

TECHNICAL NOTE

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The Detection of Opiate Drugs in Nontraditional Specimens (Clothing): A Report of Ten Cases

REFERENCE: Tracqui, A., Kintz, P., Ludes, B., Jamey, C., and Mangin, P., "The Detection of Opiate Drugs in Nontraditional Specimens (Clothing): A Report of Ten Cases," *Journal of Forensic Sciences*, JFSCA, Vol. 40, No. 2, March 1995, pp. 263–265.

ABSTRACT: We present a series of 10 fatalities involving opiate overdose, in which morphine, codeine, and 6-monoacetylmorphine were identified and quantified, not only in postmortem biological samples, but also in pieces of underwear taken from the bodies. Small tissue samples (about 1 g) were cut off from several parts of the underwear, stored at ambient temperature until analysis, then extracted by agitation in a mixture of chloroform/2-propanol/*n*-heptane (60:14:26, v/v/v) and assayed using GC/MS in the single ion monitoring mode. Morphine, codeine and 6-monoacetylmorphine concentrations were in the range 0.02 to 9.27 $\mu\text{g/g}$. These results indicate that the impregnation of underwear by sweat and sebaceous secretions and/or urine provides detectable levels of the drugs excreted by these ways. Even in the absence of biological samples, assaying pieces of clothing may bring some evidence about the drug abuser status of their owner.

KEYWORDS: toxicology, underwear, clothes, opiates, GC/MS, chromatographic analysis

The examination of clothes represents an important step of the forensic investigations done on cadavers, especially in the areas of ballistics (search for bullet perforations, gas burns, or powder tattooing), or serologic/genetic individual identification (from blood or semen stains). Up to now, however, very few had been reported on the potential interest of garment samples in the specific field of forensic toxicology.

The aim of this preliminary study was to determine whether the toxicological analysis of underwear may be able to bring some retrospective evidence about a previous intake of pharmaceuticals or drugs of abuse.

Materials and Methods

Subjects

The study was carried out on a population of ten known drug abusers (eight males and two females), aged 19 to 37, deceased

Received for publication 4 March 1994; revised manuscript received 18 July 1994; accepted for publication 19 July 1994.

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under circumstances suggesting opiate overdose, and consecutively autopsied at the Medicolegal Institute in Strasbourg between April 15th, 1993 and January 20th, 1994. Apparition of putrefactive features on the body or presence of biological stains (blood, vomit) on the garment were rejection criteriae. In addition to the classical samples (blood, urine, bile, gastric contents, and hair), small tissue samples (about 1 g each) were systematically cut off with clean stainless steel scissors from several parts of the underwear (front and/or lateral parts in underpants or panties; axillary zones in shirts or T-shirts), weighed, then stored under plastic cover at ambient temperature until the day of analysis.

Materials

Chloroform, 2-propanol and *n*-heptane were HPLC grade and purchased from Merck (F.R.G.); all other chemicals were analytical grade and provided by Merck (F.R.G.) and Prolabo (France). The deuterated standards D₃-morphine, D₃-codeine, and D₃-6-monoacetylmorphine were obtained from Radian (U.S.A.).

Chromatography

All analyses were performed on a Hewlett-Packard (U.S.A.) GC/MS system, consisting of a HP 5890 chromatograph equipped with a HP 7693 autosampler and a HP 5972 mass selective detector operated at 70 eV (ion source temperature 180 to 190°C). The electron multiplier voltage was set at +400 V above autotune voltage; the detector was daily autotuned with perfluorotributylamine. The column was a HP-5 MS (5% phenyl/95% methyl siloxane; 30 m \times 0.25 mm, i.d.). The carrier gas was He (purity grade N55; flow rate 1.8 mL/min). Injection was performed in the splitless mode. Injector and detector temperatures were 270°C and 280°C, respectively; the column oven temperature was programmed to rise from an initial value of 60°C (maintained 1 min) up to 295°C (ramp + 30°C/min), then kept at 295°C for the final 8 min. The ions monitored and typical retention times (RT) for the different drugs assayed and the deuterated internal standards were as follows: morphine and D₃-morphine, m/z 429 and 432, respectively (RT = 11.21 min); codeine and D₃-codeine, m/z 371 and 374, respectively (RT = 10.92 min); 6-monoacetylmorphine (6-MAM) and D₃-6-MAM, m/z 399 and 402 (RT = 11.58 min). Analytes were identified and quantified based upon comparison of retention times and relative abundance of two confirming ions to the deuterated internal standards.

TABLE 1—Opiate concentrations in the biological and textile samples. (All values in µg/L fluid or µg/kg dry tissue.)

Subject (sex, age)	Cause of Death	Delay autopsy tissue analysis	Sample	Morphine	Codeine	6-MAM
No 1 (M, 27)	Heroin overdose	13 days	Blood	214	66	...
			Urine	1458	337	112
			Underpants (2 pieces)	24–37
No 2 (M, 32)	Codeine overdose	6 days	Blood	...	2104	...
			Urine	780	1277	...
			Shirt	...	1020	...
			Underpants	...	634	...
No 3 (M, 27)	Heroin overdose	9 days	Blood	346	21	...
			Urine	1287	147	354
			Underpants (2 pieces)	8074–1917	358–119	9269–844
No 4 (M, 23)	Heroin overdose	1 day	Blood	293	31	...
			Urine	4357	344	156
			Underpants (2 pieces)	4321–2347	4330–448	6341–7951
No 5 (M, 32)	Heroin overdose	75 days	Blood	311	67	...
			Urine	8427	356	814
			T-Shirt	35	1567	...
			Underpants	22	377	...
No 6 (F, 37)	Heroin overdose	65 days	Blood	188	38	...
			Urine	4321	443	281
			T-shirt	239
No 7 (M, 30)	Heroin overdose	261 days	Blood	450	100	...
			Urine	68	148	244
			T-Shirt	2387	684	581
			Underpants (2 pieces)	68–487	0–358	0–212
No 8 (F, 19)	Heroin overdose	14 days	Blood	292	53	...
			Urine	1869	218	110
			T-Shirt (2 pieces)	27–147	231–531	163–288
No 9 (M, 24)	Heroin overdose	24 days	Blood	145	26	...
			Urine	2741	1246	387
			Underpants	48	26	39
No 10 (M, 25)	Heroin overdose	16 days	Blood	389	37	...
			Urine	2731	562	399
			T-Shirt	89	27	644
			Underpants	681	67	231

Procedure

To approximately 1 g of textile sample or 1 mL biological fluid (blood, urine) in 30 mL Pyrex centrifuge tubes were added the deuterated opiate standards (D_3 -morphine, D_3 -codeine, D_3 -6-MAM; 200 ng each), 2 mL of a saturated $(NH_4)_2HPO_4$ buffer, pH 8.4, and 10 mL of the extracting solvent (chloroform/2-propanol/*n*-heptane, 60:14:26, v/v/v). The mixture was vigorously shaken on a horizontal agitator for 10 min, then centrifuged at 2800 g for 10 min. After purification of the organic phase by an additional acid extraction (5 mL of 0.2 M HCl), the aqueous layer was re-extracted with 2 mL $(NH_4)_2HPO_4$ buffer, pH 8.4, 1 mL 1M NaOH, and 5 mL chloroform. After agitation and centrifugation, the final organic phase was removed and evaporated to dryness at 45°C in a rotary evaporator (Speed Vac Concentrator mod. A 290, Savant Instruments, U.S.A.). Derivatization was performed by adding 20 µL BSTFA + 1% TMCS to the dry extract (70°C, 20 min), then 2 µL of the obtained sample were injected into the GC column.

Hair samples were prepared and extracted according to previously reported procedures [1].

Results and Discussion

The results of the toxicological investigations are summarized in Table 1. As shown, blood and urine analyses clearly ruled the

cause of death to be opiate overdosage for the 10 subjects (heroin: 9 cases; codeine: 1 case). In addition, hair analysis revealed in all cases a past history of chronic drug abuse.

Opiate (morphine and/or codeine and/or 6-MAM) were shown to be present in all pieces of garment tested, with concentrations ranging from 0.02 to 9.27 µg/g of dry tissue sample; in particular, 6-MAM could be detected in 7 from the 9 subjects deceased from heroin overdosage (Fig. 1). In subjects 5, 6, and 7 the toxicological analyses (that had to be delayed due to necessities of police inquiries) gave positive results although they had been performed respectively 75, 65, and 261 days after garment sampling, which may suggest some long-term stability of the drugs tested after they got trapped onto the textile fibers.

At least 2 excretory mechanisms seem to be implicated: It is likely that the presence of drugs in underpants samples constitutes a result of ante or postmortem urine losses mainly; the finding of the same drugs in shirts or T-shirts (subjects 2, 5–8, 10) clearly shows that a sweat/sebaceous excretory component is also involved. In fact, the ability of sweat glands to excrete drugs taken orally or intravenously has been established for a long time, for various compounds such as sulphur, iodine, copper, quinine [2], ethanol [3], salicylic acid, antipyrine, methylene blue [4], sulfonamides [5,6], methadone and metabolites [7,8], phenobarbitone [9], morphine [10], cocaine and metabolites [11,12], and cannabinoids [13].

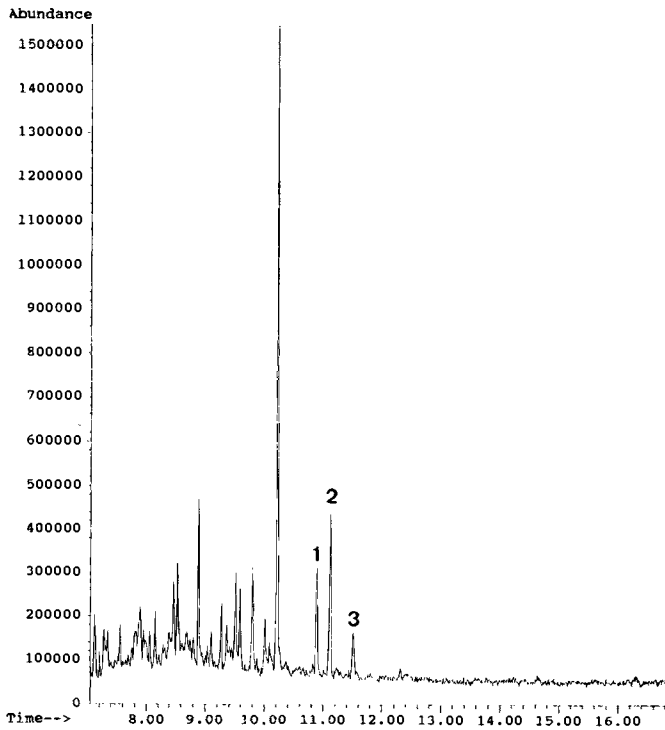


FIG. 1—Total ion chromatogram of the T-shirt extract from subject Nb. 10. Peak 1: codeine (27 ng/g) + D_3 -codeine; Peak 2: morphine (89 ng/g) + D_3 -morphine; Peak 3: 6-MAM (644 ng/g) + D_3 -6-MAM.

The quantitative data appear inconsistent, notably no relationship seems to exist between drug levels in biological fluids and textile samples. This is in fact not surprising, since 1) tissue levels represent the result of a cumulative drug excretion over a variable period of time from one subject to another, 2) several excretory mechanisms are probably involved, 3) the nature (thus the binding capabilities?) of the textile fibers was very variable. In addition, little is known about the mechanisms and rates of sweat/sebaceous excretion, but large interindividual variations probably exist as for other metabolic pathways.

Conclusion

These preliminary results indicate that impregnation of underwear by sweat and sebaceous secretions and/or urine provides detectable levels of opiate excreted by these ways. Even in the absence of biological samples, assaying pieces of garment (for example, underwear found in an abandoned habitation) may bring some evidence about the drug abuser status of their owner—a somewhat exciting prospect. Further studies under experimental

conditions (healthy volunteers, known amount of drug taken and standardized sampling material for example, skin patches [12]) are necessary in order to minimize the different bias and allow quantitative interpretation of the analytical data.

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