

研究论文

固相萃取-在线凝胶渗透色谱-气相色谱/质谱法测定松子仁中的28种有机氯农药和拟除虫菊酯农药

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摘要 建立了松子仁中28种有机氯农药和拟除虫菊酯农药多残留的在线凝胶渗透色谱-气相色谱/质谱(GPC-GC/MS)分析方法。样品以乙腈-水(体积比为4:1)为提取剂高速匀浆提取,提取液经Aluminium-N固相萃取柱净化,除去样品中大部分的脂肪和甾醇等干扰基质,再经在线GPC进一步除去样液中的色素和脂肪等大分子干扰物质,有效地降低了样品复杂基质带来的背景干扰。加标水平为0.05 mg/kg时,大部分农药的回收率为70%~120%,相对标准偏差小于15%。28种农药的检出限为0.002~0.05 mg/kg。采用外标法定量,方法的线性关系和回收率结果均令人满意。实验证明,该方法是一种快速、准确、灵敏度高的同时检测松子仁中农药多残留的检测方法。

关键词 [固相萃取](#) [在线凝胶渗透色谱-气相色谱/质谱](#) [有机氯农药](#) [拟除虫菊酯农药](#) [松子仁](#)

Determination of 28 organochlorine and pyrethroid pesticides in pine nuts using solid-phase extraction and on-line gel permeation chromatography-gas chromatography/mass spectrometry

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Abstract

An analytical method has been developed for the determination of 28 organochlorine pesticides and pyrethroid pesticides in pine nuts. The sample was extracted With acetonitrile-water (4:1, v/v) as the extraction solution by means of high-speed homogenization. The crude extract was purified by an Aluminium-N solid phase extraction column to remove most of the fat and sterols in the sample, then on-line gel permeation chromatography-gas chromatography/mass spectrometry (GPC-GC/MS) analysis was performed. The recoveries for the most of pesticides in the sample spiked with the standards of 0.05 mg/kg were 70%-120%, and the relative standard deviations were less than 15%. The limits of detection of 28 organochlorine pesticides and pyrethroid pesticides were 0.002-0.05 mg/kg. The linear relationship and the recovery results were satisfactory. The method is rapid, accurate, highly sensitive, and can be used for the simultaneous determination of pesticide residues in pine nuts.

Key words [solid-phase extraction \(SPE\)](#) [on-line gel permeation chromatography-gas chromatography/mass spectrometry \(GPC-GC/MS\)](#) [organochlorine pesticides](#) [pyrethroid pesticides](#) [pine nuts](#)

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