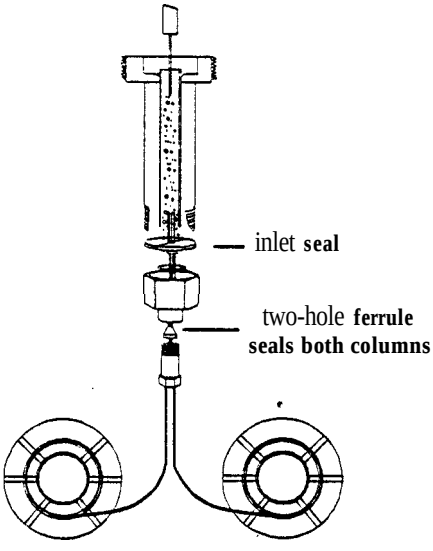
Hints for the Capillary Chromatographer

Confirmational Analysis Using Dual Capillary Columns

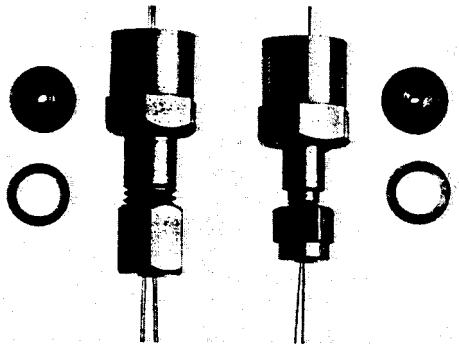
While capillary columns offer high resolution, they do not necessarily separate all of the components contained in a complex mixture. Coeiutions can occur which decrease the quantitative and qualitative accuracy of an analysis. This is particularly a problem for detectors such as FIDs, ECDs, NPDs, and other detectors that do not give positive identification for each peak. Even mass spectrometers cannot differentiate between structural isomers and must rely on the capillary column to separate isomers for proper quantitation.

Anaiyzing the same sample on two columns of different polarity can. increase both the qualitative and quantitative reliability. However, having to repeat the analysis on a second column will significantly reduce sample throughput. The simple solution to improving analytical reliability without reducing sample throughput is to use a simultaneous dual column technique. This technique involves connecting two capBary columns to one GC inlet and connecting each column to its own detection system (Figure 1). Both columns are usually of the same internal diameter so the flow rates are balanced.and similar amounts of the analytes are directed onto each column. This approach will result in confirmational analysis without reducing sample throughput. Simultaneous dual column analysis has become a more routine technique in laboratories involved with complex analyses in complicated matrices.

Figure 1 - Dual column analysis increases the quantitative and qualitative accuracy of an analysis.



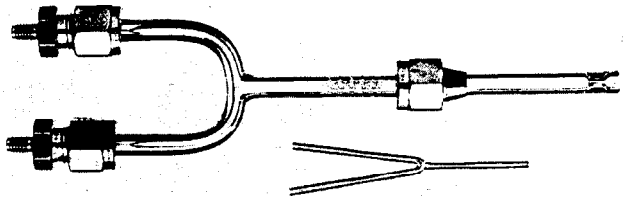
Using Split or Splitless Injectors



Narrow bore capillary columns can use two-hole capillary femdes in standard 1/16"fittings. 0.53mm ID columns require 1/8' inletfinings and l/8* two-hale ferrules to accommodate the larger column ODs.

Spiit or splitless injections are the easiest dual column analyses to perform. Both columns can be inserted into the split/splitless inlet fitting and terminate in the inlet sleeve. Columns with internal diameters of 0.3mm ID or less (or 0.5mm OD) can be inserted directly into the l/16" standard capillary fitting by using a two-hole capillary ferrule. Columns with internal diameters of 0.53mm ID cannot be inserted into a standard l/16" capillary fitting because the outside column diameter (0.8mm) is too large for both to fit simultaneously. Special inlet fittings which use a 1/8" fitting and l/8" two-hole ferrule can be used for 0.53mm ID column.

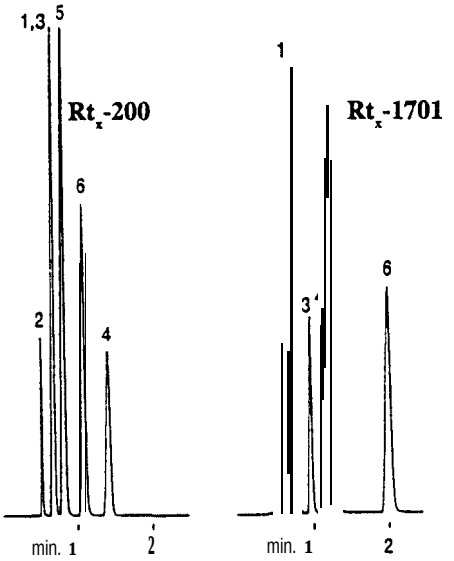
Using On-column or Direct Injectors



A Direct Injection Tee compared to a Universal "Y" Press-Tight Connector.

On-column or direct injections require a Press-Tight'cormection to the inlet sleeve. Usually a section of 0.53mm ID tubing is attached to one leg of a Press-Tight connector. Analysts must be careful that the flow through both legs of the the "Y" is similar or the detector response will differ. Another

Figure 2 - A dual column system improves qualitative and quantitative accuracy of a blood alcohol analysis.



Compounds	
1	acetaldehyde
2	methanol
3	ethanol
4	acetone
5	isopropanol
6	N-propanol (IS)

30m. 0.53mm ID, 3.0um Rtx-200 (cat.#15085)
30m. 0.53mm ID. 3.0um Rtx-1701 (cat.#12085)
Oven temp.: 40C isothermal
In]. temp.: 125C
temp.: 250°C
Carrier gas: hydrogen @
Sample: lOOml headspace
direct injection
Detector: FID

approach is to use a Direct Injection Tee which is installed into the injector and each column can be connected to the remaining legs of the Tee. The Direct Injection Tee has a vaporization chamber to reduce sample backflash and mixing a device, such as a glass screw or cyclone, to ensure sufficient vaporization and reduce discrimination of preferential splitting.

Choosing a Confirmational Column

The selection of a confirmational column is based on three factors: elution order change, thermal stability, and analysis time. The columns should be of differing polarity so the elution orders are significantly different on both columns. Usually nonpolar or intermediately polar columns are chosen because both have similar thermal stabilities. Highly polar columns often have limited maximum operating temperatures which can restrict the analytical molecular weight range. Sometimes a column coated with a thinner film for an intermediate or polar stationary phase is chosen to complement a thicker film nonpolar column in an effort to match analysis times more closely. The analysis of blood alcohols demonstrates the power of dual column confirmational analysis. Figure 2 shows this analysis on a 30m, 0.53mm ID, 3.0um Rtx-200 and 1701. Individually, neither column can resolve all components in the sample mixture. Acetaldehyde coelutes with ethanol on the Rtx-200 making quantitation impossible. The R-t-1701 fully resolves the acetaldehyde from ethanol but allows acetone and isopropanol to elute closely. By comparing the summed areas of the peaks that coelute on one column to the individual areas that are fully resolved on the second column, an accurate quantitative determination can be made.

Simultaneous dual column systems increase the quantitative and qualitative accuracy for complex analyses. They also increase laboratory productivity by providing twice the amount of data without the added expense of additional instrument time.

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