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HPLC测定大鼠血浆中盐酸川芎嗪及其代谢物

Determination of Tetramethylpyrazine and its Active Metabolite in Rat Plasma by HPLC

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中文关键词: [川芎嗪](#) [血药浓度](#) [高效液相色谱法](#)

英文关键词: [tetramethylpyrazine](#) [serum concentration of drug](#) [HPLC](#)

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作者	单位	E-mail
袁芳 ¹ , 陈卓佳 ² , 陈杰 ^{3*}	1. 广东药学院药科学院, 广州 510006; 2. 中山大学药学院临床药理研究所, 广州 510080; 3. 中山大学附属第一医院药剂科, 广州 510006	chenjiezs@163.com

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中文摘要:

目的 建立川芎嗪(TMP)及其代谢物2-羟甲基-3, 5, 6-三甲基吡嗪(HTMP)大鼠血浆中的高效液相色谱测定方法。方法 血浆碱化后用氯仿-1-氯正丁烷(3:1)萃取, 采用HPLC检测, 色谱柱为Hypersil BDS C₁₈(4.6 mm×250 mm, 5 mm), 流动相为20 mmol·L⁻¹磷酸二氢钾缓冲液(pH 5.6)-甲醇(72:28), 2-甲基吡嗪为内标, 流速: 1.0 mL·min⁻¹。检测波长为285 nm, 柱温为室温。结果 TMP大鼠血浆浓度测定方法在0.031 25~50 μg·mL⁻¹内线性良好($r=0.999\ 5$); HTMP线性范围为0.031 25~5 μg·mL⁻¹($r=0.999\ 8$)。TMP、HTMP的定量下限均为0.031 25 μg·mL⁻¹, 提取回收率为86.2%~93.0%(TMP)、73.8%~95.1%(HTMP), 相对回收率为96.9%~117.7%(TMP)、97.5%~104.9%(HTMP), 批内和批间RSD均<9%($n=5$)。结论 该方法简便、准确, 重复性好, 灵敏度高, 适合于TMP及其主要代谢产物HTMP血药浓度测定及药动学研究。

英文摘要:

OBJECTIVE To establish an HPLC method for the determination of concentration of tetramethylpyrazine (TMP) and its active metabolite, 2-hydroxymethyl-3,5,6-trimethylpyrazine (HTMP) in rat plasma and apply this method to the pharmacokinetic study of TMP. METHODS 2-Methylpyrazine was used as internal standard. The alkalified serum samples were extracted with a chloroform-1-chloro-butane (3:1) induced liquid-liquid extraction. The target analytes were quantitatively determined by HPLC. Hypersil BDS C₁₈ column(4.6 mm×250 mm, 5 mm) was used. The mobile phase consisted of 20 mmol·L⁻¹ potassium dihydrogen phosphate buffer(pH 5.6)-methanol(72:28). The flow rate was 1.0 mL·min⁻¹, 15 mL sample was injected and detected by the ultra-violet detector at 285 nm. RESULTS The linearity was obtained over the concentration ranges of 0.031 25-50 μg·mL⁻¹ for TMP($r=0.999\ 5$) and 0.031 25-5 μg·mL⁻¹ for HTMP($r=0.999\ 8$). The lower limit of quantitation (LLOQ) was 0.031 25 μg·mL⁻¹ for TMP and HTMP. The inter- and intra-batch precisions (RSD%) were less than 9% for both analyses. The accuracies and extracted recoveries were 86.2%-93.0% for TMP and 73.8%-95.1% for HTMP. The relative recoveries were 96.9%-117.7% for TMP and 97.5%-104.9% for HTMP. CONCLUSION The method has good selectivity, acceptable accuracy, precision and sensitivity. It can be applied to pharmacokinetic study of TMP in rats.

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