Continuous Improvement in Petroleum/Petrochemical Analysis—HP's Family of Innovative PLOT Columns

Brochure Brief

Porous Layer Open Tubular (PLOT) columns have been replacing traditional packed columns used for gas analysis because of advantages in resolution, speed and reproducibility. PLOT columns are fused silica columns coated with a homogenous layer of adsorbent particles.

Agilent technologies now offers a comprehensive line of PLOT columns for analysis of fixed gases, low molecular weight hydrocarbon isomers, and volatile polar compounds (**Table 1**). The Agilent family of PLOT columns includes a variety of dimensions to suit your application. For applications requiring high capacity, select wide bore 530 µm columns. If speed is a factor, or you are using GC/MS, select a 250 µm or 320 µm column.

Table 1. HP-PLOT Column Applications

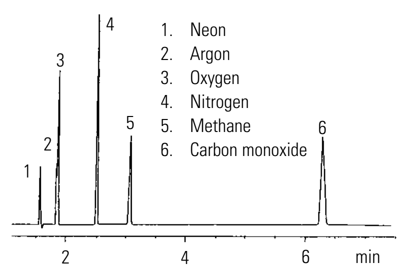
Column	Stationary Phase	Typical Applications
HP-PLOT MoleSieve	Molecular Sieve 5A Zeolite	Permanet gases, noble gases
HP-PLOT Al_2O_3	Aluminum Oxide deactivated with KCl, Na_2SO_4 or a proprietary deactivation	C1–C6 hydrocarbon isomers including refinery gas, ethylene, propylene, and butadiene
HP-PLOT Q	Polystyrene-divinylbenzene	All C1–C3 isomers, alkanes to C14, CO_2, methane, air/CO, water, oxygenated compounds, sulfur compounds, solvents

HP-PLOT MoleSieve

HP-PLOT MoleSieve columns are recommended for the analysis of permanent and noble gases. HP-PLOT MoleSieve columns have a layer of 12–50 µm thick molecular sieve 5A zeolite immobilized inside the tubing. This provides excellent separation of small molecules such as nitrogen, oxygen, argon, methane, and carbon monoxide.

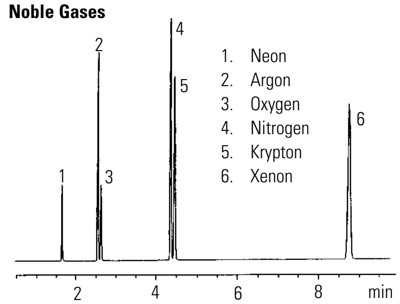
The absorbent is bound to the inner surface of the fused silica tubing, reducing the chance of signal spikes due to particles breaking free and invading the system valve or detector. This results in improved sensitivity of detectors and overall system accuracy.

Figure 1. Gas analysis using thin-film HP-PLOT MoleSieve column



Column: HP-PLOT MoleSieve, 30 m x 0.32 mm x 12 µm (Part no. 19091P-MS4)
Carrier: Helium, 2 ml/min
Oven: 40°C
Sample: 250 µl, Split (ratio 75:1)
GC System: 5890 Series II GC with EPC and TCD

Figure 2. Resolution of argon and oxygen at 35°C



Column: HP-PLOT MoleSieve, 30 m x 0.53 mm x 50 µm (Part no. 19095P-MSO)
Carrier: Helium, 4 ml/min
Oven: 35°C (3 min) to 120°C (5 min) at 25°C/min
Sample: 250 µl, Split (ratio 50:1)
GC System: 5890 Series II GC with EPC and TCD

The analysis of permanent gases is a common use of molecular sieve columns. HP-PLOT MoleSieve columns provide ample column efficiency and loading capacity to separate N_2, O_2, CH_4, and CO gases completely (**Figure 1**). This column demonstrates retention of the gases necessary for many analyses requiring timing of gas sample valves. In the isothermal run shown at 40°C, there is partial resolution of argon and oxygen. For complete resolution of argon and oxygen, without cryogenic cooling, the thick-film HP-PLOT MoleSieve columns are available. **Figure 2** shows the analysis of ambient noble gases at near ambient temperatures. In less than 10 minutes, all noble gases plus oxygen and nitrogen, were well separated at 35°C.

HP MoleSieve columns are available in 0.32 and 0.53 dimensions. For applications requiring the resolution of argon and oxygen without expensive cryogenic cooling, select the thick film HP-PLOT MoleSieve/5A columns. The economical thin-film columns are ideal for many application including routine air monitoring and analysis in less than 10 seconds. The thin-film columns resolve argon and oxygen at subambient temperatures.

HP-PLOT Al_2O_3

The HP-PLOT Al_2O_3 family consists of columns using Al_2O_3 particles and a different deactivation. All HP-PLOT Al_2O_3 columns are suitable for resolving C1–C6 hydrocarbon isomers in hydrocarbon streams. Each has particular advantages noted in **Table 2**.

These columns are available in ids ranging from 0.25 mm to 0.53 mm. The 0.53 mm id is most popular, due to its high sample capacity and compatibility with large volume samples requiring gas sampling valves. **Figure 3** shows analysis of ethylene and propylene streams using a 0.53 mm HP-PLOT Al_2O_3 KCl; detection limits for trace hydrocarbon components of less than 10 ppm can be achieved with HP-PLOT Al_2O_3 columns.

All three columns have an upper temperature limit of 200°C for the 0.32 mm and 0.53 mm id columns. The 0.25 mm id columns can be used up to 250°C for short periods. Because of the improved efficiency and higher temperature limit of these columns, the 0.25 mm id column can provide fast, baseline resolution of hydrocarbon isomers up to C10 (**Figure 4**). The 0.25 mm id column is also the most suitable for GC-MS work.

HP-PLOT Al_2O_3 KCl

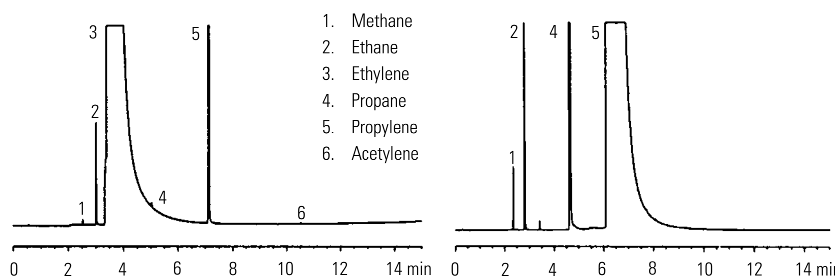
Because HP-PLOT Al_2O_3 KCl is the least polar of the Al_2O_3 columns (as measured by RI of olefins), it will show minimal adsorption of and provide accurate quantitation of dienes.

Table 2. HP-PLOT Al_2O_3 Columns

Column	Deactivation Type	Polarity	Application Strength
HP-PLOT Al_2O_3 KCl	KCl	Least polar Al_2O_3 phase (lowest retention of olefins versus comparable paraffin)	Column of choice for accurate quantitation of dienes, especially propadiene and butadiene from ethylene and propylene streams
HP-PLOT Al_2O_3 "S"	Sodium Sulphate	More polar than Al_2O_3 KCl	Excellent general use column Optimized C2–C4 isometer separation Excellent reproducibility Best for resolving acetylene from butane and propylene from iso-butane*
HP-PLOT Al_2O_3 "M"	Proprietary	Most polar of Al_2O_3 PLOTs (in general—highest retention of olefin versus comparable paraffin)	Excellent general use column Optimized C2–C4 isomer separation Excellent reproducibility Best for resolving cyclopropane from propylene*

** except for differences noted above, the "S" and "M" type columns will show very similar chromatographic performance.*

Figure 3. Analysis of ethylene and propylene



Column: HP-PLOT Al_2O_3, "KCl", 50 m x 0.53 mm x 15 µm (Part no. 19095P-K25)
Oven: 35°C (2 min), 10°C/min to 190°C
Carrier: Hydrogen, 10 ml/min
Inlet: 250°C Split ratio 30:1
Detector: FID, 250°C
Sample: 0.25 cc Sampling Loop

Column: HP-PLOT Al_2O_3, "KCl", 50 m x 0.53 mm x 15 µm (Part no. 19095P-K25)
Oven: 35°C (2 min), 10°C/min to 180°C
Carrier: Hydrogen, 10 ml/min
Inlet: 250°C Split ratio 30:1
Detector: FID, 250°C
Sample: 0.25 cc Sampling Loop

The peak area ratios in **Figure 5** demonstrate that HP-PLOT KCl has little activity for butadiene.

Because of its low polarity, commercially available Al_2O_3 KCl type columns can exhibit some drawbacks, especially under overload conditions: 1) poor resolution of cyclopropane and propylene, 2) co-elution of acetylene and/or propadiene with propylene and butane isomers.

HP-PLOT Al_2O_3 "S" and "M"

These columns are excellent general use columns. Unlike KCl-based columns, the HP-PLOT Al_2O_3 "S" and "M" columns are ***optimized for hydrocarbon isomer separation*** and ***selectivity***. These columns permit baseline separation of C1–C6 isomers in hydrocarbon streams, even under overload conditions; they provide excellent selectivity for ethylene, propane, cyclopropane,

Figure 4. Fast C1 to C10 hydrocarbon separation

1. Methane
2. Ethane
3. Ethylene
4. Propane
5. Cyclopropane
6. Propylene
7. iso-butane
8. n-Butane
9. Propadiene
10. Acetylene
11. trans-2-Butene
12. Butene-1
13. isobutylene
14. cis-2-Butene
15. iso-Pentane
16. n-Pentane
17. 1,3-Butadiene
18. Propyne
19. trans-Pentene
20. Pentene-1
21. iso-Pentane
22. cis-2-Pentene
23. Hexane
24. Benzene
25. Heptane
26. i-Octane
27. n-Octane
28. Cyclohexene
29. Toluene
30. Nonane
31. Ethylbenzene
32. p-Xylene
33. Decane

Columns: HP-PLOT Al_2O_3, "S", 30 m x 0.25 mm x 5 µm (Part no. 19091P-S33)
Oven: 120°C (1 min), 25°C/min to 250°C (2 min)
Carrier: Hydrogen, 28 psi @ 120°C
Inlet: 250°C, Split flow 120 ml/min
Detector: FID, 250°C
Sample: 5 µl headspace gases

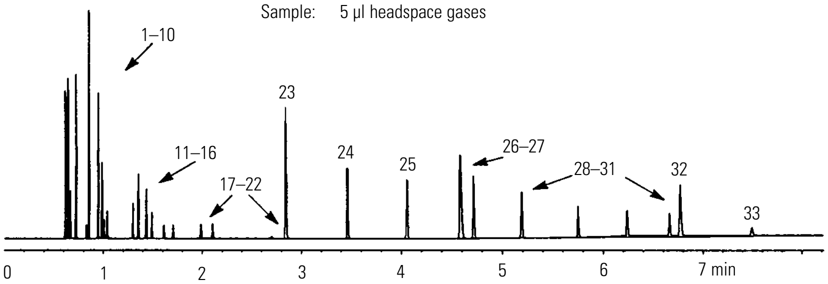
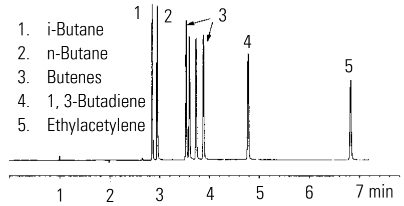


Figure 5. C4 isomers including butadiene at low levels

1. i-Butane
2. n-Butane
3. Butenes
4. 1, 3-Butadiene
5. Ethylacetylene



Columns: HP-PLOT Al_2O_3, "KCl", 50 m x 0.53 mm x 15 µm (Part no. 19095P-K25)
Oven: 120°C (3 min), 10°C/min to 180°C
Carrier: Hydrogen, 5 psi @ 120°C
Inlet: 250°C, split flow 200 ml/min
Detector: FID, 250°C
Sample: 0.25 cc sampling valve

propylene, acetylene, propadiene, and 1,3 butadiene.

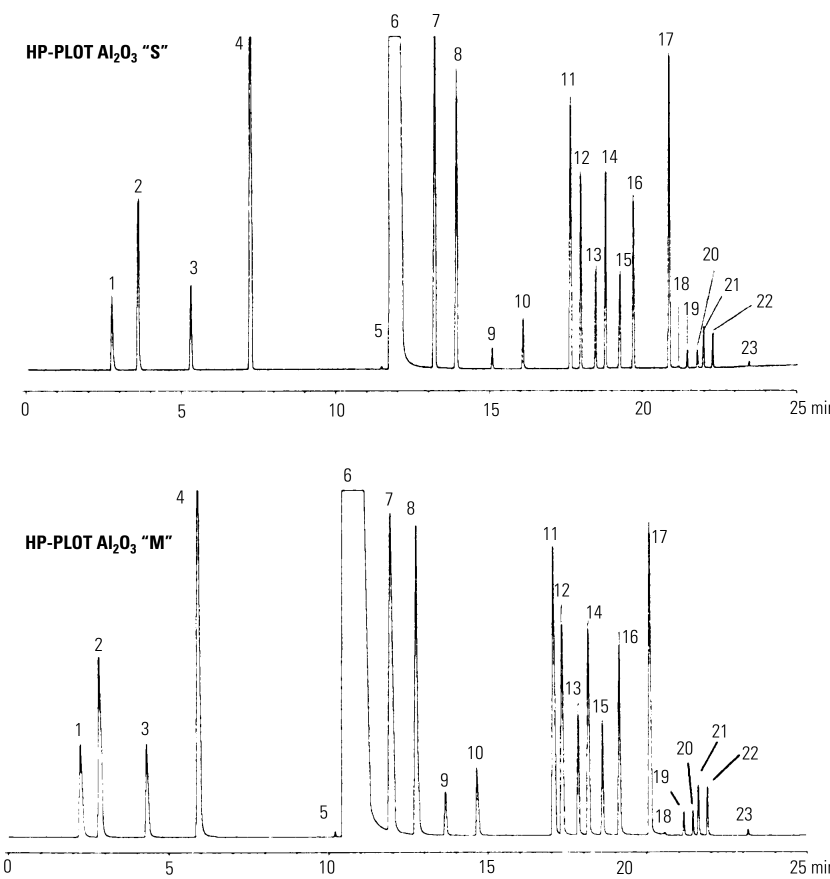
Optimized Separation of Ethylene and Propylene Samples

In **Figure 6** the analysis of 92% pure propylene is shown using HP-PLOT Al_2O_3 "S" and "M" columns. Twenty-three components were baseline resolved in 24 minutes with both cyclopropane and iso-butane being resolved from propylene, without the trace cylopropane (60 ppm) being masked by overload.

In **Figure 7** the important impurities in a 98.5% ethylene sample are resolved on the same "S" column. The high capacity of this column is reflected in the quantitation of propane which shows no peak distortion even under severe overload conditions (1.5 cc injected). All of the chromatograms show sharp, symmetrical peaks attributable to the excellent resolution and unique selectivity of the columns.

Figure 8 shows the analysis of a competitive PLOT Al_2O_3 column under overload conditions. Note: 1) propylene tails, reducing the resolution of propylene and iso-butane and 2) ethylene distorts the propane peak and splits it into two peaks. The reason for these difference versus the HP-PLOT Al_2O_3"S" or "M" columns

Figure 6. Analysis of propylene on HP-PLOT Al_2O_3 columns



Columns: HP-PLOT Al_2O_3, "S" and "M", 0.53 mm x 50 m (Part no. 19095P-S25 and 19095P-M25)
Carrier: Hydrogen constant flow mode, column flow 6 ml/min
Oven: 35°C (2 min), 5°C/min to 100°C, 10°C/min to 180°C (5 min)
Inlet: Split/splitless 250°C
Detector: FID, 250°C
Sample: **Figure 6:** 5 µl splitless of propylene blending with C1 to C5 hydrocarbons
Figure 7: 1.5 cc of ethylene blending with C1 to C5 hydrocarbons, split ratio 30:1

can be seen in the RI values for these compounds (**Table 3**).

The HP-PLOT “S” and “M” columns have RI values around 250 for ethylene, which reduce the impact of ethylene overload on impurities eluting immediately afterward. Higher RIs for ethylene, like that demonstrated by the competitive column, will move ethylene closer to the propane peak, resulting in possible masking or peak distortion.

RIs should also be between 350 and 380 to permit excellent resolution of cyclopropane and iso-butane from propylene. RIs lower than 350 will result in poor resolution of cyclopropane and propylene (as seen on the alumina KCl column). Higher RIs will cause propylene to begin to merge with iso-butane.

Note that the RI of acetylene on a KCl column is lower than 400, which can result in acetylene co-eluting with either propylene and/or butane isomers. This has not been observed with HP Al_2O_3 “S” or “M,” as the RI of acetylene exceeds 400. Furthermore, acetylene selectivity on a KCl column has been demonstrated to be sensitive to moisture; acetylene selectivity is less sensitive to moisture when using HP Al_2O_3 ”S” and “M” columns.

Other Applications

Refinery Gas:

Figure 9 shows a separation of refinery gas on an HP Al_2O_3 “S” column. Effective separation of hydrocarbons in refinery gas requires good selectivity for butenes. Because of the unique selectivity of the Agilent column, the separation of trans-2-butene from 1-butene and cis-2-butene from iso-pentane are achieved. The latter pair co-elutes on the competitive column under the conditions used.

Transformer Oil:

Transformer oil samples require low-level detection of acetylene and ethylene. **Figure 10** shows a 1000 ppm sample of these

Peak identification for Figures 6–11

1. Methane
2. Ethane
3. Ethylene
4. Propane
5. Cyclopropane
6. Propylene
8. n-Butane
9. Propadiene
10. Acetylene
11. trans-2-Butene
12. 1-Butene
13. Isobutylene
14. Cis-2-Butene
15. iso-pentane
16. N-Pentane
17. 1,3-Butadiene
18. Propyne
20. 2-Methyl-Butene
21. 1-Pentene
22. Cis-Pentene
23. Hexane

Table 3. Retention Indexes

Column	RI (Ethylene)	RI (Propylene)	RI (Acetylene)
Alumina/KCl	248	349	372
HP “S”	248	361	418
HP “M”	254	369	407
Competitive Al_2O_3 Column	263.5	380	417

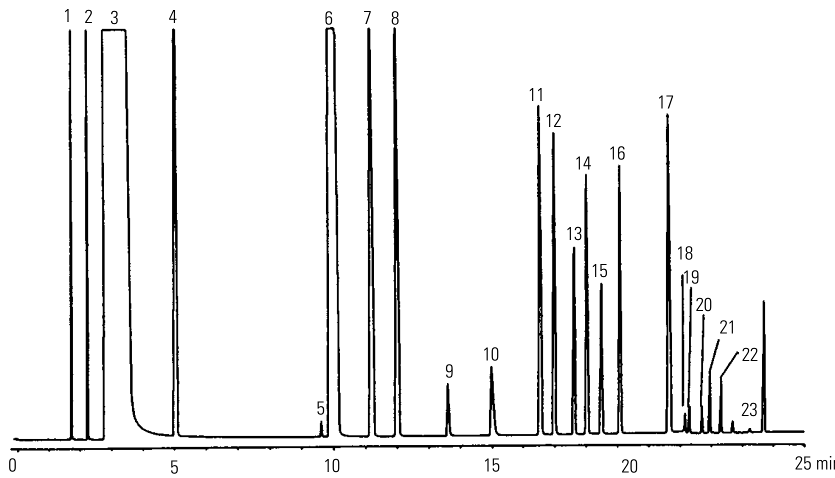
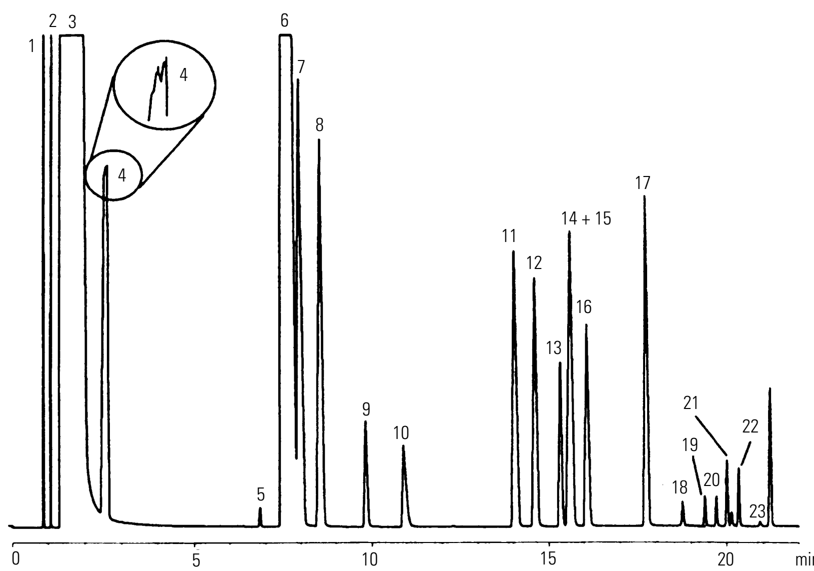


Figure 7. Analysis of ethylene on HP-PLOT Al_2O_3 “S” column



Column: Competitive Al_2O_3, PLOT Column, 0.53 mm x 30 m
Carrier: Helium constant flow mode, column flow 6 ml/min
Oven: 35°C (2 min), 5°C/min to 100°C, 10°C/min to 180°C (5 min)
Inlet: Split/splitless 250°C
Detector: FID, 250°C
Sample: 0.5 cc of ethylene blending with C1 to C5 hydrocarbons, split ratio 20:1

Figure 8. Competitive Al_2O_3 column under overload conditions

compounds at 5 µl, equivalent to 5 ppm in a 1 ml sample.

1,3-Butadiene

1,3-butadiene streams require resolution of the major component from n-pentane and propyne. Because of the unique selectivity of the HP Al_2O_3 “S” and “M” columns, these components could be resolved.

HP-PLOT Q

HP-PLOT Q is Agilent’s most versatile PLOT column. HP-PLOT Q is suitable for resolutions of:

- Hydrocarbons (all C1–C3 isomers, alkanes up to C14, natural gas, refinery gas, ethylene, propylene streams)
- CO_2, methane, air/CO, water
- Polar solvents, oxygenates and sulfur compounds.

HP-PLOT Q Features:

HP-PLOT Q columns feature baseline separation starting at 60°C for alkane hydrocarbons up to C6, all C1–C3 isomers, air/CO, water, CO_2, and other polar compounds resulting in enhanced resolution and decreased cycle times.

In addition, HP-PLOT Q columns (**Table 4**) provide:

- excellent mechanical stability with little or no spiking, making these columns ideal for valved and MS analyses
- low bleed decreasing conditioning times and improving sensitivity
- outstanding reproducibility, saving rework time and repurchasing cost
- a maximum isothermal operating temperature of 270°C

For temperature programmed runs, the maximum operating temperature for the HP-PLOT Q capillary is 290°C and this temperature should be used only for short periods of time to elute high-boiling impurities up to C14.

Enhanced Resolution and Decreased Cycle Time:

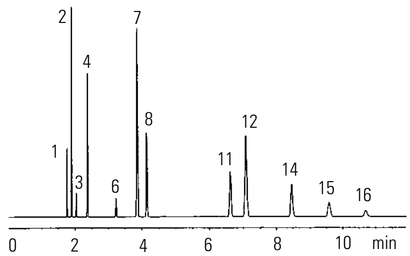
The separation of C1–C5 hydrocarbons beginning at 60°C with baseline resolution of all C1–C3 isomers is shown in **Figure 12**.

In the separation of C1–C5 hydrocarbons shown on a competitive PLOT Q column (**Figure 13**), ethylene co-elutes with acetylene; cyclopropane co-elutes with propadiene; isobutene co-elutes with n-butatane; and baseline rise starts at approximately 165°C.

Natural gas provides an excellent example of PLOT Q ability to resolve hydrocarbons from a variety of polar compounds (**Figure 14**). Note the baseline separation of air/CO and CO_2 from methane; H_2S and H_2O and neo-pentane and iso-pentane. The excellent resolution of the HP-PLOT Q capillary in comparison to other commercially available PLOT-Q columns is demonstrated in the resolution of air/CO and CO_2 from methane starting at 60°C (**Table 5**).

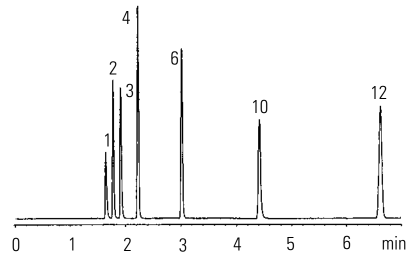
This resolution can result in up to 30% savings in GC cycle time versus analyses on comparable columns which require starting at 40°C.

Figure 9. Refinery gas separation on a 50 m x 0.53 mm id Al_2O_3 PLOT “S” type column



Column:	HP-PLOT Al_2O_3, “S”, 0.53 mm x 50 m (Part no. 19095P-S25)
Carrier:	Helium Constant flow mode, column flow 6 ml/min
Oven:	100°C
Inlet:	Split/splitless 250°C
Detector:	FID, 250°C
Sample:	5 µl of refinery gas sample split ratio 20:1

Figure 10. 5 µL transformer oil gas injected splitless



Column:	HP-PLOT Al_2O_3, “S”, 0.53 mm x 50 m (Part no. 19095P-S25)
Carrier	Helium Constant flow mode, column flow 6 ml/min
Oven:	100°C
Inlet:	Split/splitless 250°C
Detector:	FID, 250°C
Sample:	5 µl of transform oil gas sample, 1000 ppm, splitless

Table 4. HP-PLOT Q Columns

Features	Advantages	Benefits
Separations starting at higher temperatures	Baseline separation at 60°C for alkane hydrocarbons up to C6, all C1 to C3 isomers, air, water, CO_2, and other polar compounds	Enhanced resolution Cycle time improvements up to 30%
Excellent mechanical stability	No spiking No traps required	Suitable for valved, online, and MS work
Low bleed		Improved sensitivity Shortest conditioning time of any DVB-type phase
Excellent chemical stability	Hundreds of injections of alcohols with no loss of peak shape or retention time	Outstanding column lifetime Excellent reproducibility
Enhanced versatility	Wide range of separations	Separations of parafins, olefins aromaticsulfur compounds Decreased cycle time
High temperature limit	Expanded operating range Possible elimination of valve steps	
Same selectivity as other DVB phases		Easy method transfer

Stability for Valved, On-Line or MS Analyses

The proprietary process used by Agilent to bind polymeric particles to the HP-PLOT Q column wall results in excellent mechanical stability making the use of a particle trap unnecessary.

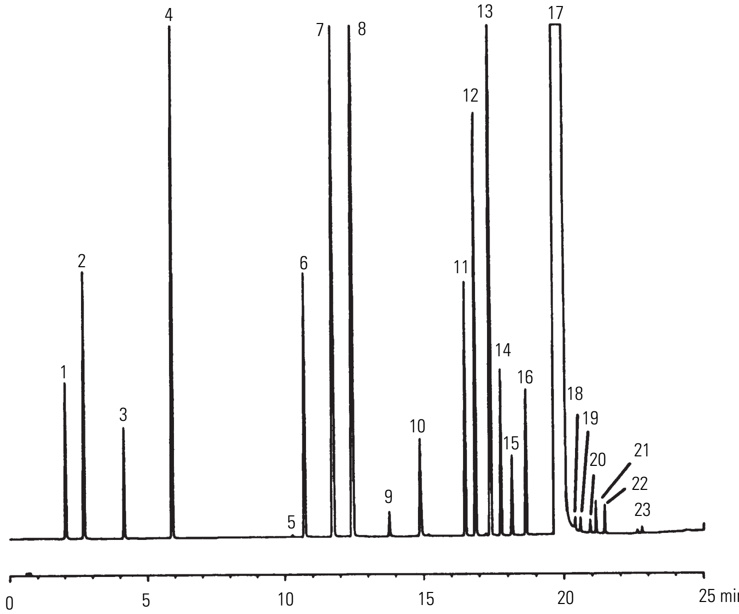
A customer reported that “even without a particle trap, we observed absence of spiking. This demonstrated that the HP-PLOT Q immobilization process is superior to commercially available DVB technologies. The lack of spiking was observed even with pressure swings as high as 5-10 psig/minute making this column suitable for use in valved applications, in an on-line environment, and for MS applications.” In fact, each HP-PLOT Q column is tested using a valved operation.

Outstanding Reproducibility and Lifetime

Until now, the analysis of samples containing hydrocarbons and polar compounds, including water and alcohol, has been problematic when using PLOT columns. This was due to the loss of reproducibility and column lifetime associated with moisture and alcohol induced hydrolysis, active site formation, and peak tailing. The bonded DVB-based HP-PLOT Q column exhibits excellent stability over hundreds of injections for unmatched reproducibility and column lifetime.

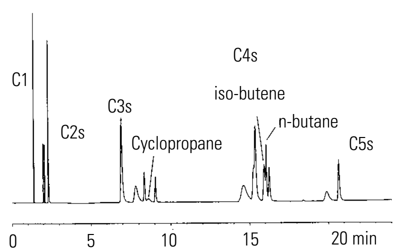
This is demonstrated in the analysis of alcohols shown (over 200 alcohol injections, **Figure 15**). These samples were injected using headspace (70°C) onto a column with an initial temperature of 50°C to recondense the sample then ramped to 250°C. These conditions were selected to thoroughly test PLOT Q inertness to alcohol.

The HP-PLOT Q column did not show any sign of peak shape deterioration even after 1800 injections. Other



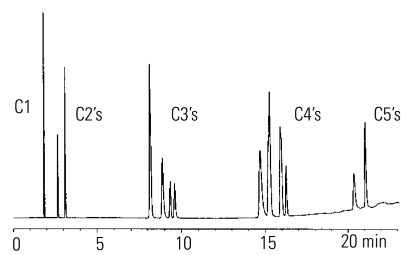
Column: HP-PLOT Al_2O_3, “S”, 50 m x 0.53 mm x 15 µm (Part no. 19095P-S25)
Carrier: Helium, 30 cm/sec (35°C), 4.6 ml/min constant flow
Oven: 35°C (2.5 min) to 100°C at 5°C/min, to 180°C at 10°C/min
Injection: Splitless; inlet 250°C
Detector: FID (250°C), 5890 Series II GC
Sample: 5 µl of 1, 3-butadiene blending with C1 to C5 hydrocarbons

Figure 11. 1, 3-Butadiene



GC: Agilent 6890
Column: HP-PLOT “Q”, 0.53 mm x 30 m (Part no. 19095P-Q04)
Oven: 60°C (8 min), 7.5°C/min to 180°C
Carrier: H_2, 8 ml/min @ 60°C
Inlet: 250°C, Split ratio 12:1
Detector: FID, 250°C
Sample: 17 HC, 5 µl (C1-20%, C2 to C5-1% to 14%)

Figure 12. Hydrocarbons C1–C5 using an HP-PLOT Q column



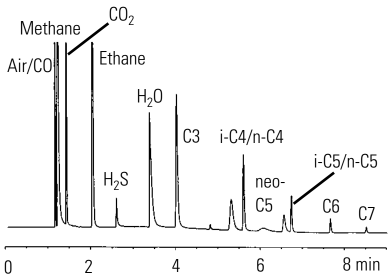
GC: Agilent 6890
Column: 0.53 mm x 25 m Brand X
Oven: 60°C (3 min), 7.5°C/min to 180°C (1 min)
Carrier: H_2, 7 ml/min (25°C)
Inlet: 250°C, 5 µl, Split mode
Split flow: 150 ml/min
Detector: FID, 250°C
Sample: 17 HC C1 to C5

Figure 13. Hydrocarbons C1–C5 using a competitive PLOT column

commercially available DVB columns are reported to show degradation after 30–60 injections.

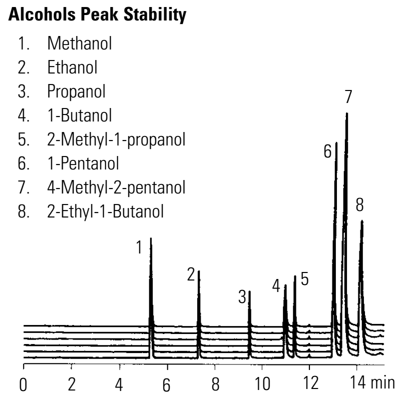
Table 6 provides additional proof of the resistance of the HP-PLOT Q column to water and alcohol contamination. Retention time and area ratios are shown for 1 µl of aqueous sample with 500 ppm of alcohols injected on-column for 150 sequential runs.

Figure 14. Baseline resolution of air/CO, CO_2, and methane in a natural gas sample. C7 concentration is approximately 200 ppm



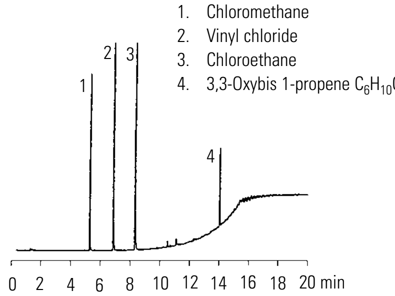
Column:	HP-PLOT Q, 0.53 mm x 30 m (Part no. 19095P-Q04)
Carrier:	Helium, 8.6 ml/min @ 60°C
Oven:	60°C (2 min), 30°C/min to 240°C (1 min)
Detector:	TCD 250°C
Injection:	250°C Split mode (12:1)
Sample:	0.25 cc Natural gas sample, Methane, 80% +

Figure 15. Consecutive runs for up to 200 injections of alcohol



GC:	6890
Column:	HP-PLOT Q, (Part no. 19095P-Q04)
Oven:	50°C (2 min), to 250°C at 15°C/min
Carrier:	Helium, 25 psig
Detector:	FID
Analyte concentration:	100 ppm (v/v)

Figure 16. Chlorinated hydrocarbons



GC:	6890
Column:	HP-PLOT Q (Part no. 19095P-Q04)
Oven:	50°C (2 min), to 250°C at 15°C/min
Carrier:	Helium, 25 psig
Detector:	FID
Analyte concentration:	100 ppm

Table 5. Comparative analyses: Resolution of PLOT-Q, Brand X, and Brand Y columns

Resolution	HP-PLOT Q	Brand X	Brand Y
R (Air/CO, Methane, 40°C)	2.56	2.38	1.76
R (Air/CO, Methane, 60°C)	1.76	1.60	1.34
R (CO_2, Methane, 40°C)	11.2	8.1	10.1
R (CO_2, Methane, 60°C)	7.2	5.1	5.3

Column size: 0.53 mm x 30 m x 40 µm. Sample size: 0.25 cc.

Table 6. Sequential runs of alcohol retention time and area ratio

Retention Time (min)	Average	RSD%	Area Ratio	Average	RSD%
Methanol	1.947	0.316	MeOH/EtOH	0.845	1.641
Ethanol	2.893	0.109	IPA/EtOH	1.181	2.066
IPA	3.880	0.071	Peak Width (IPA)	0.0508	4.02

HP-PLOT Q Applications

Hydrocarbons

HP-PLOT Q columns are used for the analysis of hydrocarbon gases such as refinery gas, ethylene and propylene, natural gas, and polar compounds. The new HP-PLOT-Q column can be used for the separation of hydrocarbons up to C14, as well as all C2 and C3 isomers.

Chlorinated hydrocarbons are used as catalyst moderators in industrial reactors, and these hydrocarbons can also be degraded by other commercial PLOT capillaries.

In contrast, HP-PLOT Q columns exhibit excellent peak shape for chlorinated hydrocarbons with no evidence of degradation (**Figure 16**).

Refinery Gas

In the GC/TCD analysis of refinery gas shown (**Figure 17**), PLOT-Q columns show good separation of analytes even with a 0.25 ml sample size and COS is well separated from propylene. The water peak shape can be improved by elevating the column temperature (100 ppb COS has been reported to be resolved from 99% propylene using 1 ml gas phase direct injection SCD).

Natural Gas

In the GC/TCD analysis of natural gas, HP-PLOT Q columns show good separation of water and H_2S, air/CO and CO_2 from methane, and neopentane and iso-pentane with a starting temperature as high as 60°C (**Figure 14**).

1,3 Butadiene and Aromatic

In the analysis of 1,3, butadiene and aromatics, 1,3 butadiene exhibits no polymerization (**Figure 18**) with the HP-PLOT Q column. Polymerization is sometimes seen during identical separations with other commercially available DVB-based columns.

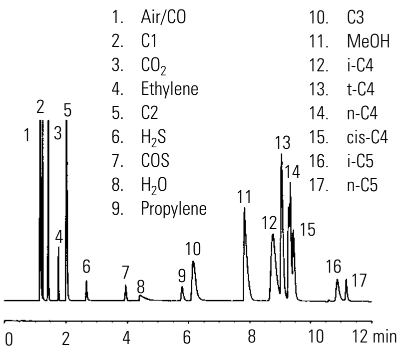
Oxygenates

The HP-PLOT Q column has proven to be effective in the resolution of oxygenates other than alcohols and water. This is demonstrated in the separation of allyl chloride (2-chloropropene) from ethylene oxide synthetic standard (**Figure 19**) and the separation of various oxygenated compounds in hydrocarbons (**Figure 20**).

Polar Solvents

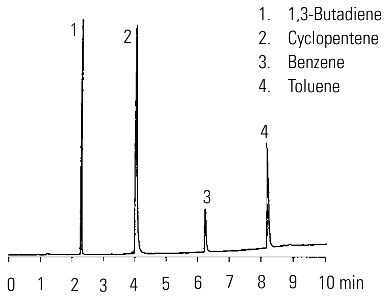
HP-PLOT Q columns also provide good separation for polar solvents such as methanol, acetone, and methylene chloride as well as low boiling point alcohols (**Figure 21**).

Figure 17. Separation of refinery gas sample on HP-PLOT Q



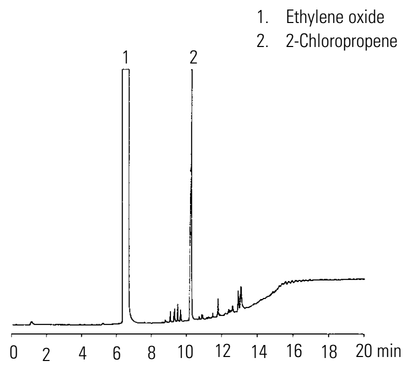
GC: 6890
Column: HP-PLOT Q, 0.53 mm x 30 m (Part no. 19095P-Q04)
Oven: 60°C (5 min), 20°C/min to 200°C (1 min)
Carrier: Helium p=9.0 psi @ 60°C
Inlet: 250°C Split mode 0.25 cc valve
Split flow: 100 ml/min
Detector: TCD, 250°C
Sample: Refinery gas and others

Figure 18. Butadiene and aromatics



GC: 6890
Column: HP-PLOT Q, (Part no. 19095P-Q04)
Oven: 150°C (2 min), to 250°C at 15°C/min
Carrier: Helium, 25 psig
Detector: FID
Butadiene concentration: 200 ppm

Figure 19. Ethylene oxide synthetic standard



GC: 6890
Column: HP-PLOT Q (Part no. 19095P-Q04)
Oven: 50°C (2 min), to 250°C at 15°C/min
Carrier: Helium, 25 psig
Detector: FID
Injection: 1 µl liquid injection, 1:40 split
Analyte concentration: 2000 ppm

This chromatogram demonstrates the breadth and clean separation of polar solvents, alcohols, ketones, esters, aldehydes, and aromatics using PLOT-Q columns.

Sulfur Compounds

Sulfur components can be well separated using HP-PLOT Q columns, including mercaptans, H_2S, COS, and disulfides (**Figures 22** and **23**). COS shows excellent peak symmetry. Note however, that DVB columns are typically not used for low-level analysis of sulfur-containing compounds due to adsorption. This is also true for HP-PLOT Q. However, detection of COS at 100 ppb has been reported with HP-PLOT Q using 1 ml gas-phase direct injection, Sulfur Chemiluminescence Detector.

Same Selectivity as Other DVB-based Columns

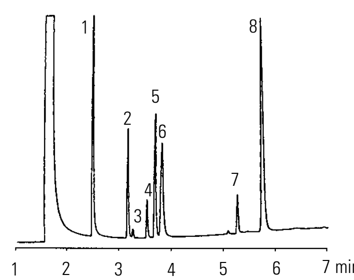
Because selectivity is maintained with PLOT Q versus other commercially available DVB-phases, transferring your method to HP-PLOT Q (**Table 7**) is easy.

Hewlett-Packard has been manufacturing high-quality gas chromatographs, columns, and supplies for decades. And we build the same quality and excellence into our expanded range of capillary PLOT columns.

HP columns are manufactured to ISO 9001 registered processes. Each column is individually tested to exacting specifications for capacity, retention, base/acid ratio, bleed, and plates/meter. Column construction and detail are monitored with the same attention to detail that we spend on our chromatography instruments. That's why Agilent columns produce repeatable, reliable results from run to run and column to column.

Figure 20. Oxygenates in hydrocarbon sample

1. Ethanol
2. 2-Propanone
3. Dichloromethane
4. Acetic acid, methyl ester
5. Diethyl ether
6. Pentane
7. Acetic acid, ethyl ester
8. Hexane



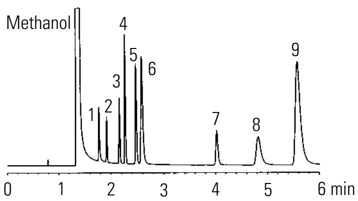
GC: 6890
Column: HP-PLOT Q, (Part no. 19095P-Q04)
Oven: 150°C (2 min), to 250°C at 15°C/min
Carrier: Helium, 25 psig
Detector: FID
Butadiene concentration: 1000 ppm in methanol

Table 7. RI comparisions of HP-PLOT Q and other commercially available DVB-based columns

	RI Ethylene (60°C)	RI Propylene (60°C)	RI Ethyl Acetate (150°C)
HP-PLOT Q	170.2	291.8	576.5
Vendor A	170.6	291.5	575.1
Vendor B	172.2	290.1	576.6

Figure 21. Separation of solvents on HP-PLOT Q

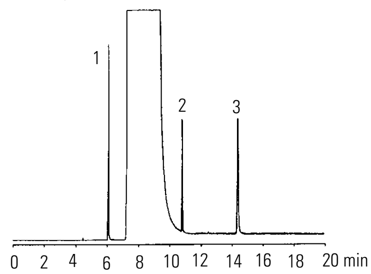
1. Ethanol
2. Acetonitrile
3. Acetone
4. Dichloromethane
5. Diethyl ether
6. Pentane
7. Ethyl acetate
8. Hexane
9. Benzene



GC:	6890
Column:	HP-PLOT Q, 0.53 mm x 30 m (Part no. 19095P-Q04)
Oven:	150°C
Carrier:	Helium, 9 ml/min
Injection:	250°C Split mode (12:1)
Detector:	FID, 250°C
Sample:	1 µl 0.1–0.3% solvents in methanol

Figure 22. Sulphur compounds in hexane

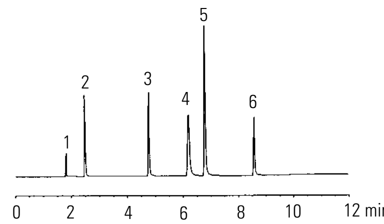
1. Propyl mercaptan
2. Dimethyl disulfide
3. Dietyl disulfide



GC System:	6890/FID (Part no. 19095P-Q04)
Oven:	10°C (2 min), 15°C/min to 250°C
Carrier:	10 psi He head pressure
Analyte concentration:	50 ppm

Figure 23. Odor additives—separation of mercaptans and other sulfur compounds on HP-PLOT Q

1. Hydrogen Sulfide
2. Carbonyl Sulfide
3. Ethanelthiol
4. iso-propyl mercaptan
5. n-propyl mercaptan
6. n-butyl mercaptan



Column:	HP-PLOT Q, 0.53 mm x 30 m (Part no. 19095P-Q04)
Oven:	105°C (1 min), 15°C/min to 240°C at (2 min)
Carrier:	Hydrogen p=3.8 psi @ 60°C
Inlet:	250°C Split mode, 0.25 cc injection
Split flow:	100 ml/min
Detector:	FID, 250°C
concentration:	H_2S, COS: <1% mercaptans: <10%

HP-PLOT Columns Ordering Information

HP-PLOT MoleSieve Columns

Description*	HP-PLOT MoleSieve Part No.
Thin Film	
15 m x 0.32 mm x 12 µm	19091P-MS3
30 m x 0.32 mm x 12 µm	19091P-MS4
15 m x 0.53 mm x 25 µm	19095P-MS5
30 m x 0.53 mm x 25 µm	19095P-MS6
Thick Film	
15 m x 0.32 mm x 25 µm	19091P-MS7
30 m x 0.32 mm x 25 µm	19091P-MS8
15 m x 0.53 mm x 50 µm	19095P-MS9
30 m x 0.53 mm x 50 µm	19095P-MS0

HP-PLOT Al_2O_3, "S," "M," and "KCl" Columns

Description*	HP-PLOT Al_2O_3 "S"	HP-PLOT Al_2O_3 "M" Part No.	HP-PLOT Al_2O_3 "KCl" Part No.
15 m x 0.25 mm x 5 µm	19091P-S31	19091P-M31	19091P-K31
30 m x 0.25 mm x 5 µm	19091P-S33	19091P-M33	19091P-K33
50 m x 0.32 mm x 8 µm	19091P-S15	19091P-M15	19091P-K15
15 m x 0.53 mm x 15 µm	19095P-S21	19095P-M21	
30 m x 0.53 mm x 15 µm	19095P-S23	19095P-M23	19095P-K23
50 m x 0.53 mm x 15 µm	19095P-S25	19095P-M25	19095P-K25

HP-PLOT Q Columns

Description*	HP-PLOT Q Part No.
15 m x 0.32 mm x 20 µm	19091P-Q03
30 m x 0.32 mm x 20 µm	19091P-Q04
15 m x 0.53 mm x 40 µm	19095P-Q03
30 m x 0.53 mm x 40 µm	19095P-Q04

** Length x Internal Diameter x Film Thickness*

Additional Reading

Additional reading is described below. These pieces can be ordered by calling your local HP office, or by contacting us at *http://www.agilent.com*

1. *Trace Level Hydrocarbon Impurities in Ethylene and Propylene* (5965-7824E)
2. *New Look at Light Hydrocarbon Separations on Commercial Alumina PLOT Columns: Column Selectivity and Separation.* Zhenghua Ji. J. High Resolution Chromatography-Vol 19, January 1996. Pgs 32-36 (Not on HP Web Site)
3. *Optimized Determination of C1–C6 Impurities in Propylene and Ethylene Using HP-PLOT Al_2O_3 Columns* (5962-8417E)
4. *HP-PLOT Q: Next Generation Columns for Light Hydrocarbons and Polar Compounds* (5965-7872E)
5. *GC/TCD Analysis of a Natural Gas Sample on a Single HP-PLOT Q Column* (5966-0978E)
6. *Agilent Chemical/Petrochemical Solutions Catalog (5968-6124E)*

Printed in USA 4/00
5968-1763E

Agilent Technologies
Innovating the HP Way