色谱 2009 27 (1): 34-38 ISSN: 1000-8713 CN: 42-1226/O

研究论文

高效液相色谱-串联质谱法测定蜂蜜中残留的19种喹诺酮类药物

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收稿日期 2008-7-24 修回日期 2008-9-30 网络版发布日期 2009-2-2 接受日期 2008-10-2

摘要 建立了高效液相色谱-电喷雾串联质谱联用测定蜂蜜中恩诺沙星、环丙沙星、诺氟沙星、氧氟沙星、双氟沙星、恶喹酸、氟甲喹、沙拉沙星、司帕沙星、丹诺沙星、氟罗沙星、马波沙星、伊诺沙星、奥比沙星、吡哌酸、培氟沙星、洛美沙星、西诺沙星和萘啶酸等19种喹诺酮类药物残留的方法。比较酸性溶液阳离子固相萃取(PCX柱)、近中性缓冲溶液反相固相萃取(HLB柱)和碱性溶液阴离子固相萃取(PAX柱)3种不同提取净化方法的提取效果，最终选择使用碱性溶液溶解蜂蜜样品，强阴离子固相萃取柱一步富集净化。以甲醇和0.1%甲酸溶液作为流动相，C18作为分析色谱柱，采用梯度洗脱方式进行液相色谱分离，选择离子反应监测模式检测19种喹诺酮类药物，内标方法定量。在1～100 μg/L范围内，19种喹诺酮类药物的线性相关系数均大于0.991。通过实际样品的添加回收试验，方法的定量限(S/N=10)为1.0 μg/kg，3个添加水平的回收率为71%～118%，相对标准偏差为4.2%～6.7%。

关键词 高效液相色谱-串联质谱 喹诺酮类药物 蜂蜜

Simultaneous determination of 19 quinolone residues in honey using high performance liquid chromatography-tandem mass spectrometry

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Abstract

A method for the simultaneous analysis of 19 quinolone residues, enrofloxacin, ciprofloxacin, norfloxacin, ofloxacin, difloxacin, oxolinic acid, flumequine, sarafloxacin, sparfloxacin, danofloxacin, fleroxacin, marbofloxacin, enofloxacin, orbifloxacin, pipemidic acid, pefloxacin, lomefloxacin, cinofloxacin, and nalidixic acid in honey was developed by high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS). In comparison of the three different extraction methods, i.e. acid solution coupled with cation-exchange solid-phase extraction cartridge (PCX), neutral buffer solution coupled with a reversed-phase extraction cartridge (HLB) and alkali solution coupled with a strong anion-exchange solid-phase extraction cartridge (PAX), the third method was finally used. The cartridge was then applied to accumulate and purify the target analytes from the sample matrices in one step. The HPLC separation was performed on a C18 column with a linear gradient elution program of methanol and 0.1% formic acid solution as the mobile phase. Selective reaction monitoring (SRM) was used for the selective detection of 19 quinolones. The linearity of all the 19 quinolones in the range from 1 μg/L to 100 μg/L had correlation coefficient greater than 0.991. In the detection of spiked samples, the detection limit of the method was 1.0 μg/kg for all the 19 quinolones, and the recoveries were 71%-118% with the relative standard deviations of 4.2%-6.7%. Internal standard calibration was used for the quantitative analysis.

Key words high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) quinolone drugs honey

DOI:

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