研究论文

超高效液相色谱法测定水果和饮料中残留的氟嘧菌酯和嘧螨酯维丽丽1,薄海波2*,毕阳1,吴永隆1

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摘要 建立了多种水果和饮料中氟嘧菌酯和嘧螨酯残留的超高效液相色谱测定方法。样品用乙酸乙酯—环己烷(体积比为1:1)超声波萃取,凝胶渗透色谱法净化,超高效液相色谱—二极管阵列检测器检测,外标法定量。采用BEH C18色谱柱(50 mm×2.1 mm, 1.7 μ m),流动相为水—乙腈(体积比为3:7),流速为0.3 μ min,柱温为40 °C,紫外检测波长为251 nm。实验结果表明:氟嘧菌酯和嘧螨酯在0.05~2 μ mg/L范围内线性关系良好(r>0.999),在不同的基质中添加3个浓度水平(0.01,0.05,0.1 μ mg/kg)的氟嘧菌酯和嘧螨酯,两者的回收率均在82.60%~101.11%之间,相对标准偏差为5.4%~15.3%;检出限不大于6 μ mg/kg,定量限不大于20 μ mg/kg。

关键词 超高效液相色谱 凝胶渗透色谱 氟嘧菌酯 嘧螨酯 农药残留 水果 饮料

Determination of fluoxastrobin and fluacrypyrim residues in fruits and beverages by ultra performance liquid chromatography

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Abstract

A method was developed for the determination of fluoxastrobin and fluacrypyrim residues in fruits and beverages by ultra performance liquid chromatography with photo-diode array (UPLC-PDA) detection. The sample was extracted with ethyl acetate-cyclohexane (1:1, v/v) by ultrasonic, cleaned-up by gel permeation chromatography (GPC), and then determined by UPLC-PDA. The quantification was performed by external standard. A BEH C18 colunm (50 mm×2.1 mm, 1.7 μ m) was used, and water-acetonitrile (3:7, v/v) was used as mobile phase at a flow rate of 0.3 mL/min. The column temperature was set at 40 °C, and ultraviolet absorption wavelength was set at 251 nm. The calibration curves were linear between the peak area and the concentration in the range of 0.05-2 mg/L for fluoxastrobin and fluacrypyrim, the correlation coefficients were greater than 0.999. The average recoveries spiked in fruit and beverage matrices at the three concentration levels of 0.01, 0.05, 0.1 mg/kg ranged from 82.60% to 101.11% with the relative standard deviations of 5.4%-15.3%. The limits of detection (LOD) were not greater than 6 μ g/kg and the limits of quantification (LOQ) were not greater than 20 μ g/kg in fruit and beverage matrices for fluoxastrobin and fluacrypyrim.

Key words <u>ultra performance liquid chromatography (UPLC)</u> <u>gel permeation chromatography (GPC)</u> <u>fluoxastrobin</u> <u>fluacrypyrim</u> <u>pesticide residues</u> <u>fruit</u> <u>beverage</u>

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