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Applications note

Bonded PLOT Columns for Dual-Column GC Analysis of Gases and Volatiles

Porous Layer Open Tubular (PLOT) columns have been used widely in the gas chromatography (GC) analysis of gases and volatile compounds. Their strong retention and unique selectivity for these analytes of interest make them ideal for a variety of environmental and petrochemical applications. They are more sensitive and efficient, and provide faster analyses of gases and volatiles than packed or gas-liquid columns.

Restek's bonded **Rt-Msieve™ 5A** PLOT column, made from molecular sieve 5A, provides the most effective separation of all the permanent gases and is capable of separating critical pairs such as He/Ne and Ar/O_2 to baseline at 30°C or above. But, this column is very reactive and retentive for CO_2, resulting in poor chromatographic peak shape.

Restek's bonded porous polymer-based **Rt-QPLOT™** column provides tremendous opportunities for tuning chromatographic selectivities in the analysis of gaseous and volatile hydrocarbons, and various solvents. It is very sensitive for CO_2 but does not separate the permanent gases as well as the molecular sieve 5A. Therefore, coupling the Rt-Msieve™ 5A and Rt-QPLOT™ columns dramatically enhances the separation effectiveness for the analysis of permanent gases and CO_2. Two segments of Rt-QPLOT™ column (50cm each, cut from the end of the analytical column cat.# 19716) were connected to a 30m x 0.32mm Rt-Msieve™ 5A column and a 30m x 0.53mm Rt-QPLOT™ column in parallel using two universal Press-Tight® 'Y' connectors (Figure 1).

Using this configuration at an oven temperature of 30°C, all the gases are well separated in a single run (see Figure 2 on back). Under the listed analysis conditions, all gases including CO_2 elute from the Rt-QPLOT™ column and reach the detector first. Components not separated by the Rt-QPLOT™ column (i.e., He, Ar, O_2, N_2, CH_4, and CO) are separated by the Rt-Msieve™ 5A column and reach the detector next. It should be noted that Ar and O_2 are baseline resolved, and that CO elutes after 18 minutes with good peak shape.

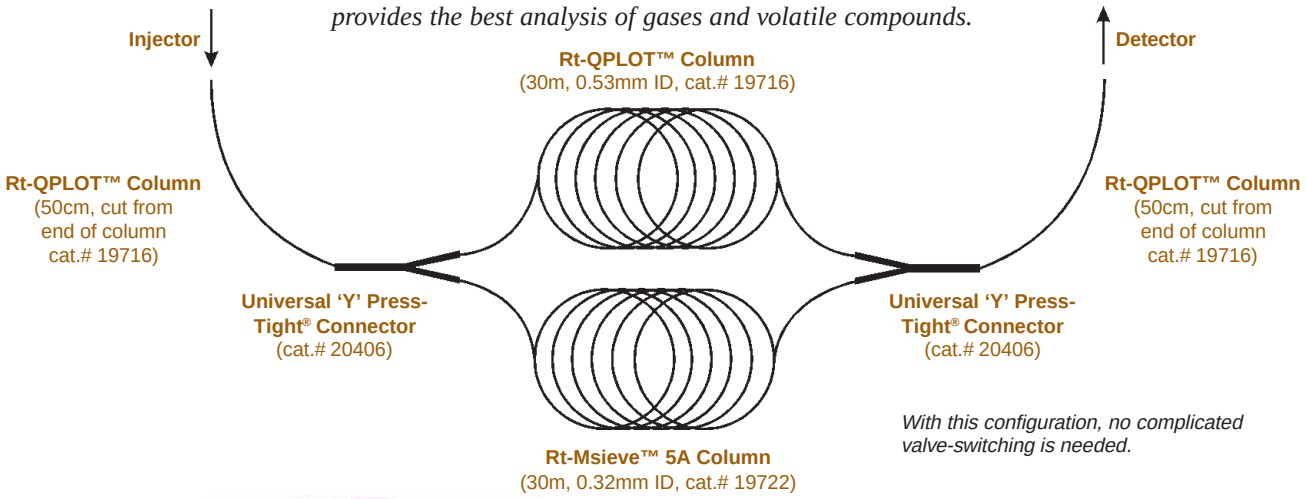
Quantitation of this analysis can be done a couple of ways: The external standard approach assumes that the sample split at the head of the two columns is consistent from run to run, which is common practice for dual-column methods. The analytical system is initially calibrated with known standards and the subsequent sample areas are compared to the calibration curve. The concentrations of the compounds are reported directly from the calibration curve.

Another method is to report the compounds using area percent. In order to obtain accurate results, the area counts of the compounds eluting from both columns can be normalized.

The mixture of He, Ar, O_2, N_2, CH_4, CO, and CO_2, is impossible to completely separate using any single column, unless cryogenic cooling is applied. Multiple separation mechanisms are needed to pull these compounds apart at normal analysis conditions. This Applications Note focuses on a solution to this difficult analysis using Restek's bonded PLOT columns.

Figure 1

A dual-column configuration of an Rt-Msieve™ 5A and an Rt-QPLOT™ column provides the best analysis of gases and volatile compounds.



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Because the sample splits prior to the two analytical columns, the CO_2 area count from the Rt-QPLOT™ column must be normalized to the other compounds eluting from the Rt-Msieve™ 5A column. One way to normalize the area counts is to use methane as an internal reference compound. Divide the CO_2 area by the methane area obtained from the Rt-QPLOT™ column. The peak areas of the compounds eluting from the Rt-Msieve™ 5A column are divided by the associated methane area. The normalized areas from both columns then can be combined to calculate percent levels for each of the compounds.

A second method to normalize the CO_2 response on the Rt-QPLOT™ column to the areas of the compounds on the Rt-Msieve™ 5A column is to use the summation of the permanent gas (He, Ar, O_2, N_2, and CO) areas from each column. The relationship is:

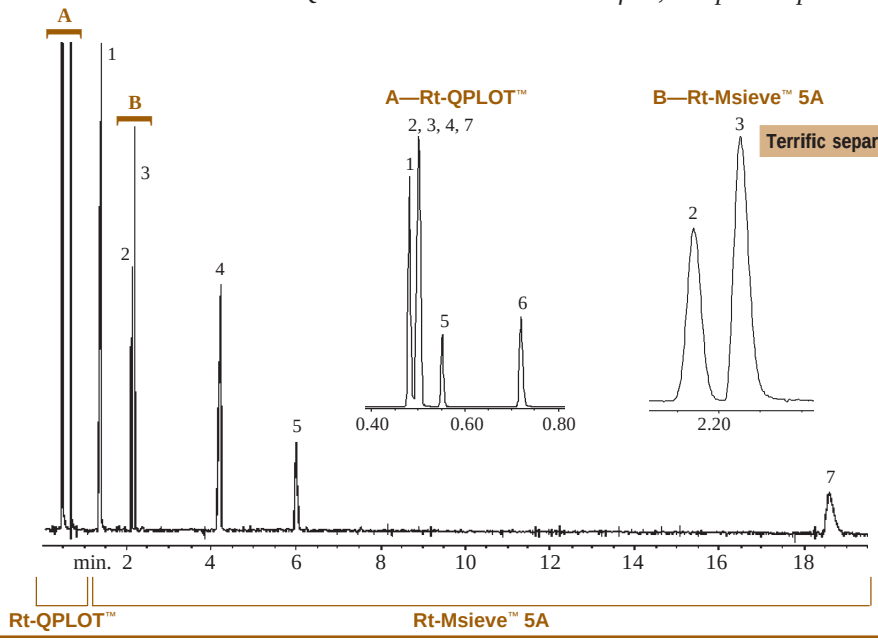
$$\frac{\text{(area of } CO_2 \text{ from the Rt-QPLOT™ column)}}{\text{(total area perm. gases from the Rt-QPLOT™ column)}} \times \text{(total area perm. gases from the Rt-Msieve™ 5A column)} = \text{(relative area } CO_2 \text{ on the Rt-Msieve™ 5A column)}$$

Using the relative area of CO_2 with the other compound areas on the Rt-Msieve™ 5A column, the percent levels of the compounds can be calculated.

The analysis of permanent gases including CO_2 has previously required valve switching with two column systems. Now, with Restek's innovative analytical approach using the Rt-Msieve™ 5A and the Rt-QPLOT™ columns in parallel, this analysis can be accomplished without cooling or complicated valve switching and with less expensive equipment.

Figure 2

Use the Rt-Msieve™ 5A and Rt-QPLOT™ columns to achieve fast, complete separation of gases and volatile compounds.



1. helium
2. argon
3. oxygen
4. nitrogen
5. methane
6. carbon dioxide
7. carbon monoxide

See Figure 1 for column data.
Oven temp.: 30°C isothermal;
Inj.: 20µL, split injection (10:1), 5% each compound;
Carrier gas: hydrogen, 8psi;
Det.: HP µTCD.

Product Listing

Rt-Msieve™ 5A Columns

ID	df (µm)	Temp. Limits	15m	30m
0.32mm	30	up to 300°C	19720	19722
0.53mm	50	up to 300°C	19721	19723

Rt-QPLOT™ Columns

ID	df (µm)	Temp. Limits	15m	30m
0.32mm	10	up to 250°C	19717	19718
0.53mm	20	up to 250°C	19715	19716

Universal 'Y' Press-Tight® Connectors

20405, each	20406, 3-pk.

Universal Angled 'Y' Press-Tight® Connectors

20403, each	20404, 3-pk.

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