

Synthesis and application of novel β -CD derivatives as chiral stationary phases for supercritical fluid chromatography

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Synthesis and Application of Novel β -CD Derivatives as Chiral Stationary Phases for Supercritical Fluid Chromatography

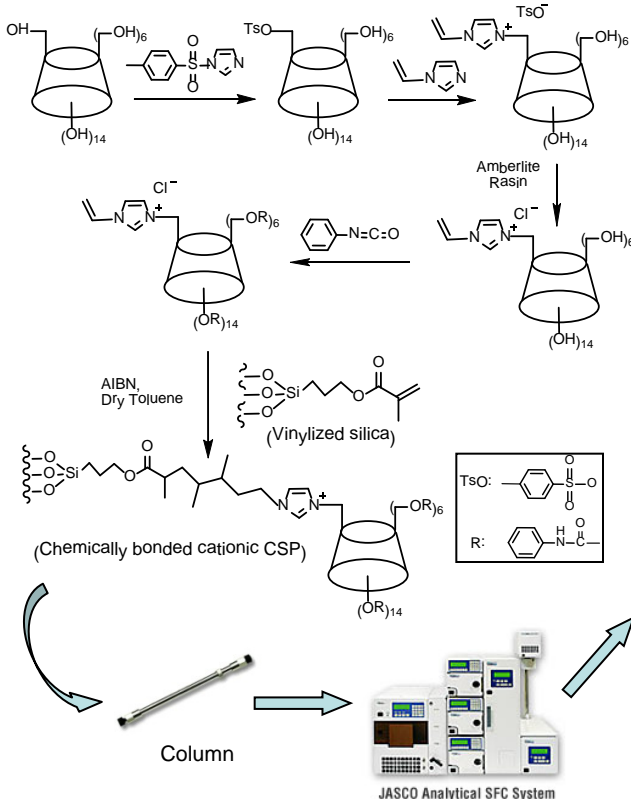
Introduction

Enantioseparation finds its greatest significance in pharmaceutical industry. Enantiomers of chiral drugs tend to show markedly different pharmacological activities. Currently, many commercial drugs are obtained as a mixture of two enantiomers through synthesis methods. In order to meet the demand of enantiometric pure drugs, chromatographic methods have been used extensively in analysis, separation and purification of chiral compounds.

Objective

- Synthesize novel chemically bonded cationic chiral stationary phase (CSP) based on β -cyclodextrin derivative.
- Investigate CSP's enantioseparation efficiency on Supercritical Fluid Chromatography (SFC) using a range of racemic samples.

Synthesis of CSP



SFC Enantioseparation Results

Racemate	k_1'	k_2'	α	R_s	Mobile Phase
Althiazide	21.06	25.23	1.20	1.43	a
Bendroflumethiazide	13.01	19.17	1.47	3.48	a
Naringenin	28.98	30.16	1.04	0.49	b
4'-Hydroxyflavanone	6.21	7.08	1.14	0.89	b
Hesperetin	23.78	25.65	1.08	0.72	b
Indapamide	29.98	32.79	1.09	0.83	b
4-Chromanol	8.47	9.77	1.15	0.93	c
7-Methoxyflavanone	8.33	10.13	1.22	1.42	c
6-Methoxyflavanone	4.85	5.86	1.21	1.00	c
2-phenoxypropionic acid	9.03	10.54	1.17	0.63	d
2-(3-chlorophenoxy)propionic acid	16.12	17.00	1.05	0.29	d
Dansyl-DL-Norvaline	7.61	8.33	1.09	0.71	e
Dansyl-DL- α -amino-n-butyric acid	9.41	9.72	1.03	0.35	e
Dansyl-DL-phenylalanine	12.80	14.06	1.10	0.71	e
Dansyl-DL-Norleucine	7.23	8.03	1.11	0.70	e
Dansyl-DL- α -aminocaprylic acid	6.49	7.35	1.13	0.86	e

SFC condition: temperature=40°C, BPR pressure=15MPa, flowrate=2ml/min, mobile phase: a) CO₂/Methanol=80/20; b) CO₂/Methanol=90/10; c) CO₂/Methanol=99/1; d) CO₂/IPA=70/30; e) CO₂/IPA (1%TEA, 1% acetic acid)=70/30.