

论文

吡啶离子液体双水相-高效液相色谱法同时测定牛奶中3种喹诺酮药物残留

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摘要:

建立了吡啶离子液体双水相-高效液相色谱同时测定牛奶中噁喹酸、萘啶酸及氟甲喹3种喹诺酮药物残留的方法. 牛奶样品经氯化钠和磷酸混合溶液提取后, 采用吡啶离子液体*N*-乙基-2-甲基吡啶溴化盐([EMPy]Br)和K₂HPO₄形成的双水相体系萃取富集, 以0.1%磷酸水溶液-乙腈为流动相, 梯度洗脱, 紫外检测. 该方法对噁喹酸、萘啶酸和氟甲喹测定的线性范围分别为0.3~15, 0.5~20和0.5~25 μg/mL, 相关系数(*r*)均大于0.9997, 3种药物的检出限在8~10 μg/kg之间. 对不同加标浓度的牛奶样品测定, 绝对回收率均在86.4%~94.8%范围内, 相对标准偏差为3.6%~8.3%. 该方法对牛奶中喹诺酮药物残留的检测具有简单、快速、环保和灵敏度高等优点.

关键词: 双水相; 吡啶离子液体; 高效液相色谱; 喹诺酮药物检测; 牛奶

Simultaneous Determination of Three Quinolones Residues in Milk by Pyridinium Ionic Liquid-based Aqueous Two-phase Systems Coupled with High Performance Liquid Chromatography

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Abstract:

A method was developed for the analysis of three quinolones(oxolinic acid, nalidix acid and flumequine) in milk by pyridinium ionic liquid-based aqueous two-phase systems coupled with high performance liquid chromatography. Milk samples were extracted with the mixed solutions consisting of sodium chloride and phosphoric acid. Then extracts were enriched by aqueous two-phase systems composed of the ionic liquid pyridine *N*-ethyl -2-methyl-pyridine bromide salt([EMPy]Br) and K₂HPO₄. The separation of the analytes was achieved via 0.1% phosphoric acid and acetonitrile with gradient elution under UV detection. The linear ranges are 0.3—15 μg/mL for oxolinic acid, 0.5—20 μg/mL for nalidixic acid and 0.5—25 μg/mL for flumequine. The correlation coefficient(*r*) is more than 0.9997. The limits of detection are 8—10 μg/kg. Based on detecting spiked quinolones concentrations in milk samples, the absolute recoveries ranged from 86.4% to 94.8% with relative standard deviation from 3.6% to 8.3%. The proposed method of determining quinolones residues in milk is simple, rapid, friendly to environment and high sensitive.

Keywords: Aqueous two-phase systems; Pyridinium ionic liquid; High performance liquid chromatography; Determination of quinolone; Milk

收稿日期 2009-03-09 修回日期 网络版发布日期

DOI:

基金项目:

浙江省科技计划(批准号: 2007C23071, 2007F70011)、浙江省自然科学基金(批准号: Y4080252)和嘉兴市科技计划(批准号: 2008AY2016)资助.

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