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高效液相色谱-串联四级杆质谱法测定鱼体中河豚毒素

Determination of tetrodotoxin in fish by liquid chromatography tandem mass spectrometry



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中文摘要:

建立高效液相色谱-串联四级杆质谱联用仪多反应监测技术(MRM)测定鱼体中的河豚毒素(TTX)含量的方法。方法 海鱼样品用80%酸化甲醇水溶液(含0.1%乙酸)超声提取,二氯甲烷脱脂净化,超滤管离心,取滤液过膜后进行分析。目标物TTX以pH=3.5的乙腈-0.06%甲酸铵溶液(65:35,V/V)为流动相,经HILIC色谱柱(100 mm×2.1 mm,3 μm)等梯度洗脱分离,采用电喷雾离子源,选择质谱多反应监测正离子模式,以保留时间和河豚毒素的二级质谱特征碎片离子予以双定性确证,基质标准曲线校正,外标法定量。结果 河豚毒素与杂质能良好分离,在0.10~2.0 mg/kg范围内线性关系良好($r=0.997$),方法检出限为0.012 mg/kg,定量限为0.041 mg/kg,加标回收率为75.7%~108.1%,相对标准偏差为1.4%~5.1%。应用该方法对20份实际样品进行检测,均未检出TTX。结论 本方法准确、快捷、简便,可应用于食物中毒因子识别与鉴定。

Abstract:

To establish a method of multiple reaction monitoring technology (MRM) by high performance liquid chromatography tandem mass spectrometry (HPLC-MS/MS) to detect tetrodotoxin (TTX) content in fish. Methods Fish samples were ultrasonic extracted by 80% acidified methanol solution (containing 0.1% acetic acid), and purified by dichloromethane, centrifuged by ultrafiltration, and analyzed after membrane filtration. TTX was separated on a HILIC column, with acetonitrile-0.06% ammonium formate solution (V/V=65:35, pH=3.5) as the mobile phase for equal gradient elution. Multiple reaction monitoring (MRM) in positive ion mode was used, and qualitative confirmation was achieved from retention time and secondary mass characteristic ions of TTX. The matrix-matched external standard calibration curves were used for quantitative analysis. Results Tetrodotoxin and impurity could be well separated, and it showed good linearity in the range of 0.10-2.0 mg/kg ($r=0.997$). The detection limit of the method was 0.012 mg/kg, the limit of quantification was 0.041 mg/kg, the average recovery was 75.7%-108.1%, and the precision was 1.4%-5.1%. The method was applied to 20 fishes, while TTX was not detected. Conclusion The method was accurate, efficient and simple, and could be applied to the recognition and identification of factors of food poisoning.

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