An Excellent Gas Chromatographic Stationary Phase for Separation of Phenol and Cresol Isomers --Heptakis(2,6-di-O-pentyl-3-O-allyl)-β-Cyclodextrin

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Abstract: Heptakis(2,6-di-O-pentyl-3-O-allyl)-β-cyclodextrin as an excellent gas chromatographic stationary phase separating phenol and cresol isomers is described.

Keywords: Gas chromatographic stationary phase, phenol and cresol isomers, heptakis(2,6-di-O-pentyl-3-O-allyl)-β-cyclodextrin.

The analysis of phenols in environmental samples is of great importance because of their widespread occurrence in the environment. Especially it is hard to separate *p*-cresol and *m*-cresol[1,2]. In this paper, eight kinds of β-cyclodextrin derivatives were studied for the separation of phenol and cresol isomers. The result showed that the capillary column coated with heptakis(2,6-di-O-pentyl-3-O-allyl)-β-cyclodextrin was excellent for separating these compounds. The method is simple, quick, accurate and can be successfully used in environmental and industrial analysis.

Experimental

SP-3700 gas chromatograph(Beijing Analytical Instrument Factory, Beijing), equipped with flame ionization detector was used. The oven temperature was 250℃. Data collection was performed with a LDC/Milton Ron integrator. Nitrogen served as the carrier gas. β-CDs were synthesized by our studying group. Other reagents were of analytical grade.

Eight kinds of β-cyclodextrin derivatives were tested to find a suitable stationary phase for the separation of phenol and cresol isomers. The capillary columns coated with these stationary phases are listed in **Table I**

Results and discussion

The relative retention $\alpha_{\text{phenol/}o\text{-cresol}}$ and $\alpha_{p\text{-cresol/}m\text{-cresol}}$ were determined by using eight kinds of β-CDs. The results are listed in **Table II**. It shows that the columns coated with heptakis(2,6-di-O-pentyl)-β-CD (No.1) and heptakis(2,6-di-O-pentyl-3-O-

allyl)-β-CD(No.2) are the best stationary phases for the separation of the four phenols. The chromatograms are shown in **Figure 1**. It is apparent that the column coated with heptakis(2,6-di-O-pentyl-3-O-allyl)-β-cyclodextrin is an excellent column for the separation of phenol and cresol isomers.

Table I The columns and stationary phases

Col. No.	Stationary phase	Size (m×mm i.d.)	Velocity (cm/s)	Temp. (℃)	k	Efficiency (plates/m)	Compound tested
1	Heptakis(2,6-di-O-pentyl)-β-CD	10×0.25	30.86	120	10.37	370	dodecane
2	Heptakis(2,6-di-O-pentyl-3-O-allyl)-β-CD	20×0.25	19.61	120	3.10	2170	dodecane
3	Heptakis(3-O-allyl)-β-CD	10×0.25	18.73	120	4.40	1510	tetradecane
4	Heptakis(2,6-O-trifluoroacetyl-3-O-allyl)-β-CD	10×0.25	15.72	120	2.66	3400	undecane
5	Permethyl-β-CD	10×0.25	17.01	140	4.36	750	phenol
6	Bridgelinked-β-CD	17×0.25	17.71	120	5.14	800	dodecane
7	PEG-20M-β-CD	20×0.25	16.67	120	1.14	2510	heptanol
8	Permethyl -β-CD: allyl -β-CD 1:2 mixed	10×0.25	17.36	120	3.24	3040	dodecane

Table II. Relative retention of phenol/*o*-cresol and *p*-cresol/*m*-cresol

Col.No.	1	2	3	4	5	6	7	8
Temperature/℃	120	130	110	130	130	140	120	100
α 2/1	1.11	1.26	1.23	1.30	1.00	1.11	1.00	1.14
α 4/3	1.05	1.09	1.06	1.00	1.14	1.03	1.03	1.06

α 2/1= α phenol/*o*-cresol α 4/3= α *p*-cresol/*m*- cresol

Figure 1. Chromatograms of phenol and three cresol isomers

(a) Column No.1 column temperature:140℃, carrier gas linear velocity: 17.71cm/s
(b) Column No.2 column temperature:120℃, carrier gas linear velocity: 58.48cm/s
1: acetone, 2: phenol, 3: *o*-cresol, 4: *p*-cresol, 5: *m*-cresol

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