Analysis of ketamine & norketamine in Urine by automatic Solid-Phase Extraction (SPE) and Positive ion chemical ionization–gas chromatography-mass spectrometry (PCI-GC-MS)

EUNMI KIM, JUSEON LEE, SANGKIL CHOI, MIAE LIM and HEESUN CHUNG

National Institute of Scientific Investigation, Korea

BACKGROUND: Ketamine(KT) is widely abused for hallucination and also misused as a "date-rape" drug in recent years. An analytical method using positive ion chemical ionization-gas chromatography-mass spectrometry(PCI-GC-MS) with an automatic solid-phase extraction(SPE) apparatus was studied for the determination of KT and its major metabolite, norketamine(NK), in urine.

METHODS: 6 KT suspected urine samples were provided by the police. For the research of KT metabolism, KT was administered to SD rats by i.p. at a single dose of 5, 10 and 20 mg/kg, respectively, and urine samples were collected during 24, 48 and 72 hr after administration. For the detection of KT and NK, urine samples were extracted on an automatic SPE apparatus(Rapid Trace, Zymark) with mixed mode type cartridge, Drug-Clean™(200 mg, Alltech). The identification for KT and NK was used by PCI-GC-MS. M/z 238(M+1), 220 for KT, m/z 224(M+1), 207 for NK and m/z 307(M+1) for Cocaine-D_3 as internal standard were extracted from the full scan mass spectrum and the underlined ions were used for quantitation.

RESULTS: Extracted calibration curves were linear from 50 to 1000 ng/mL for KT and NK with correlation coefficients exceeding 0.99. The limit of detection(LODs) was 25 ng/mL for KT and NK. The limit of quantitation(LOQs) was 50 ng/mL for KT and NK. The recoveries of KT and NK at 3 different concentrations(86, 430 and 860 ng/mL) were 53.1 to 79.7% and 45.7 to 83.0 %, respectively. The intra- and inter-day run precisions(CV) for KT and NK were less than 15.0 %, and the accuracies(bias) for KT and NK were also less than 15% at the 3 different concentration levels (86, 430 and 860 ng/mL). The analytical method was also applied to real 6 KT suspected urine specimens and KT administered rat urines, and the concentrations of KT and NK were determined. And dehydronorketamine(DHNK) was also confirmed by these urine samples, however the concentration of DHNK was not calculated. SPE is simple, and need less organic solvent than liquid-liquid extraction(LLE), and PCI-GC-MS can offer both qualitative and quantitative information for urinanalysis of KT in the forensic science.

KEYWORDS: *Urinanalysis of ketamine, SPE, PCI-GC-MS*

Corresponding author: emkim@nisi.go.kr