

论文

基于PA6纳米纤维膜固相萃取-液相色谱法检测牛奶中的邻苯二甲酸酯

殷雪琰, 许茜^{1,2}, 吴淑燕¹, 王敏¹, 顾忠泽²

1. 东南大学公共卫生学院, 南京 210009;
2. 东南大学生物电子学国家重点实验室, 南京 210096

摘要:

采用静电纺丝法制备了聚酰胺6(PA6)纳米纤维膜, 结合固相萃取技术-液相色谱法(HPLC-UV)检测了市售牛奶样品中的6种邻苯二甲酸酯(PAEs)的含量. 对影响实验的各种因素, 如提取溶剂的种类及用量、超声时间、洗脱溶剂的种类及用量、纳米纤维膜的用量、pH及过样速度等进行了考察. 在最优化条件下, 邻苯二甲酸二甲酯(DMP)、邻苯二甲酸二乙酯(DEP)、邻苯二甲酸丁基苄酯(BBP)、邻苯二甲酸二丁酯(DBP)、邻苯二甲酸二(2-乙基己)酯(DEHP)和邻苯二甲酸二正辛酯(DOP)的检出限分别为0.02, 0.01, 0.05, 0.05, 0.10和0.25 ng/mL. 将该方法应用于不同品牌不同包装牛奶样品的检测, 只需2.5 mg PA6纳米纤维膜, 即可完全萃取样品中的PAEs, 相对标准偏差(RSD)小于5.82%, 回收率为93.40%~104.83%. 该方法测定牛奶中PAEs环境雌激素, 检出限低, 灵敏度高, 结果准确可靠, 重现性好.

关键词: 邻苯二甲酸酯; PA6纳米纤维膜; 高效液相色谱-紫外检测; 固相萃取; 牛奶

Determination of Phthalate Esters in Commercial Milk Samples Using PA6 Nanofibers Mat-based Solid-phase Extraction Coupled with Liquid Chromatography

YIN Xue-Yan¹, XU Qian^{1,2*}, WU Shu-Yan¹, WANG Min¹, GU Zhong-Ze²

1. School of Public Health, Southeast University, Nanjing 210009, China;
2. State Key Laboratory of Bioelectronics, Southeast University, Nanjing 210096, China

Abstract:

A new method for simultaneous determination of six phthalate esters in commercial milk was developed by the combination of latest proposed PA6 nanofibers mat-based solid-phase extraction with high-performance liquid chromatography-ultraviolet detector(HPLC-UV). Conditions for obtaining optimum extraction efficiency such as extraction solvents and its volume, extraction time, eluant and its volume, amount of adsorbent, pH, flow rate of sample were investigated and optimized in detail. Under optimized conditions, the detection limits found for dimethyl phthalate(DMP), diethyl phthalate(DEP), butylbenzyl phthalate(BBP), dibutyl phthalate(DBP), diethyl hexylphthalate(DEHP) and dioctyl phthalate(DOP) were 0.02, 0.01, 0.05, 0.05, 0.10 and 0.25 ng/mL, respectively. The proposed method was applied to the analysis different brands and packaging of milk samples. PAEs in commercial milk samples can be completely extracted by 2.5 mg PA6 nanofiber mat. The satisfactory spiked recoveries were obtained in the range of 93.40%—104.83% with relative standard deviation(RSD) of below 5.82% were achieved. The main advantages of the method are low detection limits, high sensitivity and selectivity and the results are accurate and reliable, reproducible.

Keywords: Phthalate ester; PA6 nanofibers mat; High-performance liquid chromatography ultraviolet detector(HPLC-UV); Solid-phase extraction; Milk

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通讯作者: 许茜, 女, 副教授, 主要从事食品和药品分析研究. E-mail: q_xu@163.com

作者简介:

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