

## 高效液相色谱法同时测定青菜中环丙氨嗪和三聚氰胺残留

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## Simultaneous Determination of Cyromazine and Melamine in Green Vegetables With HPLC

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摘要

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摘要 研究并建立了同时测定青菜中环丙氨嗪和三聚氰胺残留量的高效液相色谱紫外检测分析方法。冷冻干燥并均质后的青菜样品经V(氨水):V(甲醇)=5:95的混合溶液超声辅助浸提、离心、旋转蒸发、固相萃取等浓缩净化处理后,用高效液相色谱定量。分离色谱柱为Agilent NH<sub>2</sub>柱,流动相为V(乙腈):V(水)=90:10的混合溶液,流速为1 mL·min<sup>-1</sup>,检测波长为214 nm。环丙氨嗪和三聚氰胺的标准曲线在0.05~10.0 mg·L<sup>-1</sup>范围内与其峰面积线性良好,相关系数不低于0.9995,在0.2~4.0 mg·kg<sup>-1</sup>添加范围内,环丙氨嗪和三聚氰胺平均回收率分别为81.26%~87.69%和78.24%~82.33%,相对标准偏差分别为2.75%~6.18%和3.99%~6.67%,方法的最低检测浓度分别为11.30和20.38 μg·kg<sup>-1</sup>。该方法操作简单且灵敏度高,适用于青菜样品中环丙氨嗪和三聚氰胺残留检测。

关键词: 环丙氨嗪 三聚氰胺 青菜 固相萃取 高效液相色谱

Abstract: An analytical method has been developed for simultaneous determination of cyromazine and metabolite melamine in green vegetables using a high performance liquid chromatograph (HPLC) coupled with an ultraviolet detector. Fresh green vegetable samples were freeze-dried, homogenized and subjected to extraction in solution containing 95% methanol and 5% aqueous ammonia assisted with ultrasonics. The extracts were concentrated and purified through centrifugalization, rotary evaporation and solid phase extraction, and quantified using a high performance liquid chromatography-ultraviolet detector. It used an Agilent NH<sub>2</sub> column as chromatographic column for separation, solution consisting of 90% acetonitrile and 10% water as carrier with a flow rate of 1 mL·min<sup>-1</sup> and worked with a wavelength of 214 nm. It was found that the standard curves of cyromazine and melamine in the range of 0.05-10 mg·L<sup>-1</sup> showed a good linear relationship with their peak areas with correlation coefficients not less than 0.9995, and the recovery rate of cyromazine and melamine at fortified levels of 0.2-4.0 mg·kg<sup>-1</sup> varied in the range of 81.26%-87.69% and 78.24%-82.33% with relative standard deviation being 2.75%-6.18% and 3.99%-6.67%, respectively. The limit of quantification (LOQs) was 11.30 μg·kg<sup>-1</sup> for cyromazine and 20.38 μg·kg<sup>-1</sup> for melamine. This method is simple, sensitive and suitable for determination of cyromazine and melamine in green vegetables.

Keywords: cyromazine melamine green vegetables solid phase extraction HPLC

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