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Molecular Sieve PLOT Columns for the Capillary GC Separation of Permanent Gases

W. Betz, M. Keeler

PLOT capillary columns containing Carboxen carbon molecular sieves or molecular sieve 5A have sufficient capacity for directly injected, highly concentrated samples of permanent gases and C2 and C3 hydrocarbons. The pore size ranges and bonded layering of the particles provide effective separations of these analytes.

Porous solids, such as molecular sieves, are traditionally used in packed bed separation processes ranging from bulk-scale to bench-scale. Processes at the bench scale include gas-solid chromatography (GSC), using molecular sieves consisting of either carbon or aluminosilicate. Advancements in chromatographic techniques, including improvements in wall-coated capillary columns and molecular sieves, have enhanced efficiency and led to the development of a family of porous layer open tubular (PLOT) columns.

Carboxen-1006■ and Molecular Sieve 5A PLOT capillary columns exhibit adsorption strengths that allow the separation of analytes with subambient boiling points. Table 1 illustrates the reproducibility of these two sets of columns.

The carbon molecular sieve particles in the PLOT layer (30µm) are spherical, and allow an extended path length through the intact particle. These particles also provide a large internal surface area

Table 1. Reproducibility Data for Molecular Sieve PLOT Columns

Description	k′	Standard Deviation
Carboxen-1006 (n = 24)	5.58 (CO_2)	0.54
Mol Sieve 5A (n = 20)	4.97 (CO)	0.36

(725m²/g). A proprietary process is used to adhere these carbon particles to the walls of the column. This provides a stable porous solid surface, eliminates particle loss from the walls, and allows rapid temperature programming of the GC oven for fast-run analyses.

Macropores have a positive effect on the kinetic behavior of a porous solid such as Carboxen™-1006 adsorbent. The plots in Figure A indicate poor kinetic behavior of a micropores-only particle. The presence of macropores allows separation of analytes at linear velocities well above optimum.

Figure A. van Deemter Plot Shows the Effect of Porosity on Efficiency

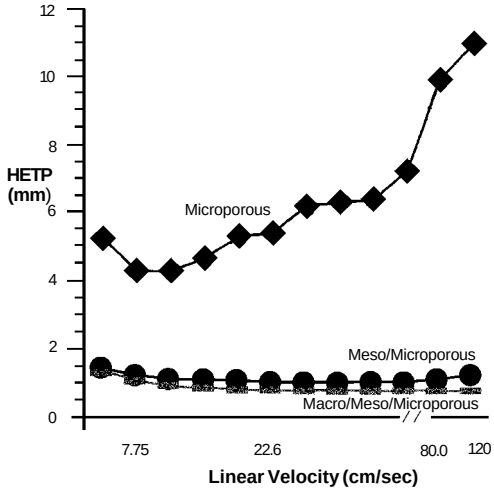

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Figure B. Light Gases on a Carboxen-1006 PLOT Column

Column: **Carboxen-1006 PLOT, 30m x 0.53mm ID**
Cat. No.: **25461**
Oven: 35°C (1 min) to 250°C at 24°C/min
Carrier: helium, 10mL/min
Det.: TCD, 250°C
Inj.: 70µL, direct, 230°C

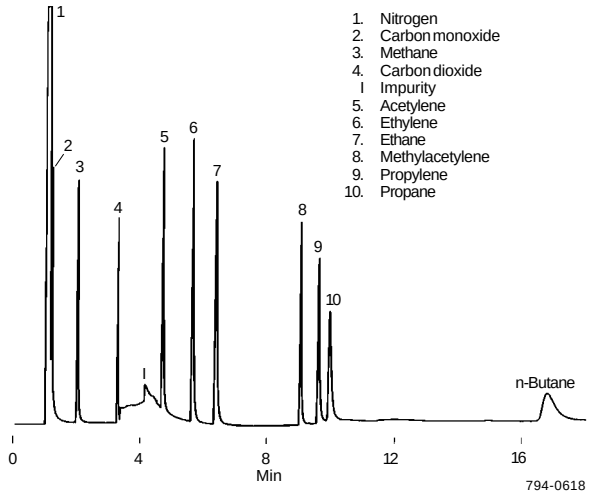

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Figure B illustrates the ability of the Carboxen-1006 molecular sieve to separate nitrogen, carbon monoxide, and other light gases. Note the excellent baseline resolution. Figures C and D show the use of the Carboxen-1006 column for the separation of formalin solutions and impurities in propylene.

The aluminosilicate PLOT column incorporates a molecular sieve 5A in the sub-micron particle size range. The process used to prepare this column is similar to that for the Carboxen-1006 column. Thus, similar optimization procedures are available. Figure E illustrates the use of the Molecular Sieve 5A column.

If you currently are using packed columns and traditional adsorbents for GSC separations of permanent gases or light hydrocarbons, consider the high efficiency of our PLOT capillary columns.

Figure C. Formalin Components on a Carboxen-1006 PLOT Column

Column: **Carboxen-1006 PLOT, 30m x 0.53mm ID**
Cat. No.: **25461**
Oven: 220°C
Carrier: helium, 22cm/sec
Det.: TCD, 230°C
Inj.: 0.2µL formalin solution, direct, 230°C

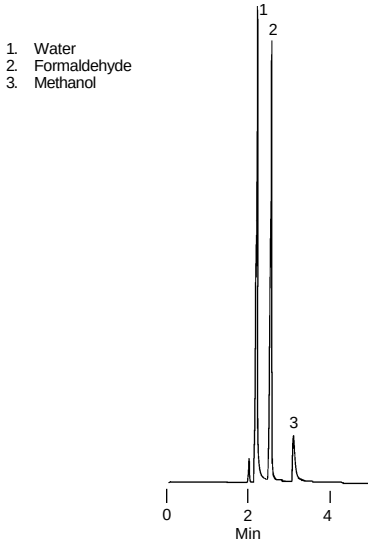

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Figure D. Impurities in Propylene on a Carboxen-1006 PLOT Column

Column: **Carboxen-1006 PLOT, 30m x 0.32mm ID**
Cat. No.: **24241-U**
Oven: 200°C (4 min) to 250°C at 24°C/min
Carrier: helium, 76cm/sec
Det.: FID, 230°C
Inj.: 20µL propylene, approx. 1% each contaminant, direct, 230°C

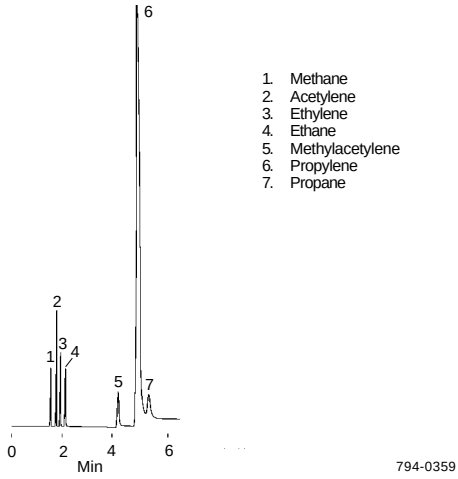

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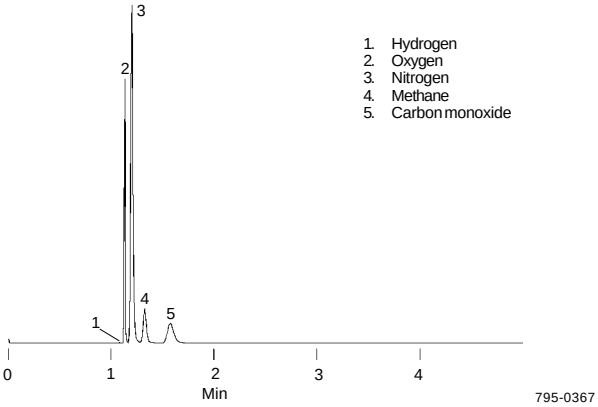
Figure E. Small Molecules on a Molecular Sieve 5A PLOT Column

Column: **Mol Sieve 5A PLOT, 30m x 0.53mm ID**
Cat. No.: **25463**
Oven: 65°C
Carrier: helium, 10mL/min
Det.: TCD, 260°C
Inj.: direct, 260°C



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Ordering Information:

Description	Max. Temp. (°C)	Cat. No.
Carboxen-1006 PLOT Columns		
30m x 0.32mm ID	250	**24241**
30m x 0.53mm ID	250	**25461**
Mol Sieve 5A PLOT Columns		
30m x 0.32mm ID	300	**24243**
30m x 0.53mm ID	300	**25463**

■ US Pat. No. 4,839,331.

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