

# MICROGRAM

Laboratory Operations Division  
Office Of Science And Drug Abuse Prevention

BUREAU OF NARCOTICS & DANGEROUS DRUGS / U.S. DEPARTMENT OF JUSTICE / WASHINGTON, D.C. 20537

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Vol. III, No. 3

May, 1970

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## THE WHITE HOUSE

DRUG ABUSE PREVENTION WEEK, 1970

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BY THE PRESIDENT OF THE UNITED STATES OF AMERICA

### A PROCLAMATION

The past decade has seen the abuse of drugs grow from essentially a local police problem into a serious threat to the health and safety of millions of Americans. The number of narcotics addicts in the United States is estimated to be in the hundreds of thousands and the effects of their addiction spread far beyond their own lives.

Statistics tell but part of the tragedy of drug abuse. The crippled lives of young Americans, the shattered hopes of their parents, the rending of the social fabric -- as addicts inevitably turn to crime in order to supply a costly habit -- these are the personal tragedies, the human disasters that tell the real story of what drug abuse does to individuals and can do to our nation.

NOW, THEREFORE, I, RICHARD NIXON, President of the United States of American, do hereby designate the week beginning May 24, 1970, as Drug Abuse Prevention Week.

I call upon officials of the Federal government, particularly in the Departments of Justice and Health, Education, and Welfare, to join with educators and administrators of the academic community at large in establishing meaningful programs for the promotion of drug abuse prevention among

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Analytical methods in **Microgram** do not have official status. Use of funds for printing this publication approved by the Bureau of the Budget, April 8, 1969. **CAUTION:** Use of this publication is restricted to forensic scientists serving law enforcement agencies.

young people. I urge State and local governments, as well as business, professional, and civic groups, to cooperate in such programs and to exercise their initiative in exploring new methods by which the potential dangers of drug experimentation can be communicated to the entire nation. The communications media can provide invaluable assistance in this regard, and I request their full support of this endeavor.

I encourage members of the clergy, and all those whose activities interrelate with young people, to make a special effort during this week to discourage drug abuse, to end drug experimentation, and to eliminate illegal drug traffic.

IN WITNESS WHEREOF, I have hereunto set my hand this twenty-eighth day of April, in the year of our Lord nineteen hundred and seventy, and of the Independence of the United States of America the one hundred and ninety-fourth.

RICHARD NIXON

BNDD FORENSIC CHEMISTS' SEMINARS for Fiscal Year 1971 are tentatively planned as follows:

September 14-18, 1970	February 8-12, 1971
October 26-30, 1970	April 12-16, 1971
December 7-11, 1970	June 14-18, 1971

For further information or application forms write to:

Chief, Special Programs Division (TRNS)  
Bureau of Narcotics & Dangerous Drugs  
U.S. Department of Justice  
Washington, D. C. 20537

The last seminar in Fiscal Year 1970 was held April 27-May 1.

ON-THE-JOB TRAINING FOR FORENSIC CHEMISTS is available in BNDD Regional Laboratories. This training is designed to suit a particular problem or individual need of the trainee. To make arrangements, the director or chief chemist of the local laboratory should contact the chief chemist of the BNDD regional laboratory serving his area. Addresses of the regional laboratories are attached.

Heroin (Diacetylmorphine HCl) was identified by Northern Illinois Police Crime Laboratory in a foil packet containing a white powder. The contents of the exhibit was alleged to be "PEE," reportedly taken by sniffing.

Marijuana cigarette was also analyzed by the Northern Illinois Police Crime Laboratory. Close examination revealed blue particles of pentobarbital. A yellow-colored material, insufficient in quantity to be analyzed, adhered to some of the blue particles. It is believed that the material probably was a pulverized Desbutal tablet with the methamphetamine removed.

para-Methoxymethylnitrostyrene, a yellow powder, was recently encountered in the BNDD San Francisco Regional Laboratory. This substance can easily be reduced to para-methoxyamphetamine.

A. K. Dixon, Experientia, 24, 743 (1968) indicates that para-methoxyamphetamine has a potency in rats exceeded only by LSD (LSD equals 4600 mescaline units). Shulgin, Nature, 221, 537 (1969) has human data which shows a potency of five mescaline units for para-methoxyamphetamine.

Heroin has been encountered in the BNDD Washington Regional Laboratory in one milliliter, plastic disposable syringes filled with clear, colorless solution. Analysis of two syringes showed 6.1 and 6.4 milligrams heroin.

Lavender (Lavandula officinalis) tubular calyx containing seeds was identified by the BNDD Special Testing and Research Laboratory. The exhibit, probably a form of incense, was submitted as suspected hashish.

"IMMENOCTAL" Tablets were recently identified by the BNDD Special Testing and Research Laboratory. The tablets were in dispensing foil labeled "IMMENOCTAL," "10," and "ne pas dépasser la dose prescrite," On the opposite side, the manufacturer's symbol showing the letters "ISH."

The product, manufactured in France by the Laboratoires de l' ISH, contains secobarbital sodium 0.1 gram. The excipients were potato starch (large amount), and lactose monohydrate (moderate amount).

The tablets were white, round, flat, flat-beveled on both sides, and single-scored. The side opposite the score had "IM 10" engraved on it. Average weight: 347 milligrams, diameter 11.09 to 11.12 millimeters, thickness 2.96 to 3.00 millimeters. Bevel angles were about 16° on one side and about 13° on the other.

Cinquefoil (Potentilla recta L.) was identified in material submitted to the Special Testing and Research Laboratory. Cinquefoil is encountered as alleged marihuana. Species of Potentilla were used in medicine because of high content of tannin in their roots.

LSD Tablets containing brass-colored flecks were recently examined by the Special Testing and Research Laboratory. The flecks, having a metallic luster, were uniformly distributed throughout the tablet matrix.

The tablets were light-grey, round, flat-convex, unscored, 6.59 to 6.61 millimeters in diameter, 3.61 to 3.81 millimeters thick at center and 1.4 to 1.7 millimeters thick at the edge. Average weight of ten tablets was 105.6 milligrams, and each tablet contained approximately 100 micrograms of LSD. The flat face of each tablet was approximately 4.2 millimeters in diameter, with a wide flat bevel sloping steeply to the ridged edge. Both faces had faint concentric striations. The flat face had a series of tiny, characteristic lumps and gouges at the edge, and convex face had a series of large characteristic lumps at the edge, identical on each tablet. The tablets were made with a single pair of punches.

Analysis showed a large amount of spray-dried proteinaceous material and a small amount of amorphous opaque flecks. These were 1 millimeter or less in length. X-ray diffraction and microchemical tests showed the presence of copper, and X-ray diffraction showed zinc. The flecks are probably brass, estimated at approximately 5 per cent of the tablet weight.

Benactyzine hydrochloride and N-Methyl-3-piperidyl benzilate ("LBJ") are sometimes identified incorrectly, according to reports. Joseph E. Koles, BNDD Forensic Chemist, Special Testing and Research Laboratory, states that the ultraviolet spectra of the two are identical and that they give the same color reactions with Marquis reagent and hot concentrated sulfuric acid. In addition, their infra-red spectra are very close in appearance and the compounds show little or no separation in many thin-layer chromatography systems.

In order to facilitate the correct identification of the two substances, according to Koles, it is recommended that the infra-red spectra of the

known and unknown be critically examined peak by peak, and that standards of both compounds be run on each thin-layer chromatogram.

In addition, a fast check on the identification can be made by taking the melting point of the unknown. (Benactyzine M.P. 51<sup>o</sup>, Benactyzine hydrochloride M.P. 177-178<sup>o</sup>, N-methyl-3-piperidyl benzilate is a liquid; N-methyl-3-piperidyl benzilate hydrochloride M.P. 210-218<sup>o</sup>).

The hydrochlorides of the two compounds can also be readily identified by X-ray diffraction, Koles reports.

alpha-Methyltryptamine (Hydroxyamphetamine) has been encountered by our Special Testing and Research Laboratory in a small amount of white powder from the East Coast. See Usdin, Earl and Daniel H. Efron, Psychotropic Drugs and Related Compounds, p.98 (Public Health Service Publication No. 1589, Superintendent of Documents, Washington, D. C.); Stecher, Paul G., Ed., The Merck Index, 8th Ed. p.547 (Merck & Co., Inc., Rahway, N. J.); Osol, Arthur, Ed., The United States Dispensatory, 26th Ed., pp. 1120 and 1125; (J. P. Lippincott Co., Philadelphia); Microgram, I, 4, p.42 and I, 6, p.80; Clarke, E. G. C., Ed., Isolation and Identification of Drugs...p.373 (The Pharmaceutical Press, London). alpha-Methyltryptamine is not a federally controlled drug.

p-Chlorophenylalanine (PCPA) caused "hypersexuality," increased aggression and perceptual disorientation in cats, according to a report in Science, 168,499-501 (April 24, 1970). The behavior of the cats is considered to be sequelae of the chronic administration of the drug. Pronounced changes developed rapidly after a latent period of 3 to 5 days from the first injection.

The compound is a likely candidate for trial as a drug by the "street pharmacologist." It is listed in at least one chemical supplier's catalog, for those wishing to obtain an analytical standard.

#### MEETINGS:

Second World Meeting on Medical Law, Washington, D. C., August 18-21, 1970.  
Contact: R. Dierkens, Dr. Jur., Agregé Law Faculty, Secretary General,  
5 Apotheekstraat B-9000, Ghent, Belgium.

Association of Official Analytical Chemists (A.O.A.C.) will sponsor a symposium entitled "Analytical Methods in Forensic Science" at the A.O.A.C., October 15, 1970, at the Marriott Motor Hotel, Twin Bridges, Washington, D.C.

Speakers will be scientists from government agencies, industry and universities. Techniques of major interest to crime laboratories will be discussed by scientists engaged in the examination of physical evidence.

There will be a panel discussion of forensic science methods, the need to form committees to study instrumentation and methodology, and to discuss future forensic science programs at the A.O.A.C.

Contact: Richard L. Brunelle, Association of Official Analytical Chemists  
Box 540 Benjamin Franklin Station, Washington, D. C.

American Academy of Clinical Toxicology annual meeting in San Francisco, October 24-26, 1970. Will include a one-day symposium on "The Clinical Toxicology of Substances of Abuse" (other than narcotics) and one-day symposium on "Legal Aspects of Clinical Toxicology." Address:

P. O. Box 2565  
Houston, Texas 77001

### GLOSSARY

#### B.

BAMMIES	Poor quality marihuana
BANG	One injection of narcotic; to inject drugs
BAMBITA	Desoxyn
BANGING	Under influence of drugs
BARBS	Barbiturates
BATTED OUT	Arrested
BEAT THE GONG	Smoke opium
BEDBUGS	Fellow addicts
BELONGS	On the habit
BELTED	Under influence of drugs
BEHIND THE IRON HOUSE	In jail
BENDING AND BOWING	Under the influence of drugs
BENNIES	Benzedrine; amphetamines
BERNICE	Cocaine
BERNIES FLAKE	Cocaine

(To be continued.)

## IDENTIFICATION OF CANNABIS

by

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### INTRODUCTION

The higher incidence of abuse of Cannabis in recent years has necessitated identification of larger numbers of Cannabis samples. This in turn has caused workers in the field (e.g. Turk & others 1969; Maunder 1969) to search for more rapidly performed tests with a view to reducing the time of analysis to the minimum which allows certainty of identification. We describe here a procedure for analysis which is advantageous in combining two independent techniques for the detection of three Cannabis components and which offers positive identification in a reasonably short time. It consists of extraction of the suspected Cannabis or Cannabis resin sample with a stock solution of dibenzylphthalate in petroleum ether, the extract then being analysed without further purification by gas chromatography and also by paper chromatography. Both chromatographic systems offer good resolution of the three Cannabis components.

### EXPERIMENTAL METHODS

#### Preparation of the Extract

The Cannabis or Cannabis resin is shaken vigorously for one minute with sufficient stock solution of dibenzylphthalate (10mg/ml) in petroleum ether (40 - 60°) to produce a mixture containing approximately 20% w/v of Cannabis or 10% w/v of Cannabis Resin. The supernatant solution is used, without further purification, for chromatography.

#### GAS CHROMATOGRAPHY

In our experiments a Pye 104 Gas Chromatograph equipped with a flame ionisation detector and a Kelvin Electronics servoscribe recorder was used. The column was glass, 5ft x 4mm internal diameter, packed with 80-100 mesh acid-washed,

siliconised Diatomite C which was coated with 1% cyclohexahedimethanol succinate (CDMS). A hydrogen pressure of 18lb/sq in, air 7lb/sq in, and a nitrogen flow rate of 50ml/min was used throughout. The operating temperature was 220°. 1  $\mu$  l of the extract was injected onto the column at an appropriate attenuation and the retention times of cannabidiol (CBD),  $\Delta^1$ -3, 4-trans tetrahydrocannabinol (THC), and cannabinol (CBN), were calculated relative to dibenzylphthalate, (DBT) the internal standard. The total analysis time was approximately 15 mins. Retention times of the cannabinoids relative to dibenzylphthalate are given in Table I.

#### PAPER CHROMATOGRAPHY

Whatman SG81 paper (7 cms x 25 cms) is immersed in a 15% w/v solution of silver nitrate in distilled water, the excess solution is allowed to drain off, and the paper is then air dried. After applying spots of the extract of suspected Cannabis or Cannabis Resin, and of  $\Delta^1$ -3, 4-trans tetrahydrocannabinol, the paper is developed in chloroform using the ascending technique. Location of the cannabinol is by spraying successively with a 1% solution of Fast Blue Salt B in water and then 2N sodium hydroxide. Development time is 10 minutes for a 5 cm. run. Rf values are given in Table I.

#### DISCUSSION

A number of gas chromatographic systems for the analysis of Cannabis samples have been reported, the most recent of which (Lerner 1969) has described the use of dl-methadone hydrochloride as an internal standard, allowing quantitation of Cannabinols in samples should this be necessary. The method which we have described also allows quantitation of Cannabinols but differs from that of Lerner in utilising an internal standard which has a longer retention time than that of any of the Cannabis components. This, in our view, is advantageous in reducing the probability of one of the components of Cannabis having the same retention time as the internal standard.

The use of silver nitrate impregnated media for separation of cannabinoids has previously been reported by Caddy and Fish (1967) Hively & others (1966) and by Turk & others (1969) and it is our experience that these systems offer satisfactory resolution of the cannabinoids. However, the modification which we have described is ideally suited to routine analysis



in that a large number of silver nitrate impregnated papers can be prepared in one batch and conveniently stored ready for use in an envelope. Papers may be stored thus, in the dark, for up to one month.

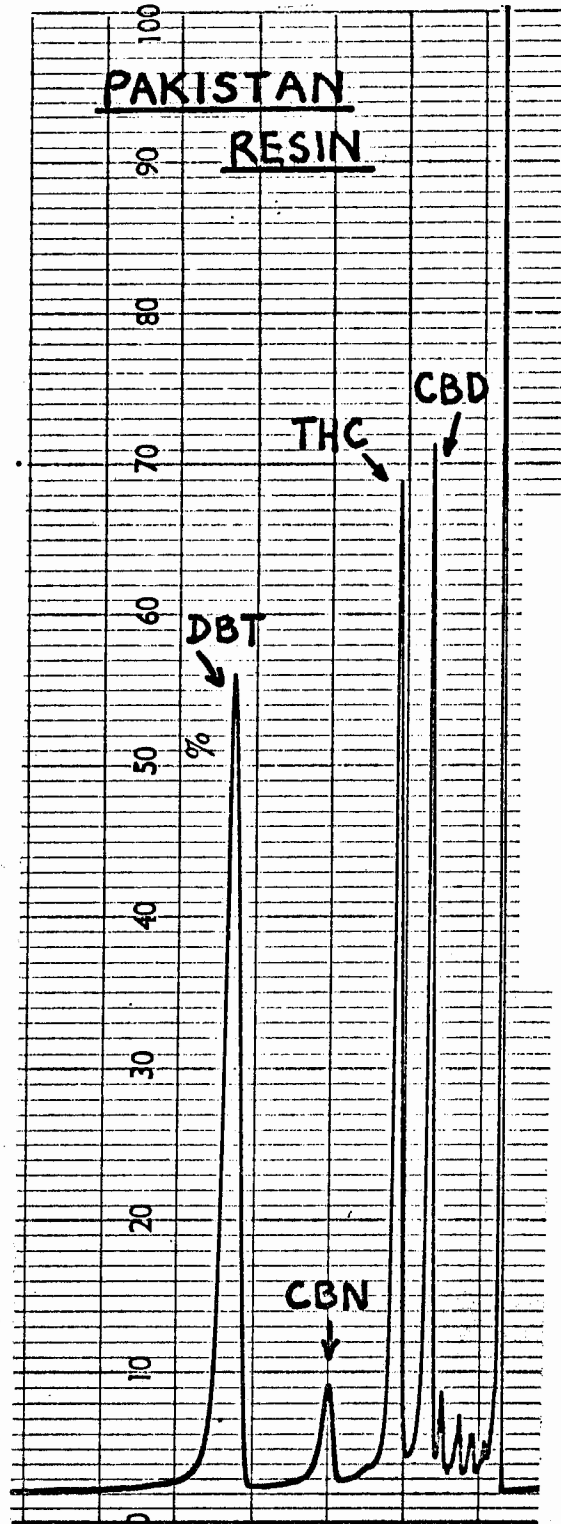
19 February 1970

TABLE I- Relative Retention Times and Rf's of Cannabis Components

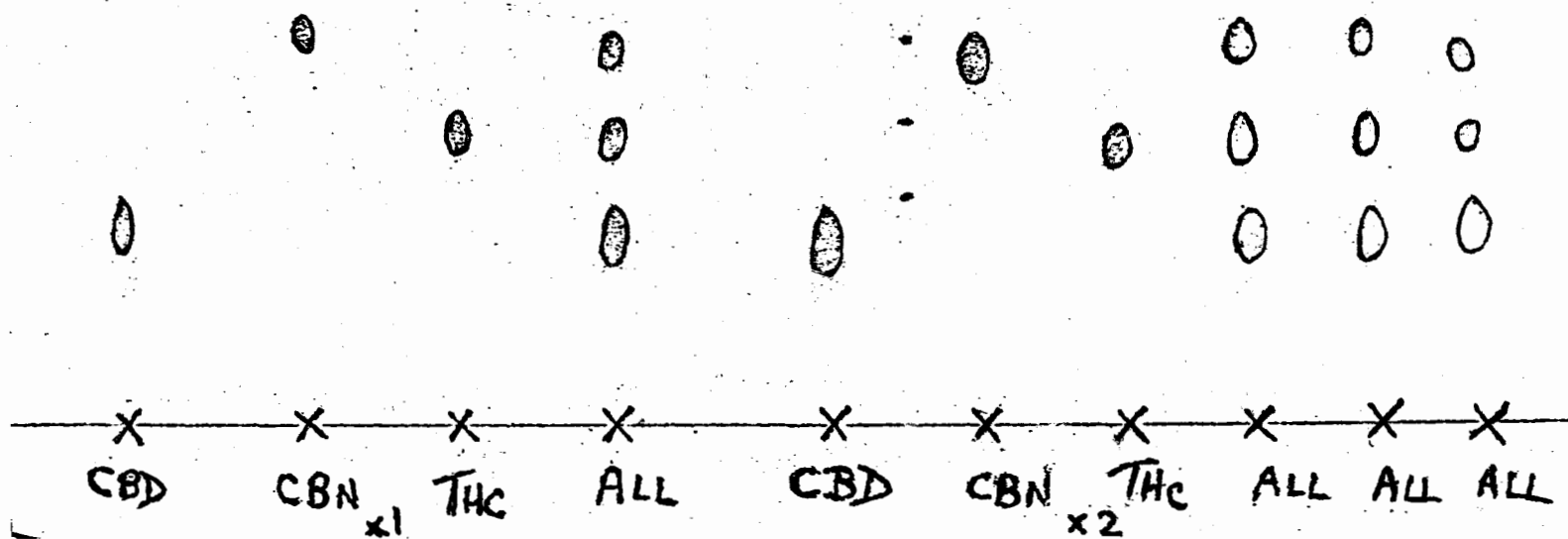
Cannabis Component	Retention time relative to DBT	Rf on SG 81 Paper
Cannabidiol	0.26	0.3
$\Delta^1$ -3, 4-trans Tetrahydrocannabinol	0.39	0.6
Cannabinol	0.64	0.8

REFERENCES

- Caddy, B. and R. Fish (1967), J Chromatog., 31, 584 - 587
- Hively, R.L., Mosher, W.A., and F.W. Hoffman (1966), J. Amer. Chem. Soc., 88, 1832 - 1833
- Lerner, P.(1969), Bull. Narcot., XXI, 3, 39 - 42
- Maunder, M. J. de Faubert (1969), J. Ass. Pub. Analyst, 7, 24 - 30
- Maunder, M. J. de Faubert (1969), Bull. Narcot., XXI, 4, 37 - 43
- Turk, R.F., Dharir, H.I., and Forney, R.B. (1969), J. For. Sci., 14, 3, 389 - 392



SG 81 + AgNO<sub>3</sub>  
Development: CHCl<sub>3</sub>  
Time 15 mins.



## The Analysis of Cocaine In Illicit Preparations

by James Moore  
Washington Regional Laboratory  
Bureau of Narcotics and Dangerous Drugs

The majority of illicit cocaine samples received in this laboratory are either cocaine-sugar or cocaine-procaine-sugar mixtures. Method I described herein was developed for the separation, identification and quantitation of cocaine-procaine mixtures. Procaine, which interferes in the ultraviolet determination of cocaine, is trapped on a 0.1N HNO<sub>3</sub> column while cocaine is passed through. After quantitation the isolated cocaine may be identified by infrared and/or T.L.C. techniques. Method II describes a procedure for the analysis of an unusual mixture of cocaine, tetracaine & quinine received by this laboratory. In this latter sample the concentration of each component was found to be less than 1%. In most cases the major diluent found in the cocaine samples was a sugar, such as lactose, mannitol, or dextrose.

## Procedure

### Method I

#### Cocaine - Procaine Mixture<sup>1</sup>

Apparatus - A glass chromatographic column 250mm. long by 22mm. i.d., with one end constricted to a stem 60mm. long.

Reagents - Diatomaceous earth<sup>2</sup>; HNO<sub>3</sub>, 0.1N aqueous solution; H<sub>2</sub>SO<sub>4</sub>, 0.5N aqueous solution; water-saturated chloroform, prepared by vigorously mixing equal volumes of chloroform and water and allowing the phases to separate; ammoniacal chloroform, prepared by vigorously mixing 2 ml. of ammonia and 100 ml. of chloroform and allowing the phases to separate.

Sample Preparation - Weigh accurately a portion of powdered sample equivalent to about 5mg. of cocaine (usually about 50-100mg. powder) into a 100-ml. beaker. Add 2 ml. of 0.1N HNO<sub>3</sub> and swirl beaker until powder is completely wetted. Add 3g. of diatomaceous earth and mix with a spatula until fluffy. To another 100-ml. beaker add 1 ml. of 0.1N HNO<sub>3</sub>, 2 g. of diatomaceous earth and mix until fluffy.

<sup>1</sup>Analysis based on a 5% cocaine - 5% procaine - 90% sugar mixture.

<sup>2</sup>Celite 545, acid - washed, Johns Manville, New York, N.Y.

Add this latter mixture to a glass chromatographic column with a plug of glass wool in the stem. Pack moderately. Add to the same column the sample mixture and again pack moderately. Dry wash the sample beaker with about 0.5 g. of diatomaceous earth wetted with a few drops of water and transfer to column. Pack moderately and overlay with a plug of glass wool.

Procedure - Place a 125-ml. separatory funnel directly beneath the prepared chromatographic column. Elute the cocaine from the column with 100 ml. of water-saturated chloroform. After the elution is complete add 20.0 ml. of 0.5N  $H_2SO_4$  to the separatory funnel and shake it vigorously for one minute. Allow the layers to separate and settle and then discard the chloroform. Prepare a standard cocaine solution in 0.5N  $H_2SO_4$  saturated with chloroform having a concentration of about 0.25mg./ml. Scan the sample acid layer and standard solution in the ultraviolet between 320  $\mu$  and 240  $\mu$  against a blank consisting of 0.5N  $H_2SO_4$  saturated with chloroform. Use the sample and standard absorption at 275  $\mu$  for quantitation.

The procaine is eluted from the column into a 500-ml. volumetric flask by passing 100 ml. of ammoniacal chloroform through the column followed by 100 ml. of water-saturated chloroform. Add 5 ml. of methanol to the flask and dilute to volume with ammoniacal chloroform. Prepare a standard procaine solution in 1% methanol-ammoniacal chloroform solvent having a concentration of about 0.010mg./ml. Scan the sample and standard solutions in the ultraviolet between 370 and 240 mu. against a blank consisting of 1% methanol-ammoniacal chloroform solvent. Quantitate using the absorption maximum at 280 mu.

#### Cocaine Identification

(A) Infrared - make the sample solution basic with ammonia and extract the cocaine free base with successive portions of chloroform, filtering each extract through cotton into a beaker. Evaporate the chloroform extracts to a concentration of about 2 mg./5-ml. Transfer a volume of chloroform equivalent to a 1-2 mg. of cocaine to a mortar containing about 200 mg. KBr. Evaporate the chloroform gently to dryness and prepare a KBr disk in the usual manner. Compare spectra to that of a standard cocaine prepared in a similar manner.



(B) Thin layer Chromatography - proceed as in (A), except evaporate chloroform extracts to give a concentration of about 1mg./1 ml. Use this solution for T.L.C. identification. Proceed using the T.L.C. systems described in (1).

Method II

Cocaine - Quinine - Tetracaine Mixture<sup>3</sup>

Apparatus - See Method I.

Reagents - Diatomaceous earth; sodium bicarbonate;  $\text{HNO}_3$ , 0.2N aqueous solution; HCl-NaCl solution, 1N and 0.1N aqueous solution with respect to HCl and NaCl, respectively; HCl, 0.1N aqueous solution; ammoniacal chloroform;  $\text{H}_2\text{SO}_4$ , 0.5N aqueous solution.

Sample Preparation - Column A - Weigh accurately about 100 mg. of powdered sample into a 100-ml. beaker, add 2 ml. of  $\text{H}_2\text{O}$  and enough  $\text{NaHCO}_3$  to make solution distinctly basic to litmus. Add 3 g. of diatomaceous earth and mix until fluffy. Transfer quantitatively to column as described in Method I.

Column B - Into a 100-ml. beaker add 3 ml. of 0.2N  $\text{HNO}_3$  and 5 g. of diatomaceous earth and mix until fluffy. Transfer to column and pack moderately.

Column C - Into an 100-ml. beaker add 2 ml. of the HCl-NaCl solution and 3 g. of diatomaceous earth and mix until fluffy. Transfer to column and pack moderately.

<sup>3</sup>Analysis based on a 5% cocaine-5% quinine-5% tetracaine and 85% sugar mixture.

Column D - Into a 100-ml. beaker add 2 ml. of 0.1N HCl and 3 g. of diatomaceous earth and mix until fluffy. Transfer to column and pack moderately.

Arrange columns so that elution will proceed from column A through B, C, and D.

Procedure - Place a 400-ml. beaker beneath column D. Elute column A with 100 ml. of water-saturated chloroform and allow eluant to pass through columns B, C, and D into beaker. Elute column B with 50 ml. of water-saturated chloroform and allow eluant to pass through columns C and D into beaker.<sup>4</sup> Elute column C with 25 ml. of water-saturated chloroform and allow eluant to pass through column D into beaker. Discard chloroform in beaker upon completion of elutions. Separate columns and proceed with individual component analysis as follows:

(A) Cocaine - Elute column D into a 250-ml. separatory funnel with 50 ml. of ammoniacal chloroform followed by 75 ml. of water-saturated chloroform. Upon completion of elution add 20.0 ml. of 0.5N H<sub>2</sub>SO<sub>4</sub> to the separatory funnel and proceed as in method I beginning with "and shake it vigorously for one minute"

<sup>4</sup>Observe column B under a fluorescent hand light; if column is functioning properly the fluorescent quinine will remain on column during initial elution.

(B) Tetracaine - Elute column C into a 500-ml. volumetric flask with 100 ml. of ammoniacal chloroform followed by 100 ml. of water-saturated chloroform. Add 5 ml. of methanol to the flask and dilute to volume with ammoniacal chloroform. Prepare a standard tetracaine solution in 1% methanol-ammoniacal chloroform solvent having a concentration of about 0.010mg./ml. Scan the sample and standard solutions in the ultraviolet between 380 and 240mu. against a blank consisting 1% methanol-ammoniacal chloroform solvent.

(C) Quinine - Elute column B into a 500-ml. volumetric flask as for tetracaine. Evaporate solvent in flask on a steam bath under a current of air. Dilute residue in flask to volume with 0.5N H<sub>2</sub>SO<sub>4</sub>. Prepare a standard quinine solution in 0.5N H<sub>2</sub>SO<sub>4</sub> having a concentration of about 0.010mg/ml. Scan the sample and standard solutions in the ultraviolet between 370 and 220mu. using the absorption maximum at 250mu. for quantitation.

#### Cocaine Identification

See Method I

#### Reference

- (1) Internal Revenue Service, "Methods of Analysis", Rev. 6-67, pp. 92-94.

THE IDENTIFICATION OF HASHISH  
BY  
THIN LAYER CHROMATOGRAPHY

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The forensic chemist may be required to identify various concentrates of the resinous material from *Cannabis sativa* (marijuana) known as hashish, kif or bhang. The evidence encountered in law enforcement is found in a variety of shapes, and colors. Four known cannabinols, soluble in methanol, may be separated by micro thin layer chromatography. The absorbent used is basic silica gel G supported on 1 x 3 glass microscope slides. Developer used is 1,1,1 trichlorethane and methanol (9:1). Visualization by acid vanillin shows four blue-green spots. This chemical approach to the identification of hashish is suggested as a supplement to microscopic examination.

**APPARATUS:**

Microscope slides - plain glass, 1" x 3" , cleaned in strong detergent, rinsed, allowed to stand in acid dichromate cleaning solution overnight, rinsed and stored in 50% methanol solution. Just before use, they are wiped dry with a lint-free towel.

Capillary tubes -- 75 mm. x 0.5-0.9 mm. diameter. Coating chamber and developing chambers -- two ounce, large mouth, screw capped bottles, 86 mm x 33 mm. TLC sprayer. Electric hot plate.

**SOLUTIONS:**

Absorbent --	50 grams silica gel G in 100 ml. 0.1 N NaOH
Developer --	5 ml. 1,1,1 trichlorethane/methanol (9:1)
Visualizer --	3 g. vanillin in 100 cc absolute ethanol plus 0.5 ml. concentrated H <sub>2</sub> SO <sub>4</sub>
Standard Control --	3.4 mg/cc. synhexl (Parahexyl, Abbott) in abs. ethanol or methanol extract of <i>Cannabis sativa</i>

**PROCEDURE:**

The suspected hashish is examined microscopically for the characteristic cystolith hairs or other plant particles from *Cannabis*

sativa. A 5% (saturated) solution of the suspected drug is made in 1 ml. absolute methanol by grinding in a mortar and pestle or simply by crushing with a glass rod in the bottom of a small test tube and stirring vigorously on a mechanical stirrer. Solution is assisted by warming. The small test tube is set aside in a vertical position, allowing the plant residues to settle to the bottom while the plates are being coated and activated.

A two ounce, wide mouth jar should be kept almost filled with absorbent solution. After mixing the slurry, two dry micro slides are dipped into the slurry, back to back, to a depth of 60 mm. Removing the slides, the excess slurry is allowed to drip back into the coating jar until a thin, but still liquid film, coats the slide. The two slides are separated, placed on their backs on a flat surface, bumped slightly to obtain a smooth thin film and allowed to air dry until the slurry has lost its shine. The micro plates are then activated on an electric hotplate at 52° C. for ten minutes and the excess silica gel wiped from the edges of the slide. The supernatant extract of hashish, free of plant material, is spotted 12 mm. from the bottom of the plate with capillary tube. Up to four overlay spots may be helpful in producing more distinct cannabinol spots, providing the total spotting area is held to 3 mm. or less and each spot is allowed to dry before a subsequent overlay is made. A reference and unknown are spotted on each slide.

In the meantime, 5 ml. developing solution has been placed in a 2 oz. wide mouth, screw capped, jar and allowed to saturate for 1 minute. When the spots are dry, the micro plate is inserted in the developing chamber and allowed to develop to a height of 50 mm. The micro plate is removed and allowed to air dry.

Visualization is accomplished with a fine vanillin spray soaking the suspected areas by repeated spraying. The micro plate is placed on its back on an electric hot plate at 52°C and observed for color development. A large reddish spot at Rf 75 indicates the system is operating. When 3 or 4 blue-green spots develop at Rf 40, 50, 68 and 88, the slide is removed from the hotplate. The blue-green colors fade. Many spots of different colors may develop from various plant pigments but only blue-green spots indicate a cannabinoid. Old or weak hashish may show only 2 or 3 blue-green spots.

**RULES AND REGULATIONS**

7069

**Chapter II—Bureau of Narcotics and Dangerous Drugs, Department of Justice**

**PART 320—DEPRESSANT AND STIMULANT DRUGS; DEFINITIONS, PROCEDURAL AND INTERPRETATIVE REGULATIONS**

**Listing of MDA, MMDA, TMA, JB-318, and JB-336 and Their Salts as Subject to Control**

In the matter of listing MDA, MMDA, TMA, JB-318, and JB-336 and their salts as "depressant or stimulant" drugs within the meaning of section 201(v) of the Federal Food, Drug, and Cosmetic Act, because such drugs have a potential for abuse because of their hallucinogenic effect.

Comments were received concerning JB-318 and JB-336, their salts, and all their position isomers, and all the salts thereof, on the proposal in this matter published in the FEDERAL REGISTER of March 10, 1970 (35 F.R. 4305), from the Colgate-Palmolive Co. through its Division, Lakeside Laboratories, and its wholly owned subsidiary, Lakeside Laboratories, Inc., Milwaukee, Wis., suggesting that the compounds N-ethyl-3-piperidyl benzilate methobromide and N-methyl-3-piperidyl benzilate methobromide which are Lakeside Laboratories' drug products Piptal and Cantil, respectively, not be listed as "depressant or stimulant" drugs within the meaning of 21 U.S.C. 321(v) because of the inability of these products to penetrate the blood-brain barrier and produce a hallucinogenic effect. Lakeside further requests that the bases, N-ethyl-3-piperidyl benzilate and N-methyl-3-piperidyl benzilate, not to be controlled since they are required to produce Piptal and Cantil.

It is the decision of the Director of the Bureau of Narcotics and Dangerous Drugs that the compounds N-ethyl-3-piperidyl benzilate methobromide and N-methyl-3-piperidyl benzilate metho-

bromide are not salts of the bases N-ethyl-3-piperidyl benzilate and N-methyl-3-piperidyl benzilate, and therefore are not encompassed by the subject proposal. It is the opinion of the Bureau of Narcotics and Dangerous Drugs that these compounds are properly designated as piperidinium bromide benzilates. Further, to insure proper control, it is necessary to place the bases under the control requirements of the Drug Abuse Control Amendments of 1965 as hallucinogenic drugs.

Therefore, pursuant to the provisions of the Federal Food, Drug, and Cosmetic Act (secs. 201(v), 511, 701, 52 Stat. 1055, as amended, 79 Stat. 227 et seq.; 21 U.S.C. 321(v), 360a, 371) and under the authority vested in the Attorney General by Reorganization Plan No. 1 of 1968 (33 F.R. 5611), and redelegated to the Director, Bureau of Narcotics and Dangerous Drugs (28 CFR 0.100), § 320.3 (c) (2) is amended by inserting the following:

**§ 320.3 Listing of drugs defined in section 201 (v) of the Act.**

\* \* \* \* \*

(c) \* \* \*

(3) Hallucinogenic effect:

<i>Established name</i>	<i>Some trade and other names</i>
MDA, its salts, and all its isomers, such as optical and position, and all the salts thereof.	3,4-methylenedioxy amphetamine (MDA) or 4,5-methylenedioxy amphetamine. 2,3-methylenedioxy amphetamine or 5,6-methylenedioxy amphetamine.
MMDA, its salts, and all its isomers, such as optical and position, and all the salts thereof.	5-methoxy-3,4-methylenedioxy amphetamine (MMDA) or 3-methoxy-4,5-methylenedioxy amphetamine. 6-methoxy-3,4-methylenedioxy amphetamine or 2-methoxy-4,5-methylenedioxy amphetamine. 2-methoxy-3,4-methylenedioxy amphetamine or 6-methoxy-4,5-methylenedioxy amphetamine. 6-methoxy-2,3-methylenedioxy amphetamine or 2-methoxy-5,6-methylenedioxy amphetamine. 5-methoxy-2,3-methylenedioxy amphetamine or 3-methoxy-5,6-methylenedioxy amphetamine. 4-methoxy-2,3-methylenedioxy amphetamine or 4-methoxy-5,6-methylenedioxy amphetamine.
TMA, its salts, and all its isomers, such as optical and position, and all the salts thereof.	3,4,5-trimethoxy amphetamine (TMA). 2,4,5-trimethoxy amphetamine or 3,4,6-trimethoxy amphetamine. 4,5,6-trimethoxy amphetamine or 2,3,4-trimethoxy amphetamine. 2,3,5-trimethoxy amphetamine. 3,5,6-trimethoxy amphetamine. 2,3,6-trimethoxy amphetamine. 2,5,6-trimethoxy amphetamine. 2,4,6-trimethoxy amphetamine.
JB-318, its salts, and all its position isomers, and all the salts thereof.	N-ethyl-3-piperidyl benzilate (JB-318). N-ethyl-2-piperidyl benzilate. N-ethyl-4-piperidyl benzilate.
JB-336, its salts, and all its position isomers, and all the salts thereof.	N-methyl-5-piperidyl benzilate (JB-336). N-methyl-2-piperidyl benzilate. N-methyl-4-piperidyl benzilate.

Any person who will be adversely affected by the foregoing order may at any time within 30 days from the date of its publication in the FEDERAL REGISTER file with the Chief Counsel, Bureau of Narcotics and Dangerous Drugs, Department of Justice, Room 613, 1405 I Street NW., Washington, D.C. 20537, written objections thereto. Objections shall show wherein the person filing will be adversely affected by the order and specify with particularity the provisions

of the order deemed objectionable and the grounds for objections. If a hearing is requested, the objections must state the issues for the hearing, and such objections must be supported by grounds legally sufficient to justify the relief sought. Objections may be accompanied by a memorandum or brief in support thereof. All documents shall be filed in six copies.

*Effective date.* This order shall become effective 31 days from the date of its

publication in the FEDERAL REGISTER, except as to any provision that may be stayed by the filing of proper objections. Notice of the filing of objections or lack thereof will be announced by publication in the FEDERAL REGISTER.

Dated: April 30, 1970.

JOHN E. INCERSOLL,  
Director, Bureau of  
Narcotics and Dangerous Drugs.

[F.R. Doc. 70-5502; Filed, May 4, 1970;  
8:45 a.m.]



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