The Reporter

Reprinted from Volume 15, No. 6, 1996

T296036

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Alumina PLOT Columns for Rapid and Consistent Separations of Light Hydrocarbons by Gas Solid Chromatography

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Supelco, Bellefonte, PA USA

Incorporation of alumina, carbons, porous polymers, and other adsorbents into porous layer open tubular columns (PLOT columns) with relatively small diameters has considerably improved gas-solid chromatographic separations of many small analytes. 30-meter PLOT columns containing sub-micron, granular, activated alumina particles elute unsaturated hydrocarbons after the saturated hydrocarbons of the same carbon number. A patented adhesive technology immobilizes the adsorbent to the column wall – no particles are lost during routine analyses or rapid temperature programming. The high temperature limit of the adhesive, >360°C, makes the operating temperature limit of these columns a function of the particles used, not a function of the adhesive. The columns effectively separate mixtures of C1-C7 hydrocarbons.

Carbons, porous polymers, alumina, and other adsorbents have been used for decades for gas-solid chromatographic separations of small analytes. The evolution of GSC to the degree that these adsorbents can be incorporated into porous layer open tubular columns (PLOT columns) with relatively small diameters has considerably improved resolution for many key analytes.

The choice of adsorbent depends on the properties of the analytes to be separated. Aluminum oxide provides a unique retention pattern for saturated and unsaturated light hydrocarbons, hence its application as a gas-solid adsorbent. We prepare 30-meter PLOT columns with sub-micron, granular, activated alumina particles with a surface area of 350m^2/g. The use of sodium sulfate as a desiccant and activating agent ensures that the activity of the alumina surface is effective in eluting (distilling) unsaturated hydrocarbons after the saturated hydrocarbons of the same carbon number.

In preparing these PLOT columns we incorporate a proprietary, patented adhesive technology through which the adsorbent particles are immobilized, or cemented, to the wall of the capillary column. Because the adhesive also bonds the particles to one another, no particles are lost during routine analyses or rapid temperature programming. A significant feature of the adhesive is its high temperature limit, greater than 360°C, which makes the operating temperature limit of the columns a function of the particles used, not a function of the adhesive.

We also optimize column preparation to ensure that the adhesive does not interfere with the pores in the particles. This, in turn, ensures that analyte molecules are transferred, unrestricted, to the internal micropores in the particles, and that there is no adverse effect on the external working surface. We have used this adhesive technology to prepare PLOT columns containing carbon, Tenax®, Porapak® N, U, and T, HayeSep® A, C, D, N, and T, polymer G-45, Mol Sieve 5X and 13X, and alumina/KCl adsorbents.

C1 to C4 hydrocarbons are separated effectively by a 30-meter alumina PLOT column, using a temperature program (Figure A). The elution pattern desired for analyses of trace impurities in unsaturated hydrocarbons – trace impurities eluting before major peaks – is a significant benefit of this column. The C4 isomers in

Figure A. C1-C4 Hydrocarbons on an Alumina PLOT Column

Column: **Alumina PLOT, 30m x 0.53mm ID**
Cat. No.: **25464**
Oven: 35°C (2.5 min) to 180°C at 5°C/min
Carrier: helium, 4mL/min
Det.: FID, 200°C
Inj.: 10µL, 1% each analyte in nitrogen, direct injection, 200°C

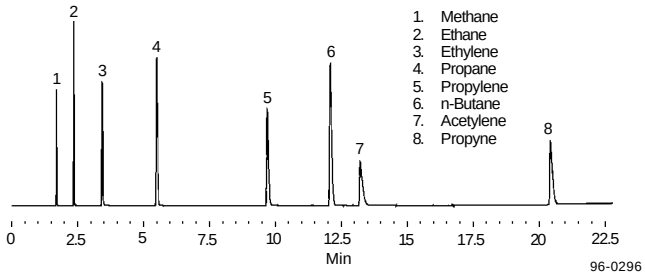
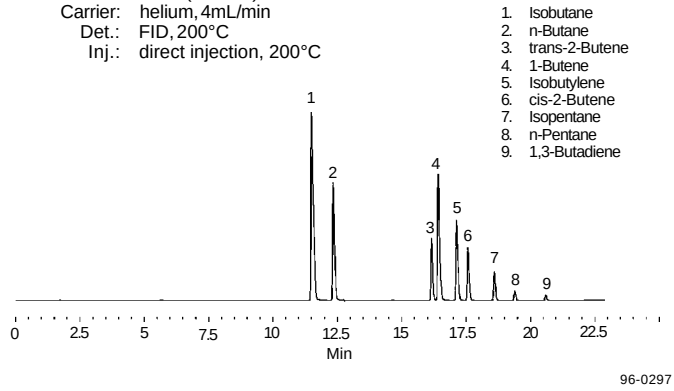


Figure B. Natural Gas Standard (C4 and C5 Hydrocarbons)

Column: **Alumina PLOT, 30m x 0.53mm ID**
Cat. No.: **25464**
Oven: 35°C (2.5 min) to 180°C at 5°C/min
Carrier: helium, 4mL/min
Det.: FID, 200°C
Inj.: direct injection, 200°C



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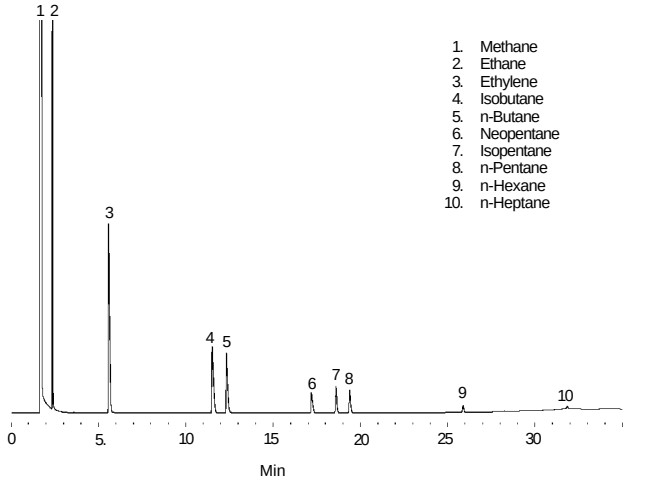
American Society for Testing and Materials (ASTM) natural gas reference standard blend 6 also are effectively separated, using the same column and temperature program (Figure B).

Mixtures containing C1 to C7 hydrocarbons are analyzed by programming the column to 200°C. Figure C shows an analysis of a calorimetric natural gas reference standard comprised of two permanent gases (nitrogen and carbon dioxide, which are not shown), methane (the major component of the sample), and ethane, propane, isobutane, n-butane, isopentane, neopentane, n-hexane, and n-heptane.

Our PLOT columns prepared with carbon molecular sieves, zeolite molecular sieves, porous polymers, and activated alumina provide options for a wide range of analyses, including permanent gases, light hydrocarbons, Freon® gases, sulfur gases, and many polar compounds. Our proprietary adhesive process eliminates particle loss from the column wall and allows rapid temperature programs to be used. Further, our production processes ensure reproducible performance from column to column. We recommend our 30-meter alumina PLOT columns for GSC separations of small hydrocarbons.

Figure C. Natural Gas Standard (C1-C7 Hydrocarbons)

Column: **Alumina PLOT, 30m x 0.53mm ID**
Cat. No.: **25464**
Oven: 35°C (2.5 min) to 200°C at 5°C/min
Carrier: helium, 4mL/min
Det.: FID, 200°C
Inj.: natural gas standard (Cat. No. 3-03-101), direct injection, 200°C



96-0298

Ordering Information:

Description	Cat. No.
Alumina PLOT Columns	
30m x 0.32mm	**24244**
30m x 0.53mm	**25464**
Light Hydrocarbon Mixes*	
C1-C4 Straight Chain	**22566**
C4 Saturates/Unsaturates	**22567**
Natural Gas Standard**	**303101**

For PLOT columns of other dimensions, please inquire.

For an extensive selection of other chemical standards for light hydrocarbons analyses, refer to the Supelco catalog.

*10-20ppm each analyte in nitrogen in a 4-liter SCOTTY® I cylinder. Cat. No. 22566 includes the 8 compounds in Figure A; Cat. No. 22567 is similar to the mix in Figure B, but includes ethylacetylene (1-butyne), does not include n-pentane or isopentane. For 37-liter cylinders, please see our general catalog.

**Includes the components in Figure C, plus helium, nitrogen, and carbon dioxide (which do not elicit a response from a flame ionization detector), in a 14-liter SCOTTY II cylinder. For mole percent composition, please see our general catalog.

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