色谱 2009 27 (1): 91-95 ISSN: 1000-8713 CN: 42-1226/O

研究论文

气相色谱-负化学离子源质谱测定大豆和玉米中12种三唑类杀菌剂的残留量

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收稿日期 2008-7-7 修回日期 2008-10-2 网络版发布日期 2009-2-2 接受日期 2008-10-6

摘要 建立了一种可用于大豆和玉米中12种三唑类杀菌剂残留量测定的分散固相萃取-气相色谱-负化学离子源质谱方法。样品经含1%冰醋酸的乙腈提取, 分散固相萃取法净化, 采用气相色谱-负化学离子源质谱分时段选择离子监测技术进行测定与确证, 外标法定量。12种农药在50～1000 μg/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8 μg/kg;在10，20和40 μg/kg 3个添加水平下所有农药的回收率为70%～130%, 相对标准偏差(RSD)≤13.9%。该方法在检测大豆和玉米基质时无干扰现象出现。

关键词 气相色谱-负化学离子源质谱 选择离子监测 分散固相萃取 三唑类杀菌剂 大豆 玉米

Determination of 12 azole fungicide residues in beans and corn by offline disperse solid phase extraction and gas chromatography-negative chemical ionization mass spectrometry

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Abstract

A confirmatory method is proposed for the determination of 12 azole fungicide residues in beans and corn with the technique of offline disperse solid phase extraction (DSPE) and gas chromatography-negative chemical ionization mass spectrometry (GC-NCI/MS). The pesticides interested were extracted from the samples with acetonitrile containing 1% acetic acid and simultaneous liquid-liquid partitioning formed by adding anhydrous magnesium sulfate plus sodium acetate followed by a simple cleanup step known as dispersive solid-phase extraction. The aliquot was determined and confirmed by GC-NCI/MS using external standard method. The recoveries of all pesticides were between 70% and 130% at the three spiked levels, 10 μg/kg, 20 μg/kg and 40 μg/kg. The relative standard deviations were less than 13.9%. The linearity of method was good from 50 to 1000 μg/L. The limits of quantification (LOQ) were less than 8 μg/kg. The method is selective with no interference and is suitable for the confirmatory of pesticide residues in beans and corn.

Key words gas chromatography-negative chemical ionization mass spectrometry (GC-NCI/MS) selected ion monitoring disperse solid phase extraction azole fungicides beans corn

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