Hints for the Capillary Chromatographer

Techniques for Dual Capillary Column Confirmational Analysis

While capillary columns offer high resolution, they do not necessarily separate all components contained in complex mixtures. Coelutions can occur which decrease the quantitative and qualitative accuracy of an analysis. This is particularly a problem for ECDs, FIDs, NPDs and other detectors which do not give a positive identification for each peak. Even mass spectrometers cannot differentiate between structural isomers and must rely on the column for complete separation. Dual column confirmational analysis using two columns of different polarity can increase the reliability of GC data. If two peaks coelute on the first column, they can usually be separated on a second column of different polarity enhancing qualitative results. Quantitative results can be confirmed since the areas of the coeluting peaks on the first column should equal the combined areas of separated peaks on the second column.

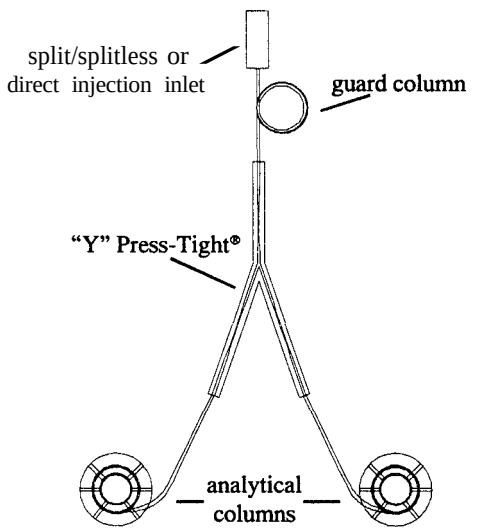
There are three types of single inlet/dual column connection techniques commonly used. The technique chosen will depend on whether split/splitless or direct injections are performed. Only the '7"' Press-Tight@ connector/guard column combination can be used with either split/splitless or direct injection techniques. The two-hole ferrule technique works best with split/splitless injections, whereas the direct injection tee is designed to function in a 1/4" packed column injection system operated in the direct injection mode. All three techniques will be described separately.

"Y" **Press-Tight @ Connector with Guard Tubing**
Figure 1 shows the "Y" Press-Tight@ configuration for dual column confirmational analysis. A five-meter guard column is connected to the base of the "Y" Press-Tight@ with the two analytical columns connected to each outlet leg of the connector. The guard column can be connected to either a split/splitless or direct injection inlet depending on the analyst's preference. The vaporized sample initially travels through the guard column until it reaches the "Y" Press-Tighta where the sample stream splits and a portion travels onto each column. The sample continues to travel through each analytical column until it reaches the detector and provides individual chromatograms.

Press-Tight@ "Y"s connect fused silica tubing in the same fashion as a straight Press-Tight" connector. A square cut using a sapphire blade or ceramic scoring wafer is essential to forming a good seal. Examine the column end to make sure it is square and insert it into the Press-Tight@ connector, pushing firmly until a uniform brown polyimide "ring" forms. In addition, a small amount of polyimide glue can be used to strengthen the connection.

Usually, the inside diameter of the guard tubing is chosen to match the analytical columns. However, 0.53mm ID guard tubing can be used with two 0.32mm ID analytical columns if the flow rate through the guard tubing is high enough to avoid band broadening. The combined flow rate through each analytical column should equal or exceed the carrier gas optimum flow rate through the larger bore guard tubing.

Figure 1 - The "Y" Press-TightaD configuration allows dual columns to be used in either a split/splitless or direct injection inlet.

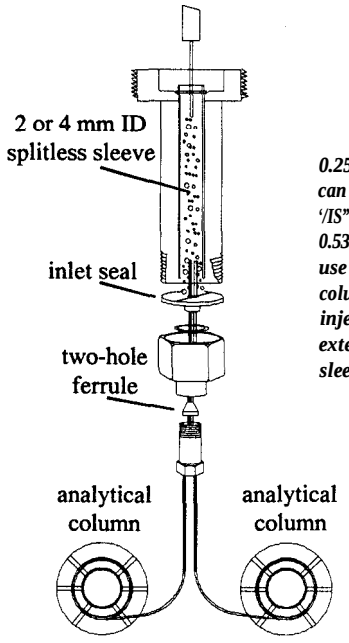


The "Y" Press-TighP con&ration offers versatility since it allows any diameter column or guard column to be connected to any inlet such as spliusplitless or direct.

Two Hole Ferrule for Split/Splitless Injectors
Dual column confirmational analysis can also be performed by connecting two columns simultaneously to the same split/splitless inlet via a two-hole ferrule (Figure 2). Most i/16" capillary inlet fittings will accommodate two 0.25 or 0.32mm ID capillary columns. However, two 0.53mn-r ID columns are too large to fit a standard 1/16" capillary inlet fitting and require a special l/g" capillary inlet fitting with a l/s" two-hole ferrule. Use a split or splitless liner with at least a 4mm ID to ensure that both column ends will fit into the sleeve. If 2mm ID inserts are used, the analyst runs the risk of thecolumn end sitting too close to the sleeve wall which increases split/splitless mass discrimination effects. Standard gooseneck sleeves can not be used because the restriction is less than lmm

and does not accommodate both columns side by side. Recently, extended goosenecks have become available which are designed with a 4mm internal base to accommodate even two 0.53mm ID columns simultaneously.

Figure 2 - Two hole ferrules can be used to allow dual column confirmational analysis in the same split/splitless inlet.



0.25 and 0.32mm ID column bores can be used with standard '/IS" inletfittings. However, 0.53mm ID columns require the use of '/~"fittings to allow both columns to fit side by side in the injector. Either straight or extended gooseneck split/splitless sleeves can be used.

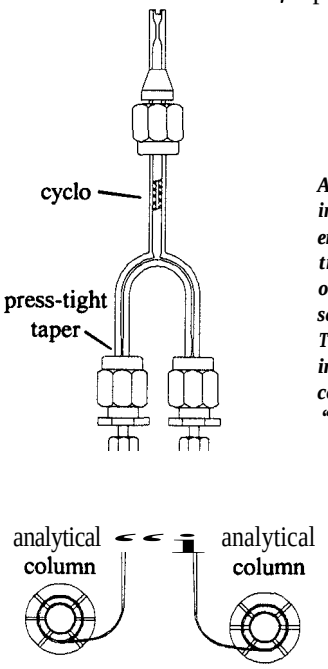
Direct Injection Tee

Many analysts prefer to perform dual column confirmational analysis using direct injection into a 1/4" packed column injection port. Special glass inlet "T"s are available to allow direct connections into two 0.32 or 0.53mm ID columns (Figure 3). The connection from the column inlet to the "T" is made via a Press-Tight@ taper as the primary sealing mechanism and a 7/4" to 1/16" reducing fitting as the secondary sealing mechanism. A proper Press-Tight@' seal between the column and glass inlet Direct Injection "T" is essential to prevent peak tailing and can be visually observed in Restek's Dual Direct Injection Tee. For the direct injection '7" to function properly, the sample must be thoroughly vaporized prior to the "T" splitting point. Glass wool can be used but may detract from the inertness of the system. Devices such as inverted cups or glass screws (cycles) can also be incorporated into the inlet leg to ensure complete sample vaporization. These devices also ensure a high degree of inertness since they can be deactivated as a complete unit.

Uniform Sample Splitting

Regardless of which type of dual column system you choose, both column diameters and lengths should be the same. This will ensure that the same amount of sample reaches each column. Slight differences in flow rates between each analytical column are acceptable. However, large flow differences cause an excessive amount of sample to be delivered preferentially onto one column resulting in lower sensitivity for the other column.

Figure 3 - A dual column direct injection '7" allows two 0.32 or 0.53mm ID columns to be securely connected to one 1/4" packed column inlet.



A dual column direct injection "T" incorporates a glass screw to ensure complete samule vaooriza-tion prior to ` sample onto two columns. The dual sealing mechanisms of a Press-Tight@ taper and reducing fitting increase the ease of use and confidence over the Press-TighP "Y" configuration.

Simultaneous dual column confirmational analysis increases qualitative and quantitative reliability without increasing analysis time. The "Y" Press-Tighta can be used with any injection mode. The two column ferrule technique can only be used for split/splitless injectors, whereas the dual column direct injection '7"' must be used in 1/4" packed column injection ports. No conclusive evidence exists that favors one technique over the other when analyzing adsorptive compounds. However, the two-hole ferrule technique used in the splitless injection mode exhibited the highest amount of molecular weight discrimination. Direct Injection is preferred over splitless injection when analyzing high molecular weight compounds, because it minimizes molecular weight discrimination. (For more information on molecular weight discrimination, request Restek's ***Guide to Direction-column Flash Vaporization Injection.)*** Therefore the direct injection tee or the"Y" Press-Tight@ in the direct injection mode is recommended over the two-hole ferrule when analyzing high molecular weight compounds or samples with a wide boiling point range. Otherwise, the choice depends on the analyst's personal preference and inlet limitations.

See Restek's ***Chromatography Products Catalog*** under Dual Column Analysis or call your local distributor for more information. ■