Journal of Chromatography, 115 (1975) 678–681

CHROM. 8681

Note

Rapid screening procedures for some street drugs by thin-layer chromatography

An evaluation

WARREN JAMES WOODFORD

Department of Chemistry, Emory University, Atlanta, Ga. 30322 (U.S.A.)

(Received July 28th, 1975)

The author was employed by an attorney to identify a white powder confiscated by the State of Georgia. The State's crime laboratory facilities and access to authentic drug standards were provided by court order.

An initial screen of the unknown powder using thin-layer chromatography (TLC) was deemed appropriate, and the methodology presented in two recent publications[1,2] was selected. The two articles describe the characteristics indicative of several drugs of high abuse potential after TLC.

The experimental technique, common to both articles, consists of extracting selected drugs (pure and mixed authentic standards) into 95% ethanol. After application of a quantity of a drug or mixture to a pre-coated thin layer of silica gel, the plate is eluted with a prescribed solvent mixture. Finally, spots are detected and identified by their R_F values and color reactions with indicator sprays or ultraviolet light. The results of those experiments are reproduced in Tables I and II. Note that Table II lacks color reaction data of the "caine" drugs with iodoplatinic acid (IPA) and no acidic iodoplatinic acid (AIPA) colors are reported for the drugs in Table I.

TABLE I

CHARACTERISTICS OF DRUGS REPORTED IN REF. 1 AFTER TLC

Drug	R_F	*Color with*		
		*IPA**	*PDAB***	*UV (254 nm)*
Psilocybin	0.00	purple-brown	purple-blue	—
N-Monomethyltryptamine	0.21	purple-blue	blue	—
N,N-Dimethyltryptamine	0.48	blue	blue	—
LSD tartrate	0.69	purple	blue	blue
Strychnine sulfate	0.32	purple-blue	—	—
Mescaline sulfate	0.27	purple	—	—
DL-Methamphetamine	0.37	blue	—	—
D-Amphetamine	0.49	blue	—	—
Phencyclidine	0.79	dark purple	—	—

* IPA = iodoplatinic acid.

** PDAB = *p*-dimethylaminobenzaldehyde.

TABLE II

CHARACTERISTICS OF DRUGS REPORTED IN REF. 2 AFTER TLC

Drug	R_F	*Color with*	
		*AIPA**	*PDAB*
Heroin	0.45	dark brown	—
Tetracaine	0.55	grey-violet	—
Procaine	0.70	blue-violet	yellow
Cocaine	0.79	dark purple	—
Lidocaine	0.87	light blue	—
Butacaine	0.89	dark blue	yellow
Benzocaine	0.89	—	yellow
Halocaine	0.93	purple-violet	—

* AIPA = acidic iodoplatinic acid.

EXPERIMENTAL

Authentic samples of phencyclidine, cocaine and procaine were provided by the crime laboratory.

The TLC plates were a 0.25 mm thickness of silica gel G, without fluorescent indicator (Uniplate). The 20 × 20 cm glass plate was factory prescored for easy breaking in order to provide smaller plates.

The solvent system[1,2] was ethyl acetate–*n*-propanol–28% ammonium hydroxide mixture (40:30:3).

The reagents[1,2] used for the detection of spots were: IPA: equal parts of 0.3% hexachloroplatinic acid and 6% aqueous potassium iodide; *p*-dimethylaminobenzaldehyde (PDAB): 0.8 g of PDAB in a mixture of 10 ml of 98% sulfuric acid and 90 ml of 95% ethanol; AIPA: 5 ml of 5% aqueous solution of hexachloroplatinic acid, 45 ml of 10% aqueous potassium iodide, 50 ml of distilled water and 100 ml of 2 *N* hydrochloric acid.

Procedures

The unknown powder (*ca.* 25 mg) was agitated with 10 drops of 95% ethanol in a small test tube. A 20 × 20 cm TLC plate was marked off into three sections. Each section received aliquots from the unknown sample's supernatant liquid and from chloroform solutions of cocaine, phencyclidine and procaine. The unbroken plate was eluted in an equilibrated chromatotank containing 73 ml of the solvent system.

After removal of the plate from the tank, the solvent was eliminated from the silica by evaporation in an exhaust hood at room temperature. The dry plate was viewed under ultraviolet light (254 nm) and then broken into three separate sections. Every section contained a sample of the unknown plus each of the authentic standards.

RESULTS

Table III summarizes the characteristics of the unknown powder's three constituents (A, B and C) after TLC analysis.

TABLE III

CHARACTERISTICS OF THE UNKNOWN POWDER'S CONSTITUENTS AFTER TLC

Component	R_F	*Color with*			
		IPA	*PDAB*	*AIPA*	*UV (254 nm)*
A	0.05	—	—	orange-brown	—
B	0.13	light blue	—	orange-brown	—
C	0.78	purple	—	orange-brown	—

The data presented in Tables I and II lead one to suspect that component C ($R_F = 0.78$) is phencyclidine. The negative result with AIPA serves to exclude cocaine from consideration. Moreover, a positive identification of components A and B is not possible from the data in Tables I and II.

A plate section containing authentic samples of phencyclidine, cocaine, procaine and the unknown was sprayed with IPA reagent. The three known drugs produced indistinguishable purple colors. Their R_F values were in reasonable agreement with those reported in Tables I and II.

Another section prepared similarly to the one above was sprayed with AIPA solution. The authentic drugs and the unknown's components all turned orange-brown. No purple hue was noted at any time up to 8 h after spraying. Thus, contrary to the results in Table II, a purple color was not observed with procaine and cocaine.

The third section was heated in an oven at 105° for 10 min, cooled to room temperature, then sprayed with a PDAB solution until the plate was just wet. A yellow spot was observed for the procaine standard.

CONCLUSIONS

The data presented in refs. 1 and 2 were of no value in identifying any component of the unknown powder suspected of containing an illicit drug. The R_F value of component C is consistent with either phencyclidine or cocaine. A dark purple color with IPA does not serve to distinguish the two because both drugs produce this color (although only phencyclidine is cited in Table I). We also find that both drugs give an orange-brown color with AIPA which is in direct contradiction to the cocaine color test in Table II. Cocaine is reported elsewhere[3] as giving a purple color with IPA spray reagent. In fact, most of the drugs in Table II give a blue to purple color with IPA[4]. Had all the drugs been consolidated into one publication, the shortcomings of this method would have become immediately obvious.

The negative result obtained from the use of AIPA on authentic samples of cocaine and procaine was particularly disturbing. This experiment was repeated, special care being taken to prepare fresh solutions of AIPA, cocaine hydrochloride (Merck, Darmstadt, G.F.R.), and phencyclidine. A generous amount of each drug was applied to the TLC plate and eluted as described above. Again, only orange-brown spots were observed upon spraying with AIPA solution. An AIPA solution provided by the crime laboratory likewise yielded no purple colors.

A procedure appearing in the scientific literature purporting to screen successfully drugs of a high abuse potential may become routine practice by any forensic

laboratory. A laboratory with a limited budget may not test experimentally the reliability and limitations of its methods. It is therefore particularly important that forensic procedures be checked carefully prior to publication. The results of these experiments present a glaring example of an unreproducible color test for a drug of high abuse potential.

Strict adherence to the procedures outlined in refs. 1 and 2 incorrectly suggests that component C of the powder is phencyclidine and not cocaine.

It was definitely established that component C is not phencyclidine by the following experiment: The unknown powder was extracted with ^2H_2O leaving a substantial portion of insoluble residue (*ca.* 50% by weight). TLC of the solution showed it to contain mostly component C. A nuclear magnetic resonance spectrum of the soluble material was essentially identical to a reference spectrum of cocaine hydrochloride.

NOTE BY THE EDITOR

Most publications in this field stress that a spot on a chromatogram is an indication only which must be confirmed by other methods (see for example ref. 5). It is in any case insufficient for court evidence.

REFERENCES

1 J. K. Brown, L. Shapazian and G. D. Griffin, *J. Chromatogr.*, 64 (1972) 129.
2 J. K. Brown, R. H. Schingler, M. G. Chaubal and M. H. Malone, *J. Chromatogr.*, 87 (1973) 211.
3 F. Connie and J. Paul, *Mikrochimica Acta*, (1974) 443.
4 G. Zweig and J. Sherma (Editors), *Handbook of Chromatography*, Vol. II. CRC Press, 1972, p. 179.
5 I. Sunshine, *J. Chromatogr.*, 82 (1973) 125.