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Identification of Street Drugs by Thin-Layer Chromatography and a Single Visualization Reagent

A number of reports on the use of thin-layer chromatography (TLC) in the analysis of street drugs have appeared [*1–3*]. Colors with various visualization agents applied in sequence are used for identification in conjunction with R_f values, spot tests [*4*], and crystal tests [*5*]. This paper reports the use of a rapid TLC screening procedure that utilizes a single developing solvent and a single visualization reagent, *N*,2,6-trichloro-*p*-benzoquinone imine (TCBI), for the preliminary identification of the drugs of abuse. In addition, other TLC developing solvents are outlined that can be used for further confirmation.

TCBI dissolved in various organic solvents has been used as a TLC visualization reagent for cannabinoids [*6*] and barbiturates [*7*]. Vinson and Hooyman [*8*] have found that a formulation of TCBI with dimethyl sulfoxide and sodium bicarbonate allows its use as a visualizer for a wide variety of drugs.

Materials

Solvents and chemicals, Baker Analyzed® grade, J. T. Baker Chemical Co.

Flexible TLC sheet, Baker-flex®, Silica Gel IB2, 20 by 20 cm, 200 μm thick, J. T. Baker Chemical Co.

TLC developing tank, 8½ by 4 by 9 in., Kontes Glass Co.

Ultraviolet visualization chamber, Chromato-Vue®, Model CC-20, Ultraviolet Products, Inc.

Drummond Microcaps®, Arthur H. Thomas Co.

Visualization reagent (TCBI), 0.1 g of *N*,2,6-trichloro-*p*-benzoquinone imine dissolved in 90 ml of chloroform with 10 ml of dimethyl sulfoxide previously saturated with sodium bicarbonate added

Procedure

A solution material to be identified was spotted 1.5 cm from the bottom of an unactivated 20 by 20-cm silica gel plate. The spots were dried at room temperature using a hair drier. The plates were developed to a height of 10 cm in an unsaturated tank.

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The developing solvents were Solvent A, ethyl acetate/methanol/ammonia, 100:18:1.5; Solvent B, methanol/ammonia, 100:1.5; Solvent C, chloroform/diethyl ether, 85:15; and Solvent D, benzene/dioxane/acetone/ammonia, 25:10:14:1. After development the plates were removed and dried in an oven at 110°C for 5 min. The plates were observed under short ultraviolet (UV) illumination and the colors noted. The plates were then lightly sprayed, until they were just wet, with TCBI visualization reagent and placed in a 110°C oven for 1 to 2 min. Occasionally, respraying was necessary in order to optimize the development of color.

Results

The colors of the various drugs under shortwave UV illumination and with the TCBI reagent are shown in Table 1. In addition, the R_f values for 3 μg (3 μl of 1 mg/ml solution) of each drug are listed for the various developing solutions. Each R_f value is the average of R_f determinations on two different plates. The limits of detection, using 1 μl of standard solutions appropriately diluted, are also tabulated. Spots for drugs at low levels are best seen by viewing from the back of the plate with transmitted light.

Discussion

Solely on the basis of color with TCBI, some distinction can be made between the classes of drugs. Barbiturates yield a blue color with TCBI, phenothiazines gray to purple, and other basic drugs usually green to brown. Amphetamines all are greenish in color, with the exception of amphetamine itself, which is gray purple.

Barbiturates all have R_f values above 0.9 in developing Solvent A and can readily be distinguished from the other drugs. Solvent A should be used as a general screening solvent for all types of drugs, with Solvent B used for further confirmation of basic drugs. Barbiturates, because of their similar structures, require the two confirmation Solvents C and D for identification.

Under the procedural conditions, the limits of detection of a majority of drugs are better for TCBI, as determined in this work, than those listed for commonly used visualizers [*9*]. The sensitivity of amphetamine and methamphetamine with TCBI is superior to that of ninhydrin, 0.25 μg versus 1 μg, respectively. With morphine, down to 0.25 μg can be detected with TCBI, but only 2 μg with iodoplatinate as the visualizer. Sedatives-hypnotics are usually visualized with a combination of mercuric sulfate and diphenylcarbazone with a detection limit of 0.1 to 1 μg; with TCBI, most of these drugs can be seen below 0.05 μg. The background of the plates after spraying with TCBI and heating is a very light tan color that darkens slightly on storage. The light background provides a good contrast for the colored spots. This is not the case with many of the commonly used spray reagents, notably iodoplatinate.

In contrast to some of the other spray reagents, the TCBI formulation is stable for months if kept in a brown bottle in the refrigerator. The spray, however, can become discolored from its customary light yellow by contact with ammonia or other basic chemicals and should then be discarded. The TCBI-visualized spots are stable indefinitely, although the colors tend to change to brown during long-range storage.

Summary

TCBI has been shown to be an extremely sensitive visualizer that gives a wide

TABLE 1—*TLC of drugs with various developing solvents.*

Drug	R_f Values				Color Under Short UV	Color with TCBI	Detection Limit, μg
	Solvent A	Solvent B	Solvent C	Solvent D			
Chloroquine	0.03	0.24	...	...	...	green	5
Strychnine	0.04	0.20	...	...	...	green	0.25
Atropine	0.05	0.17	...	...	...	green	...
Epinephrine	0.06	0.30	...	...	yellow	brown	0.1
Morphine	0.07	0.35	...	...	...	brown green	0.25
Bufotenine	0.07	0.23	...	...	...	brown green	...
Ephedrine	0.07	0.25	...	...	...	orange brown	0.25
Mescaline	0.07	0.20	...	...	blue	pink brown	0.25
Methamphetamine	0.07	0.26	...	...	...	green	0.25
Dextromethorphan	0.08	0.26	...	...	...	gray	...
Acetophenazine	0.10	0.62	...	...	orange	yellow green	0.1
Codeine	0.10	0.35	...	...	...	brown green	0.25
Dimethyltryptamine	0.10	0.32	...	...	blue	brown green	0.25
Nortryptilene	0.10	0.26	...	...	blue	brown	0.1
Phentermine	0.13	0.57	...	...	...	green	...
Phenylephrine	0.13	0.30	...	...	...	green	0.1
Quinine	0.14	0.55	...	...	blue	gray green	...
Amphetamine	0.18	0.39	...	...	...	gray purple	0.25
Trifluoperazine	0.18	0.54	...	...	blue	blue purple	0.25
Heroin	0.20	0.46	...	...	...	yellow green	0.25
Perphenazine	0.20	0.63	...	...	orange	gray purple	0.05
Phenmetrazine	0.20	0.57	...	...	yellow	yellow green	0.25
Quinidine	0.20	0.61	...	...	blue	green	...
Promazine	0.25	0.44	...	...	...	purple	0.25
Promethazine	0.25	0.54	...	...	...	purple	0.25
Meperidine	0.27	0.53	...	...	...	green	...
Nicotine	0.28	0.63	...	...	...	gray purple	0.05
Lysergic acid diethylamide	0.33	0.77	...	...	blue	gray brown	0.25
Chlorpromazine	0.33	0.52	...	...	...	gray purple	0.25
Mephentermine	0.35	0.52	...	...	blue	green	5
Thioridazine	0.36	0.44	...	...	blue	gray brown	0.25
Phendimetrazine	0.36	0.67	...	...	...	gray	...

Diphenhydramine	0.37	0.57	...	...	blue	blue green	0.25
Methadone	0.41	0.43	...	...	...	blue green	0.1
Sulfathiazole	0.41	0.89	...	...	...	brown blue	...
Ethinamate	0.43	0.72	...	...	...	orange brown	...
Pentazocine	0.49	0.66	...	...	blue	gray green	0.25
Methylphenidate	0.52	0.69	...	...	...	gray green	0.25
Apomorphine	0.54	0.75	...	...	...	blue green	0.25
Procaine	0.54	0.67	...	...	...	brown green	0.1
Thiopropazate	0.58	0.71	...	...	blue	blue	0.25
Trimeprazine	0.59	0.61	...	...	...	blue gray	0.1
Chlordiazepoxide	0.64	0.79	...	...	...	green	5
Sulfamerazine	0.67	0.89	...	...	...	gray	...
Cocaine	0.73	0.70	...	...	...	green	...
Propoxyphene	0.74	0.74	...	...	...	blue gray	0.1
Dicyclomine	0.77	0.78	...	...	...	green	...
Anileridine	0.79	0.81	...	...	...	brown green	0.1
Oxazepam	0.81	0.80	...	...	...	blue	...
Noludar	0.86	0.91	...	...	...	blue	0.5
Ibogaine	0.90	0.67	...	...	...	brown green	0.25
Pentobarbital	0.91	...	0.36	0.88	...	blue	<0.05
Diphenylhydantoin	0.91	...	0.19	0.71	...	blue	<0.05
Allobarbital	0.91	...	0.34	0.72	...	blue	<0.05
Benzocaine	0.92	0.83	...	...	...	orange brown	0.50
Phenobarbital	0.92	...	0.26	0.59	...	blue	0.01
Barbital	0.92	...	0.24	0.62	...	blue	0.04
Aprobarbital	0.92	...	0.33	0.80	...	blue	<0.05
Diethylpropion	0.93	0.79	...	...	...	gray brown	...
Hexital	0.93	...	0.46	0.93	...	blue	<0.05
Secobarbital	0.93	...	0.41	0.90	...	blue	<0.05
Methapyrilene	0.93	0.55	...	...	...	brown green	0.25
Butabarbital	0.93	...	0.29	0.83	...	blue	<0.05
Butalbital	0.93	...	0.41	0.84	...	blue	0.01
Mephobarbital	0.94	...	0.60	0.90	...	blue	<0.05
Methohexital	0.95	...	0.81	0.98	...	blue	<0.05
Tetrahydrocannabinol (Δ^9)	0.95	0.75	...	...	...	red brown	...
Amobarbital	0.95	...	0.34	0.86	...	blue	<0.05
Glutethimide	0.98	...	0.81	0.96	...	gray green	0.1
Lidocaine	0.98	0.87	...	...	...	green	0.1

variety of colors with the drugs of abuse. The combination of TCBI as a visualization reagent and manual TLC, using flexible silica gel sheets, can provide a rapid means for the preliminary identification of street drugs.

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