

研究论文

分散固相萃取-气相色谱-串联质谱法测定蔬菜中107种农药的残留量

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摘要 采用分散固相萃取-气相色谱-串联质谱(QuEChERS-GC-MS/MS)建立了蔬菜中107种农药残留量的分析方法。样品由含1%冰醋酸的正己烷饱和乙腈提取、分散固相萃取法净化,采用气相色谱-串联质谱方法在分时段选择反应监测模式下进行测定,外标法定量。所有农药在0.05~1 mg/L范围内线性关系均良好;所有农药的方法定量限(LOQ)均低于10 µg/kg;在10 µg/kg的添加水平下,大蒜、青刀豆、萝卜和菠菜4种基质中绝大多数农药的平均回收率处于60%~130%之间,相对标准偏差(RSD)不大于15.3%。该方法不仅能用于多种蔬菜基质中107种农药残留的检测,而且还能较好地解决本底成分相当复杂的大蒜基质极易出现的干扰问题。

关键词 [气相色谱-串联质谱](#) [选择反应监测](#) [分散固相萃取](#) [多农药残留](#) [蔬菜](#)

Determination of 107 pesticide residues in vegetables using off-line dispersive solid-phase extraction and gas chromatography-tandem mass spectrometry

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Abstract

A screening method was developed for the determination of 107 pesticide residues in vegetables using off-line dispersive solid-phase extraction (DSPE) and gas chromatography-tandem mass spectrometry (GC-MS/MS). The pesticides interested were extracted from the samples with acetonitrile (saturated by n-hexane) containing 1% acetic acid and simultaneously separated by liquid-liquid partitioning with adding anhydrous magnesium sulfate plus sodium acetate following by a simple cleanup step known as dispersive solid-phase extraction. The extracts were determined by GC-MS/MS using external standard method. The method was reliable and stable that the recoveries of almost all pesticides were in the range from 60% to 130% at the spiked level of 10 µg/kg into four vegetable matrixes (garlic, green bean, radish and spinach) and the relative standard deviations (RSDs) were all not more than 15.3%. The linearity of the method was good between 0.05 mg/L and 1 mg/L, and all limits of quantification (LOQs) less than 10 µg/kg. The method is selective with no interference, especially in the complicated garlic matrix.

Key words [gas chromatography-tandem mass spectrometry \(GC-MS/MS\)](#) [selected reaction monitoring \(SRM\)](#) [dispersive solid-phase extraction \(DSPE\)](#) [multiple pesticide residues](#) [vegetables](#)

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