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论文

新型表面聚合联萘胺型手性固定相的制备及评价

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摘要:

采用(*R*)-(+)-1,1'-联萘2,2'-二胺(DABN)作为手性单体, 以键合在3-氨基硅胶上的4,4'-偶氮-二(4-腈基)戊酸(ACVA)作为引发剂, 通过直接引发表面聚合反应, 制备了一种新型HPLC刷型手性固定相(CSP-a). 采用庚烷-醇-有机调节剂-有机酸为流动相, 在柱温30 ℃和UV 254 nm检测条件下实现了在CSP-a上联萘酚衍生物、3,5-二硝基苯甲酰-氨基酸甲酯和乙酯以及一种反式环氧乙烷衍生物对映体的色谱拆分, 考察了有机调节剂和有机酸对样品在CSP-a上拆分的影响. 结果表明, CSP-a对于*π*-酸及*π*-碱类化合物都有明显拆分效果, 三氟醋酸调节作用优于冰醋酸, 流动相中加入CH_2Cl_2和$CHCl_3$可增强保留, 改善分离度.

关键词: 手性固定相 1,1'-联萘2,2'-二胺 对映体拆分 联萘酚衍生物 DNB-氨基酸酯 环氧乙烷衍生物

Preparation of (*R*)-(+)-1,1'-Binaphthyl-2,2'-diamine Polymer as a Novel Chiral Stationary Phase by Surface-initiated Polymerization and Chromatographic Evaluation

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Abstract:

A novel brush-type chiral stationary phase(CSP-a) for HPLC was prepared by surface-initiated polymerization with (*R*)-(+)-1,1'-binaphthyl-2,2'-diamine as a chiral monomer and dichloride of 4,4'-azobis-4-cyanopentanoic acid bonded on the surface of silica gel as a radical initiator. The resolutions of the enantiomers of binaphthol derivatives, *N*-3,5-dinitrobenzoyl-*D*,*L*-amino acid methyl esters and isopropyl esters, and a *trans*-ethylene oxide derivative on CSP-a were carried out using heptane-alcohol-organic modifier-organic acid as the mobile phases under the detection conditions of the column temperature of 30 ℃ and UV wavelength of 254 nm. The effects of organic modifier and organic acid on the resolutions of the analytes on CSP-a were examined. The results show that CSP-a prepared was effective for the resolutions of the enantiomers of π-acid and π-base derivatives. Trifluoroacetic acid was a better organic modifier than acetic acid. Adding CH_2Cl_2 and $CHCl_3$ to the mobile phase could increase retention and resolution of the samples on CSP-a.

Keywords: Chiral stationary phase 1,1'-Binaphthyl-2,2'-diamine Enantiomeric resolution Binaphthol

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参考文献:

1. Zhong Q. Q., Han X. X., Armstrong W. D., *et al*.. J. Chromatogr. A[J], 2005, 1066: 55—70
2. Gasparrini F., Misiti D., Rompietti R., *et al*.. J. Chromatogr. A[J], 2005, 1064: 25—38
3. LIU Xu-Dong(刘旭东), DING Jin-Ying(丁金英), GAO Lian-Xun(高连勋). Chinese J. Chromatogr.(色谱)[J], 2005, 23(2): 146—151
4. Guido H. S., Jr . Robert P Cross. J. Am. Chem. Soc.[J], 1950, 72(5): 2299—2300
5. TAN Duan-Ming(谭端明), YAO Jun-Hua(姚骏骅). Acta Chimica Sinica(化学学报)[J], 2002, 60(10): 1854—1859
6. Brown J., Berry S., Murdoch R.. J. Org. Chem.[J], 1985, 50(22): 4345—4349
7. CHEN Guang-Ming(陈光明), CHEN Fu-Heng(陈馥衡), ZHOU Chang-Hai(周长海). Chem. J. Chinese Universities(高等学校化学学报)[J], 1995, 16(2): 216—219
8. SONG Rui-Juan(宋瑞娟), GUO Ying(郭瑛), FU Yu(富玉), *et al*.. Chem. J. Chinese Universities(高等学校化学学报)[J], 2007, 28(1): 56—58
9. XU Zhi-Guang(徐志广), GU Guo-Bang(古国榜), LIU Hai-Yang(刘海洋). Chemistry Online(化学通报)[J], 2007, 70(10): 782—786
10. Kyba E. P., Gokel G. W., Cram D. J., *et al*.. J. Org. Chem.[J], 1977, 42(26): 4173—4184
11. Lingenfelter D. S., Helgeson R. C., Cram D. J., *et al*.. J. Org. Chem.[J], 1981, 46(2): 393—406
12. Perrin S. R., Pirkle W. H.; Ed.: Ahuja S.. Chiral Separations by Liquid Chromatography, ACS Symposium Series 471[C], Washington DC: American Chemical Society, 1991: 43—66
13. YU Shi-Lin(于世林). Method and Application of High Performance Liquid Chromatography(高效液相色谱方法及应用)[M], Beijing: Chemical Industry Press, 58—91
14. Zief M., Crane L. J.. Chromatographic Chiral Separations[M], New York: Marcel Dekker Inc., 1988: 315

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2. 姚金水, 张献, 魏明星 .带有酰胺侧基的中空手性高分子微球的合成及其分离性能[J]. 高等学校化学学报, 2007,28(8): 1606-1608
3. 明永飞,赵亮,张红丽,师彦平,李永民 .戊唑醇对映体在新型纤维素键合手性固定相上的拆分[J]. 高等学校化学学报, 2007,28(2): 258-260

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