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

### II. COCAINE, HEROIN, LOCAL ANESTHETICS AND MIXTURES

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#### SUMMARY

The extraction of illicit cocaine and heroin (diacetylmorphine) samples with 95% ethanol and subsequent analysis on pre-coated thin layers of silica gel (Merck) made the tentative identification of cocaine, heroin and some local anesthetics possible. Two detecting reagents were used, *p*-dimethylaminobenzaldehyde and acidic iodoplatinate. *p*-Dimethylaminobenzaldehyde was used for the detection of procaine, butacaine and benzocaine, acidic iodoplatinate for cocaine, heroin and some local anesthetics. The thin-layer chromatographically derived characteristics of cocaine, heroin, benzocaine, procaine, and lidocaine were unique, allowing for the relatively easy detection of these five compounds. The procedure is useful for the rapid screening of illicit cocaine and heroin samples for the presence or absence of the compounds listed. The unequivocal identity of these compounds is not established but the procedure does permit tentative identification.

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#### INTRODUCTION

The increased number of samples alleged to be cocaine being submitted to "street-drug" monitoring programs<sup>1-6</sup> and law enforcement agencies<sup>7</sup> would suggest that the non-medical user of illicit drugs is beginning to favor the stimulant cocaine.

The number of alleged cocaine samples submitted to our laboratory for screening increased from two samples in 18 months (November 1970 to May 1972) to an average of four samples a month. The adulteration of cocaine with local anesthetics<sup>2-6</sup>, and the substitution of other drugs such as heroin (diamorphine) mixed with procaine<sup>2,5</sup> made it necessary that a rapid screening procedure be devised to extract, separate and identify these compounds.

Investigation in our laboratory showed that a single extraction with 95% ethanol and a thin-layer chromatographic (TLC) evaluation<sup>9</sup> of the resultant ethanolic extract was satisfactory for the extraction, separation and tentative identification of cocaine, heroin and selected local anesthetics.

The compounds selected to illustrate this method were: cocaine hydrochloride, tetracaine hydrochloride, procaine hydrochloride, butacaine sulfate, lidocaine hydrochloride, benzocaine, holocaine hydrochloride, and a known sample of "Mexican Brown" heroin<sup>8</sup>.

## EXPERIMENTAL

*Materials and methods*

The following standard solutions were prepared: 5 mg/ml in 95% ethanol each of tetracaine hydrochloride, butacaine sulfate, benzocaine, and holocaine hydrochloride; 10 mg/ml in 95% ethanol of cocaine hydrochloride; 10 mg/ml of lidocaine hydrochloride in saline (Xylocaine\*); 10 mg/ml of procaine hydrochloride\*\* in saline; 10 mg/ml of "Mexican Brown" heroin (of unknown concentration) in 95% ethanol.

The TLC was carried out on activated<sup>9</sup> pre-coated silica gel, without fluorescent indicator, plates of 0.25 mm thickness (Merck, Darmstadt, G.F.R.). The developing solvent was ethyl acetate-*n*-propanol-28% ammonium hydroxide solution (40:30:3) (ref. 9).

The reagents used for detection were acidic iodoplatinate (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), and *p*-dimethylamino-benzaldehyde (PDAB)<sup>9</sup>.

*Procedures*

*Thin-layer chromatography.* The TLC procedure as previously described<sup>9</sup> was found to be satisfactory for separation of the selected compounds when applied either singly or in known mixtures. The areas after visualization were discrete and well separated.

Cocaine hydrochloride was used as a marker, the standard solution was applied to the TLC plate with the local anesthetics and the ethanolic extracts of street-samples alleged to be cocaine. The  $R_F$ ,  $R_C$  (relative to cocaine = 1) values, minimum amounts detectable and colors with the two reagents are summarized in Table I.

*Extraction.* Known solid mixtures were prepared of cocaine with selected local anesthetics (Table II) to establish the efficiency of the single extraction procedure using 95% ethanol. A previously identified "street" mixture of heroin and procaine was also used.

## RESULTS AND DISCUSSION

The solvent mixture used for the separation of these compounds was effective, the  $R_F$  values were sufficiently different (Table I),  $R_C$  values were constant, and the spots which appeared after spraying with the detecting reagents were discrete and well defined. The color reaction of procaine, butacaine, and benzocaine with PDAB and the distinctive color of each of the compounds (benzocaine excepted) with AIPA were used as evidence to identify these compounds. Amounts of each compound required for unequivocal detection were sufficiently small (Table I) that one would expect to extract an identifiable quantity from 25–75 mg of illicit cocaine or heroin samples.

*Heroin.* Distinctive characteristics of this compound were the  $R_F$  and  $R_C$  values, characteristic dark brown color with AIPA and no reaction with PDAB.

\* Astra Laboratories, Worcester, Mass., U.S.A.

\*\* Abbott Laboratories, Chicago, Ill., U.S.A.

TABLE I  
CHARACTERISTICS OF COCAINE, HEROIN AND SELECTED LOCAL ANESTHETICS  
AFTER TLC

Drug	Minimum amount for detection ( $\mu\text{g}$ )	Color with		$R_F$	$R_C$
		PDAB*	AIPA**		
Heroin***	?	—	dark brown	0.45	0.57
Tetracaine	2	—	grey-violet	0.55	0.69
Procaine	1	yellow	blue-violet	0.70	0.89
Cocaine	10	—	dark purple	0.79	1.00
Lidocaine	1	—	light blue	0.87	1.10
Butacaine	1	yellow	dark blue	0.89	1.12
Benzocaine	1	yellow	—	0.89	1.12
Holocaine	5	—	purple-violet	0.93	1.18

\* Color immediate upon spraying with PDAB.

\*\* Colors *ca.* 5 min after spraying.

\*\*\* A known sample of "Mexican Brown" heroin of unknown strength.

TABLE II  
CHARACTERISTICS OF HEROIN, COCAINE, PROCAINE, BENZOCAINE, AND LIDO-  
CAINE EXTRACTED FROM KNOWN MIXTURES, AFTER TLC

Mixture	Amount applied ( $\mu\text{l}$ )	$R_F$	Color with	
			PDAB	AIPA
Heroin +	5	0.45	—	dark brown
Procaine*	5	0.70	yellow	blue-violet
Cocaine +	2	0.79	—	dark purple
Procaine**	2	0.70	yellow	blue-violet
Cocaine +	2	0.79	—	dark purple
Procaine +	2	0.70	yellow	blue-violet
Benzocaine +	2	0.89	yellow	—
Lidocaine***	2	0.87	—	light blue

\* Extracted from 75 mg of brown powder, concentration of each constituent unknown.

\*\* Extracted from a mixture of two parts of cocaine to one part of procaine; final concentration *ca.* 10 mg cocaine and 5 mg procaine per milliliter of 95% ethanol.

\*\*\* Extracted from a mixture of two parts of cocaine to one part of each of the listed local anesthetics, final concentration as in above.

**Cocaine.** The tentative identity of this compound was achieved by considering the  $R_F$  value, absence of color with PDAB reagent and the characteristic dark purple color with AIPA reagent.

**Local anesthetics.** The tentative identity of these compounds was established by considering  $R_F$  and  $R_C$  values, presence or absence of color after spraying with PDAB reagent and characteristic color (or absence of color for benzocaine) with

AIPA reagent. The tentative identification of benzocaine and butacaine, both having the same  $R_f$  value, was accomplished by spotting these compounds in duplicate and spraying each with PDAB and AIPA. Benzocaine was PDAB-positive and AIPA-negative; butacaine was both PDAB- and AIPA-positive.

*Extraction of mixtures.* Heroin, procaine hydrochloride, cocaine hydrochloride, benzocaine, and lidocaine hydrochloride were extracted from extraneous carrier material with 95% ethanol. TLC analysis of these extracts yielded data (Table II) identical to those of the standard compounds.

## CONCLUSIONS

A rapid qualitative analysis of street-drugs, containing common local anesthetics but alleged to be pure cocaine or heroin, can be effected by single extraction with 95% ethanol and TLC analysis of the extract, provided the TLC-derived criteria are judiciously applied. Procaine, lidocaine and benzocaine, the most common adulterants<sup>2-6</sup> of illicit cocaine, and cocaine itself can be readily detected. The tentative identification of heroin is made possible due to the unique TLC characteristics of this compound.

The procedure described is satisfactory for screening illicit cocaine and heroin samples, both alone or adulterated with local anesthetics, for the presence or absence of these compounds. The unequivocal identity of these compounds is not claimed but it does allow one to make working assumptions as to the possible identity of these compounds. For legal purposes further tests 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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