#### 研究论文

液相色谱-串联质谱法测定蔬菜水果中的吡丙醚残留量赵广宇1,郭德华2\*,赵善贞2,盛永刚2,邓晓军2

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摘要 建立了测定蔬菜、水果中吡丙醚残留量的液相色谱—串联质谱 (LC-MS/MS) 分析方法。样品在醋酸钠缓冲液下用酸性乙腈提取,取1 mL提取液用PSA(N-丙基乙二胺)填料净化后,采用CAPCELL PAK C18色谱柱 (50 mm×2.0 mm, 3  $\mu$ m) 分离,以含0.1%甲酸的乙腈溶液和含0.1%甲酸的2 mmo1/L乙酸铵溶液作为流动相进行梯度洗脱,以电喷雾电离三重四极杆串联质谱在正离子多反应监测 (MRM) 模式下进行测定。吡丙醚在2.5~50  $\mu$ g/L范围内呈线性关系,相关系数为0.999 9, 在5, 50, 100  $\mu$ g/kg 3个添加水平下的回收率为84.7%~91.5%,相对标准偏差 (RSD, n10) 低于10%。该方法操作简便,稳定性和选择性好,灵敏度高(检出限为5  $\mu$ g/kg),适用于蔬菜、水果中吡丙醚残留量的测定。

关键词 液相色谱-串联质谱法 吡丙醚 水果 蔬菜;杀虫剂

# Determination of pyriproxyfen residue in vegetables and fruits by liquid chromatography-tandem mass spectrometry

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#### Abstract

A method of liquid chromatography-tandem mass spectrometry (LC-MS/MS) was established for the determination of pyriproxyfen residue in vegetables and fruits. The pyriproxyfen residue in the sample was extracted by acidified acetonitrile with the presence of sodium acetate buffer, and cleaned up by PSA (primary secondary amine) sorbent, and the separation was performed by ultrafast liquid chromatography (UFLC) on a CAPCELL PAK C18 column (50 mm×2.0 mm, 3  $\mu$ m) and the gradient elution of acetonitrile (containing 0.1% formic acid) and 2 mmol/L ammonium acetate solution (containing 0.1% formic acid) at a flow rate of 0.3 mL/min. The calibration curve was linear between the peak area and the concentration of 2.5~50  $\mu$ g/L with the correlation coefficient more than 0.999 9. The limit of quantification of pyriproxyfen was 5  $\mu$ g/kg. The average recoveries spiked at three concentrations of 5, 50 and 100  $\mu$ g/kg ranged 84.7% ~91.5%. The relative standard deviations (n10) were all less than 10%. The method is selective without interference and suitable for the determination of pyriproxyfen residue in vegetables and fruits.

**Key words** <u>liquid chromatography-tandem mass spectrometry (LC-MS/MS)</u> <u>pyriproxyfen</u> <u>fruits</u> <u>vegetables</u> <u>pesticide</u>

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