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Note

A simplified procedure for the identification of drugs from the illicit street market by thin-layer chromatography

In the last few years, some methods for the analysis of drugs of abuse have been described[1–6]. Thin-layer chromatography (TLC) is most useful for the rapid identification of these drugs. An extensive review article on this subject was published by KAISTHA[7]. A rapid screening procedure by TLC was introduced by BROWN *et al.*[8], in which the screening is restricted to the amphetamines and psychoactive compounds. Recently, FILEDT KOK AND KAMP[9] described a procedure for the identification of drugs and the results of an illicit drug analysis programme. They used several solvent systems: a general screening system[10], a system to determine the purity of LSD samples with specially prepared alkaline TLC plates[11] and one particularly suitable for the analysis of opiates[12]. In addition to these methods, a system for the separation of barbiturates was developed in this laboratory[13]. We succeeded in preparing a solvent system that combines the possibilities of all four of these systems.

Preparation of plates

The TLC plates (20 × 20 cm) were coated with Silica Gel G (Merck) to a thickness of 0.25 mm according to STAHL's method[14]. For activation, the plates were heated at 110° for 2 h and stored in a desiccator.

Solvent system

The solvent system developed had the following composition: chloroform–diethyl ether–methanol–25 % ammonia solution (75:25:5:1).

Spray and colour reagents

Iodide. A 1 % solution in methanol (Imeth).

Sulphuric acid–ethanol spray reagent. A mixture of equal parts of concentrated sulphuric acid and 96 % ethanol.

Iodoplatinate spray reagent (IPA). A volume of 10 ml of 10 % hexachloroplatinic acid mixed with 250 ml of a 4 % potassium iodide solution and diluted with distilled water to a final volume of 500 ml.

p-Dimethylaminobenzaldehyde spray reagent (DMBA). An amount of 0.5 g of *p*-dimethylaminobenzaldehyde dissolved in a mixture of 53 ml of concentrated sulphuric acid and 50 ml water. To this solution, 0.5 ml of iron(III) chloride solution (10.5 % aqueous iron(III) chloride in water) was added.

Diphenylcarbazone–mercury(II) chloride[15] *(DCHg).* Equal parts of 2 % mercury (II) chloride in ethanol and 0.2 % diphenylcarbazone in ethanol.

Sulphuric acid–formaldehyde test. Ten drops of 40 % formaldehyde solution added to 10 ml of concentrated sulphuric acid (Marquis reagent).

Alkaline cobalt test. A 2 % cobalt nitrate solution in methanol and 25 % isopropylamine solution in methanol.

Standard solutions

D-Amphetamine sulphate, DL-methylamphetamine hydrochloride, phenmetrazine hydrochloride, ephedrine hydrochloride: 1 mg/ml. Lysergic acid diethylamide (LSD) tartrate, N,N-dimethyltryptamine (DMT), N,N-diethyltryptamine (DET): 0.5 mg/ml. DL-1-Methyl-2-(2,5-dimethoxy-4-methylphenyl)ethylamine (STP, DOM), DL-1-methyl-2-(2,5-dimethoxy-4-ethylphenyl)ethylamine (DOET): 2 mg/ml. All of these were used as solutions in 1 % tartaric acid. Psilocybin (Indocybin, ampoules of 3 mg/ml, Sandoz). Phencyclidine (Sernylan, bottles of 20 mg/ml, Parke-Davis). Allobarbital, cyclobarbital, phenobarbital, methylphenobarbital, butobarbital, barbital: 5 mg/ml in 96 % ethanol. The salts of the other compounds listed in Table I were dissolved in 1 % tartaric acid (2 mg/ml).

Procedure

A little of the solid sample was dissolved in one drop of 1 % tartaric acid. If the result of the alkaline cobalt test was suspect for a barbiturate, some of the sample was dissolved in one drop of 96 % ethanol. The samples were applied 2 cm from the bottom together with standard solutions. The choice of the standard solutions depended on the results of the previously performed sulphuric acid–formaldehyde test. The spots were air-dried and the plates were placed in the developing tank. The solvent was allowed to travel a distance of 10 cm (*ca.* 40 min). The plates were removed and dried under a stream of warm air. The dried plates were examined under UV light (366 nm) and the fluorescent areas marked. The plates were then sprayed with different reagents. The amphetamines and phenmetrazine were sprayed with Imeth; the spots of LSD, DMT, DET and psilocybin were made visible with DMBA reagent; the barbiturates were sprayed with DCHg. Other drugs were treated with the sulphuric acid–ethanol spray reagent and subsequently with IPA. The results are summarized in Table I.

Results and discussion

The separation obtained with the solvent mixture was adequate for the identification of drugs that are commonly offered on the illicit market (Table I). The procedure takes only 1 h. To obtain reproducible results, it is necessary to prepare the solvent system accurately.

It should be pointed out that the amphetamines and phenmetrazine gave a colour after treatment with sulphuric acid–ethanol and IPA if the samples were used as pure substances. However, amphetamine and phenmetrazine are frequently used as tablets and in these cases only a poor colour developed with IPA. However, satisfactory results were obtained with Imeth.

The opium alkaloids, heroin and 6-monoacetylmorphine were distinctly separated. The colour of morphine was a unique bright greyish blue.

The spot of LSD showed a bright blue fluorescence under UV light, before spraying with DMBA. The green fluorescence of DMT and DET appeared after treatment with DMBA. The green colour of these spots changed into dark blue within a few hours.

In many preparations of LSD, one to three other DMBA-positive compounds

TABLE I

CHARACTERISTICS OF DRUGS OF ABUSE

Drug	*Sulphuric acid–formaldehyde test*[a]	*Volume of standard solution applied (µl)*	*UV (366 nm)*	*Primary location reagent*	*Colour*	*R_F value*
Amphetamine	Or→Br	10		Imeth		0.58
				IPA	Bl	
Methylamphetamine	Or→Br	10		Imeth		0.50
				IPA	Bl	
Phenmetrazine	Gr	10		Imeth		0.66
				IPA	Bl	
Ephedrine	Ye	10		IPA	Pu	0.30
Methyl phenidate		10		IPA	PuRe	0.84
Caffeine		10		IPA	Pi	0.82
Cocaine		10		IPA	Pu	0.94
Procaine		10		IPA	PuBl	0.70
Morphine	Pu	10	Bl	IPA	Bl	0.18
Codeine	PuBl	10		IPA	PuBr	0.31
Thebaine	BrYe	10		IPA	PuBr	0.80
Noscapine	Ye→Pu	10	Bl	IPA	Pu	0.97
Papaverine	Pu→BrYe	10		IPA	Pu	0.92
Narceine	BrYe→Br	10		IPA	Pi	0.06
Heroin	Pu	10		IPA	dark Bl	0.79
6-Monoacetylmorphine[b]	Pu			IPA	dark Bl	0.54
LSD[c]	Grey	10	Bl	DMBA	Bl[f]	0.85
Lysergamide[d]			Bl	DMBA	Bl[f]	0.67
Psilocybin	Or	4		DMBA	Bl[f]	0.03
Psilocine[e]				DMBA	PuBl[f]	0.58
DMT	GrYe	10	Gr	DMBA	Gr[f]	0.43
DET	GrYe	10	Gr	DMBA	Gr[f]	0.58
Mescaline	Or	10		IPA	PuRe	0.28
STP	Gr	10		IPA	PuRe	0.42
DOET	Gr	10		IPA	PuRe	0.44
Phencyclidine		1		IPA	Pu	0.97
Strychnine		10		IPA	Pu	0.48
Diphenhydramine	Ye	10		IPA	BlPu	0.55
Allobarbital		3		DCHg		0.46
Barbital		3		DCHg		0.47
Butobarbital		3		DCHg		0.58
Cyclobarbital		3		DCHg		0.50
Methylphenobarbital		3		DCHg		0.79
Phenobarbital		3		DCHg		0.33

[a] Pu = purple; Bl = blue; Re = red; Or = orange; Br = brown; Pi = pink; Ye = yellow; Gr = green. The colours with Imeth and DCHg are not characteristic.
[b] Degradation product of diacetylmorphine in heroin samples from the illicit market.
[c] R_F values of other DMBA-positive compounds in illicit LSD preparations: 0.71, 0.64 and 0.53.
[d] Extracted from morning glory seeds (*Rivea corymbosa*) with 96% ethanol.
[e] From psilocybin during the run of the solvent mixture.
[f] Colour develops faster during heating.

could be detected (Table I). These samples are not suitable for colorimetric or fluorimetric determination of the LSD content.

Phencyclidine showed a purple spot after treatment with sulphuric acid–ethanol and IPA. After spraying with IPA only, the spot had a pink colour with a blue rim.

Conclusions

After the identification of 200 samples obtained from the illicit market during the last 7 months, the chromatographic system described in this paper has proved to be a suitable and reliable screening method. This method offers the possibility of the chromatographic analysis of the following drugs of different chemical natures with one solvent mixture: amphetamines and other central stimulants, psychoactive drugs, impurities in LSD samples, opium alkaloids, heroin and 6-monoacetylmorphine, barbiturates and some other compounds offered as an amphetamine, a psychoactive compound, and cocaine. Furthermore, a particular solvent system for the examination of hashish samples contaminated with opium, as used up to now, is no longer necessary, although it is advisable to use other methods in addition for definite identifications.

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