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研究论文

免疫亲和柱净化-柱后光化学衍生-高效液相色谱法同时检测粮谷中的黄曲霉毒素、玉米赤霉烯酮和赭曲霉毒素A

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摘要 建立了同时检测粮谷中黄曲霉毒素(B1、B2、G1和G2)、玉米赤霉烯酮和赭曲霉毒素A的免疫亲和柱净化-柱后光化学衍生-高效液相色谱方法。样品经过甲醇-水(体积比为80∶20)提取,通过免疫亲和柱富集和净化,采用Waters Nova-Pak 色谱柱(3.9 mm i.d.×150 mm,4 μm),以甲醇、乙腈和1%的磷酸溶液为流动相,梯度洗脱,柱后光化学衍生、改变波长荧光检测。黄曲霉毒素(B1、B2、G1和G2)、玉米赤霉烯酮和赭曲霉毒素A检出限分别为0.24,4.0和0.5 μg/kg,标准曲线的线性范围分别为0.24~6.0,4.0~100.0和0.5~40.0 μg/L;在小麦、玉米、黑麦样品中,平均加标回收率为70.8%~94.0%,相对标准偏差为2.79%~9.38%。

关键词 免疫亲和柱 光化学衍生 高效液相色谱 真菌毒素 粮谷

分类号

Simultaneous Determination of Aflatoxins, Zearalenone and Ochratoxin A in Cereal Grains by Immunoaffinity Column and High Performance Liquid Chromatography Coupled with Post-Column Photochemical Derivatization

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Abstract

A method was developed for the simultaneous determination of aflatoxins (AFs) (B1, B2, G1 and G2), zearalenone (ZEA) and ochratoxin A (OTA) in cereal grains by high performance liquid chromatography （HPLC） with fluorescence detection after immunoaffinity column clean-up and post-column derivatization. Cereal grain sample was extracted with methanol-water (80∶ 20, v/v). The extract was purified by immunoaffinity column and the toxins were separated by reversed-phase HPLC, and quantified with fluorescence detection after photochemical derivatization. The separation was performed on a Nova-Pak column (3.9 mm i.d.×150 mm, 4 μm, Waters) with a linear gradient of methanol-acetonitrile-1%phosphoric acid mixture. The calibration curves for mycotoxins were made in the concentration range of 0.24-6.0 for AFs (B1,B2,G1 and G2), 4.0-100.0 for ZEA and 0.5-40.0 μg/L for OTA. Recoveries of the different cereal grains (wheat, rice, rye) spiked with mycotoxins at levels ranged from 0.24-1.0 μg/kg for AFs (B1, B2, G1 and G2), 4.0-16.0 μg/kg for ZEA and 0.5-3.0 μg/kg for OTA were from 70.8% to 94.0% and relative standard deviations were between 2.79% and 9.38%. The limits of detection were 0.24 μg/kg for AFs (B1, B2, G1 and G2), 0.5 μg/kg for OTA and 4.0 μg/kg for ZEA.

Key words immunoaffinity column photochemical derivatization high performance liquid chromatography (HPLC) mycotoxin cereal grain

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