

研究论文

高效液相色谱-串联质谱联用测定蜂王浆中的四种硝基呋喃类药物的代谢物

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摘要 报道了高效液相色谱-串联质谱联用测定蜂王浆中呋喃唑酮、呋喃西林、呋喃妥因和呋喃它酮4种硝基呋喃类药物的代谢物残留的方法。以三氯乙酸作为蜂王浆的蛋白质沉淀剂, 同时提供衍生化反应所需的酸性环境; 使用4种同位素内标, 补偿了衍生化效率、衍生后样品溶液的pH值及光照对定量结果所产生的影响, 极大地提高了定量的准确性。实验结果表明, 呋喃它酮代谢物的检测下限可以达到0.03 $\mu\text{g}/\text{kg}$, 其他3种硝基呋喃类药物的代谢物的检测下限可以达到0.05 $\mu\text{g}/\text{kg}$ (S/N大于5); 呋喃它酮代谢物的定量下限可以达到0.20 $\mu\text{g}/\text{kg}$, 其他3种硝基呋喃类药物的代谢物的定量下限可以达到0.25 $\mu\text{g}/\text{kg}$ (S/N大于10); 线性范围为0.4~20 ng/mL, 添加回收率为97.7%~104.8% (内标校正), 相对标准偏差 (RSD) 为2.7%~9.7%。

关键词 [高效液相色谱](#) [串联质谱](#) [同位素内标](#) [硝基呋喃类药物; 代谢物](#) [蜂王浆](#)

分类号

Determination of Metabolites of Nitrofurantoin Antibiotics in Royal Jelly by High Performance Liquid Chromatography-Tandem Mass Spectrometry

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Abstract

Nitrofurans are a group of widely used veterinary antibiotics that have been banned in many countries. This has generated a great deal of interests and demands for assay of nitrofurans in animal food products. To our knowledge, this is the first time that a high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) method has been successfully developed for simultaneously analyzing the metabolites of four nitrofurantoin antibiotics (furazolidone, furaltadone, nitrofurazone and nitrofurantoin) in royal jelly. Trichloroacetic acid solution was used both to precipitate proteins and to provide acidic reaction condition. Four isotope internal standards were utilized to improve the quantitative precision. The limits of detection (LODs) were 0.03 $\mu\text{g}/\text{kg}$ for the metabolite of furaltadone and 0.05 $\mu\text{g}/\text{kg}$ for the other three metabolites. The limits of quantitation were 0.20 $\mu\text{g}/\text{kg}$ for the metabolite of furaltadone and 0.25 $\mu\text{g}/\text{kg}$ for the other three metabolites. The linear range was 0.4-20 ng/mL for all the target analytes. The recoveries calibrated by internal standard were in the range of 97.7%-104.8% with the relative standard deviations (RSDs) of 2.7%-9.7%. It showed that this method could meet the requirements of national monitoring plan in China and the Minimum Required Performance Limits (MRPL) set by the European Union.

Key words

[high performance liquid chromatography \(HPLC\)](#) [tandem mass spectrometry \(MS/MS\)](#) [isotope internal standard](#) [nitrofurans](#) [metabolites](#) [royal jelly](#)

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