

## 高效液相色谱-离子阱质谱法测定尿液中 $\beta_2$ -受体激动剂及 $\beta$ -受体阻断剂

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## Determination of $\beta_2$ -agonists and $\beta$ -blockers in urine using high performance liquid chromatography-ion trap mass spectrometry

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摘要

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**摘要** 建立了尿液中23种 $\beta_2$ -受体激动剂及5种 $\beta$ -受体阻断剂的高效液相色谱-离子阱质谱(HPLC-IT-MS)测定方法。尿液样品采用冷沉淀蛋白,上清液过Extrelut<sup>TM</sup>硅藻土柱,用乙酸乙酯洗脱后,洗脱液经旋转蒸发仪浓缩并复溶待测。HPLC分离采用AtlantisT3-150 mm 甲醇和含0.1%甲酸的水溶液为流动相梯度洗脱,IT-MS采用电喷雾离子源在多反应离子监测模式下测定。定量分析选择9种经过氘代的 $\beta_2$ -受体激动剂为内标。各化合物的线性范围为0.005~0.16 mg/L,尿液中的检出限均能达到0.2  $\mu$ g/L。空白尿液样品中不同加率为57.1%~127.1%,相对标准偏差为1.1%~31.1%。该方法简便快速,灵敏度高,适用于人或动物尿液中23种 $\beta_2$ -受体激动剂及阻断剂的定性和定量分析。

**关键词:** 高效液相色谱-质谱法  $\beta_2$ -受体激动剂  $\beta$ -受体阻断剂 尿液

**Abstract:** A method has been developed for the determination of 23  $\beta_2$ -agonists and 5  $\beta$ -blockers in urine samples using high performance liquid chromatography-ion trap mass spectrometry (HPLC-IT-MS). Urine samples were deproteinized by high-speed frozen centrifugation, and the supernatants were loaded on an Extrelut<sup>TM</sup> diatom column for clean-up. The analytes were eluted by ethyl acetate and concentrated for further analysis. The analysis and separation was performed on an AtlantisT3-150 mm chromatographic column with the gradient elution using methanol and water (containing 0.1% formic acid). The detection was carried on a linear ion trap mass spectrometer using multiple reaction monitoring (MRM) mode with the source operated in positive mode of electrospray ionization. Nine deuterium labeled  $\beta_2$ -agonists were used as internal standards for quantitative analysis. The results showed that the linear ranges for 23  $\beta_2$ -agonists and 5  $\beta$ -blockers were 0.005–0.16 mg/L, and the limits of detection were 0.2  $\mu$ g/L. The mixed standard solution was added into the blank urine samples, and the recoveries of 23  $\beta_2$ -agonists and 5  $\beta$ -blockers were ranged from 57.1% to 127.7% with the relative standard deviations of 1.1%-31.1%. This study demonstrates that the method is easy, fast, sensitive, and suitable for the confirmation and quantification of 23  $\beta_2$ -agonists and 5  $\beta$ -blockers in urine samples.

**Keywords:** high performance liquid chromatography-mass spectrometry (HPLC-MS)  $\beta_2$ -agonists  $\beta$ -blockers; urine

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