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CZE-ESI-TOF MS applied to hair analysis in the screening for drugs of abuse

ROSSELLA GOTTARDO, AMERIGA FANIGLIULO, JENNIFER P. PASCALI, FRANCO TAGLIARO

Department of Medicine and Public Health, Unit of Forensic Medicine, University of Verona, Verona, Italy

INTRODUCTION AND AIM: The combination of the high efficiency, simplicity and rapidity typical of capillary zone electrophoresis (CZE) with the high sensitivity and the accuracy of mass determination offered by time-of-flight (TOF) mass spectrometry (MS) is very attractive in forensic toxicological analysis. In addition, TOF MS providing "full scan" data, allows for the post-run retrieval of compounds which had not been targeted before the analysis, thus meeting a fundamental need of the forensic toxicological investigation. On the other hand, hair analysis has become a well established analytical tool to investigate retrospectively drug abuse histories.
The purpose of the present work was the development of a rapid CZE-TOF-MS method for the determination of the major drugs of abuse and their metabolites (namely, 6-monoacetyl-morphine, morphine, amphetamine, methamphetamine, MDA, MDMA, benzoylecgonine, ephedrine and cocaine) in hair samples.

MATERIALS AND METHODS: Hair samples (100 mg) from drug users, subjects under chronic treatment with psychoactive drugs, from corpses subjected to autopsy and negative control subjects were washed, cut and incubated overnight in 0.25 M HCl at 45ºC then neutralized with NaOH and extracted by LLE. CZE separations (PACE 5500, Beckman Coulter, Fullerton, CA) were carried out in a 100 cm × 75 µm (ID) uncoated fused silica capillary. The separation buffer was composed of 25 mM ammonium formate, pH 9.5; the separation voltage was 15 KV. Electrokinetic injections were performed at 7 KV for 20 seconds. ESI-TOF MS detection (MicroTOF, Bruker Daltonics, Bremen, Germany) was performed in the ESI positive ionization mode using the following conditions: capillary voltage 4 KV, nebulizer gas (nitrogen) pressure 0.6 bar, source temperature 200ºC and drying gas (nitrogen) flow rate 5 L/min. The sheath liquid was composed of isopropanol-water (50:50, v/v) with 0.5% formic acid, delivered at a flow rate of 4 µL/min. Nalorphine was used as internal standard.

RESULTS AND DISCUSSION: Under the described conditions, the separation of 6-monoacetylmorphine, morphine, amphetamine, methamphetamine, MDA, MDMA, benzoylecgonine, ephedrine, cocaine, nicotine, methadone and several other compounds of toxicological interest was achieved in 20 minutes. The TOF mass spectrometer provided excellent selectivity based on accurate mass determinations (average accuracy < 5ppm) suitable for drug identification, which was also confirmed by the recording of the isotopic pattern. The limits of detection were lower than that required for determining accurately drug concentrations in hair around the established cut-offs (i.e. 0.1 ng/mg). Separation efficiency ranged from 0.5 to 1.7X106 theoretical plates, clearly much higher than that obtained with the commonly used LC-MS methods.

On these grounds, the described method can be proposed for rapid, selective and accurate toxicological screening of hair for both clinical and forensic purposes.

KEYWORDS: *Hair analysis, Drugs of abuse, Capillary electrophoresis, TOF*

Corresponding author: gotross@inwind.it