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Chiral Gas Chromatography of Citronellal, β -Citronellol and Linalool on a β -DEX 225 Column

Y. Belov, Gas Separations; A. Kumar, Chemical Standards, Supelco, Bellefonte, PA, USA

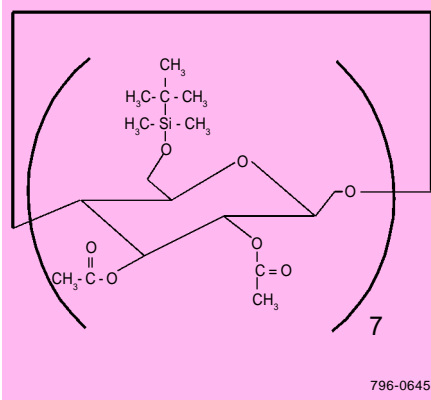
Our new β -DEX 225 columns separate many optical isomers that are poorly separated on other chiral columns.

Citronellal, β -citronellol and linalool are constituents of essential oils. Used principally in perfumery, they are of considerable commercial importance. These compounds also are chiral molecules, and the two configurations of each molecule must be monitored, because each can have a distinctive odor.

Our β -DEX™ 225 capillary columns are chiral GC columns designed for separating enantiomers and other isomers. These fused silica columns are coated with a solution of heptakis(2,3-di-O-acetyl-6-O-tert-butylidimethylsilyl)- β -cyclodextrin in SPB™-20 poly(20% diphenyl/80% dimethylsiloxane). β -DEX 225 columns provide good separation of many enantiomers that are not separable or poorly separable on other chiral columns.

Cyclodextrins are chiral cyclic oligomers composed of six or more D-glucose units bonded through α -(1-4) linkage. β -Cyclodextrin consists of seven glucose residues. The mouth of the torus-shaped cyclodextrin molecule has a larger circumference than the base. Secondary hydroxyl groups at C₂ and C₃ atoms of the glucose units are located around the mouth. Primary hydroxyl groups at C₆ atoms of the glucose units are located around the base.

Figure A. Heptakis (2,3-di-O-acetyl-6-O-tert-butylidimethylsilyl)- β -cyclodextrin



Heptakis(2,3-di-O-acetyl-6-O-tert-butylidimethylsilyl)- β -cyclodextrin is synthesized from native β -cyclodextrin. Primary hydroxyl groups are selectively substituted with tert-butylidimethylsilyloxy groups. Secondary hydroxyl groups are converted into acetyl groups (Figure A).

Figure B. Enantioseparation of Citronellal

Column: β -DEX 225, 30m x 0.25mm ID, 0.25 μ m film
 Cat. No.: 24348
 Oven: 95°C
 Carrier: helium, 20cm/sec
 Det: FID, 300°C
 Inj.: 1 μ L methylene chloride containing 1mg/mL mixed enantiomers, split 100:1, 220°C

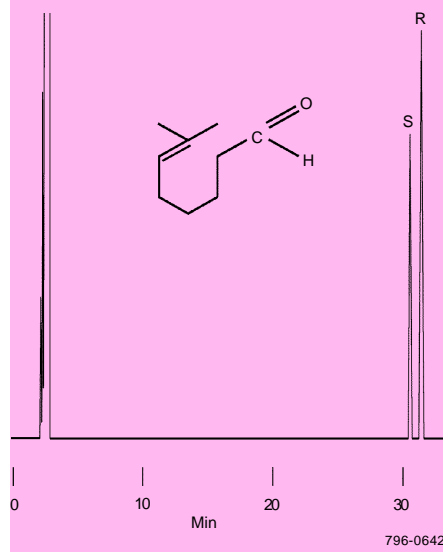


Table 1. Enantioseparation of Citronellal, Citronellol and Linalool*

	T, °C	k' ₁	α	R _s
Citronellal	95	13.8(S)	1.029	2.5
β -Citronellol	95	19.4(S)	1.019	1.5
Linalool	90	12.8(R)	1.029	2.0

*k'₁ = retention factor for first peak
 α = separation factor
 R_s = resolution factor

We analyzed citronellal isomers using a 30-meter x 0.25mm ID x 0.25 μ m film β -DEX 225 column (Figure B). Note the baseline separation. Enantioseparation factors for citronellal are compared with those for β -citronellol and linalool in Table 1.

Ordering Information:

Description	Cat. No.
β -DEX 225 Capillary Column 30m x 0.25mm ID, 0.25 μ m film	24348
Linalool Synthetic Standard 500mg, neat	48-2635

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

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