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Abstract A calix[6]arene bonded stationary phase was prepared by modifying silica gel with 3-aminopropyltriethoxysilane and then coupled with p-tert-butylcalix[6]arene ester. The chromatographic behavior of polycyclic aromatic hydrocarbons (PAHs) positional isomers of nitroanilines, phthalic esters, and some monofunctional compounds of benzene on this phase was described. In comparing with conventional reversed-phase C18 columns it showed predominant reversed-phase character and obvious priority for separation of positional isomers. The possible separation mechanism has been investigated.

Key words calix[6]arene, high performance liquid chromatography, stationary phase

1. Experimental
1.1. Reagents and Samples
Silica gel (200 300 mesh, 7 m2/g, from Anhui), bonded phases (Agela, Shanghai); aminopropyltriethoxysilane (CNT, Shanghai); 1,10-decanediol diacrylate (Tianjin Chemical Reagent); soda ash (analytical grade); sodium chloride; sodium nitroprusside (all from Guangdong Chemical Reagent); methanol (analytical grade); ethyl acetate (analytical grade); hydrochloric acid (analytical grade)

Zhong [6], Park [31]...
1.2 Waters Millennium III, Vaivo Bruker FT-IR etc.

1.3

10 g 10 mL 50 mL 0. g N2 24 h 6 mL Gutsche NaH THF

1.4

150 mm × 4.0 mm i.d. 40 mL 10 mL CCl4 50 MPa

2

2.1

6

Fig. 1 IR spectrum of 3-aminopropyl silica

Fig. 2 IR spectrum of the new p-tolyl-butylicl[6] arene-bonded silica gel stationary phase

Fig. 3 Retention values of a mixture of benzene[1] toluene[2] naphthalene[3] biphenyl and phenanthrene with different contents of methanol in mobile phase

2.2.2  

Fig. 4  Typical chromatogram of a mixture of PAHs
Mobile phase [methanol-water] 60:40 [v/v] flow rate 1.0 ml/min.
1. benzene 2. toluene 3. xylene 4. naphthalene 5. 2-methyl-

Fig. 5  Chromatogram of a mixture of phthalates
Conditions as in Fig. 4.
1. methyl phthalate 2. ethyl phthalate 3. n-buty phthalate 4. n-amyl phthalate 5. cyclohexyl phthalate.

2.2.3  

Fig. 6  The chromatogram of m-nitroaniline
Mobile phase [0.02 mol/L NaH₂PO₄ solution pH 3.80] methanol 70:30 [v/v] flow rate 1 ml/min.
1. m-nitroaniline 2. p-nitroaniline 3. o-nitroaniline.

Fig. 7  Chromatogram of toluene and its derivatives
Mobile phase [0.02 mol/L NaH₂PO₄ solution pH 3.5] acetonitrile 70:30 [v/v] flow rate 1 ml/min.
1. benzonic alcohol 2. benzaldehyde 3. benzoic acid 4. methyl-
phenolate 5. toluene.
本文考察了多种类型分析物在自制的对叔丁基杯芳烃键合固定相上的保留行为，首次在该固定相上分离了邻苯二甲酸酯类和同碳数的芳香化合物。发现该类型固定相具有显著的反相特征，分析物的保留主要受杯芳烃上缘对其包结识别的强弱所控制。今后的工作，应致力于完善这类固定相；对杯芳烃进行不对称修饰而用于分离及识别特殊的有机化合物。

参考文献：