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气相色谱-串联质谱技术分析烟草中49种农药残留

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Determination of 49 pesticide residues in tobacco by gas chromatography-tandem mass spectrometry

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摘要 采用改进的QuEChERS(quick, easy, cheap, effective, rugged and safe)前处理法,结合气相色谱-串联质谱(GC-MS/MS)技术建立了检测烟草中49种农药残留的分析方法。样品用含0.1%乙酸的乙腈溶液提取,提取液被氮吹至干后,残渣用乙腈-乙酸乙酯(1:1, v/v)溶液溶解,溶解液经N-丙基乙二胺(PSA)吸附剂、无水MgSO4、C18吸附剂净化后,直接进行GC-MS/MS测定,内标法定量。实验结果表明,49种农药在低质量浓度(0.05 μg/L)的加标水平下的平均加标回收率为60.4%～104.8%,高质量浓度(5 μg/L)的平均加标回收率为70%～115%,相对标准偏差均小于15%;其中16种农药的方法检出限(LOD)分别为0.01～0.03 μg/kg,其余33种农药的LOD均小于0.01 μg/kg;相关系数都大于或等于0.991。该方法样品前处理简单、分析时间短、灵敏度和精密度均符合农药多残留痕量检测技术的要求,适用于烟草中多种农药残留的检测。

关键词： 气相色谱-串联质谱 农药残留 烟草

Abstract： A method was developed for rapid determination of 49 pesticide residues in tobacco based on gas chromatography-tandem mass spectrometry (GC-MS/MS). Tobacco was extracted with 0.1% acetic acid-acetonitrile solution. The supernatant was quantitatively transferred and dried with nitrogen. The concentrated extract was dissolved with acetonitrile-ethyl acetate(1:1, v/v) solution and cleaned up by primary secondary amine (PSA) sorbents, MgSO4 and C18 sorbents, then determined by GC-MS/MS with mirex as internal standard. The ranges of spiked recoveries of 49 pesticides at 0.05 μg/L and 5 μg/L were 60.4%~104.8% and 70%~115%, respectively. The relative standard deviations were below 15%. The detection limits of 16 pesticides were 0.01~0.03 μg/kg and those of the other 33 pesticides were less than 0.01 μg/kg; the correlation coefficients were larger than 0.991. This method is simple, rapid and characterized with acceptable sensitivity and accuracy to meet the requirements for the analysis of multiple pesticide residues in tobacco.

Keywords： gas chromatography-tandem mass spectrometry (GC-MS/MS) pesticide residues tobacco

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