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A tendency for re-offending in drug-facilitated crime☆

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ABSTRACT

The authors present 3 cases that demonstrate a return to DFC following periods of inactivity. The offences occurred in Paris and its suburbs and in each of the cases there were two distinct periods of activity by the offenders with 2, 8 and 22 victims attributed to each of the perpetrators.

To 20 mg of decontaminated and cut hair, 100 pg/mg of clonazepam-d4 was added as internal standard. Hair specimens were extracted with CH_2Cl_2/ether after incubation overnight at 56 °C in pH 7.6 buffer. Extractions were performed on blood and urine using Toxi-tube A® with 5 ng/mL of clonazepam-d4. The residues were analyzed by LC–ESI-MS/MS. Calibration curves in blood and urine (0.5–500 ng/mL) were prepared by spiking aliquots of blank fluids ($r^2 > 0.9816$ for all drugs). LOD in body fluids ranged 0.5–10 ng/mL. Calibration curves in hair (0.5–100 pg/mg) were prepared by spiking aliquots of blank hair ($r^2 > 0.9877$ for all drugs). LOD in hair ranged 0.5–5 pg/mg.

Case #1: Two young women were raped with an interval of approximately 1 year between the incidents. Lorazepam (present, <2 pg/mg) was detected in hair obtained from the first victim, and zolpidem (19 pg/mg) in hair of the second one. The offender was in jail between the two offences. *Case #2*: The offender approached a total of 8 men and women who were aged over 50 years. The offender was in jail between the two series of respectively 3 and 5 victims. Zopiclone was detected in victims' hair ($n = 7$) at concentrations 13–42 pg/mg. *Case #3*: The offender stole thousands of Euros using credit cards obtained from 22 different wealthy victims. He employed a cocktail of up to 6 drugs made up of: flunitrazepam, clonazepam, doxylamine, cyamemazine, zolpidem and lorazepam. Drugs were detected in all victims' hair ($n = 18$) at concentrations in the range 1–81 pg/mg for all drugs. Between the two series (of respectively 4 and 16 victims) the offender spent 6 months in jail, and then police spent 6 months looking for him while he was under judiciary control prior to his judgment.

Segmental hair analysis permits retrospective information on drug exposure and should be considered in the investigation of drug-facilitated crimes not only to prove single exposure but also when there has been any appreciable delay in samples being obtained for analysis. Indeed, in 56% cases reported in this paper, due to the long time that elapsed between offences and the opportunity to obtain samples for analysis hair analysis was considered the only viable matrix to investigate the possibility of drug involvement in the crimes.

Our experience demonstrates that the incidence of re-offending in DFC after a period of inactivity (often due to imprisonment) may be of concern, notably in big cities.

1. Introduction

Currently liquid chromatography–tandem mass spectrometry with electrospray ionization (LC–ESI-MS/MS) has proven to be the most appropriate analytical method for the qualitative and quantitative analysis of drug traces in hair as well as in blood and urine. We have employed this method for several years in the segmental hair analysis as this can provide retrospective information that can prove useful in the assessment of drug-facilitated crimes (DFC) in forensic case reports [1]. The authors present 3 cases that demonstrate a return to DFC following periods of inactivity.

The offences occurred in Paris and its suburbs and in each of the cases there were two distinct periods of activity by the offenders with respectively 2, 8 and 22 victims attributed to each of the perpetrators. Each perpetrator demonstrated their own *modus operandi* – sexual abuse, money robbery, and credit cards pin code extortion. The victims were also of the same kind in each group, respectively: women, more than 50-year-old people, and young men.

In most of the cases, hair was considered the only viable matrix to investigate the possibility of drug involvement in the crimes, given the long time that elapsed between offences and the

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opportunity to obtain samples for analysis. For this reason, this paper focuses mainly on hair analysis.

The aim of this paper is also to report on drugs administrated without one's knowledge for criminal purpose, thus analytical results for voluntary reported alcohol or illicit drugs intake, and other psychotropic declared treatment are not reported.

2. Materials and methods

Biological samples and toxicological analysis were performed based on request of the police, a public prosecutor or a judge. When victims were examined soon after the offence by a general practitioner in a forensic emergency unit, blood and urine were collected. The information recorded included the details of the complaint and sometimes the toxicological screening findings performed at the hospital (e.g. urine analysis by immunoassay).

2.1. Blood and urine collection

Plain blood and urine were collected just after the medical examination, according to the consensus of the French Society of Analytical Toxicology (*Société Française de Toxicologie Analytique*) [2]. All the samples were refrigerated at +4 °C before analysis. After analysis, the remaining specimens were kept frozen.

2.2. Hair samples collection

When possible, scalp hair was collected between 4 and 8 weeks after the alleged DFC, and cut close to the skin in the vertex posterior position. When scalp hair was not available or too short, pubic or leg hair was collected.

2.3. Methods

2.3.1. Standards and reagents

Drug standards were obtained from Cerilliant (LGC standards, France). Ammonium formate (min. 98%), formic acid (99%), methanol and 0.1N sodium hydroxide for analysis were obtained from Carlo Erba, France. Acetonitrile (HPLC grade RS Plus) was obtained from Carlo Erba, France. Dichloromethane and ether (Chromanorm HPLC grade) were obtained from Prolabo, France. PTFE filters (0.2 μm × 25 mm) were obtained from Alltech, France. Soerensen buffer was adjusted at pH 7.6, as previously described [3].

2.3.2. Instrumentation

Specific screening for DFC drugs was performed by LC–ESI-MS/MS. As previously described [3] and briefly summarized hereunder, the liquid chromatographic separation was carried out using a ThermoElectron Surveyor system, fitted with an Uptisphere ODB C18 column (150 mm × 2 mm, 5 μm) (Interchim, France). The mobile phase consisted of a mixture of acetonitrile and 2 mM formate buffer (pH 3). It was delivered at a flow rate of 200 μL/min under the following conditions: 15:85 (v/v) held 0.5 min, then increased to 10:90 (v/v) up to 10 min and re-equilibrated for 5 min at initial conditions.

The detection was performed on a triple stage quadrupole (TSQ Quantum Ultra, ThermoFisher), fitted with an electrospray ionization (ESI) "Ion Max" source, and allowed the simultaneous detection of benzodiazepines, minor tranquilizers, antihistaminics and neuroleptics, in positive polarity and selected reaction monitoring mode [3,4].

2.3.3. Extractions

Blood and urine: For each type of specimen, 1 mL was extracted with Toxi-tube A® (Varian), adding 5 ng clonazepam-d_4 as internal standard (IS). After horizontal agitation and centrifugation, the extract was evaporated to dryness. The residues were reconstituted by 100 μL of MeOH/ACN/formate buffer pH 3. Ten microliters were injected into the chromatographic system.

Hair: For the screening of DFC drugs procedure, hair strands were segmented in 2-cm-long segments, then washed twice with dichloromethane for cleaning fat residues, and finely cut with scissors. An intensive wash is not generally employed for traces level detection in hair.

Two nanograms clonazepam-d_4 (IS) were added to 20 mg of sample, followed by incubation 14 h at 56 °C in Soerensen buffer pH 7.6. Liquid–liquid extraction was carried out at ambient temperature for 10 min with 2 mL of dichloromethane/ether (80/20, v/v). After centrifugation, the organic layer was filtered through a 0.2 μm PTFE filter, and then evaporated. Each residue was dissolved in 100 μL of MeOH/ACN/formate buffer pH 3.

2.3.4. Method validation

Blood and urine: For each analyte, calibration curves were prepared with blank blood and urine at concentrations ranging from 0.5 to 500 ng/mL. Curves were linear in that range and r^2 were always higher than 0.9816. When out of the calibration range, real urine samples were diluted before re-analysis.

The limits of detection (LOD) were evaluated with decreasing concentrations of the drugs spiked in drug-free blood and urine, until a response equivalent to three times the background noise was observed. LOD in body fluids ranged 0.5–1 ng/mL for all analytes. The limits of quantification (LOQ) were considered the concentration of analyte at which the signal-to-noise ratio of the quantification ions was at least 10. LOQ in body fluids ranged 1–5 ng/mL.

Intra-day ($n = 6$) and inter-day ($n = 18$) precision were less than 20% in the whole range except at the LOQ where precision was accepted at less than 25%. Accuracy was less than 30%. Specificity was tested with 6 different blank matrix, a blank spiked with the internal standard, and a blank spiked with the internal standard plus the analytical standards.

Hair: Drug-free hair samples were obtained from laboratory staff and relatives. The determination of benzodiazepines and analogs in hair was fully validated according to the French and European Norm (NF EN) ISO/CEI 17025 quality standards. Calibration curves were prepared with drug-free hair spiked at concentrations ranging from 0.5 to 100 pg/mg. Curves were linear in that range and r^2 were always higher than 0.9877. LOD in hair ranged 0.5–2 pg/mg for benzodiazepines and analogs, and ranged 1–5 pg/mg for other sedatives. LOQ in hair ranged 0.5–5 pg/mg for benzodiazepines and analogs, and ranged 5–10 pg/mg for other sedatives.

Intra-day ($n = 6$) and inter-day ($n = 18$) precision were less than 20% in the whole range except at the LOQ where precision was accepted at less than 25%. Accuracy was less than 40%. Specificity was tested with 6 different blank matrix, a blank spiked with the internal standard, and a blank spiked with the internal standard plus the analytical standards.

3. Results from case reports and discussion

For each individual, hair measurements were done on a relevant growth area with at least three 2-cm-long segments, and focused around the alleged time of the offence, estimated with an approximate growth rate of 1 cm/month. No segmentation was done on body hair. The following reported concentrations were from single measurement for each individual's hair.

Case #1: Two young women, aged 25 and 27 years, were raped with an interval of approximately 1 year between the separate incidents. Each victim reported eating a strawberry tartlet which they suspected had been adulterated with a psychotropic drug. Both victims had met the perpetrator recently before the offence and both had fallen asleep suddenly after eating the tartlet. Lorazepam was detected in the blood and the urine collected at +24 h on the first victim (Table 1) at concentrations in agreement with the recent intake of lorazepam. Lorazepam was detected at trace level (<2 pg/mg) in her hair only in one segment, matching to the period of the offence. Blood and urine were not collected for the second victim because she lodged her complaint more than 5 days after the offence. However a low concentration of zolpidem was detected in the segment of hair corresponding to the period of the offence (Table 1).

The offender was in jail between the two offences. He admitted both crimes.

Table 1
Results for screening of DFC drugs in blood, urine and/or hair specimens of the 2 victims, case #1.

Case #1		Blood	Urine	Head hair
Victim #1	Delay before sampling	24 h	24 h	2 months
	Lorazepam	16 ng/mL	1041 ng/mL	Traces <2 pg/mg
Victim #2	Delay before sampling	ns	ns	1.5 months
	Zolpidem	–		19 pg/mg

ns: not sampled.