色谱 » 2011, Vol. 29 » Issue (01) :26-30 DOI: 10.3724/SP.J.1123.2011.00026

正相液相色谱-串联质谱法分离普萘洛尔对映体

张娟红1,2, 王荣1,2*, 谢华1, 孟宪栋1, 贾正平1,2*, 马骏1, 张军莉1, 王娟1

1. 兰州军区兰州总医院全军临床药理基地, 甘肃 兰州 730050; 2. 兰州大学药学院, 甘肃 兰州 730000

Enantiomeric separation of propranolol by normal phase chiral liquid chromatography coupled with tandem mass spectrometry

ZHANG Juanhong1,2, WANG Rong1,2*, XIE Hua1, MENG Xiandong1, JIA Zhengping1,2*, MA Jun1, ZHANG Junli1, WANG Juan1

1. Base for Clinical Pharmacology, Lanzhou General Hospital of Lanzhou Military Region, Lanzhou 730050, China; 2. Pharmacy College, Lanzhou University, Lanzhou 730000, China

摘要 参考文献 相关文章

Download: PDF (174KB) HTML 1KB Export: BibTeX or EndNote (RIS) Supporting Info

摘要 建立正相液相色谱-串联质谱(LC-MS/MS)分离普萘洛尔对映体的方法,并用于盐酸普萘洛尔片对映体含量测定。样品使用甲醇进行简单提取,采用Chiralcel OD-H手性柱,以正己烷-乙醇-氨水(70:30:0.4, v/v/v)为流动相,流速为0.4 mL/min。在正离子模式下,通过电喷雾离子化(ESI+),采用多反应监测(MRM)方式进行检测,用于定量分析检测的离子对为m/z 260.2→116.0,在20 min内完成普萘洛尔对映体定量分析。盐酸普萘洛尔对映体在2.5～1000 μg/L质量浓度范围内线性关系良好,定量限为2.5 μg/L;日内及日间测定的相对标准偏差小于2.64%。两种对映体的加样回收率范围分别为99.08%～102.58%和100.21%～103.16%。该方法准确、简便、可靠、有效,可用于盐酸普萘洛尔片对映体的质量控制。

关键词： 正相液相色谱-串联质谱 手性分离 测定 普萘洛尔对映体

Abstract： A rapid and sensitive method was developed and validated using a normal phase liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) for determination of propranolol enantiomers in pharmaceuticals. Sample preparation involved a single extraction step by the addition of methanol. Separation of propranolol enantiomers was achieved on a Chiralcel OD-H chiral column using a mobile phase consisting of n-hexane-ethanol-ammonia (70:30:0.4, v/v/v), and the flow rate was 0.40 mL/min for 20 min. The analyte was monitored by tandem mass spectrometry with electrospray positive ionization in multiple reaction monitoring (MRM) mode, using the transitions of m/z 260.2→116.0. Propranolol enantiomers can be completely separated. The linear range was 2.5~1000 μg/L, and the limit of quantification (LOQ) was 2.5 μg/L. The values for within day and between day precisions and accuracies were well within the generally accepted criteria for analytical methods. The relative standard deviations (RSDs) were less than 2.64%, and the recoveries of the two enantiomers were 99.08%~102.58% and 100.21%~103.16%, respectively. The separation method is accurate, convenient, reliable, efficient, and can be subsequently used for quality control of propranolol enantiomers in pharmaceuticals.

Keywords： normal phase liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) chiral separation determination propranolol enantiomers

Received 2010-09-05; published 2011-01-21

Fund:

国家自然科学基金项目(No. 20775089)和甘肃省科技支撑计划项目(No. 1011FKCA144).

Corresponding Authors: .1123.2011.00026 *通讯联系人: 王荣,教授,主要研究方向为手性药物分析及体内药物分析. E-mail: wangrong-69@163.com. 贾正平,教授,博士生导师,主要研究方向为手性药物分析及分子药理学. Email: wangrong-69@163.com

Service
把本文推荐给朋友
加入我的书架
加入引用管理器
Email Alert
RSS
作者相关文章
王荣

引用本文:

张娟红1,2, 王荣1,2*, 谢华1, 孟宪栋1, 贾正平1,2*, 马骏1, 张军莉1, 王娟1.正相液相色谱-串联质谱法分离普萘洛尔对映体[J] 色谱, 2011,V29(01): 26-30

ZHANG Juanhong1,2, WANG Rong1,2*, XIE Hua1, MENG Xiandong1, JIA Zhengping1,2*, MA Jun1, ZHANG Junli1, WANG Juan1.Enantiomeric separation of propranolol by normal phase chiral liquid chromatography coupled with tandem mass spectrometry[J] Chinese Journal of Chromatography, 2011,V29(01): 26-30

链接本文:

http://www.chrom-china.com/CN/10.3724/SP.J.1123.2011.00026 或 http://www.chrom-china.com/CN/Y2011/V29/I01/26