

论文

液相色谱-串联质谱法测定大鼠脑透析液中CTN986及其脱糖产物的含量

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摘要:

建立了测定CTN986及其脱糖产物芦丁和陆地棉苷在大鼠脑透析液中含量的高效液相色谱-串联质谱(HPLC-MS/MS)法. 采用微透析技术结合HPLC-MS/MS, 以甲醇-异丙醇-水-甲酸(体积比20:10:70:0.1)为流动相, 用Zorbax C8柱分离, 待测物通过电喷雾离子化四极杆串联质谱, 以多反应监测方式(MRM)进行检测. 采用外标法分析给予CTN986后大鼠脑透析液中CTN986及其脱糖产物的浓度, 经过体内回收率校正后, 计算出脑透析液中待测物的浓度. CTN986、芦丁和陆地棉苷的线性范围为2~500 ng/mL, 日内及日间精密性(RSD)均小于15%, 准确度(RE)在-4%~13%之间. 本方法专属性强、灵敏度高, 适用于脑透析液中CTN986的药代动力学分析, 为药代动力学研究提供了新的方法学参考.

关键词: CTN986; 液相色谱-串联质谱法; 微透析

Liquid Chromatography-Tandem Mass Spectrometric Assay for the Determination of CTN986 and Its Deglycosylation Products in the Rat Brain Dialysate

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Abstract:

An HPLC-MS/MS method was developed for the determination of CTN986 and its two deglycosylation products, rutin and hirsutin, in the rat brain dialysate. The dialysate samples were analyzed by offline liquid chromatography-tandem mass spectrometry. The separation was performed using a Zorbax C8 column with an isocratic mobile phase consisted of methanol/isopropanol/water/formic acid (20:10:70:0.1, volume ratio). An API 3000 tandem mass spectrometer equipped with electrospray ionization source was used as detector and was operated under the positive ion mode. Multiple reactions monitoring(MRM) mode was used to quantify three analytes. Calibration was performed by external standardization and regression curves were constructed ranging from 2 to 500 ng/mL for each compound. The intra- and inter-day precision values were below 15% and accuracy was between -4% and 13% for all quality control samples. As a result, the procedure proved to be very suitable for routine analysis. This quantitation method was applied to the pharmacokinetic study of CTN986 in the brain dialysate samples from rats following oral administration.

Keywords: CTN986; Liquid chromatography-tandem mass spectrometry; Microdialysis

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