Chinese Chemical Letters Vol. 16, No. 2, pp 233-236, 2005 233
http://www.imm.ac.cn/journal/ccl.html

Perpentylated (2, 3, 6-Tri-*O*-pentyl)-β-cyclodextrin Used as Capillary Gas Chromatographic Stationary Phase Prepared by Sol-gel Technology

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Abstract: Capillary column preparation using perpentylated (2,3,6-tri-*O*-pentyl)-β-cyclodextrin as stationary phase by sol-gel technology with simplicity and rapidity is described. Multiple preparation steps in conventional column technology were avoided. The prepared columns exhibit satisfactory chromatographic performances and pronounced selectivity for a wide range of test solutes, and have been successfully used for the separation of nitrotoluene, dimethoxybenzene, alcohols, alkanes, dimethylphenol and cresol isomers.

Keywords: Sol-gel technology, perpentylated -β-cyclodextrin, gas chromatography.

Sol-gel column technology effectively combines with capillary surface treatment, deactivation, coating and stationary phase immobilization into one single step and provides efficient incorporation of organic components into the inorganic polymeric structures in solution under extremal mild thermal conditions. It has been used for the fused silica capillary column preparation of such hydroxy-terminated stationary phases as PDMS[1], PEG[2] and crown ether[3, 4]. Cyclodextrin derivatives have found their wide applications in the separation of some isomers and complex mixtures. Few recent studies have been made concerning the capillary column preparation using cyclodextrin derivatives as stationary phases by sol-gel method. In this paper, the capillary columns coated with perpentylated-β-cyclodextrin by the sol-gel technology was investigated.

Experimental

Perpentylated-β-CD was provided by china Agricultural University. Fused-silica capillary was obtained from Yong Nian Optical Fibre factory. Trifluoroacetic acid, methyltriethoxysilane (MTEOS)and methanol were purchased from Beijing Chemical Reagents Company (Beijing, China). The experiment was carried out on a SP-3700 gas chromatograph (Beijing Analytical Instrument Factory, Beijing, China).

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A fused silica capillary was first rinsed with methylene chloride followed by a brief nitrogen purge for 20 min, and then the column was successively rinsed with 1.0 mol/L NaOH solution, deionized water, 0.1 mol/L HCl solution for 30 min. Afterwards, the column was conditioned for 2 h at 120 ℃. The sol-gel coating solution was prepared as follows: 0.1108 g perpentylated-β-cyclodextrin was dissolved in 800μL methylene choride, then 120μL of MTEOS was added to this solution and mixed for 5 min. Then, 80μL TFA was added to the solution, and the mixture was thoroughly vortexed. The clear top portion of the resulting solution was introduced into the capillary using a nitrogen pressure of 0.4 MP. After a set of period of in-capillary residence time, the solution was expelled from the capillary under the same pressure and the capillary was purged with nitrogen for another 30 min, then heated from 40 ℃ to 180 ℃ at a rate of 1℃/min and holding at 180℃ for 6 h. While conditioning, the column was purged with nitrogen.

Results and Discussion

The column performances were determined and the results are listed in **Table 1.**

Table 1 The column performances of the prepared three columns

Column No	Stationary phase	Column dimension (m×mm i.d.)	Retention factor，k*	Column temperature (℃)	Column efficiency (plate/m)
1	Perpentylated-β-CD	10×0.25	2.82	140	2621
2	Perpentylated-β-CD	10×0.25	2.71	150	2874
3	Perpentylated-β-CD	10×0.25	2.67	160	2763

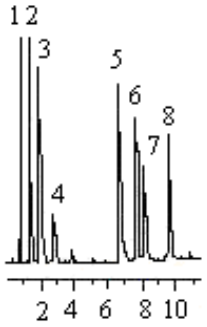
**n*-tridecane was used as the test reagent.

The sol-gel column showed excellent solvent stability, which was tested by comparing the solute retention times before and after rinsing the column with methylene chloride **(Table 2)**.

Table 2 Retention time repeatability for Grob mixture before and after rinsing the column with methylene chloride (n=3)

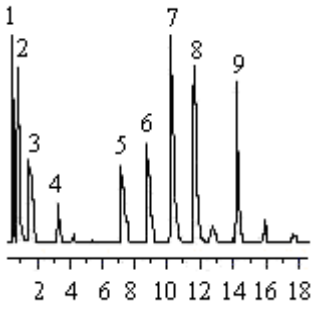
	Before rinsing (av)		After rinsing (av)	
Solute	Retention time(min)	RSD (%)	Retention time (min)	RSD (%)
acetone	0.420	0.11	0.410	0.24
n-butanol	1.060	0.26	1.020	0.31
n-nonane	1.170	0.14	1.080	0.26
decane	1.320	0.30	1.270	0.41
undecane	1.680	0.08	1.560	0.20
dodecane	2.010	0.27	1.980	0.32
1,4-butanediol	2.810	0.18	2.640	0.26
naphthalene	3.280	0.22	3.170	0.33
tridecane	4.250	0.41	4.160	0.44
2,6-xylidine	4.440	0.13	4.310	0.25
tetradecane	5.130	0.21	5.100	0.31
2,6-dimethylphenol	5.680	0.14	5.620	0.21

Figure 1 GC separation of alcohols



(1)methanol (2) ethanol (3) *n*-butanol, (4) *iso*-butanol (5) *n*-heptanol
(6) *n*-octanol (7) 1,4-butanediol (8) *n*-decanol

Figure 2 GC separation of alkanes



(1)hexane (2) heptane 3) octane (4) nonane (5) undecane
(6) dodecane (7) tridecane (8) tetradeca (9) hexadecane

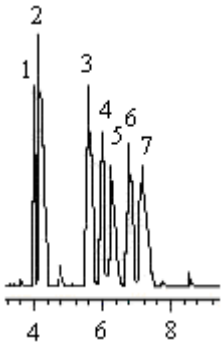
Deactivation is a critically important task in GC column technology. The deactivation quality of the sol-gel coated column was evaluated by using alcohols and alkanes as test solutes (shown in **Figure 1** and **Figure 2)**.

Figure 3 showed that dimethylphenol and cresol were separated and good peak shapes were also obtained. **Figure 4** and **Figure 5** showed the high efficiency separation of the benzene isomers.

Conclusion

Sol-gel technology offers great convenience for capillary column preparation. The capillary column coated with perpentylated (2,3,6-tri-*O*-pentyl)-β-CD by sol-gel technology provided satisfactory separations of compounds, including alcohols, alkanes and benzene isomers. This study demonstrates that the capillary column using perpentylated (2,3,6-tri-*O*-pentyl)-β-CD as stationary phase made by sol-gel technology is efficient.

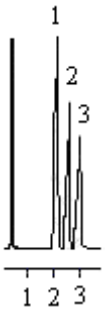
Figure 3 GC separation of cresol isomers and dimethylphenol



T /min

(1)2, 6-dimethylphenol (2)*p*-cresol (3)*m*-cresol (4)*o*-cresol (5)2, 5-dimethylphenol, (6)3, 5-dim-ethylcydphenol (7)3, 4-dimethylphenol

Figure 4 GC separation of nitrotoluene



T /min

(1) *o*-nitrotoluene
(2) *m*-nitrotoluene
(3) *p*-nitrotoluene

Figure 5 GC separation of dimethoxybenzene

T /min

(1) *m*-dimethoxybenzene
(2) *p*-dimethoxybenzene
(3) *o*- dimethoxybenzene

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Received 17 December, 2003