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固相萃取-超高效液相色谱-串联质谱法检测粮食及其制品中的玉米赤霉烯酮

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Determination of zearalenone and related mycotoxins in grain phase extraction coupled with ultra performance liquid chrom spectrometry

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摘要 建立了粮食及其制品中6种玉米赤霉烯酮类物质(α-玉米赤霉醇、β-玉米赤霉醇、α-玉米赤霉烯醇、β-玉米赤霉烯醇、玉米赤霉烯酮)的超高效液相色谱-串联质谱(UPLC-MS/MS)检测方法。样品用84%(体积分数)乙腈水溶液提取,通过ENVI-Carb石墨化炭黑萃取柱进行富集净化,用6 mL二氯甲烷-甲醇(7:3, v/v)溶液洗脱,采用UPLC-MS/MS进行测定。在ACQUITY UPLCTM BEH C18反梯度洗脱,流动相为水和乙腈;质谱采集模式为电喷雾负离子多反应监测模式。以α-玉米赤霉烯酮-d4为内标,6种目标物的线性范围为μg/L,相关系数(R2)大于0.99,检出限为0.1～0.2 μg/kg, 3个不同水平的加标平均回收率为79.9%～104.0%,相对标准偏差不大于用该方法对北京市的粮食及相关产品进行了分析,结果发现玉米赤霉烯酮的检出率最高,含量为0.42～220.7 μg/kg;此外还检出了α霉烯醇。该方法具有操作简单、灵敏度高、重现性好等特点,符合食品样品中痕量污染物的检测要求。

关键词： 固相萃取 超高效液相色谱-串联质谱 玉米赤霉烯酮 真菌毒素 粮食 粮食制品

Abstract： A method was established for the determination of 6 zearalenonic compounds (α-zearalanol, β-zea zearalenol, β-zearalenol, zearalanone and zearalenone) in grain and its products based on solid-phase extrac coupled with ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS). The sample extracted by 84% (v/v) acetonitrile-water solution and further purified by an ENVI-Carb graphite carbon black cartridge, which was eluted by 6 mL dichloromethane-methanol (7:3, v/v) solution. The target compounds we by UPLC-MS/MS. The chromatographic separation was performed on an ACQUITY UPLCTM BEH C18 column wit elution using acetonitrile and water as mobile phases. The mass spectrometric acquisitions were carried out b multiple reaction monitoring (MRM) in electrospray negative ionization mode. The good linearities (R2＞0.99) w achieved for the 6 compounds over the range of 0.1-50 μg/L based on the internal standard calibration of α-z d4. The detection limits of the method were 0.1-0.2 μg/kg. The mean recoveries of the 6 target compounds (s three concentration levels) ranged from 79.9% to 104.0%, with the relative standard deviations (RSDs) no m 10%. It has been applied in the analysis of grain and related products taken from Beijing. As a result, zearale presented a highest detectable frequency, with a concentration range of 0.42-220.7 μg/kg. In addition, α-zea β-zearalenol were also detected in this survey. This proposed method is simple, sensitive, reproducible, and c with the regulations for the determination of trace contaminants residues in food matrices.

Keywords： solid-phase extraction (SPE) ultra performance liquid chromatography-tandem mass spectrome MS/MS) zearalenone mycotoxin grain grain products

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