高效液相色谱法测定食品中氟啶脲的残留量

杜利君

摘 要: 建立了高效液相色谱测定食品中氟啶脲残留量的方法。样品中的氟啶脲经正己烷或乙腈提取,弗罗里硅土净化后,以乙腈-水(4:1)混合溶液为流动相,经<色谱柱分离,紫外检测器测定。结果表明:氟啶脲在0.05~2.0 mg/L范围内线性良好(相关系数为0.999 8),定量限(以信噪比为3计)为0.05 mg/kg。在0.05、0.1、1.0 mg/kg添加水平下的加标回收率为82.1%~102.5%,相对标准偏差为3.00%~6.25%。该方法操作简便、快速,稳定性好,样品和溶液用量少,可用于水果、蔬菜、粮谷、动物源性等食品中氟啶脲残留量的日常检测。

关键词: 高效液相色谱法;氟啶脲;食品

中图分类号: 文献标识码: 文章编号:
2.2 HPLC

The HPLC system consisted of an Agilent 1200 series pump, a diode array detector (DAD), and a G6225AA autosampler. The column was a Waters Symmetry C18 (5 μm, 250 mm × 4.6 mm). The mobile phase was a gradient mixture of water (A) and methanol (B), with the following gradient: 0-5 min, 10% B; 5-10 min, 10% B; 10-15 min, 10% B; 15-20 min, 10% B; 20-25 min, 10% B; 25-30 min, 10% B; 30-35 min, 90% B; 35-40 min, 90% B; 40-45 min, 90% B; 45-50 min, 90% B. The flow rate was 0.8 mL/min, and the injection volume was 20 μL. The column temperature was 40 °C. The detection wavelength was 260 nm. The chromatograms were analyzed using Agilent ChemStation software.

2.3 Results and Discussion

The regression equation of the calibration curve was y = 0.0502x + 0.0037, with a correlation coefficient of R² = 0.9998. The linearity range was 0.001-2 mg/L, and the limit of quantification (LOQ) was 0.0005 mg/L. The precision was evaluated by analyzing the standard solution at five concentrations (0.001, 0.005, 0.01, 0.05, and 0.1 mg/L) in triplicate. The relative standard deviation (RSD) was less than 2%. The stability was evaluated by analyzing the standard solution at three concentrations (0.001, 0.01, and 0.1 mg/L) at 0, 24, and 72 hours. The RSD was less than 2%.

The recoveries were evaluated by spiking the standard solutions into the samples at three levels (0.001, 0.01, and 0.1 mg/L) and analyzing each sample in triplicate. The average recoveries were 88.9%, 101.2%, and 98.5%, respectively. The extraction recovery was evaluated by analyzing the standard solution at two levels (0.001 and 0.01 mg/L) in triplicate. The extraction recovery was 98.5%.

Trichlorfon, Techny, and 1,2,4-trichlorobenzene were used as internal standards. The retention times were 6.8, 13.2, and 16.4 min, respectively. The peak purity was checked using the 3-D model and full-scan MS. No interfering peaks were observed in the extracted ion chromatograms (XIC) of the target compounds.

The chromatograms of the standard solution and the blank matrix were compared. The blank matrix was prepared by gravel, sand, soil, and leaf. The chromatograms were analyzed using Agilent ChemStation software. The retention times were 6.8, 13.2, and 16.4 min, respectively. No interfering peaks were observed in the XIC of the target compounds.
2.5 The correlation coefficient $r = 0.999$ and $0.05 \sim 2.0$ mg/L, indicating that the S/N ≥ 10. The detection limit was 0.05 mg/kg.

Table 1 Recoveries and precisions (RSDs) of chlorfluazuron spiked in foods $n = 10$

<table>
<thead>
<tr>
<th>Spiked/ mg/kg</th>
<th>Apple Recovery/%</th>
<th>RSD/%</th>
<th>Buckwheat Recovery/%</th>
<th>RSD/%</th>
<th>Beef Recovery/%</th>
<th>RSD/%</th>
<th>Spinach Recovery/%</th>
<th>RSD/%</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>98.7</td>
<td>3.93</td>
<td>82.1</td>
<td>6.35</td>
<td>85.1</td>
<td>6.25</td>
<td>89.6</td>
<td>5.37</td>
</tr>
<tr>
<td>0.1</td>
<td>96.5</td>
<td>4.26</td>
<td>86.5</td>
<td>5.57</td>
<td>84.6</td>
<td>5.80</td>
<td>102.5</td>
<td>4.98</td>
</tr>
<tr>
<td>1</td>
<td>97.6</td>
<td>3.47</td>
<td>89.2</td>
<td>4.18</td>
<td>83.8</td>
<td>3.31</td>
<td>98.1</td>
<td>3.00</td>
</tr>
</tbody>
</table>

Fig. 1 Chromatogram of chlorfluazuron standard

Fig. 2 HPLC chromatograms of a blank asparagus sample and b the asparagus spiked with chlorfluazuron standard at 0.05 mg/kg

Fig. 3 HPLC chromatograms of a blank scallion sample and b the scallion spiked with chlorfluazuron standard at 0.05 mg/kg
能满足国内外残留限量要求，并可节约大量的有机溶剂，经济实用，可以在实验室中广泛推广。

参考文献: