色谱 » 2010, Vol. 28 » Issue (11) :1038-1042 DOI: 10.3724/SP.J.1123.2010.01038

超高效液相色谱-电喷雾串联质谱法测定动物饲料中的大环内酯类和林可胺类抗生素

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Simultaneous determination of macrolide and lincosamide antibiotics in animal feeds by ultra-performance liquid chromatography-electrospray ionization tandem mass spectrometry

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摘要 建立了动物饲料中竹桃霉素、红霉素、吉他霉素、交沙霉素、罗红霉素、泰乐菌素6种大环内酯和林可霉素、克林霉素2种林可胺抗生素的超高效液相色谱-电喷雾串联质谱(UPLC-ESI-MS/MS)检测方法。饲料样品采用甲醇提取,Oasis HLB固相萃取柱富集净化,Waters Acquity UPLC BEH C18色谱柱分离,以0.1%甲酸和乙腈为流动相进行梯度洗脱,流速为0.3 mL/min,正离子模式扫描,多反应监测模式检测,外标法定量。实验结果表明,8种药物在1～100 μg/L范围内具有良好的线性关系。在空白饲料样品中分别添加1、10和100 μg/kg 3个加标水平的8种药物,其平均回收率为68.6%～95.2%,相对标准偏差(RSD)为4.9%～11.8%,定量限均为1 μg/kg。结果表明,该方法简便快速、灵敏度高,适用于动物饲料中大环内酯类和林可胺类抗生素的同时检测。

关键词： 超高效液相色谱-串联质谱 大环内酯类抗生素 林可胺类抗生素 饲料

Abstract： A method for the simultaneous determination of six macrolide antibiotics (oleandomycin, erythromycin, kitasamycin, josamycin, roxithromycin and tylosin) and two lincosamide antibiotics (lincomycin and clindamycin) in animal feeds by ultra-performance liquid chromatography-electrospary ionization tandem mass spectrometry (UPLC-ESI-MS/MS) was developed. The macrolide and lincosamide antibiotics were extracted from the feeds with methanol followed by enrichment and clean-up with an Oasis HLB cartridge. The UPLC separation was performed on a Waters Acquity UPLC BEH C18 column by a gradient elution using 0.1% formic acid and acetonitrile as the mobile phase at a flow rate of 0.3 mL/min. The identification of eight drugs was carried out by positive electrospray ionization in multiple reaction monitoring (MRM) mode, and the quantification analysis was performed by external standard method. The calibration curves showed good linearity in the range of 1~100 μg/L. The average recoveries of the eight drugs from the feeds spiked at 1, 10 and 100 μg/kg levels were between 68.6% and 95.2%, and the relative standard deviations (RSD) were between 4.9% and 11.8%. The limits of quantification (LOQ) of the drugs in the feeds were 1 μg/kg. The method is simple, rapid, sensitive and suitable for the simultaneous determination of macrolide and lincosamide antibiotics in animal feeds.

Keywords： ultra-performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) macrolide antibiotics lincosamide antibiotics feeds

Received 2010-07-21; published 2010-11-25

Fund:

国家质量监督检验检疫总局科研项目(2008IK167)、质量检验公益性行业科研项目(200910145)和国家科技支撑计划项目(2009BADB9B02).

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引用本文:

严丽娟1*, 张峰2, 方恩华1, 郭彦妮1, 周昱1, 林立毅1, 储晓刚2.超高效液相色谱-电喷雾串联质谱法测定动物饲料中的大环内酯类和林可胺类抗生素[J] 色谱, 2010,V28(11): 1038-1042

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