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高效液相色谱-串联质谱法测定葱中5种双酰肼农药残留

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Determination of five diacylhydrazine insecticide residues in Welsh onion using high performance liquid chromatography-tandem mass spectrometry

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摘要 建立了同时分析葱中5种双酰肼农药的高效液相色谱-串联质谱方法。样品经乙腈高速匀浆提取,盐析后浓缩,弗罗里硅土柱色谱净化,采用液相色谱-串联质谱分析5种化合物。质谱分析采用电喷雾电离,正离子扫描,多反应监测模式。对双酰肼农药主要碎片的裂解方式进行了剖析。实验证明,柱净化后无明显的基质效应,样品中添加0.002～0.2 mg/kg的5种双酰肼,其回收率为72.6%～95.5%,相对标准偏差(n=5)小于15%;检出限为0.5 μg/kg,定量限为2 μg/kg。该方法提取效果好,具有良好的灵敏度、回收率和重复性。

关键词： 高效液相色谱-串联质谱 双酰肼农药 残留 葱

Abstract： A high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) method was established for the determination of RH-5849, methoxyfenozide, chromafenozide, fufenozide and tebufenozide residues in Welsh onion. The residues in Welsh onion were extracted by blending with acetonitrile, concentrated after salting out and purified with a Florisil column. The mobile phase was a mixture of methanol and water (containing 0.1% formic acid) （60:40, v/v） at a flow rate of 0.2 mL/min. The mass spectrometry was operated with electrospray in positive ionization mode and five diacylhydrazine insecticides were identified in multiple reaction monitoring (MRM) mode. The fragmentation pathways for the main product ions were analyzed. No significant matrix effect was found when the five diacylhydrazines were detected after the samples were purified by Florisil columns. The calibration curves showed good linearity within the concentrations of 2~400 μg/L with the correlation coefficients more than 0.999. The recoveries of the five diacylhydrazines spiked in Welsh onion were 72.6%~95.5% at spiked levels of 0.002~0.2 mg/kg. The relative standard deviations (RSDs) were less than 15%. The limit of quantitation (LOQ) was 2 μg/kg for each diacylhydrazine. This method is sensitive and accurate in the determination of the five diacylhydrazine insecticides in Welsh onion.

Keywords： high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) diacylhydrazine insecticide residue Welsh onion

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