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#### 论文

枸杞果酒中赭曲霉毒素A液相质谱串联法测定

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摘要:

目的 建立测定枸杞果酒中赭曲霉毒素A(OTA)的高效液相色谱-串联质谱法。方法 样品用免疫亲和柱净化,以 Agilent Zorbax SB-C<sub>18</sub>色谱柱(2.1 mm×50 mm,3.5µm)分离;梯度洗脱流动相:水(含0.1%甲酸)和乙腈(含0.1%甲酸);流速:0.4 mL/min;进样量:10µL;电喷雾正离子多反应监测扫描模式(MRM)检测。结果 该方法的定量限为0.02 ng/mL,在0.02~20 ng/mL内线性关系良好,加样水平分别为0.2、2、20 ng/mL时,其回收率分别为71.0%、83.7%、86.0%,相对标准偏差(RSD)分别为8.0%、9.0%、6.1%;在随机购买的12份市售枸杞果酒中测得OTA的含量为0.03~0.18 ng/mL,检出率为50.0%。结论 赭曲霉毒素A在样品中的测定结果均低于欧盟限量;该方法简便、灵敏、准确,可应用于枸杞果酒中赭曲霉毒素A的测定。

关键词: 高效液相色谱-串联质谱法 赭曲霉毒素A(OTA) 枸杞果酒

Determination of ochratoxin A in wolfberry fruit wine with high performance liquid chromatography-tandem mass spectrometry

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Abstract:

Objective To develop a sensitive and accurate method of high performance liquid chromatography-tandem mass spectrometry for the quantification of ochratxoin A(OTA)in wolfberry fruit wine. Methods Samples were cleaned up by immunoaffinity column(IAC)and then separated on Agilent Zorbax SB-C<sub>18</sub> column(2.1 mm×50 mm, 3.5µm). Gradient mobile phase was prepared with water (containing 0.1% formic acid) and acetonitrile (containing 0.1% formic acid). Flow rate was set to 0.4 mL/min and the injection volume was 10µL. Liquid chromatography-electrospray ionization-MS/MS(LC-ESI-MS/MS)in multiple reaction monitoring (MRM) was operated under positive electrospray ionization mode. Results The limit of quantification (LOQ) of the method was 0.02 ng/mL. The calibration curves showed a good linearity in the range of 0.02-20 ng/mL. The spiked levels were set at 0.2,2,and 20 ng/mL with the average recoveries of 71.0%,83.7%, and 86.0% and the relative standard deviations (RSDs) of 8.0%,9.0%, and 6.1%, respectively. OTA concentrations in the 12 samples investigated were between 0.03 ng/mL and 0.18 ng/mL, and the detection rate was 50.0%. Conclusion OTA concentrations in the samples investigated were all below the maximum allowable limit established by the European Community. The method was proved to be simple, rapid, sensitive, and suitable for the quantification of OTA in wolfberry fruit wine.

Keywords: high performance liquid chromatography-tandem mass spectrometry ochratoxin A wolfberry fruit wine

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