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研究论文

免疫亲和柱净化/柱前衍生化-高效液相色谱荧光检测法测定粮谷中的T-2毒素

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摘要 建立了免疫亲和柱净化/柱前衍生化-高效液相色谱荧光检测器测定粮谷中T-2毒素含量的方法。样品经甲醇-水(体积比为80∶20)混合溶剂提取,通过免疫亲和柱(IAC)净化,以氰酸蒽(1-AN)为衍生化试剂、4-二甲基氨基吡啶(DMAP)为催化剂进行衍生,以ZORBAX Eclipse XDB-C18 柱为分离柱,乙腈-水(体积比为80∶20)为流动相进行高效液相色谱分离及荧光检测,荧光检测的激发波长为381 nm,发射波长为470 nm。T-2毒素的质量浓度为0.01~1.5 mg/L时与峰高呈良好的线性,相关系数为0.9985。在0.01~1.5 μg/g添加水平下,回收率为79.7%~94.5%,相对标准偏差小于7%;检出限(S/N=3)为0.01 μg/g。该方法净化效果好,灵敏度高,操作简便快速。

关键词 免疫亲和柱 柱前衍生化 高效液相色谱法;荧光检测;T-2毒素;粮谷

分类号

Determination of T-2 Toxin in Cereal Grains by High Performance Liquid Chromatography with Fluorescence Detection after Immunoaffinity Column Clean-Up and Precolumn Derivatization

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Abstract

A method has been developed for the determination of T-2 toxin in cereal grains by high performance liquid chromatography with fluorescence detection after immunoaffinity column clean-up and precolumn derivatization.The derivatization reaction was used to develop a sensitive, reproducible and accurate method for the determination of T-2 toxin in wheat, corn, barley and rice. T-2 toxin was extracted with methanol-water (80∶ 20, v/v), purified by immunoaffinity columns containing antibodies specific for T-2 toxin, and quantified by reversed-phase high performance liquid chromatography with fluorescence detection (excitation wavelength, 381 nm; emission wavelength, 470 nm) after derivatization with 1-anthroylnitrile （1-AN） and 4-dimethylaminopyridine (DMAP). ZORBAX Eclipse XDB-C18 column and mobile phase of acetonitrile-water (80∶ 20, v/v) were used for the analysis. Recoveries from the different cereals spiked with T-2 toxin at levels ranging from 0.01 to 1.5 μg/g were from 79.7% to 94.5%, the relative standard deviations were lower than 7% and the limit of detection was 0.01 μg/g based on a signal-to-noise ratio of 3∶ 1.

Key words immunoaffinity column precolumn derivatization high performance liquid chromatography （HPLC） fluorescence detection T-2 toxin cereal grains

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