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Enantiomeric Analysis of 4-Methyloctanoic Acid by Chiral Gas Chromatography

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Chiral capillary GC columns commercially available from Supelco and other suppliers were evaluated for the direct enantioseparation of 4-methyloctanoic acid and its methyl ester. The best results were achieved on a γ -DEX 120 column.

4-Methyloctanoic acid, its homologs, and its esters are flavor components of foods, and also are pheromones. Free 4-methyloctanoic acid and its homologs contribute to the odor of cheese and meat (mutton, lamb, goat) (1-3). Male coconut rhinoceros beetles (*Oryctes rhinoceros* L.) produce 4-methyloctanoic acid and its ethyl ester as sex-specific compounds (4). Enantiomers of 4-methylalkanoic acids have different odors (5). Consequently, enantiomeric analysis and separation of 4-methylalkanoic acids is important.

Enantioseparations of 2-branched fatty acids and esters with an asymmetric center near the carboxy group on commercially available columns are well recognized (6-7). However, little is known about the enantioseparation of branched alkanic acids with the asymmetric center located farther from the carboxy group. Mosandl *et al* (8-9) separated enantiomers of 3- and 4-methylalkanoic acids and their methyl esters using heptakis-(2,3-di-O-methyl-6-O-tert-butyl-dimethylsilyl)- β -cyclodextrin and octakis(2,3-di-O-methyl-6-O-tert-butyl-dimethylsilyl)- γ -cyclodextrin as chiral selectors.

We evaluated a number of commercially available columns for the direct enantioseparation of underivatized 4-methyloctanoic acid and its methyl ester. Supelco's γ -DEX™ 120 column (30m x 0.25mm ID x 0.25 μ m film) offered the best resolution (Figures A and B).

Figure A. Enantioseparation of 4-Methyloctanoic Acid

Column: γ -DEX 120,
30m x 0.25mm ID x 0.25 μ m film
Cat. No.: 24307
Oven: 115°C
Carrier: helium, 20cm/sec
Det.: FID, 300°C
Inj.: 1 μ L methylene chloride containing
~1mg/mL racemate, split 100:1,
220°C

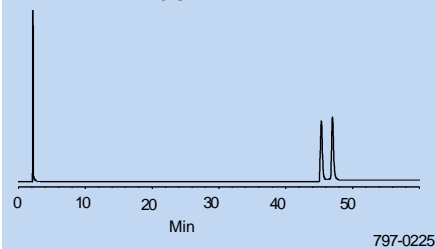
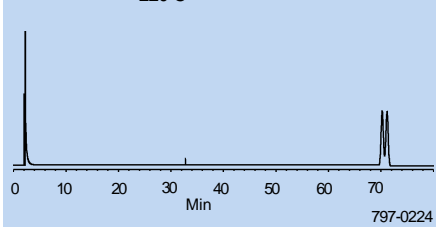


Figure B. Enantioseparation of Methyl 4-methyloctanoate

Column: γ -DEX 120,
30m x 0.25mm ID x 0.25 μ m film
Cat. No.: 24307
Oven: 75°C
Carrier: helium, 20cm/sec
Det.: FID, 300°C
Inj.: 1 μ L methylene chloride containing
~1mg/mL racemate, split 100:1,
220°C



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Only reference 6 is available from Supelco.

Ordering Information:

Description	Cat. No.
γ -DEX 120 Fused Silica Capillary Column 30m x 0.25mm ID, 0.25 μ m film	24307

The γ -DEX 120 capillary column is a member of Supelco's DEX family of chiral columns. DEX columns are an excellent choice for many chiral applications, including foods, essential oils, natural products, pharmaceuticals, and products of chemical syntheses. They exhibit low bleed, and can be used in a wide temperature range.

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