

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

Our β -DEX 225 column separates many optical isomers that are poorly separated on other chiral columns. Using this column, we observed complete separation of the isomers of three important fragrance compounds.

Key Words:

- essential oils • enantiomeric analysis • capillary GC

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.

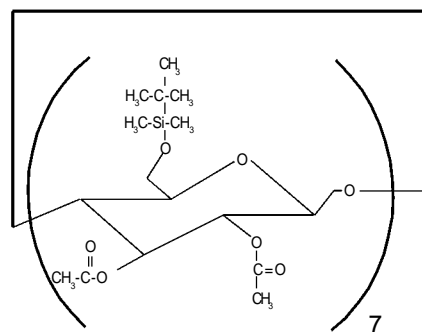
The β -DEX 225 capillary column is a chiral GC phase designed for separating enantiomers and other isomers. This fused silica column is coated with a solution of heptakis (2,3-di-O-acetyl-6-O-tert-butyl dimethylsilyl)- β -cyclodextrin in SPB™-20 poly(20% diphenyl/80% dimethylsiloxane). The β -DEX 225 column provides 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.

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

We analyzed isomers of citronellal, β -citronellol, and linalool using a 30-meter x 0.25mm ID x 0.25 μ m film β -DEX 225 column (Figures B-D). Note the complete baseline separation. Enantioseparation factors for the three compounds are shown in Table 1.

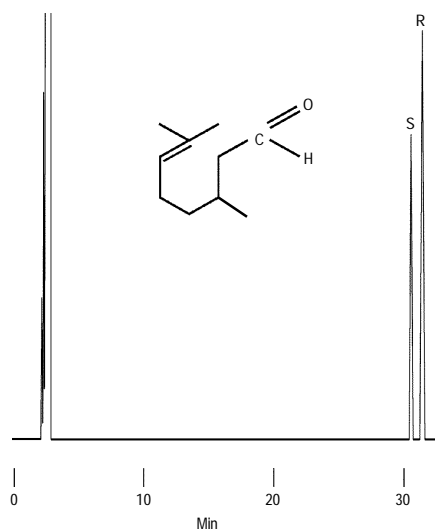
Figure A. Heptakis (2,3-di-O-acetyl-6-O-tert-butyl dimethylsilyl)- β -cyclodextrin



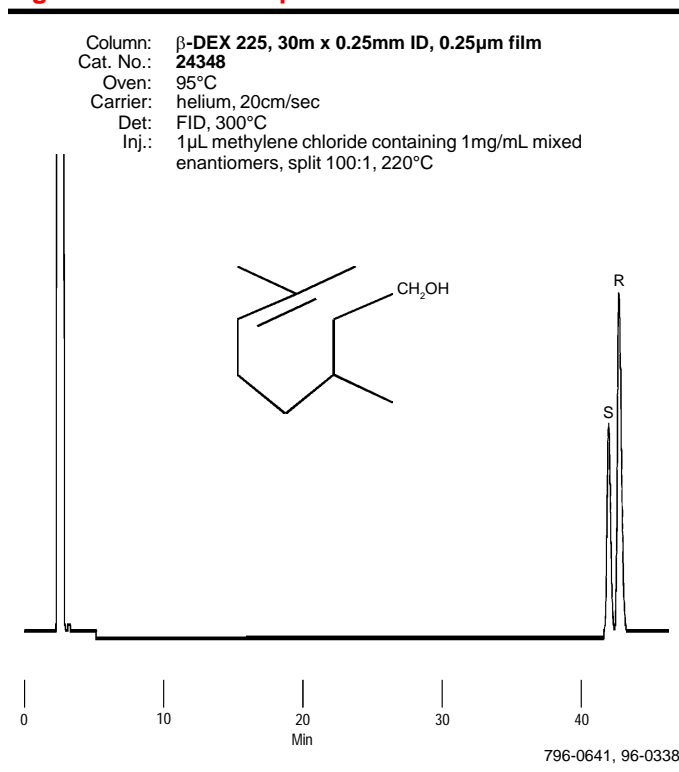
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Figure B. Enantioseparation of Citronellal

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



796-0642

Figure C. Enantioseparation of Citronellol**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

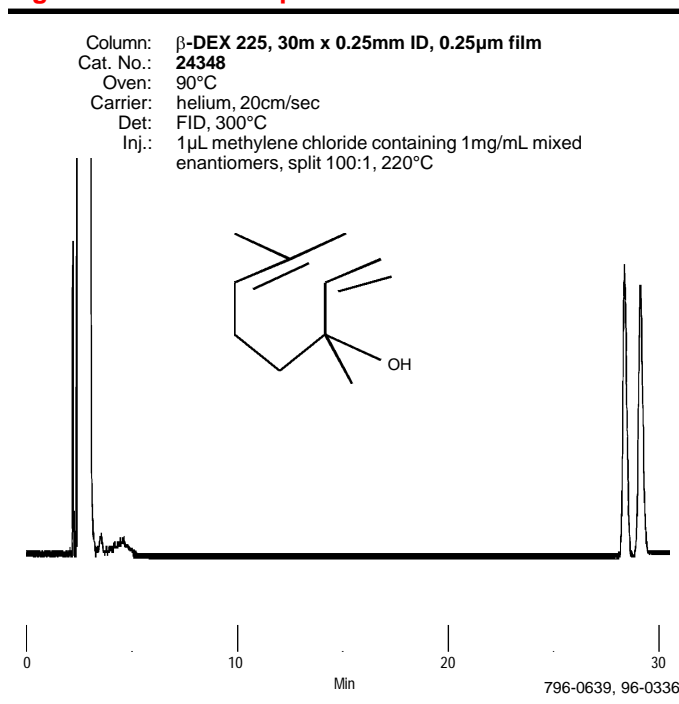
*k₁' = retention factor for first peak
 α = separation factor
 R_s = resolution factor

β -DEX columns provide unique selectivities for enantiomeric separations of other small molecules, including alcohols, aldehydes, esters, and ketones. For more information, contact your nearest Supelco representative.

Ordering Information:

Description	Cat. No.
β-DEX 225 Capillary Columns	
30m x 0.25mm ID, 0.25 μ m film	24348
30m x 0.32mm ID, 0.25 μ m film	24349
30m x 0.53mm ID, 0.50 μ m film	25442
Linalool Synthetic Standard	
500mg, neat	482635

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

Figure D. Enantioseparation of Linalool**Trademarks**

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