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论文

高灵敏度LC-MS/MS法测定犬血浆中的坦洛新

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摘要：

犬口服盐酸坦洛新控释片后血浆药物浓度C_{max}小于10 ng·mL^{-1}，需建立测定犬血浆中坦洛新的高灵敏度液相色谱-串联质谱法(LC-MS/MS)。血浆样品加入内标苯海拉明，用正己烷-二氯甲烷(2∶1)萃取后，反相C_{18}色谱柱分离，以甲醇-乙腈-甲酸铵(30∶40∶30，*v*/*v*/*v*)为流动相，流速为0.4 mL·min^{-1}。选用大气压化学离子化源(APCI)三重四极杆串联质谱仪，以选择反应监测方式进行检测，用于定量分析的离子反应分别为*m*/*z* 409→228(坦洛新)和*m*/*z* 256→167(苯海拉明)。坦洛新线性范围为0.02～50 ng·mL^{-1}，定量下限为0.02 ng·mL^{-1}。批内、批间精密度(RSD)均小于9.72%，准确度(RE)在-2.61%～8.82%。本方法灵敏度高，专属性强，用于犬口服盐酸坦洛新控释片后的药代动力学研究。

关键词： 液相色谱-串联质谱法 盐酸坦洛新 控释片 药代动力学

Determination of tamsulosin in dog plasma by a high sensitive liquid chromatography-tandem mass spectrometric method

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Abstract:

To develop and validate a liquid chromatography-tandem mass spectrometric (LC-MS/MS) method for the determination of tamsulosin in dog plasma after oral administration of controlled-release tablet of tamsulosin hydrochloride, the samples and the internal standard, diphenhydramine, were extracted from dog plasma by *n*-hexane-dichloromethane (2∶1), and separated on a Bonchrom XBP-C_{18} column using a mobile phase consisted of methanol-acetonitrile-ammonium formate (10 mmol·L^{-1}) (30∶40∶30, *v*/*v*/*v*), at a flow rate of 0.4 mL·min^{-1}. Mass spectrometric detection was operated on a triple quadrupole tandem mass spectrometer equipped with atmospheric pressure chemical ionization (APCI) source in positive mode. Quantification was performed using selected reaction monitoring (SRM) of the transitions *m*/*z* 409→228 for tamsulosin and *m*/*z* 256→167 for the internal standard, respectively. The linear concentration ranges of the calibration curves for tamsulosin were 0.02-50 ng·mL^{-1}. The lower limit of quantification was 0.02 ng·mL^{-1}. The accuracy ranged from -2.61% to 8.82% in terms of relative error (RE). The intra- and inter-day relative standard deviation (RSD) across three-run validations were lower than 9.72%. The method was proved to be highly sensitive, selective, and had been successfully applied to the pharmacokinetic study after an oral administration of 0.4 mg tamsulosin hydrochloride controlled release preparations to dogs.

Keywords: tamsulosin hydrochloride controlled release tablet pharmacokinetics LC-MS/MS

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