研究论文

高效液相色谱法同时测定蔬菜水果中的12种农药残留 李永新 孙成均 赵剑虹 杨柳桦

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摘要 建立了同时测定蔬菜水果中12种农药残留的反相高效液相色谱分析方法。将样品捣碎,用乙酸乙酯超声提取,经Florisil固相萃取柱净化、正己烷-二氯甲烷(体积比为1:1)洗脱、氮气吹干、甲醇溶解并定容后,采用高效液相色谱柱分离、紫外检测,以外标法定量。结果表明: 12种农药标准曲线的线性相关系数范围为0.9985~0.9999;检测限为0.14~2.65 ng;在水果中的平均加标回收率为62.2%~118.2%,相对标准偏差(RSD)为0.56%~11.8%;在蔬菜中的平均加标回收率为52.1%~124.6%,RSD为0.89%~18.4%。用所建立的方法成功地测定了白菜、莲白、黄瓜、苹果、梨等40份样品中的农药残留。方法具有快速、简便、准确、灵敏、重现性较好的特点,适合于蔬菜水果样品中多种微量农药残留的测定。

关键词 <u>高效液相色谱法</u> <u>固相萃取</u> <u>多农药残留; 水果</u> <u>蔬菜</u> 分类号

Simultaneous Determination of Multiresidual Pesticides in Vegetables and Fruit by High Performance Liquid Chromatography

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Abstract

A high performance liquid chromatographic (HPLC) method for simultaneous determination of 12 pesticides in vegetable and fruit samples was developed and evaluated. The analytical procedure involved in ultrasonic extraction with ethyl acetate, purification using a Florisil SPE column (6 mL, 1000 mg) and n-hexane-dichloromethane (1: 1, v/v). The eluate was blown to dryness under a stream of nitrogen and the residue was dissolved in 0.10 mL of methanol, followed by separation and quantitative analysis by using reversed-phase HPLC with gradient elution for the baseline separation of the 12 pesticides and programmed ultraviolet wavelength shift detection in 32 min. The detection limits of the 12 pesticides ranged from 0.14 to 2.65 ng. The average recoveries ranged from 62.2% to 118.2% with the relative standard deviation (RSD) range of 0.56% -11.8% for the spiked fruit samples, and from 52.1% to 124.6% with RSD range of 0.89%-18.4% for the spiked vegetables. The method was applied to determining multiresidual pesticides in vegetables and fruit for a total of 40 samples. It is concluded that this method is rapid, accurate, sensitive and reproducible for determining trace pesticides in vegetable and fruit samples.

Key words high performance liquid chromatography (HPLC) solid phase extraction (SPE) multiresidual pesticides fruit vegetables

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