

程序升温大体积进样气相色谱-负化学离子源质谱法测定白菜和苹果中103种农药残留

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Simultaneous determination of 103 pesticide residues in cabbages and apples using programmable temperature vaporizer-based large volume injection by gas chromatography-negative chemical ionization mass spectrometry

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摘要 采用程序升温大体积进样(PTV-LVI)和负化学离子源(NCI)技术建立了白菜和苹果中103种农药残留分析的气相色谱-质谱检测方法。PTV-LVI参数优化为: 初始温度45 ℃、分流排空流量20 mL/min、吹扫时间1 min和溶剂挥发温度60 ℃。样品采用QuEChERS方法进行快速处理, 在NCI方式下进样10 μL,用基质标准溶液进行定量,结果表明103种农药的方法检出限均低于5 μg/kg。在5 μg/kg和10 μg/kg添加水平下,白菜和苹果中农药的平均回收率为58.5%~113.2%,相对标准偏差为3.3%~14.5%。该方法样品处理简单快速,相比普通分流/不分流进样和电子轰击电离气相色谱-质谱法,其灵敏度和选择性明显提高,适用于日常检测工作。

关键词: 程序升温 大体积进样 负化学电离质谱 气相色谱-质谱 多农药残留 白菜 苹果

Abstract: A gas chromatography-mass spectrometric (GC-MS) method has been established for the simultaneous determination of 103 pesticide residues in cabbages and apples using programmable temperature vaporizer-based large volume injection (PTV-LVI) and negative chemical ionization (NCI). It was found that much lower detection limits for the investigated pesticides can be obtained. Prior to GC-NCI/MS analysis, the optimized parameters of PTV-LVI were as follows: inlet temperature was set at 45 ℃, split vent flow rate was 20 mL/min, evaporation time was 1 min and evaporation temperature was set at 60 ℃. The volume of injection was 10 μL. Co-extractives were removed from the acetonitrile extracts using solid phase extraction with octadecyl (200 mg) and primary secondary amine (100 mg) sorbents. Matrix matched calibration solutions were used for all the analyses. To evaluate performance of the method, validation experiments were carried out in cabbages and apples at two spiking levels (5 and 10 μg/kg). The average recoveries ranged from 58.5% to 113.2%, and the relative standard deviations (RSDs, n=6) were in the range of 3.3% ~14.5%. The limits of detection (S/N=3) were less than 5 μg/kg for all the pesticides. The results show that this method is simple, rapid, sensitive and specific. It is appropriate for the simultaneous identification and quantification of the multi-residues in cabbages and apples.

Keywords: programmable temperature vaporizer (PTV) large volume injection (LVI) negative chemical ionization (NCI) gas chromatography-mass spectrometry (GC-MS) multi- pesticide residues cabbage apple

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