

论文

LC/MS/MS的多反应监测方法定量测定灯盏乙素

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摘要:

目的: 建立一种可靠的灯盏乙素定量分析方法。方法: 用三级四极串联质谱(MS/MS)作为HPLC的检测器, 其中MS/MS使用了多反应监测(MRM)扫描方式。选择母→子离子对m/z -461→m/z -285作为MRM监测的离子对; HPLC流动相为100%甲醇, 流速0.9 mL.min⁻¹, 色谱柱Beckman ODS-1。以测定短葶飞蓬提取物的灯盏乙素含量为例, 对此方法进行了应用。结果: 灯盏乙素在短葶飞蓬提取物中含量为6.98%。方法线性范围20~160 ng.mL⁻¹ (γ=0.999); 加入灯盏乙素标准品20,60和160 ng的加样回收率分别为: 96.5%,97.4%和97.3%。检测限为1 ng,每个样品的分析时间为4 min。结论: 此法灵敏、快速、准确, 可应用于灯盏乙素的各种药剂、药代的研究。

关键词: 高效液相色谱; 串联质谱; 灯盏乙素

DETERMINATION OF SCUTELLARIN BY LC/MS/MS

Qu Jun; Wang Yiming; Luo Guoan and Wu Zhuping

Abstract:

AIM: To establish a reliable method for quantitative analysis of scutellarin. METHODS: A triple-quadrupole tandem mass spectrometer was used as a detector for HPLC to determine scutellarin. As to MS/MS, multi-reactions monitoring (MRM) scan mode was employed. Among the product ions of scutellarin, m/z -285 is the most abundant in intensity, thus the parent-daughter ion pair of m/z -461 and m/z -285 was selected as MRM ions pair. MS/MS conditions were optimized to achieve highest sensitivity. The mobile phase of HPLC was 100% methanol and the analytical column was Beckman ODS-1. The flow rate of HPLC was 0.9 mL.min⁻¹. The areas of ion flow peaks were used to determine the quantity of scutellarin. To give an example of its applications, this method was used to determine the amount of scutellarin in Erigaron breviscapus extract. RESULTS: The amount of scutellarin accounted for 6.98% ±0.11% in Erigaron breviscapus extract. The standard curve for scutellarin showed good linearity over the concentration range of 20~160 ng.mL⁻¹ (γ=0.999); the recoveries of scutellarin at added amount of 20, 60 and 160 ng are 96.5%, 97.4% and 97.3% respectively; the limit of detection is 1ng, and the analysis time of each sample was 4 minutes. CONCLUSION: This method is highly sensitive, fast, and very accurate. Therefore, it is possible to be applied to study the metabolism of scutellarin.

Keywords: MS/MS scutellarin HPLC

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