

研究论文

C18衍生的微米金颗粒的制备及其作为毛细管液相色谱和加压电色谱固定相的评价

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收稿日期 2008-12-18 修回日期 2009-2-18 网络版发布日期 2009-7-28 接受日期 2009-3-18

摘要 制备了用于色谱的微米纯金颗粒并键合上十八烷基(C18)官能团;对其进行了扫描电镜、红外光谱、元素分析、氮气吸附分析等表征。测得衍生的金颗粒的粒径、孔径以及比表面积分别为3.5 μm、5.0 nm、49.0 m²/g;红外光谱表明C18官能团已键合在金颗粒表面上;衍生后的金颗粒的含碳量为0.56%。通过电填充法得到长度为36 cm(固定相填充长度为19 cm)、内径为100 μm的毛细管色谱柱。利用极端pH的流动相(80%甲醇, pH 1.0以及pH 12.0)冲洗该色谱柱140 h,比较冲洗前后分析物的保留因子,以考察色谱柱的耐酸耐碱性能。结果表明,冲洗前后分析物的保留因子没有明显的变化,说明该色谱柱有良好的耐酸耐碱性。在毛细管液相色谱模式下,用该柱分离尿酸嘧啶、苯、萘、2-甲基萘、苊以评价色谱柱的一般性能;在碱性条件下分离咖啡因、茶碱、洛贝林以测定色谱柱分离碱性物质的能力。其分离结果表明,该色谱柱的柱效超过了50 000理论塔板/m,且色谱峰形较好。在毛细管加压电色谱模式下,施加+5 kV和~5 kV的电压均可以使苯甲酸和苯胺分离,但电场方向不同时,二者的出峰顺序不同。

关键词 [C18衍生微米金颗粒](#) [色谱固定相](#) [毛细管液相色谱](#) [加压毛细管电色谱](#)

Preparation and evaluation of octadecanethiol modified gold microspheres in capillary liquid chromatography and pressurized capillary electrochromatography as stationary phase

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Abstract

Gold microspheres modified with octadecanethiol as chromatographic stationary phase were prepared. The particles were characterized by the scanning electron micrograph (SEM), Fourier transform infrared spectroscopy (FT-IR), elemental analysis and nitrogen adsorption analysis. The average diameter, the surface area and the average pore diameter were 3.5 μm, 49.0 m²/g and 5.0 nm, respectively. The IR spectra demonstrated that C18 was bonded to the surface of gold microspheres with the carbon content of 0.56%. Using these microspheres as stationary phase, a 19 cm section of a total length of 36 cm capillary (100 μm i.d.) was packed electrokinetically, and the evaluations in capillary liquid chromatography and pressurized capillary electrochromatography were performed. The mobile phases (80% methanol) with extreme pH values (pH 1.0 or pH 12.0) were used to flush the column for 140 h. In order to investigate the chemical stability of the column, the retention factors before and after flushing were calculated and compared based on the experimental results. There was no remarkable deterioration on the retention factors after flushing, which demonstrated the column was stable under strong acidic and basic conditions. Five neutral compounds and three alkaline compounds were separated using capillary liquid chromatography to examine the retention behavior of the column, and over 50 000 theoretical plates per meter and acceptable symmetry peaks were obtained. The pressurized capillary electrochromatographic properties of the column were investigated using a separation of the mixture of aniline and benzoic acid, and the separation was obtained when a 5 kV positive or negative voltage was applied. The research work confirmed the feasibility of using the octadecanethiol modified gold microspheres as a novel stationary phase for capillary liquid chromatography and pressurized capillary electrochromatography.

Key words [octadecanethiol modified gold microspheres](#) [chromatographic stationary phase](#) [capillary liquid chromatography](#) [pressurized capillary electrochromatography](#)

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