal between the column and the liner. Carrier gas (or sample) will not escape around the column. Backflash is greatly reduced, thus the solvent front is narrow and without significant tailing.

Direct flash vaporization liners can be packed with silylated glass wool providing there is no top restriction. This restriction can be removed by cutting the liner, but the purchase of liners without a top restrictor is recommended. The glass wool should be lightly packed so unnecessary peak broadening will not occur. Do not use regular laboratory grade glass wool.

It is essential that the column end is cleanly cut and checked with a magnifying lens. The column is inserted into the liner until it fits snugly in the restriction. Too much force will crush the column end, and too little force will not seal the column in the restriction. If the column is not sealed properly, the solvent peak will tail excessively. Also, any debris in the injection liner taper (small

pieces of septa, polyimide from the last installation, etc.) will prevent a proper seal.

Hot On-column Liners: The hot on-column injection port liner has a single tapered restriction at the top of the liner. The Megabore column will seal in this region in the same manner as for the direct flash vaporization liner. The needle of a standard GC syringe (a 26 gauge) can now be inserted directly into the Megabore column. It is very important that the syringe needle is straight and that the end has no burrs or hooks. Injection volumes are limited to 0.5 µL or less.

Hot on-column Megabore injections are suitable for high boiling samples that are difficult by standard vaporization techniques. Since the injector temperatures are lower than those used for vaporization techniques, thermally labile samples can also be chromatographed with the on-column Megabore liner. A direct flash vaporization liner system will be more suitable and better in greater than 95% of all Megabore applications. Hot on-column Megabore liners should be used only when direct flash vaporization liners are not suitable.

Injector Septa

High temperature septa are recommended to minimize injector and column contamination by septa bleed materials. It is recommended that septa should be replaced after 30-50 manual injections or 75-100 autosampler injections. Leaky septa will allow air to enter the column which will rapidly damage the column. Septa will have to be replaced more frequently when using large diameter needles or needles with burrs or hooks. Over tightening the septum cap will reduce septa life also. The column temperature should be reduced to 35°C or less when changing the septa. Reduced column lifetime will result if air is introduced into the carrier gas (column) at elevated temperatures.

Guard Columns

Guard Columns/Retention Gaps

A guard column or retention gap is a short length of deactivated fused silica tubing (0.5-1.0 meter) installed between the inlet and the analytical column (Figure 11). The tubing serves two functions, one as a guard column and the other as a retention gap.

As a guard column, it catches non-volatile residues which prevent them from reaching the analytical column. As a retention gap, the tubing allows larger amounts of solvents with polarities unlike that of the stationary phase to be injected without adversely affecting peak shapes.

Glass or stainless steel unions may be used to join the analytical column to the guard column.

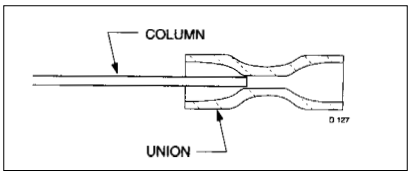


Figure 12

Unions

Glass Press-fit Unions

Universal glass press-fit unions replace stainless steel, Vespel, and resin methods of joining two pieces of fused silica tubing. The glass unions form a gas tight seal by a minor compression of the polyimide coating on the column (Figure 12). There is no need for wrenches, glue or ferrules. Since these unions are very light weight and have very little thermal mass, they track oven temperature and eliminate the possibility of a cold or hot spot in the sample path. Because the glass unions have a similar expansion coefficient to fused silica tubing, they expand and contract together and can be used over a wide temperature range (-60°C to 350°C). With proper installation, they exhibit no solvent tailing or adsorption of active compounds. Universal glass unions can be used to connect fused silica tubing with outer diameters of 0.4-0.8 mm.

Useful tips for achieving a gas tight seal are as follows: The ends of the tubing to be joined should be cut very cleanly with a column cutting tool. Examination under a 10-20X magnifier is advisable. Cleaning the ends of the tubing by swabbing with methanol and rinsing the union with methanol can help ensure a seal. Making sure there is no carrier gas flowing, the column ends are pressed firmly into the union. A visible ring of compressed polyimide inside the union and a gentle tug on the connection should confirm that the seal has been formed. If difficulties arise in making a good seal, try another glass union. In some cases, there may be some variability in the taper of the union.

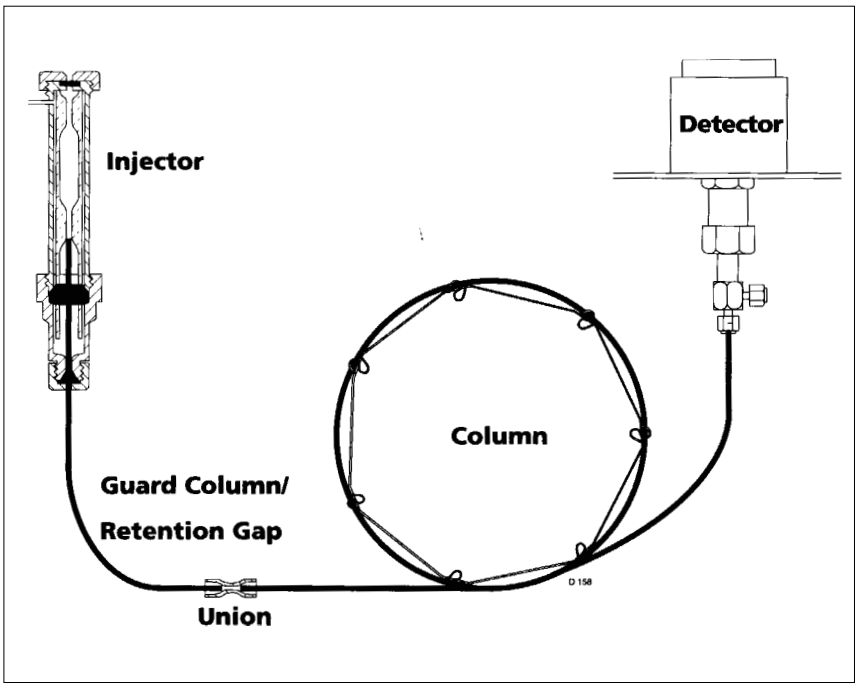


Figure 11

Traps

Traps are strongly recommended, even if high purity carrier gases are used. Trace amounts of contaminants can cause baseline irregularities, detector noise and possible column damage. Contaminated detector gases can lead to artificially high baseline readings and poor performance.

Individual traps are designed to remove moisture, oxygen, hydrocarbons and other contaminants from the gas supply. Traps are offered with either 1/4" or 1/8" Swagelok® fittings. The trap system recommended is a moisture trap, high capacity oxygen trap, and indicating oxygen trap. The moisture trap should be first in line because moisture will quickly deactivate the oxygen traps. The high capacity oxygen trap should be installed second followed by an indicating oxygen trap. ***An expired trap should be replaced immediately.***

Moisture Traps

A moisture trap should be installed in the carrier and detector gas line(s). It will remove trace levels of water, some nonpermanent gases and light hydrocarbons. Two types of absorbents are available. Both contain blue indicating Molecular Sieve 4A that turns tan at 20% humidity. Molecular Sieve 5A will trap water, some non-permanent gases and small hydrocarbons (<n-C_4H_{10}). Molecular Sieve 13X will trap larger hydrocarbons in addition to most of the impurities trapped by the Molecular Sieve 5A material.

Oxygen Traps

Trace levels of oxygen in the carrier gas can dramatically shorten the life of a gas chromatographic column.

Generally, the polyethylene glycol based phases (i.e., DB-WAX, DB-FFAP and Carbowax) are readily oxidized, especially at elevated temperatures. Polysiloxane phases can also be irreversibly damaged via oxidation at high temperatures, but much more slowly than polyethylene glycol based phases. Oxidized phases exhibit poor chromatographic performance and higher than normal bleed. Oxygen has a deleterious effect on many GC detectors: it degrades the performance of electrolytic conductivity detectors (ELCD), reduces filament lifetime in a thermal conductivity detector (TCD) and reduces the linearity and sensitivity of electron capture detectors (ECD). Both the column and detector benefit when oxygen is removed from the carrier gas supply.

Oxygen contamination can usually be attributed to small leaks in the gas lines, septum leaks or low purity carrier gas. Oxygen traps only address the last problem.

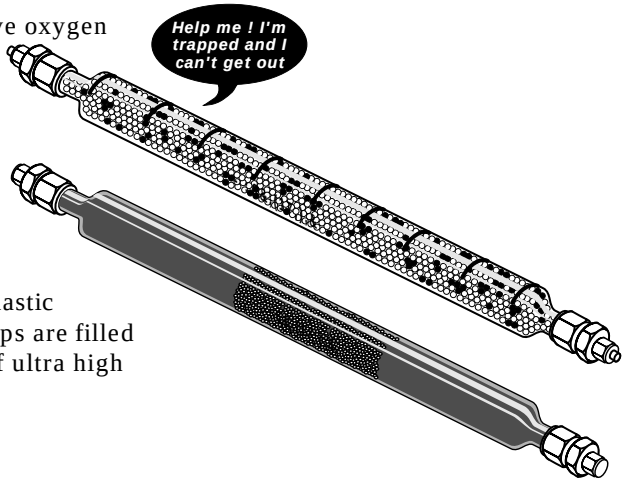
High Capacity Oxygen Traps

The most efficient oxygen traps are 99% efficient at reducing oxygen in carrier gases such as helium, nitrogen, hydrogen, argon, argon-methane, or CO_2 at 99.999% purity. A 20 to 40 fold reduction in oxygen in the gas is often obtained. These traps will effectively remove oxygen from argon-methane mixtures (used with ECDs) without disturbing the ratios of these gases.

Metal bodied traps avoid the signal noise associated with any plastic bodied trap. These traps are filled under a heated flow of ultra high purity helium instead of nitrogen. These traps will need periodic replacement. The use of an indicating oxygen trap is recommended to determine when the high capacity oxygen trap needs replacing.

Glass Indicating Oxygen Traps

A glass indicating oxygen trap should be placed downstream of the high capacity oxygen trap as a means of indicating when to replace the high capacity non-indicating trap. This will prevent premature disposal of the high capacity oxygen trap. The indicating material undergoes a vivid color change as it is depleted. The high capacity oxygen trap must be replaced immediately upon the first indication of color change in the indicating trap. (It is not unusual to partially expire the indicating trap on each end upon installation). Fully expired oxygen traps will recontaminate the gas with previously trapped materials. Unnecessary frequent maintenance of the GC system and the possibility of reduced column lifetime will be avoided by the timely changing of the high capacity oxygen trap. The indicating oxygen trap will rapidly expire if used as the solitary oxygen trap in the system or for prolonged periods after the high capacity oxygen trap has expired.



Column Contamination

One of the most common causes of column performance degradation is contamination. Usually these contaminants originate from the injected samples. Semivolatile or nonvolatile sample components accumulate in the injector and column after repeated injections of the sample. Dirty samples will contaminate the GC system at a faster rate than clean samples. Loss of resolution or separation, peak shape problems and / or baseline disturbances (artificial bleed) are common symptoms of column contamination. Active compounds such as carboxylic acids, amines, phenols and diols are particularly affected by contamination.

Solvent rinsing a capillary column will remove most contaminants and restore column performance. ***A column must have a bonded and cross-linked stationary phase to be solvent rinsed.***

A good general column rinsing can be accomplished by using methanol, methylene chloride, and hexane in series. Other solvent choices will work, but they should meet the following criteria:

- Include a polar and nonpolar solvent. Start with the most polar and finish with the least polar solvent.
- Include the injection solvent if possible.
- Each successive solvent must be miscible with the previous one.
- If aqueous-based or extracted samples were injected (biological extracts, soils, waste water, etc.), begin with water followed by methanol.
- If using an ECD, avoid halogenated solvents as the final rinse solvent, or if using an NPD, avoid acetonitrile.

Columns can be easily rinsed using a rinse kit (see Figure 13). Before rinsing the column, break off one-half meter from the injector end. Place the detector end of the column into the rinse kit. Fill the vial with the appropriate solvent. Apply 10-15 psi pressure to force the solvent through the column. For columns with 0.18-0.32 mm I.D., use 4-5 mL of each solvent. For larger I.D. columns, use 8-10 mL of each solvent. Each solvent should stay in the column for at least 10 minutes. The previous solvent does not have to completely vacate the column before rinsing with the next solvent.

After the last solvent has left the column, allow the pressurizing gas to flow through the column for 5-10 minutes. Install the column into the injector of the GC and let carrier gas flow through the column for 5-10 minutes. Install the column in the detector and check for leaks. Heat the column at
2-3°C / min until the normal conditioning temperature is reached. Condition the column as usual.

Performance Chromatogram Definitions

Partition Ratio (k)

The partition ratio (k) is a measure of how much time a solute spends in the stationary phase relative to the time it spends in the mobile phase (carrier gas). All solutes spend the same amount of time in the mobile phase, thus the partition ratio is directly related to retention caused by the stationary phase. It is a unitless number and can be calculated using Equation 5. The partition ratio is a better measure of retention than the actual solute retention time. The partition ratiois inversely proportional to column temperatures. This means that retention increases as the column temperature is decreased and retention decreases as the column temperature is increased.

Equation 5: Partition Ratio (k)

$$k = \frac{t_R - t_M}{t_M} = \frac{t'_R}{t_M}$$

where

t_R = ***solute retention time***
t_M = ***retention time of a non retained compound***
t'_R = ***adjusted retention time***

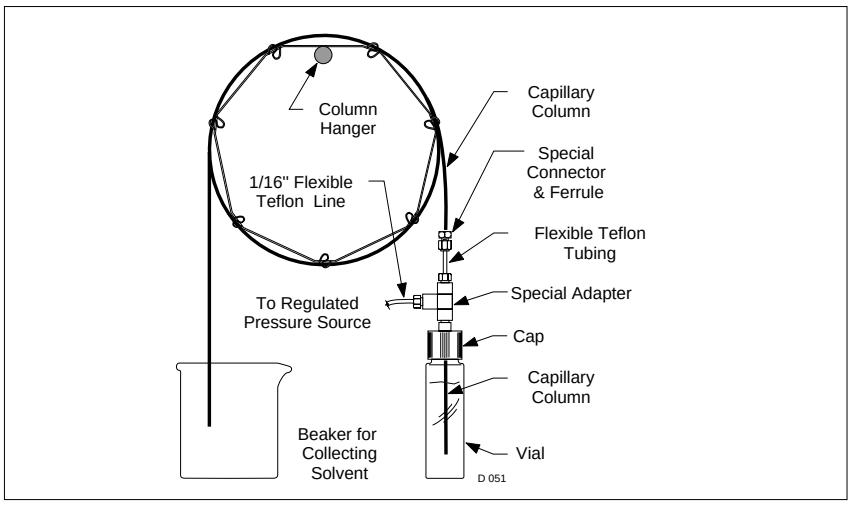


Figure 13

Performance Chromatogram Definitions

(Continued)

Column Efficiency or Number of Theoretical Plates (N)

Column efficiency is the relationship between solute retention time and the amount of band broadening (peak width increase). Symmetrical peaks with the smallest width are desired. Narrow peaks can be close together and still be resolved. Broader peaks the same distance apart will not be as well resolved and may even coelute. Column efficiency is expressed as the number of theoretical plates (N). The number of theoretical plates can be calculated using Equation 6. The higher the number of theoretical plates, the higher the columnís efficiency and its potential to resolve two closely eluting solutes. The number of theoretical plates per meter is the usual method for reporting efficiency. Decreasing column diameter will increase the number of theoretical plates, thus increasing column efficiency. In general, thinner film columns will have slightly higher efficiencies than a corresponding thicker film column.

Equation 6: Number of theoretical plates (N)

$$N = 5.545 \left(\frac{t_R}{w_h}\right)$$

where

- N = ***number of theoretical plates***
- t_R = ***retention time***
- w_h = ***peak width at half height (in units of time)***

Another measure of column efficiency is the height equivalent to a theoretical plate (H). The shorter each theoretical plate, the more that can be placed into a length of column. This translates into more theoretical plates per meter and greater efficiency. High efficiency columns will have small values of H. van Deemter curves are usually reported using h values. Equation 7 can be used to calculate H.

Equation 7: Height equivalent to a theoretical plate (H)

$$H = \frac{L}{N}$$

where

- ***L*** = ***column length (mm)***
- ***N*** = ***number of theoretical plates***

Coating Efficiency

Coating efficiency is a comparison between actual column efficiency and its theoretical maximum efficiency (Equation 8). The property actually compared is peak width. Coating efficiencies of less than 100% are departures from the theoretical behavior of a ìperfectî column (i.e., broader peaks). There are some unavoidable measurement errors, thus it is possible to obtain coating efficiencies slightly greater than 100%. Coating efficiency can be thought of as a measure of the uniformity of the stationary phase. A more uniform phase results in a column with higher efficiencies. Coating efficiency is not the amount of column coated with phase or the amount of column coated as the prescribed film thickness. Typical coating efficiencies are 85-100% for non-polar phases and 60-80% for polar phases.

Equation 8: Coating efficiency

$$CE\% = \frac{H_{theoretical}}{H_{actual}} \times 100$$

Retention Indices (I)

Retention Index (I) is a measure of the relative retention of a solute compared to normal alkanes (hydrocarbons) at a given temperature (isothermal) for a particular stationary phase. Retention indices normalize instrument variables so that retention data can be compared for different chromatographic systems. For example, a solute with a retention index of 1250 elutes between n-C12 (I=1200) and n-C13 (I=1300) under the same test conditions. Retention indices are useful when comparing relative elution orders of various solutes for a given column and conditions. Also, retention indices are good for comparing the retention or selective behavior of two columns. Retention indices can be calculated using Equation 9.

Equation 9: Retention indices (I)

$$I = 100y + 100(z-y)\frac{\log t'_{R_{(x)}} - \log t'_{R_{(y)}}}{\log t'_{R_{(z)}} - \log t'_{R_{(y)}}}$$

where

- t'_R = ***adjusted retention time***
- ***x*** = ***solute of interest***
- ***y*** = ***normal alkane with y number of carbon atoms eluting before solute x***
- ***z*** = ***normal alkane with z number of carbon atoms eluting after solute x***
- ***z-y*** = ***difference in carbon number between the two normal alkanes***

Column Test Standards

One of the best means to evaluate the performance of a capillary column is by analyzing a properly designed test standard. Test standards contain compounds that have a range of functional groups. By evaluating the shape, size and retention of the various test standard components, a substantial amount of information is obtained about the column and possibly the chromatographic system. Every J&W column is tested using a specially designed test standard. The actual chromatogram is included with every column (see Figure 14 on page 275).

It is important to save this test chromatogram for future reference. The column performance chromatogram provides additional information about the column such as temperature limits, actual dimensions and serial numbers.

Hydrocarbons

Hydrocarbon peaks are the standard to which all other peaks are compared. Due to the lack of functionality, hydrocarbons can only interact with the stationary phase. Hydrocarbon peaks should be sharp and symmetrical. Malformed hydrocarbon peaks are due to gas flow problems, poor injection technique, column contamination (especially solid or high molecular weight materials), or an extremely damaged stationary phase. Gas flow problems include dead volume, leaks in the injector, improper installation of the column, broken or improperly installed injector liner, backflash and poorly cut column ends. Straight chain hydrocarbons are used to calculate retention indices. They may also be used to determine the number of theoretical plates or coating efficiency.

Alcohols

Hydroxyl groups easily interact with any material or species in the carrier gas flow path that can hydrogen bond. Ideally the only interactions occurring is with the stationary phase. A tailing alcohol peak often indicates column activity. Silanol groups present in the column or injector liners are a source of this activity. An oxidized stationary phase will also cause tailing alcohol peaks. In most cases, stationary phase damage and the resulting active sites are permanent and cannot be reversed without substantial effort. Alcohol peak tailing can also be caused by contamination. Sample residues from previous injections or other contaminants will interact with the alcohols resulting in tailing peaks. These contaminants can be removed by solvent rinsing the column and cleaning the injector.

Acids and Bases

Usually substituted phenols (acid) and anilines (base) are used to measure a columnís behavior toward acidic or basic compounds, respectively. The presence of peak tailing for either peak indicates that there is reversible adsorption and that the column (or perhaps the injector liner) is exhibiting acidic or basic characteristics. The acid peak will tail if the column is too basic, and the base peak will tail if the column is too acidic. Columns should exhibit a ìneutralî character if they are to be applicable for a wide range of analyses. The height of the acid and base peaks are also compared to the height of a hydrocarbon peak. Hydrocarbons are not susceptible to adsorption, thus they often serve as reference peaks. The ratio of the peak height of the acid and base to a hydrocarbon is calculated. A reduction in the height ratio indicates that the column is irreversibly adsorbing the corresponding acid or base.

Others

Polynuclear aromatic hydrocarbons (PAHs) or fatty acid methyl esters (FAMEs) are often included in test mixtures. They may be used to calculate the number of theoretical plates, coating efficiencies, retention (***k***) or retention indices.

msp
MSP KOFEL
Industriestrasse 26
CH-3052 Zollikofen
mass spectrometer &
signal processing products

Telefon: 031
972 31 52
Fax: (+41) 31
971 46 43
info@msp.ch