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A RAPID SCREENING PROCEDURE FOR SOME "STREET DRUGS" BY THIN-LAYER CHROMATOGRAPHY

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SUMMARY

The extraction of some illicit drugs with 95 % ethanol and subsequent analysis on pre-coated thin layers of silica gel (E. Merck) made the tentative identification of some constituents of "street drugs"* possible. Two detecting reagents were used, *p*-dimethylaminobenzaldehyde and iodoplatinate. *p*-Dimethylaminobenzaldehyde was used for the detection of lysergic acid diethylamide, psilocybin, monomethyltryptamine, dimethyltryptamine, and iodoplatinate for strychnine, mescaline, methamphetamine, amphetamine, phencyclidine. The thin-layer chromatograms derived characteristics of lysergic acid diethylamide and phencyclidine were unique, allowing for the relatively easy detection of these two compounds. The procedure is most useful for the rapid screening of illicit drugs for the presence or absence of the listed compounds. The unequivocal identity of these compounds is not established, but the procedure does permit tentative identification.

INTRODUCTION

Ingestion of "street drugs" by some members of our society who want to take "that beautiful trip" at times causes a medical crisis. Rapid identification of the active constituent(s) of the ingested material is necessary for proper medical treatment. The alleged constituent(s) of the ingested material usually has no resemblance to the actual chemistry of the compound(s) identified upon analysis[1,2].

A number of thin-layer chromatographic (TLC) methods for identification of psychoactive compounds, usually associated with detection of drugs in urine, have been published[3–12]. These methods usually involve extended preparative extraction[3–5,7] and several developing systems[3–12]. They were found to be time-consuming and comparatively complex. The extended time required for analysis in our laboratory initiated this study to simplify procedures and reduce the time required for the tentative identification of the active constituent(s) of some "street drugs".

The compounds selected were psilocybin[a] (PSI), N-monomethyltryptamine[b] (MMT), N,N-dimethyltryptamine[b] (DMT), lysergic acid diethylamide tartrate[a] (LSD),

* The term "Street Drugs" refers to both legally and illegally manufactured drugs that are sold in the illicit street market and usually are of unknown composition.

strychnine sulfate[c] (STRY), D-amphetamine[b] (AMP), DL-methamphetamine hydrochloride[b] (MAMP), mescaline sulfate[d] (MESC), and phencyclidine hydrochloride[e] (PCP). Some of these compounds (PSI, LSD, STRY, MESC, PCP) are most frequently the alleged constituent(s) of the "street drugs" involved in a crisis situation in our area[13]. The others (MMT, DMT, AMP, MAMP) were included because they are also involved in the non-medical use of drugs.

It was recognized that "street drugs" are not usually available as pure compounds, thus it was necessary to devise a simple method of extracting the active constituent(s) for chromatographic analysis. Three samples of "street drugs" of known identity, two containing unknown amounts of LSD, the other containing PCP and commercial 5-mg tablets of amphetamine sulfate[f] and methamphetamine hydrochloride[f] were used to devise the extraction method.

EXPERIMENTAL

Materials and methods

The standard solutions used were N-monomethyltryptamine, 1 mg/ml; N,N-dimethyltryptamine, 2 mg/ml; lysergic acid diethylamide tartrate, 0.5 mg/ml; strychnine sulfate, 3 mg/ml; D-amphetamine (free base), 5 mg/ml; DL-methamphetamine hydrochloride, 5 mg/ml; all in 95 % ethanol. Mescaline sulfate, 5 mg/ml in 10 % ammonium hydroxide–95 % ethanol (1:3). Psilocybin, 1 mg/ml in 95 % ethanol–water (1:1). Phencyclidine hydrochloride, 10 mg/ml in 95 % ethanol-water (9:1).

The TLC plates were pre-coated silica gel, without fluorescent indicator, 0.25 mm thickness, 5 × 20 and 20 × 20 cm (E. Merck).

For activation the plates were heated at 105° for 60 min, stored in a dessicator over silica gel.

The solvent system was ethyl acetate–*n*-propanol–28 % ammonium hydroxide solution (40:30:3).

The reagents used for detection were iodoplatinate (IPA), equal parts of 0.3 % hexachloroplatinic acid and 6 % aqueous potassium iodide[14]; dimethylaminobenzaldehyde (PDAB), 0.8 g of *p*-dimethylaminobenzaldehyde in a mixture of 10 ml of 98 % sulfuric acid and 90 ml of 95 % ethanol.

Procedures

Thin-layer chromatography. Preliminary investigation has shown that varying amounts of each compound were required for unequivocal detection after development. Volumes of each standard solution, representing the optimum amount of compound for detection were applied to duplicate plates (20 × 20 cm) *ca.* 2.5 cm from the bottom. The spots were allowed to air dry and the plates were placed in the developing tank. The tank was lined with filter paper wetted with the solvent; the solvent mixture was added to a depth of 1 cm about 1 h prior to use. The solvent was allowed to travel a

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[a] Sandoz Pharmaceuticals, Hanover, N.J., U.S.A.
[b] K & K Laboratories Inc., Plainview, N.Y., U.S.A.
[c] Merck & Co. Inc., Rahway, N.J., U.S.A.
[d] Nutritional Biochemicals Corp., Cleveland, Ohio, U.S.A.
[e] Sernylan, Parke, Davis & Co., Detroit, Mich., U.S.A.
[f] Robinson Laboratories Inc., San Francisco, Calif., U.S.A.

distance of 10 cm (*ca.* 70 min). The plates were removed, dried in an oven at 105° for 10 min. The dried plates were examined under UV light* (254 nm), fluorescent areas marked and colors noted. One plate was lightly sprayed with IPA reagent, positive areas marked and colors noted. The other plate was treated with PDAB reagent until the plate was just wet, positive areas marked and colors noted, then heated at 105° for 10 min. Any additional positive areas after heating were marked, colors and color changes noted. The data are summarized in Table I.

Extraction. Amphetamine sulfate and methamphetamine hydrochloride tablets (5 mg) were cut in half, each half tablet was placed in a Microflex tube**, the solid material having been reduced to a fine powder with a pestle, 0.3 ml of 28 % ammonium hydroxide solution was added, the solution was mixed, and then 1 ml of 95 % ethanol was added. The resulting slurry was mixed by shaking the capped vial for approximately 5 min. The insoluble portion was allowed to settle or alternatively the tube was centrifuged. The clear supernatant was subjected to the TLC procedure described for the standard solutions. The three "street drug" samples, two with unknown amounts of LSD and one with an undetermined quantity of PCP were treated similarily, except the 28 % ammonium hydroxide solution was omitted. The solutions obtained from these extractions were subjected to preliminary TLC evaluation to determine the volumes required to give unequivocal detection of the compounds. The required volume of each extract was applied to duplicate plates and treated as described for the standard solutions. The data are summarized in Table II.

RESULTS AND DISCUSSION

The solvent mixture used for the separation of these compounds was effective, the R_F values were distinctly different (Table I), the spots which appeared after spraying with the detecting reagents were discrete and well defined. Activation of the plates and accuracy of measurement in preparing the solvent mixture were essential for reproducible results. Inspection of the developed plate with UV light (254 nm) was useful, the distinctive bright blue color of LSD at 254 nm was used as a marker when evaluating the standard solutions and extracts. The color reaction of indole derivatives with PDAB or the absence of color when treated with PDAB, separated these compounds into two distinct groups, PDAB positive and PDAB negative. The distinctive colors of phencyclidine (PCP) and strychnine (STRY) with IPA reagent were used as evidence to identify these compounds. Amounts of each compound required for unequivocal detection were sufficiently small (Table I) so that one would expect to extract an identifiable quantity from 50–100 mg of "street drug" sample.

Strychnine. Distinctive characteristics of this compound were the R_F value, and the color reactions; purple-blue changing to dark blue in a few hours with IPA reagent and absence of color with PDAB.

Amphetamine, methamphetamine, mescaline. The tentative identity of these compounds was achieved by considering the R_F values, absence of color with PDAB reagent and the blue colors with IPA reagent.

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* Chromato-Vue Portable Darkroom, Model CC-20. Ultra-Violet Products Inc., San Gabriel, Calif., U.S.A.

** Kontes of California, Berkeley, Calif. 94710, U.S.A.

Phencyclidine. The color of PCP when treated with IPA reagent was unique dark purple changing in a few hours to purple-blue. Identity was established by considering the R_F, lack of fluorescence, color with IPA and absence of color with PDAB. The presence of this drug in a sample was relatively easy to detect with the listed criteria.

TABLE I

CHARACTERISTICS OF INDOLE DERIVATIVES AND OTHER DRUGS OF MISUSE AFTER THIN-LAYER CHROMATOGRAPHY

Drug	*Amount applied* (*μg*)	R_F	*Color with*		
			UV (254 nm)	IPA[a]	PDAB[b]
PSI	5	0.00	—	PuBr	PuBl
MMT	5	0.21	—	PuBl	Blue
DMT	5	0.48	—	Blue	Blue
LSD	2	0.69	Blue	Pu	Blue
STRY	6	0.32	—	PuBl	—
MESC	15	0.27	—	Pu	—
MAMP	20	0.37	—	Blue	—
AMP	20	0.49	—	Blue	—
PCP	10	0.79	—	DkPu	—

[a] Colors *ca.* 5 min after spraying; PuBr = Purple-brown, PuBl = Purple-blue; Pu = Purple; DkPu = Dark purple.
[b] Colors after heating at 105° for 10 min; PuBl = Purple-blue.

Extraction of tablets and "street drugs". LSD and PCP were extracted from extraneous carrier material with 95 % ethanol. TLC analysis of these extracts yielded data (Table II) identical to that of the standard compounds. The detection of LSD and PCP under these conditions was unequivocal. Addition of 28 % ammonium hydroxide solution when extracting the amphetamine sulfate and methamphetamine hydrochloride tablets increased the concentration of the two drugs in the 95 % ethanol and

TABLE II

CHARACTERISTICS OF LSD, PCP, AMPHETAMINE AND METHAMPHETAMINE EXTRACTED FROM "STREET DRUGS" AND TABLETS, AFTER THIN-LAYER CHROMATOGRAPHY

Drug	*Amount applied* (*μl*)	R_F	*Color with*		
			UV (254 nm)	IPA	PDAB
LSD[a]	8	0.69	Blue	PuBr	Blue
LSD[b]	12	0.69	Blue	PuBr	Blue
PCP[c]	20	0.79	—	DkPu	—
MAMP	15	0.37	—	Blue	—
AMP	15	0.49	—	Blue	—

[a] Extracted from 105 mg of purple powder.
[b] Extracted from 95 mg of white powder.
[c] Extracted from 134 mg of white, crystalline material.

made detection possible with smaller volumes of the extracts. Approximately 2 h were required to complete all the procedures.

CONCLUSIONS

Extraction of "street drugs" with 95 % ethanol and subsequent TLC analysis made possible the rapid, tentative identification of some constituents of "street drugs" if the TLC derived criteria were judiciously applied. LSD and PCP are probably involved in most of the crises precipitated by non-medical use of drugs in our area[13] and because of unique TLC characteristics, these two compounds were relatively easy to detect. The amphetamines (MESC, MAMP, AMP) due to the similarity of their TLC derived characteristics, could only be identified with any degree of certainty as to class; identity of the individual compounds was equivocal. The increased solubility of amphetamine sulfate, methamphetamine hydrochloride and mescaline sulfate in 95 % ethanol, when ammonium hydroxide solution was added, would suggest that if the initial extract of the "street drug" gave negative TLC results, ammonia solution should be added and the basic extract re-chromatographed.

The described procedure is most useful for the rapid screening of illicit drugs for the presence or absence of the listed compounds. The unequivocal identity of these compounds is not established but it does allow one to make a valid assumption as to the possible identity of the active constituent(s) in a time of crisis. For legal purposes further tests and procedures must be used to establish beyond a doubt the chemical identity of these compounds. This procedure does not accomplish this goal.

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