

论文

HPLC-MS<sup>n</sup>法鉴定葫芦巴碱及其在大鼠体内的主要代谢产物

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摘要:

目的建立快速灵敏的LC-MS<sup>n</sup>检测葫芦巴碱及其在大鼠体内代谢物的分析方法。方法以葫芦巴碱对LC-MS<sup>2</sup>色谱及质谱条件进行优化,分析其电喷雾质谱的一级电离规律和多级质谱裂解规律,以此作为葫芦巴碱大鼠体内代谢物分析鉴定的依据。健康大鼠尾静脉注射8 mg·kg<sup>-1</sup>葫芦巴碱,收集0~48 h的尿样,经C<sub>18</sub>小柱固相萃取分离纯化后,直接采用LC-MS<sup>n</sup>方法对尿样进行测定。结果根据生物体内药物代谢转化规律及母体药物的色谱-质谱行为规律,在尿样中鉴定出母药及其N-去甲基、N-去甲基环氧化产物,以及母药及其N-去甲基环氧化物的甘氨酸衍合物。结论本方法灵敏、快速、选择性高、专属性好,可用于葫芦巴碱的代谢产物研究。

关键词: LC-MS<sup>n</sup> 葫芦巴碱 代谢物

HPLC-MS<sup>n</sup> analysis of trigonelline and its metabolites in rat urine

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Abstract:

AimTo establish a rapid and sensitive LC-MS<sup>n</sup> method for the identification of trigonelline and its main metabolites in rat urine. MethodsAfter optimizing the detection conditions of LC-MS<sup>n</sup> chromatography and mass spectrometry using trigonelline, its ionization and cleavage in ESI-MS and ESI-MS<sup>n</sup> modes were summarized, then serving as the basis for the metabolite analysis of trigonelline in rat urine. The 0-48 h urine samples of rats were collected after iv 8 mg·kg<sup>-1</sup> trigonelline, then, the samples were purified through C<sub>18</sub> solid-phase extraction cartridge. The purified samples were analyzed by LC-MS<sup>n</sup>. ResultsThe structures of trigonelline metabolites were elucidated according to the changes of the molecular weights of the metabolites ( $\Delta M$ ) and their cleavage pattern in ESI-ITMS<sup>n</sup>. As a result, two phase I metabolites and the parent drug were identified existing in rat urine, and two phase II metabolites were identified. ConclusionThe LC-MS<sup>n</sup> method is rapid and high sensitive and specific, it is suitable for the identification of trigonelline and its metabolites in rat urine.

Keywords: trigonelline metabolite LC-MS<sup>n</sup>

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