

研究论文

超高效液相色谱-电喷雾串联质谱法同时分析鸡肉中7种氟喹诺酮类药物残留

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摘要 建立了一种同时测定鸡肉中7种氟喹诺酮类药物残留的超高效液相色谱-电喷雾串联质谱确证分析方法(UPLC-ESI-MS/MS)。样品经酸化乙腈提取、正己烷脱脂和HLB固相萃取柱净化,采用ACQUITY UPLCTM BEH C18色谱柱(50 mm×2.1 mm, 1.7 μm)分离,以0.1%甲酸水溶液和乙腈作为流动相进行梯度洗脱,电喷雾质谱检测,正离子多反应监测模式进行定性和定量分析。7种药物在5~100 μg/kg范围内线性关系良好,相关系数(r^2)均大于0.99;以5, 25, 50 μg/kg 3个浓度水平进行添加回收试验,7种药物的平均回收率在79.2%~108.6%之间,相对标准偏差为4.2%~8.9%,方法的检出限(LOD)为0.2~1.4 μg/kg。方法重现性好、灵敏度高、分析时间短、确证能力强,适用于鸡肉中氟喹诺酮类药物多残留的确证检测。

关键词 [超高效液相色谱-电喷雾串联质谱](#) [固相萃取](#) [氟喹诺酮类药物](#) [鸡肉](#)

Simultaneous analysis of 7 fluoroquinolone residues in chicken muscle by ultra performance liquid chromatography-electrospray ionization tandem mass spectrometry

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Abstract

A confirmative method of ultra performance liquid chromatography coupled with electrospray ionization tandem mass spectrometry (UPLC-ESI-MS/MS) for the simultaneous determination of 7 fluoroquinolone residues (norfloxacin (NOR), ciprofloxacin (CIP), danofloxacin (DAN), enrofloxacin (ENR), ofloxacin (OFL), sarafloxacin (SAR) and marbofloxacin (MAR)) in chicken muscle was developed. The sample was extracted with acidified acetonitrile, defatted with n-hexane, and purified by an HLB solid phase extraction cartridge. The UPLC separation was performed on an ACQUITY UPLCTM BEH C18 column (50 mm×2.1 mm, 1.7 μm) utilizing a gradient elution program of acetonitrile and water (containing 0.1% formic acid) as the mobile phase. The identification and quantification were achieved by using ESI-MS/MS in positive ion mode and multiple reaction monitoring (MRM). The linear ranges were from 5 to 100 μg/kg with the correlation coefficients above 0.99 for all the 7 fluoroquinolones. The average recoveries spiked at the 3 concentration levels of 5, 25, 50 μg/kg ranged from 79.2% to 108.6% with the relative standard deviations of 4.2%~8.9%. The limits of detection were 0.2~1.4 μg/kg. The method was proved to be good reproducibility, high sensitivity, rapid, reliable and suitable for the simultaneous determination of multi-residues of fluoroquinolones in chicken muscle.

Key words [ultra performance liquid chromatography-electrospray ionization tandem mass spectrometry \(UPLC-ESI-MS/MS\)](#) [solid phase extraction \(SPE\)](#) [fluoroquinolones](#) [chicken muscle](#)

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