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论文

LC/MSⁿ法同时检测人尿液中艾司唑仑、阿普唑仑和三唑仑

顾景凯;夏荣;钟大放;孙璐

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摘要:

目的研究三氮唑苯并二氮 类药物的质谱断裂规律,建立可同时检测人尿液中艾司唑仑、阿普唑仑和三唑仑的液相色谱-质谱(LC/MSⁿ)联用法。方法用LC/MSⁿ技术,同时对3种三氮唑苯二氮 类药物进行色谱分离及质谱鉴定,并用质谱解析软件探讨该类化合物的裂解规律。结果3种药物的 $[M+H]^+$ 准分子离子均可生成脱去1分子 N_2 和1个CI原子的特征碎片离子,其最低检测限小于0.5 ng·mL⁻¹。结论该方法快速、灵敏、准确,完全适用于法医学和临床用药过量案例或病例的定性分析。

关键词: 液相色谱/电喷雾离子阱质谱法 艾司唑仑 阿普唑仑 三唑仑

IDENTIFICATION OF ESTAZOLAM, ALPRAZOLAM AND TRIAZOLAM IN HUMAN URINE BY LC/MSⁿ

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Abstract:

AIMTo investigate the fragmentation behavior of triazolobenzodiazepines and to develop a specific, sensitive and rapid LC/MSⁿ assay for simultaneous determination of estazolam, alprazolam and triazolam in human urine. METHODSAfter oral administration of a single 4 mg dose of the drugs to each of three healthy volunteers, urine samples were purified by solid-phase extraction, and then injected into an ODS column (150 mm×4.6 mm) with a mobile phase of methanol-water (8:2) for LC/MSⁿ analysis. The structures of estazolam, alprazolam and triazolam in human urine were identified by direct comparison of the observed mass spectra and the chromatographic retention time with those of the reference substance. The mass spectrometer (Finnigan LCQ) was operated in positive mode and in two scan modes including SIM and full scan MS/MS mode. The obtained mass spectra was analyzed assisted with the software Mass Frontier 1.0 for their fragmentation pathways. RESULTSThe full scan MS/MS spectra of each compound gave characteristic fragment ions of [M+H-N₂] ⁺ and [M+H-CI] ⁺. The detection limit was below 0.5 ng·mL⁻¹ for estazolam, alprazolam and triazolam in human urine. CONCLUSIONThe method is useful in forensic and clinical toxicology in which unequivocal identification of eatazolam, alprazolam and triazolam is desired.

Keywords: estazolam alprazolam triazolam LC/MSⁿ

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