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论文

HPLC-FLU法测定血清中黄藤素的含量

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1.上海市徐汇区中心医院, 上海 200031; 2.中国科学院上海药物研究所, 上海 200031 摘要:

目的 建立血清样品中黄藤素的高效液相色谱一荧光检测法。方法 色谱柱为LiChrosorb SI 60(5 μ m) 20 cm×4.0 mm ID; 流动相为二氯甲烷-甲醇-二乙胺-冰醋酸(90:9:0.4:0.5);流速为1.0 mL.min⁻¹;荧光检测 E_{χ} =365 nm, E_{m} =510 nm。血清样品加13-甲基小檗碱(内标),用三氯乙酸-氯仿提取,水浴加温、氮气吹干,用二氯乙烷溶解进样。结果 黄藤素和内标的保留时间分别为8.4 min和7.1 min。黄藤素的最低检测浓度为 0.1 ng.mL⁻¹。在0.1~10.0 ng.mL⁻¹有良好线性关系(γ =0.9999)。血药浓度测定天内、天间精密度分别为0.94%~1.85%和0.15%~6.47%。结论 用高效液相色谱-荧光检测法测定黄藤素的血药浓度,有灵敏、专一和快速的优点,可满足药代动力学研究的需要。

关键词: 黄藤素 高效液相色谱-荧光检测 血药浓度测定

DETERMINATION OF PALMATINE IN SERUM BY HPI C-FI U

ZHANG Hui; YU Chen; HONG You-Cai; JI ANG Shan-Hao; SHEN Jing-Kang; ZHU Da-Yuan

Abstract:

AIM To develop a method for analysis of palmatine in serum by high performance liquid chromatography with fluorometric detector. METHODS Separation was obtained by using a LiChrosorb SI 60 column(4.0 mm×200 mm, 5 μ m). The mobile phase consisted of a mixture of dichloromethane-methanol-diethyl amine-acetic acid(90:9:0.4:0.5) and the flow rate was 1 mL.min⁻¹. Excitation and emission wavelengths were set at 365 and 510 nm respectively. To 1.0 mL of serum containing palmatine was added 13-methylberberine (internal standard, 1S), then extracted with 5.0 mL chloroform containing trichloroacetic acid. The organic phase was removed with nitrogen and the residue dissolved with 100 μ L dichloromethane. After centrifugation, 20 μ L of the lower layer was subjected to HPLC. RESULTS The retention times of palmatine and IS were 8.4 and 7.1 min respectively. In serum the detection limit of palmatine was 0.1 ng.mL⁻¹. The extraction recoveries of palmatine and IS were over 85%. The relative standard deviations of within-day and between-day were 0.94%~1.85% and 0.10%~6.47% (n=6) respectively. CONCLUSION This method is sensitive, simple and fast, so it can fit the need of palmatine pharmacokinetic research preferably.

Keywords: HPLC-FLU serum palmatine

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