MICROGRAM
Laboratory Division
Office of Scientific Support
BUREAU OF NARCOTICS & DANGEROUS DRUGS / U.S. DEPARTMENT OF JUSTICE / WASHINGTON, D.C. 20537

Vol. V, No. 7 July, 1972

A cocaine exhibit purchased in Boston, Massachusetts was cut with benzocaine, procaine, boric acid and p-methyl aminolphenol. The latter is used in photographic developers under the trade names "Metol", "Enol" and others.

Secobarbital (9.2%), phenobarbital (8.7%) and lactose were found in combination by the New York BNDD Regional Laboratory. This exhibit was a white powder in a clear plastic bag.

Pure cocaine base, purchased as methamphetamine, is reported by New York BNDD Regional Laboratory. The exhibit was in the form of translucient crystals and was found mixed with two LSD tablets.

Cocaine HCl 40%, procaine HCl 0.9% and magnesium sulfate in combination is reported from the Mexico City area.

Cocaine 78.9% and ephedrine have been found in combination in the New York area. This is the second encounter we have had with this combination. (See Microgram, Vol. V, No. 6, page 59.)

Marihuana bricks, apparently from Mexico, were recently reported by the Chicago BNDD Regional Laboratory. These bricks measured approximately 5" X 12" X 2 1/2" and were wrapped in blue construction paper inscribed: "Verde De Michoacan"--2-3000 ft.--Mexico 72--El Armadillo Vive--Feliz Ano Nuevo".

"Liquid Hashish" (Marihuana Oil) is reported from the New York BNDD Regional Laboratory. This exhibit consisted of 879 grams of liquid containing 16% tetrahydrocannabinol (THC).

A cocaine and procaine penicillin G combination has been encountered by the BNDD Washington D.C. Regional Laboratory. The exhibit consisted of double plastic bags containing about 27 grams of grey-white powder. Analysis showed 13.7% cocaine along with the penicillin. According to our pharmacologists, an estimated 15% of the American population is presently sensitized to penicillin to a varying degree. Allergic reactions are unpredictable and vary in severity. Injection of penicillin increases the chance of an allergic reaction. However, a reaction may be brought on by any dosage form or quantity.

M E E T I N G S

The 86th Annual Meeting of the Association of Official Analytical Chemists will be held October 9 - 12, 1972, at the Marriott Motor Hotel, Twin Bridges, Washington, D.C. About 1,300 chemists, microbiologists, physicists, and their administators are expected to attend, representing Federal, state, provincial, and local government agencies, universities, and industries in North America and elsewhere. About 230 papers will be given on new techniques, methods, and instrumentation for analysis of a wide variety of substances.

California Association of Criminalists

FALL 1972

40th Semi-Annual Seminar, October 19 - 21, 1972, Mansion Inn, Sacramento, California. For further information, contact Seminar Chairman, Alan Gilmore, Sacramento County District Attorney, Criminalistics Lab, 4400 "V" Street, Sacramento, California 95817, or phone: (916) 454-5704.

SPRING 1973

41st Semi-Annual Seminar, May 17 - 19, 1973, Sheraton Inn, Harbor Island, California. For further information, contact Richard Shaw, San Diego Coroner's Office, 5555 Overland Avenue, Building 14, San Diego, California 92123, or phone: (714) 278-9600.

BNDD Forensic Chemist Seminars for the coming fiscal year are planned as follows:

1. September 18 - 22, 1972
2. November 13 - 17, 1972
3. March 5 - 9, 1973
4. June 25 - 29, 1973

All sessions will be held at the BNDD National Training Institute, Washington, D. C. For more information and application forms, write to:

Assistant Director for Training
National Training Institute
Police Training Division
Bureau of Narcotics & Dangerous Drugs
1405 Eye Street, N. W.
Washington, D. C. 20537

A N N O U N C E M E N T

Middle Atlantic Association of Forensic Sciences (MAAFS) was formed July 22, 1972, by more than twenty charter members meeting in Washington, D. C.

Officers named are:

President:	Adam J. Sobotka Bureau of Narcotics & Dangerous Drugs Washington, D. C.
President-Elect:	Joseph Gormley Federal Bureau of Investigation Washington, D. C.
Secretary-Treasurer:	John J. Tobin U.S. Customs Baltimore, Maryland

Another organizational meeting is tentatively planned for October 12, 1972 at the Twin Bridges Marriott Hotel, Washington, D. C. The first semi-annual meeting is planned for March, 1973 in Williamsburg, Virginia.

For further information, contact Adam J. Sobotka, Forensic Chemist, Washington Regional Laboratory, 460 New York Avenue, Washington, D. C. 20537.

S E L E C T E D R E F E R E N C E S

Journal of Chromatographic Sciences, Special issue, "Analysis of Drugs of Abuse," (May, 1972.)

Sadtler Research Laboratories, Inc., has announced the recent publication of a new infrared grating reference spectra collection of common abused drugs.

For further information on this collection, address inquiries to Sadtler Research Laboratories, Inc., 3316 Spring Garden Street, Philadelphia, Pennsylvania 19104, or phone: (215) 382-7800.

MARIHUANA - METHAQUALONE MIXTURE

Robert K. Simon, Ph.D., Assistant Toxicologist
Leonard R. Bednarczyk, Ph.D., Chief Toxicologist
Forensic Science Laboratory
200 South Adams Street, Wilmington, Delaware 19801

Two recent contraband drug confiscations submitted to our Office contained respectively six and one unmarked aluminum foil packets each containing approximately 0.75 grams of a brown material. Analysis revealed that the material was a mixture of marihuana and methaqualone. While analysis has confirmed the presence of methaqualone on the street as Quaalude (W. H. Rorer, white tablets), this is the first case we have seen of its mixture with marihuana.

METHODOLOGY

The material was examined by microscopy, Duquenois-Levine test and thin-layer chromatography (t.l.c.) to identify Cannabis sativa L. (Marihuana). An alkaline (pH10) extract was analyzed by ultraviolet spectrophotometry (U.V.), t.l.c. and gas-liquid chromatography (g.l.c.) to confirm the presence of methaqualone.

REAGENTS

1. Duquenois Reagent (fresh). 2.0 grams of Vanillin (4-hydroxy- 3-methoxybenzaldehyde, Fisher, A.R.), and 2.5 milliliters of acetaldehyde (Eastman) were mixed with ethyl alcohol and diluted to 100 milliliters with the later.

2. Hydrochloric Acid. (Allied Chemical, A.C.S.) concentrated.

3. TLC I (Marihuana). Petroleum ether (Fisher, A.C.S., Boiling Range 38.0 - 53.3°C). Chloroform (Fisher, A.C.S.) (6:4) mixed in 125 milliliter screw-cap glass jars; t.l.c. may be run immediately.

4. Fast Blue Salt B. (azoic Diazo Compound 48) (Matheson, Coleman and Bell). (fresh daily). Approximately 0.1 grams was dissolved in 100 milliliters of water.

5. TLC II (Basic Drugs)[1]. Benzene (Fisher, A.C.S.), Dioxane (Fisher, A.C.S.), ethyl alcohol (Publicker Industries, 95%), ammonium hydroxide (Fisher A.C.S.), concentrated. (50:40:5:5). All the solvents were mixed in a separatory funnel and then permitted to stand overnight. The ammonia water was then removed and the solvent placed in a standard Desaga t.l.c. tank (Brinkman) lined with chromatographic paper. Two fifty milliliter beakers of concentrated NH_4OH were placed in the tank. The tank was sealed and allowed to equilibrate a minimum of one hour before use.

6. TLC III (Basic Drugs)[2] Butyl ether, ethyl ether (45:45:10) and diethylamine (all Fisher, A.C.S.). The reagents were mixed and poured into a standard chromatographic paper lined Desaga tank (Brinkman). Overnight equilibration was permitted prior to use.

7. Iodoplatinate, Acidified (t.l.c. spray). 0.25 grams of platinic chloride (A.C.S.) and 5.0 grams of potassium iodide were dissolved in distilled water. Two milliliters of concentrated HCl were added and the mixture was diluted to 100 milliliters with distilled water.

① Cochin, J. and Daly, G.W., Experientia, 18, 294-295 (1962).
② Nakamura, G.R., J. A.O.A.C., 49 (5) 108 (1966).

APPARATUS

U.V. Beckman Instruments DK-2A.

G.L.C. Hewlett-Packard model 5750 gas chromatograph with dual flame-ionization detector. The column is a 1.8 meter x 6 mm. x 4 mm. glass (Pesce Glass Company, Kennett Square, Pennsylvania) packed with 1% (/W) OV-1 (Ohio Valley Specialty Company) on 80/100 mesh High-Performance Chromosorb G (Johns-Manville). The column was conditioned for one hour at 100°C, programmed at 4°C/min. to 250°C, held at 250°C for two hours prior to use.

Column:	190°C or 225°C.
Inlet:	Same as column
Detector:	250°C
Carrier:	60 cc./min. He
Flame:	40 cc./min. H_2
	300 cc./min. Air

PROCEDURE

MARIHUANA[3]

Microscopic examination of a portion of the sample was performed to determine the presence of cystolith hairs.

A petroleum ether extract of the sample was then spotted on an Eastman Chromagram Sheet (6061 silica gel without Fluorescent indicator) 8.5 x 3 cm. vs. a standard marihuana extract. The t.l.c. sheet was developed at room temperature in TLC I a distance of 8.5 cm. (13 - 15 minutes). After air drying the sheet was immersed in Fast Blue Salt B reagent and air dried.

[3] Bednarczyk, L.R. and Schweda, P., "Rapid TLC Identification of Marihuana", presented at 6th Mid-Atlantic Regional Meeting, Baltimore, Maryland, February, 1970.

PROCEDURE (Cont'd.)

The above petroleum ether extract was evaporated to dryness. 1 milliliter of Duquenois reagent and 1 milliliter of conc. HCl were added to the residue and mixed. 1 milliliter of chloroform was added and mixed.

METHAQUALONE

The brown material was acid-washed with ethyl ether (Methaqualone slightly soluble) and the ether was discarded (It was determined that no acidic drugs were present). The residue was made alkaline (pH 10) and extracted with chloroform. The chloroform was filtered through anhydrous sodium sulfate and evaporated to near dryness on a steam bath. A drop of 1 N HCl was added and the extract was allowed to evaporate to dryness in the laboratory hood. Portions of the extract were analyzed by:

U.V. double beam spectrophotometry, using both a) 0.5 N HCl and b) ethyl alcohol as solvent (reference) systems.

T.L.C. 15 cm. runs were made on Analtech silica gel GF 20 x 20 cm., 250 micron glass plates, in systems TLC II and III at room temperature. The developed plate was observed under short-wave UV (254 nm) and spots were made visible with iodoplatinate spray.

G.L.C. A 5 microliter aliquot (Unimetrics #5010, 10 microliter syringe) was screened at a column temperature of 225°C. on the 1% OV-1 column. Confirmation of the suspected methaqualone peak was run at 190°C on the 1% OV-1 column.

RESULTS AND DISCUSSION

Several intact cystolith hairs and numerous fragmented hairs suggesting marihuana, were visible under the microscope.

The t.l.c. pattern obtained was not totally identical to a standard marihuana extract (see Figure I). The t.l.c. pattern of the alkaline extract of the sample did exhibit spots for cannabidiol, tetrahydrocannabinol and cannabinol. However, a diffuse white band extended from an R_x of 0.38 to 0.86 with the cannabinol spot on the apex of the band.

The Duquenois-Levine test yielded a purple color extracted into the chloroform layer.

Based on these three tests it was concluded that the brown material was cannabis sativa L. The appearance of the diffuse

RESULTS AND DISCUSSION (Cont'd.)

t.l.c. band lead the authors to suspect that an additional substance(s) was (were) mixed with the marihuana. The sample was thus further investigated.

Table I summarizes the data resulting from analysis of the alkaline extract of the sample

TABLE I. Analytical Data On Alkaline Extract

A. U.V. a. 235, 269 (0.5 N HCl)

b. 225, 264, 304, 316 (ethanol)

increasing absorptivity ←

B. T.L.C.

Compound	R_x (codeine) t.l.c. II	t.l.c. III
Morphine	0.27	0.23
Heroin	1.7	1.6
Phencyclidine	2.3	----
Demerol	1.8	2.6
Cocaine	2.2	2.9
Unknown Extract	1.9	2.3
Methaqualone	1.9	2.3
Methadone	2.2	3.2

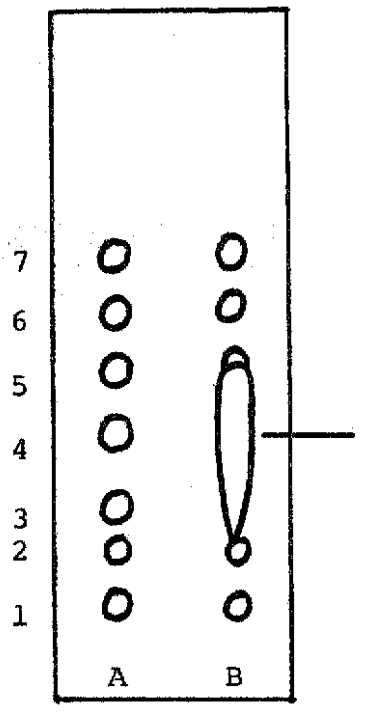
C. G.L.C.

	R.R.T. (codeine) g.l.c. (225°C)	g.l.c. (190°C)
Demerol	0.24	0.13
Methapyrilene	0.34	
Procaine	0.36	
Unknown extract	0.50	0.46
Methaqualone	0.51	0.46
Dextromethorphan	0.55	0.46
Methadone	0.56	0.45
Cocaine	0.58	
Benactyzine	0.64	
Pentazocine	0.68	
6-monoacetylmorphine	1.36	
Heroin	1.76	

Based on the above data it was concluded that the sample was a mixture of marihuana and methaqualone.

FIGURE I.

THIN-LAYER CHROMATOGRAM FROM MARIHUANA PROCEDURE



Key. A. standard petroleum ether extract of Cannabis sativa L.
B. petroleum ether extract of the sample.

Spot #	Color	Rx	Identity of Spots: Standard	Identity of Spots: Sample
1	Orange	0.0	Origin	Origin
2	Pink	0.38	Unidentified	
3	Pink	0.45	Unidentified	diffuse white band
4	Pink	0.60	Unidentified	
5	Violet	0.86	Cannabinol*	
6	Red	1.0	Δ^1 tetrahydrocannabinol*	
7	Orange	1.1	Cannabidiol*	

* Identified vs. t.l.c. of pure preparations obtained from J. Gunn, BNDD, (reference 3).

BNDD LABORATORY NOTES

DATE January 6, 1972

-80-

NO.

DRUG TYPE Narcotic

METHODOLOGY Infrared Identification

RAPID SEPARATION OF COCAINE FROM ADULTERANTS SUCH AS PROCAINE AND QUININE, AND SUBSEQUENT INFRARED IDENTIFICATION

Roger G. Fuelster
Forensic Chemist
CHICAGO REGIONAL LABORATORY, BNDD

Previous methods of separation of cocaine from adulterants involved lengthy column chromatography. The following procedure is not only rapid but yields a highly pure product.

PROCEDURE

Place sample in a small separatory funnel. Add approximately 15ml 2.8 normal hydrochloric acid. Extract with an equal volume of chloroform. Pass the extract through Whatman Phase Separating Paper #1PS. Evaporate the chloroform to dryness and dry at 105°C for 10 minutes. Obtain the IR spectra in the usual manner.

SUMMARY

This method effectively removes traces of hydrochloric acid which would otherwise hydrolize the cocaine during the final evaporating and drying stages.

FEDERAL REGISTER
Area Code 202 Phone 962-8626

DEPARTMENT OF JUSTICE

Bureau of Narcotics and Dangerous Drugs

AMPHETAMINES AND METHAMPHETAMINE

Production Quotas

On February 12, 1972, the final amphetamine and methamphetamine aggregate production quotas for 1972 were established by the Bureau of Narcotics and Dangerous Drugs and published in the FEDERAL REGISTER (37 F.R. 3194). The quota provided for a total of 989 kilograms of methamphetamine to insure adequate quantities of its anhydrous base for use in controlled methamphetamine products.

Subsequent to the establishment of the quota the Bureau has been informed that certain manufacturers require additional methamphetamine, a Schedule II controlled substance (21 CFR 308.12) for conversion in the production of noncontrolled substances. Such use was not previously called to the attention of the Bureau in considering the establishment of the total aggregate production quotas for methamphetamine. Therefore, the Director, Bureau of Narcotics and Dangerous Drugs, under the authority vested in the Attorney General by section 306 of the Comprehensive Drug Abuse Prevention and Control Act of 1970 (21 U.S.C. 826) and redelegated to the Director, Bureau of Narcotics and Dangerous Drugs by § 0.100 of Title 28 of the Code of Federal Regulations, orders that the 1972 production quotas for methamphetamine, expressed in terms of its anhydrous base, to be established as follows:

Methamphetamine (for conversion to noncontrolled substances only) (kilograms)	242

This order is effective upon the date of its publication in the FEDERAL REGISTER (6-23-72).

Dated: June 19, 1972.

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

[FR Doc.72-9493 Filed 6-22-72;8:46 am]

FEDERAL REGISTER
Area Code 202 Phone 962-8626

DEPARTMENT OF JUSTICE

Bureau of Narcotics and Dangerous Drugs

SCHEDULES OF CONTROLLED SUBSTANCES

Petition To Control Tobacco Under the Comprehensive Drug Abuse Prevention and Control Act

On May 22, 1972, the Bureau of Narcotics and Dangerous Drugs received a petition for the initiation of proceedings to control tobacco (Nicotiana Tabacum L.) under Schedule I of the Comprehensive Drug Abuse Prevention and Control Act of 1970 (Public Law 91-513). The petitioner is Woodrow A. Wallen of Seattle, Wash.

This petition was filed pursuant to the provisions of section 201(a) of the Comprehensive Drug Abuse Prevention and Control Act (21 U.S.C. 811(a)) which provides in part—

> Proceedings for the issuance, amendment, or repeal of such rules may be initiated by the Attorney General (1) on his own motion, (2) at the request of the Secretary, or (3) on the petition of any interested party.

In his petition, the petitioner states that:

> It is petitioners [sic] position, that the nonsmoking public is severely imposed upon by public smoking, and that the effects of public smoking have adverse effects on the health of nonsmokers.

The Director has determined that the petitioner has failed to demonstrate a direct personal stake in the outcome of the action proposed in his petition. In short, the petitioner has not established standing so as to require any action on his petition by the Bureau of Narcotics and Dangerous Drugs. Moreover, even had standing been established, Congress has specifically excluded "tobacco" from the definition of what may be considered a "controlled substance" (21 U.S.C. 802(6)).

For the above reasons the petition filed by Woodrow A. Wallen is denied in all respects.

This denial is effective upon the date of its publication in the FEDERAL REGISTER (7-8-72).

Dated: July 5, 1972.

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

[FR Doc.72-10603 Filed 7-7-72;10:05 am]