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Polysiloxane Capillary Column for GC Analysis of Basic Organic Compounds

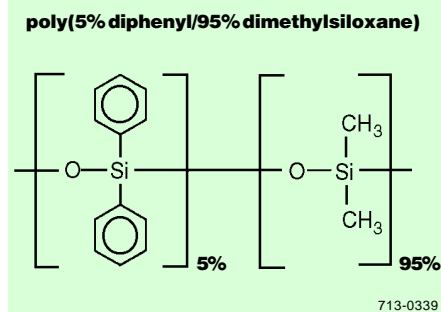
L. Sidisky, Gas Separations; K. Kiefer, Chemical Standards, Supelco, Bellefonte, PA, USA

Our new bonded phase PTA-5 column is designed for higher-temperature analyses of basic compounds.

Basic compounds are among the most difficult substances to analyze using capillary gas chromatography. Traditionally, to separate these compounds, analysts have used a polar polyethylene glycol (PEG)-based stationary phase doped with an inorganic caustic material such as potassium or sodium hydroxide. With these additives, the PEG column performs well — the basic compounds elute with good peak shape and responses. However, the PEG column is limited to a maximum temperature of about 220°C. Also, inorganic doping techniques cannot be applied to the widely used silicone phases, as caustic materials are catalysts used to prepare and degrade these polymers.

An alternative method of analyzing basic compounds includes a column with a very

Figure A. Phase Structure of PTA-5 Capillary Column



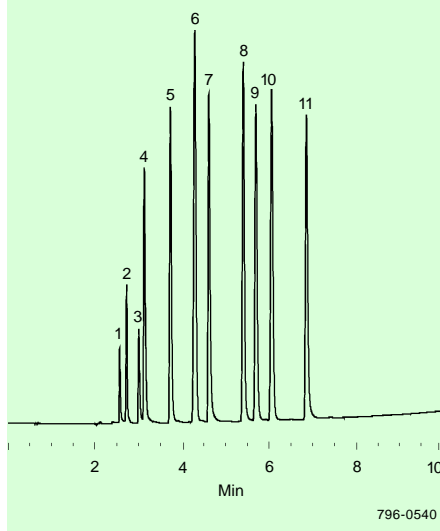
thick film of methylsilicone (4.0µm or more, phase ratio <60). Because of the thickness of the film, such a column is limited to volatile materials.

Using new technology, we recently developed a basic polysiloxane-based capillary column that is applicable to analyses of primary amines, underivatized basic drugs, and other basic compounds. Our new PTA-5 (Pre-Tested Amine) capillary column consists of a poly 5% diphenyl/95% dimethylsiloxane bonded stationary phase (Figure A). This column retains the polarity of 5% phenyl stationary

Figure B. New Column Provides Sharp, Well-Separated Peaks for Reactive Volatile Amines

Column: PTA-5, 30m x 0.53mm ID, 3.0µm film
 Cat. No.: 25439
 Oven: 35°C (2 min) to 160°C at 10°C/min
 Carrier: helium, 30cm/sec (set at 35°C)
 Det.: FID, 260°C
 Inj.: 1.0µL of volatile amine sample, split 100:1, 250°C

1. Methylamine
2. Trimethylamine
3. Dimethylamine
4. Ethylamine
5. Isopropylamine
6. tert-Butylamine
7. n-Propylamine
8. Diethylamine
9. sec-Butylamine
10. Isobutylamine
11. n-Butylamine



phases but, because of a proprietary base deactivated surface, it has an improved inertness to basic components. The maximum operating temperature of the PTA-5 column is 320°C, a significant improvement over the low maximum temperatures recommended for Carbowax®-based amine columns.

Numerous basic samples — such as primary amines, anilines, explosives, nitrosamines, and ethanolamines — can be qualitatively and quantitatively analyzed using a PTA-5 column. Figure B illustrates

the analysis of a series of volatile amines ranging from methylamine through n-butylamine on a 30m x 0.53mm ID x 3.0µm film PTA-5 column. The column provided good resolution and peak shape for these very reactive volatile amines.

Ordering Information:

Description	Cat. No.
PTA-5 Fused Silica Capillary Column 30m x 0.53mm ID, 3.0µm film	25439

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

Trademark

Carbowax — Union Carbide Corp.

