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Capillary Column Coated with Heptakis(2, 3, 6-tri-*O*-octyl)–β-cyclodextrin Using Sol-gel Technology

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Abstract: Heptakis(2, 3, 6-tri-*O*-octyl)-β-cyclodextrin was coated onto the fused silica capillary by sol-gel technology. Column efficiency and column selectivity were studied. The results indicated that high ability of separation was acquired . Positional isomers of aromatic compounds were well separated.

Keywords: Heptakis(2,3,6-tri-*O*-octyl)-β-cyclodextrin, capillary gas chromatography, sol-gel technology.

Sol-gel column technology effectively combines capillary surface treatment, deactivation, coating and stationary phase immobilization into one single step and provided efficient incorporation of organic components into the inorganic polymeric structures in solution under extraordinary mild thermal conditions. It was reported that stationary phase including hydroxy-terminated (PDMS[1] crown ether[2-3]) for gas chromatography was coated by sol-gel technology and high column efficiency was acquired. It had not been seen that β-CD derivatives for gas chromatographic stationary phase used in capillary column coated by sol-gel technology. In this paper heptakis(2, 3, 6-tri-*O*-octyl)-β -cyclodextrin derivative used as capillary gas chromatography stationary phase was described and the sol-gel column technology was applied. Column efficiency and selectivity were tested.

Experimental

Heptakis(2, 3, 6-tri-*O*-octyl)-β-cyclodextrin was provided by Institute of Chemistry, Chinese Academy of Sciences. Trifluoroacetic acid (TFA) was obtained from Beijing Chemical Factory. (Beijing, China). Methyltrimethoxysilane (MTMS) and methylene chloride were of analytical grade.

In the experiments, SP-3700 gas chromatography (Beijing Analytical Instrument Factory, Beijing) equipped with a capillary split injection system and flameionization detector (FID) was used. TL-9800 chromatography data station was used to acquire and process the data. Fused-silica capillary tubes (0.25 mm I.D.) was obtained from

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Yongnian Optical Fibre Factory (Hebei, China).

Column Preparation

To expose the maximum number of silianol groups on the silica surface, the fused silica capillary was first treated with 1.0 mol/L NaOH solution for 30 min and then washed with water for another 30 min. 0.1 mol/L HCl solution was used to neutralize the excess NaOH and the capillary was rinsed with water again. The columns were dried at about 120 ˚C for 2 h under a slow flow of nitrogen.

The sol solution for the heptakis(2, 3, 6-tri-*O*-octyl)-β-cyclodextrin was prepared as follows: 0.1869 g heptakis(2, 3, 6-tri-*O*-octyl)-β-cyclodextrin was dissolved in 0.8 mL methylene chloride . 0.1 mL of MTMS and 0.05 mL of TFA (containing 5 % water) were added to this solution, and the mixture was thoroughly vortexed. The clear top portion of the resulting sol solution was introduced into the fused silica capillary, which was pretreated in previous steps, using a nitrogen pressure of 0.6 MPa. The excess sol was expelled from the column under the same nitrogen pressure after allowing it to stay inside the capillary for 40 min. The capillary column was then purged with nitrogen for 10 min, followed by temperature-programmed heating from 50 ˚C to 180 ˚C at a rate of 1 ˚C /min under continued purging with nitrogen. The column was held under the final condition for 6 h.

Results and Discussion

Column performance

The chromatographic properties of three sol-gel heptekis(2, 3, 6-tri-*O*-octyl)-β-cyclo dextrin columns were demonstrated in **Table 1**.

Table 1 The chromatographic properties of the column coated by sol-gel technology

Stationary Phase	Column size(L×I.D.) (m×mm)	Retention factor* (*k*)	Temperature ˚C	Column efficiency (plate/m)
Heptakis(2,3,6-tri-*O*-octyl)-β-CD	10×0.25	2.25	140	3125
	10×0.25	2.19	140	2986
	10×0.25	2.23	140	3016

*tridecane was tested.

Table 2 Separation of disubstituted benzene isomers on the column coated by sol-gel method

Compound	Peak order	Temperature˚C	Retention factors k_1	k_2	k_3	Relative retention $\alpha_{2/1}$	$\alpha_{3/2}$
Cresol	*o, p, m*	140	1.34	1.79	2.32	1.29	1.29
Nitrotoluene	*o, m, p*	140	2.23	2.80	3.16	1.25	1.13
Dimethoxybenzene	*o, m, p*	140	1.56	1.91	2.78	1.22	1.45
Dichlorobenzene	*o, m, p*	140	2.30	.45	2.78	1.06	1.13
Bromotoluene	*o, m, p*	130	0.73	2.67	2.91	3.67	1.08
Xylene	*o, p, m*	90	2.56	2.83	3.31	1.11	1.17

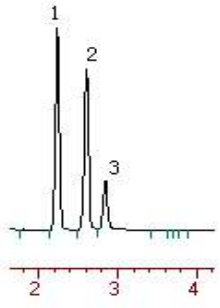
Separation Results

The coated column was used to separate. disubstituted benzene isomers. The separation results are illustrated in **Table 2**, **Figure 1**, **Figure 2**. **Table 2** showed good selectivity for positional isomers of aromatic compounds. **Figure 1** and **Figure 2** represented gas chromatographic separation of the cresol. As it can be seen in **Figure 1** and **Figure 2**, the symmetric peak shapes were obtained perfectly. It was important to examine environ- ment that cresol can be well separated either in water or in organic solvent.

Conclusion

The capillary gas chromatography column coated with heptakis(2, 3, 6-tri-*O*-octyl)-β-cyclodextrin by sol-gel method was obtained with high column efficiency and good selectivity for the separation of several mixtures of benzene isomers. This is a promising method for the coated column technology.

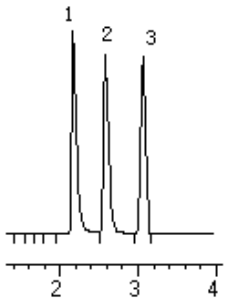
Figure 1 Chromatogram for mixture of cresol dissolved in water



T/min

Peak: 1. *o*-cresol 2. *p*-cresol 3. *m*-cresol
(10×0.25mm, I. D. column temperature 140 ˚C

Figure 2 Chromatogram for cresol in organic solvent



T/min

Peak: 1. *o*-cresol 2. *p*-cresol 3. *m*-cresol
(10×0.25mm, I. D) column temperature 140 ˚C

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