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论文

柱切换HPLC法测定血浆中氢溴酸右美沙芬的代谢物去甲右美沙芬

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摘要:

应用柱切换HPLC法建立了氢溴酸右美沙芬的主要代谢产物去甲右美沙芬的血浆浓度测定方法。去甲右美沙芬的葡萄糖醛酸结合物,经β-葡萄糖醛酸苷酶水解后,即可取血浆直接进行HPLC分析。预处理柱为30 mm×5 mm ID,内装μBondapak C_{18},37～50μm;分析柱为150 mm×5 mmID,内装YWG-C_{18},10μm。预处理流动相为0.2%的乙酸溶液,流速3 ml/min;分析流动相为乙腈—水—乙酸—三乙胺—二氯甲烷(17:82:1:0.05:0.025)的混合溶液,流速1 ml/min荧光检测波长分别为λex=290 nm和λem=315 nm。血浆浓度测定的线性范围为20～640 ng/ml,血浆中最低检测浓度为4ng/ml,方法的平均回收率为103.8%,日内及日间变异均小于10%。

关键词: 氢溴酸右美沙芬 去甲右美沙芬 柱切换 HPLC 血药浓度

COLUMN SWITCHING HPLC METHOD FOR DETERMINATION OF DEXTRORPHAN, AN ACTIVE METABOLITE OF DEXTROMETHORPHAN, IN PLASMA

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Abstract:

An HPLC method for the determination of dextrorphan, an active metabolite of dextromethorphan, in plasma was established using column switching technique. The column switching system was equipped with a per-column of 30 mm×5 mm ID, packed with μ Bondapak C_{18}, 37～50 μn, and an analytical column of 150 mm×5 mm ID, packed with YWG-C_{18}, 5 μm. A 0.2 % acetic acid solution was used as the pretreating mobile phase to wash out impurities from the per-column. The analytical mobile phase consisted of acetonitrile—water—acetic acid—triethyl-amine—dichloromethane (17:82:1:0.05:0.025). The plasma samples were directly injected into the HPLC system after enzymatic hydrolysis of dextrorphan glucuronide ester conjugate to free form with β-glucuronidase. The dextrorphan was monitored with a fluorescence detector at 290 nm (excitation) and 315 nm (emission). The method was linear within the plasma concentration range of 20～640 ng/ml (r=0.9987), and the detection limit was 4 ng/ml. The mean recoveries of the method averaged 103.8%. The relative standard deviations of the assay were less than 10% for both withinday and between-days.

Keywords: Dextrorphan Column switching HPLC Plasma drug concentration Dextromethorphan

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