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研究论文

超高效液相色谱-串联质谱法同时测定鸡肝中残留的四环素类、磺胺类和喹诺酮类药物

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摘要 采用超高效液相色谱-串联质谱(UPLC-MS/MS)在正离子模式下通过多反应监测(MRM)方式同时测定了鸡肝脏组织中3种四环素类药物、10种磺胺类药物以及8种喹诺酮类药物的残留。试样由McIlvaine缓冲液-乙腈(体积比为1:4)、乙腈提取,合并上清液并用氮气吹干,用0.05 mol/L磷酸三乙胺缓冲液-乙腈(体积比为85:15)溶解残余物,经正己烷脱脂后,采用UPLC-MS/MS进行定性、定量分析。该方法对测定的21种药物的检出限均为2 μg/kg,定量限均为5 μg/kg。在添加水平分别为5,10和50 μg/kg时,21种药物的加标回收率为66.8%～128.5%,日内测定的相对标准偏差(RSD)为0.8%～20.2%,日间测定的RSD为2.2%～15.3%。该方法可作为动物源性食品中这3类药物残留检测的确证方法。

关键词 超高效液相色谱-串联质谱法 四环素 磺胺 喹诺酮 多残留 鸡肝

Simultaneous determination of tetracyclines, sulfonamides and quinolones residues in chicken livers by ultra performance liquid chromatography-tandem mass spectrometry

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Abstract

An analytical method for the simultaneous determination of 3 tetracyclines, 10 sulfonamides and 8 quinolones in chicken livers by ultra performance liquid chromatography coupled with tandem quadrupole mass spectrometry (UPLC-MS/MS) in positive ion mode with multiple reaction monitoring (MRM) has been developed and validated. A total of 2 g homogenized sample of chicken livers was placed in a 50 mL polypropylene tube, and 1 mL of McIlvaine buffer and 4 mL of acetonitrile were added. After stirring and centrifuging for 5 min at 4 000 r/min, the supernatant was collected and the remains was extracted by 5 mL acetonitrile. The supernatant was merged together and evaporated to dryness under a steam of nitrogen at 60 ℃. The residue was dissolved with 4 mL of phosphoric acid-triethylamine buffer-acetonitrile (85:15, v/v) and 4 mL of n-hexane. After stirring for 1 min and centrifuging for 5 min at 4 000 r/min, the under layer solution was analyzed using UPLC-MS/MS. The satisfactory recoveries (66.8%~128.5%) of all the veterinary drugs were demonstrated at spiked levels of 5, 10 and 50 μg/kg with the overall relative standard deviations (RSDs) for intra-day and inter-day of the 21 analytes less than 20.5%. The limit of detection (LOD) and the limit of quantification (LOQ) were 2 μg/kg and 5 μg/kg, respectively for each drug. This method has good stability, lower detection limits and can be used as a conclusive evidence method of these drug residues in chicken livers.

Key words ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) tetracyclines sulfonamides quinolones multiresidue chicken livers

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