

基质固相分散-超高效液相色谱-串联质谱法检测蔬菜中残留的苯甲酰脲类和

韩笑¹, 娄喜山², 张莉³, 王国卿³, 马明¹, 王明林^{1**}

1. 山东农业大学食品学院, 山东 泰安 271000; 2. 烟台杰科检测服务有限公司, 山东 莱阳 265231; 3. 山东省化工研究院, 山东

Determination of benzoylurea and bishydrazide pesticide residue in vegetables by ultra performance liquid chromatography-tandem mass spectrometry with matrix solid phase dispersion

HAN Xiao¹, LOU Xishan², ZHANG Li³, WANG Guoqing³, MA Ming¹, WANG Minglin^{1**}

1. College of Food Science and Engineering, Shandong Agricultural University, Taian 271000, China; 2. Yantai J 265231, China; 3. Shandong Institute of Chemical Industry, Jinan 250014, China

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摘要 建立了基质固相分散技术结合超高效液相色谱-串联质谱(UPLC-MS/MS)测定蔬菜中苯甲酰脲类杀虫剂(除虫脲、灭幼脲、杀铃脲、氟虫脲、氟啶脲、氟铃脲)和双酰肼类杀虫剂(甲氧虫酰肼、虫酰肼)的方法。蔬菜样品经中性氧化铝和石墨化炭黑研磨,乙酸乙酯浓缩定容后,经超高效液相色谱分离,分别在正、负离子多反应监测(MRM)模式下用电喷雾电离串联质谱测定,外标法定量。结果表明,1~100 µg/L质量浓度范围内有良好的线性关系($R^2 \geq 0.99$);在1、5、10、100 µg/kg 4个加标水平上的回收率为78.5%~112.8%,标准偏差为2.3%~10.2%,检出限为0.5~1.0 µg/kg。该方法操作简便快速,样品和溶剂用量少,检出限低,可满足蔬菜中苯甲酰脲类和双酰肼类杀虫剂同时检测的要求。

关键词: 基质固相分散 超高效液相色谱-串联质谱法 苯甲酰脲类杀虫剂 双酰肼类杀虫剂 蔬菜

Abstract: A method for the determination of nine pesticides including benzoylureas (diflubenzuron, chlorobenzuron, triflumuron, teflubenzuron, flufenoxuron, chlorfluazuron, hexaflumuron) and bishydrazides (methoxyfenozide, tebufenozide) in vegetables was developed by ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) with matrix solid phase dispersion. The sample was graphitized with neutral alumina as dispersant and carbon black as purifying, and eluted with ethyl acetate. The separation was achieved by UPLC, and then the identification and quantification were performed using MS/MS with multiple-reaction monitoring and electrospray ionization in positive or negative mode. The following results were obtained: The calibration curves showed good linearity in the ranges of 1~100 µg/L with $R^2 \geq 0.99$; The recoveries were 78.5%~112.8% at four spiked levels (1, 5, 10, 100 µg/kg), and the relative standard deviations were 2.3%~10.2%; The limits of determination were 0.5~1.0 µg/kg. The method has the advantages of easy to operate, fast to perform, lower limits of quantification, consuming less sample and organic solvents. It can meet the demands of practical use for the rapid and simultaneous determination of benzoylureas and bishydrazides in vegetables.

Keywords: matrix solid-phase dispersion ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) benzoylurea pesticides bishydrazides pesticides vegetables

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Corresponding Authors: 王明林

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