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MR. FREDERICK M. GARFIELD, BNDD'S Assistant Director for Scientific Support was elected to the Executive Committee of the Association of Official Analytical Chemists at its annual meeting, October 12-15, 1970, in Washington, D. C.

Election to the eminent scientific body came one year after Mr. Garfield received the "Fellow of the AOAC" award at the 1969 convention. In making Mr. Garfield a Fellow, the association was recognizing long and notable work. To qualify, awardees must have performed major service as Associate Referee, General Referee, committeeman, or officer for a period of ten years or more.

Mr. Garfield has been an Assistant Director in the Bureau of Narcotics and Dangerous Drugs since its formation

in April, 1968. Prior to that date, Mr. Garfield was Deputy Director of the U. S. Food & Drug Administration's Bureau of Drug Abuse Control, a Bureau he planned and formed as Special Assistant to the Commissioner. While Special Assistant to the Commissioner, Mr. Garfield also served as an Advisor to the Task Force on Narcotics and Drug Abuse, The President's Commission on Law Enforcement and Administration of Justice.

A veteran of over thirty-one years government service as a chemist and an administrator, Mr. Garfield is well known in both government regulatory agencies and in industries. Recognition for his work in the U. S. Food and Drug Administration included the FDA "Award of Merit," and the U. S. Department of Health, Education and Welfare's "Superior Service Award" and the "Superior Service Group Award."

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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.

Prior to government service, Mr. Garfield worked in private industry as a control chemist and as a research chemist. He is a graduate chemical engineer, with also a major in chemistry, from Washington University in St. Louis, his home town. He has attended several courses for administrators, including the Management Course of the American Management Association, and the Conference for Federal Executives on Business Operations, Brookings Institute.

Besides the AOAC, Mr. Garfield is a member of the American Chemical Society and the Association of Food & Drug Officials of the United States.

Hand-rolled cigarettes, reportedly originating in California, were found to contain crushed mint leaves impregnated with phencyclidine by the BNDD Special Testing and Research Laboratory.

LSD on marihuana was analyzed by the BNDD Chicago Regional Laboratory. The material, submitted by a Missouri law enforcement agency, consisted of four exhibits, each containing a few grams of material. One exhibit consisted of dry, grey-green leaves, which would have been suitable for smoking. The second exhibit consisted of a small bottle filled with blackish-colored leaves and a solution. A third exhibit consisted of blackish, hashish-appearing, damp material wrapped in foil. The fourth exhibit was a black, resinous material wrapped in foil. All four exhibits were positive for marihuana and LSD.

LSD gelatin flakes, which appear to be identical to those reported from London (See Microgram, Vol. III, No. 4, page 103, June, 1970) were recently analyzed in an exhibit from a Texas police department. Gelatin flakes have since been analyzed in exhibits from midwestern and western U.S.A. In some areas, it is called "Window Glass" and if round, "Contact Lens."

Heroin hydrochloride 46.6%, containing methapyrilene and lactose was analyzed by the BNDD Chicago Regional Laboratory. The usual concentration of heroin seen by the Chicago Laboratory is 5 to 10%.

LSD-amphetamine tablets have been reported by a Nevada crime laboratory. Only three tablets were available for analysis. These were green, uncoated, unscored, 11.4 millimeters diameter, 6.0 millimeters thick, and weighed 410 milligrams. Potency of the active ingredients is not known.

LSD cardboard squares marked "Sky River" were submitted by a Washington State law enforcement agency. The BNDD San Francisco Laboratory found the 2.5 x 2.5 centimeter squares to contain 500 micrograms of LSD.

"Opium Joints" or "O.J.'s" have been analyzed by a U. S. Customs laboratory in California, by a military laboratory in Asia, and have been seen in Australia.

"O.J.'s" are marihuana cigarettes which have been dipped in or have been smeared with opium. In at least one instance, the exhibit involved a "Kent" brand cigarette repacked with marihuana, with the second paper smeared on the inside with opium.

The military laboratory reports that only a small fraction of the marihuana cigarettes that they have analyzed have contained opium.

Red coated sodium secobarbital TABLETS rapidly spread throughout the West Coast during the summer months and are still being seen. The tablets appeared to be of commercial manufacture, however, could not be identified

by ballistics examination. According to the BNDD San Francisco Laboratory, the tablets are round, biconvex, unmarked, and unscored with a solid white core. Diameter: 9.4 millimeters, thickness: 5.0 millimeters, thickness at bevel: 2.0 millimeters. Average weight: 247 milligrams.

MDA (3,4-methylenedioxyamphetamine) as the free base only, and impregnated on sucrose to form a semi-solid mass, was identified by the BNDD San Francisco Laboratory. The federally controlled drug was contained in No. 00 clear, gelatin capsules. Later, an Oregon crime laboratory has reported a quantity of free base obtained from seizure of a clandestine laboratory. This laboratory may have been the source of the MDA capsules.

Sniffing of ballpoint pen ink, deodorants, decongestants, spot removers or spray-on foot powder is on the increase, according to a report by the Council on Drug Addiction of New York City. There is also an increase of student "pushers;" in the number of intravenous heroin users in public schools in the seventh, eighth, and ninth grades; and there is 40% increase in drug arrests of 16 to 20 year-olds in New York City in 1968 over 1967. ("Information Letter," Division of Narcotic Drugs, United Nations, ii, June, 1970.)

LSD "Peace Pill" tablets have appeared in a different size. The recent tablet has the same type of "peace" symbol, however, it is 3/16" rather than the previous 1/4" diameter.

"Falling Rain" mentholated cigarettes from Thailand have been analyzed. The cigarettes were found to be filled with tobacco for about 1/4 to 1/2 of their length starting from the filter with the remainder of the cigarette filled with marihuana. (A wide variety of cigarettes are re-packed with marihuana in the Far East.)

Cortleigh cigarettes, an Australian brand that has a gold band at the junction of the filter and the tobacco portion of the cigarette, have been found containing drugs. The gold band is cut and removed. The filter and the cigarette are then separated, so that a hollow can be made in one end of either or both. Hashish, or another drug, is then placed in the hollow; the filter and cigarette are rejoined; and the band is replaced.

LSD pentagon-shaped tablets are being seen, that range in color from off-white to yellow to yellowish-green to light green to blue-green to blue. BNDD first encountered them in both federal and local police exhibits from the Gulf of Mexico area in July. In August, the tablets were being reported in the Midwest, and in September, they were seen on the West Coast. Ballistics examination of tablets from Australian authorities showed that tablets from the same source were appearing in Australia's eastern cities.

The pentagonal tablets are biconvex and unscored. The distance from any corner to the opposite side is 4.2 to 4.3 millimeters. Length of straight edge: 2.4-2.7 millimeters; distance from one corner to second corner

from it: 4.3-4.4 millimeters; center thickness: 1.65-1.72 millimeters; edge thickness: 1.0-1.2 millimeters. Edges of one surface are ridged, especially at the corners. There are characteristic lumps and gouges near the edges of this surface. The other surface has faint, generally concentric striations near edges. There are also two vertical striations (lumps) on the vertical edge - one is at one corner, the other at the opposite corner. LSD content has ranged from 40 to 184 micrograms. Tablet weight has averaged about 25 milligrams.

Secobarbital sodium in clear gelatin capsules were recently seen in the BNDD San Francisco Laboratory.

TERM "MARIHUANA" DEFINED AS CANNABIS SATIVA L. BY 26 U.S.C. 4761 (2)  
INCLUDES ALL AGRONOMIC VARIATIONS OF CANNABIS, INCLUDING CANNABIS INDICA.

United States v. John Moore (E.D. Pa., No. 69-137; June 22, 1970; D.J. 12-62-163)

After a conviction, following a jury trial, for the unlawful transfer of marihuana in violation of 26 U.S.C. 4742 (a) and the unlawful acquisition of marihuana in violation of 26 U.S.C. 4744 (a)(1), the defendant moved for judgment of acquittal and for a new trial on the principal ground that the Government's proof did not establish that the substance transferred was Cannabis sativa L., a fatal omission since "... the term 'marihuana' means ... the plant Cannabis sativa L., 26 U.S.C. 4761(2)." The defendant contended that the substance might have been Cannabis indica which he asserted was not Cannabis sativa L. He further argued Cannabis indica was regulated exclusively by 21 U.S.C. 209 which pertained to specified poisons and drugs.

At the trial, the Government chemist had testified that the substance in question was marihuana but stated that he was unable to distinguish between sativa and indica.

In its opinion, the court traced the legislative history of the marihuana statutes in issue and concluded that both from the stand-point of congressional purpose as well as botanical classification, Cannabis indica was a variety of and intended to be included within the term Cannabis sativa L. for the purpose of 26 U.S.C. 4761(2).

In reaching its conclusion, the court cited chemical and pharmaceutical definitions contained in scientific authorities and also relied upon state court decisions pointing out that Cannabis indica and marihuana were merely geographically oriented names for Cannabis sativa L. Furthermore, the 21 U.S.C. 209 regulation pertaining to the indica variety said the court "... was intended to deal solely with the regulation of this commodity within China. As such, it would not relate to the variety of Cannabis within the territorial United States."

MEETINGS

National Association of Police Laboratories - BNDD Joint Symposium on Drug Identification, November 18-20, 1970, at the New Yorker Hotel, New York City. Registration fee \$20.00 includes luncheon and cost of printing and mailing copies of techniques presented. The program is designed to present the practical aspects of drug analytical procedures in the forensic laboratory. Techniques presented will pertain to the analysis of narcotics, depressants, stimulants, and hallucinogens.

Questions relating to the subject matter can be sent in advance or submitted during the first two days of the symposium. The inquiries will be given to the appropriate lecturer, who, if present, will answer them in the concluding segment of the presentation. Questions will also be answered at the conclusion of each talk.

Contact: National Association of Police Laboratories, c/o Suffolk County Police Laboratory, Veterans Highway, Hauppauge, New York 11787.

American Academy of Forensic Sciences annual meeting, Phoenix, Arizona, February 21-26, 1971. Other organizations meeting with the Academy are the British Academy of Forensic Sciences, the Canadian Society of Forensic Sciences, the National Association of Medical Examiners, the National Association of Firearm and Tool Mark Examiners, and the new Forensic Sciences Committee of the American Society for Testing and Materials.

"Forensic Sciences and the Environment" is the general theme of this year's meeting. Topics in the plenary sessions are: "Forensic Sciences in a Closed World Ecosystem," "Forensic Sciences and the Environment of the Young Adult," and "The Forensic Sciences and the Social Order; Judicial and Administrative Reforms: Directives for the Future."

Theme for Criminalistic Section is "Narcotics and Drug Analysis and Methodology." The Jurisprudence Section's principal topics are "Environmental Control," "Alcoholism," and "Drug Addiction." Evening programs are planned on interesting topics in each of these sections. Also, full schedules on timely subjects are being planned by the Questioned Documents, Pathology and Biology, General and the Psychiatry Sections.

Registration includes the privilege to attend the meetings; a copy of the 1970 "What's New;" one free drink at the Fellowship Hour on February 21; the Academy Annual Luncheon; the barbecue (with transportation to and from); and tickets for the Annual Reception and Banquet, February 26.

Advance registration is \$50.00. Forms are available from Arthur H. Schatz, J.D., Secretary-Treasurer, American Academy of Forensic Sciences, 750 Main Street, Suite 1000, Hartford, Connecticut 06103. Registration after January 20, 1971 or at the door will be \$60.00. Early registration is advised.

Forensic toxicologists may obtain applications for membership to the International Association of Forensic Toxicologists by writing to the Association, c/o Home Office Central Research Establishment, Aldermaston, Reading, Berkshire, United Kingdom. Only practicing toxicologists are eligible.

GLOSSARY

Submissions recently submitted by readers:

WINDOW GLASS	LSD gelatin flake, square
CONTACT LENS	LSD gelatin flake, round
HAWAIIAN SUNSHINE	LSD
CALIFORNIA SUNSHINE	LSD
PURPLE HAZE	LSD
ORANGE WEDGES	LSD Tablet (triturates)
BLUE HEAVEN	Hallucinogen
WHITE LIGHTENING	Hallucinogen
BIG O	Opium
BLACK STUFF	Opium
HEROIN	Scag
HEROIN	Smack
JONES	Habit
BUMMER	Habit
DOMES	LSD Tablets
BARRELS	LSD Tablets
FLATS	LSD Tablets
CHOCOLATE CHIPS	LSD
SQUIRRELS	LSD
WEDGES	LSD Tablets (triturates)
PEACE	LSD
STRAWBERRY FIELD	LSD Tablet (triturates)
OWSLEY'S	LSD Tablets
SMEARS	LSD
CRYSTAL	Methamphetamine
SPEED	Methamphetamine
BLACK BEAUTIES	Amphetamines
DEXIES	Dexedrine (d-amphetamine sulfate)

DATE June 8, 1970

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NO. 5

DRUG TYPE Amphetamine

METHODOLOGY Microcrystalline Tests

## MODIFICATION OF THE MICROCRYSTALLINE TEST FOR d-AMPHETAMINE

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Bureau of Narcotics & Dangerous Drugs

The presence of amphetamine is determined microscopically by the gold chloride volatility test. d-Amphetamine and dl-amphetamine can be easily differentiated since they exhibit entirely different crystal types. d-Amphetamine and l-amphetamine produce similar types of crystals using this test. A modification of this test involves the addition of known l-amphetamine to a suspected d-amphetamine preparation. The subsequent formation of dl-amphetamine is positive identification for d-amphetamine since only the addition of the d and l isomers can produce racemic amphetamine. This procedure should be applicable to optical isomers of other volatile amines.

Most commercial d-amphetamine preparations contain small amounts of dl-amphetamine, and this must be taken into consideration in interpreting the test. Best results are obtained if equal amounts of d and l amphetamine are mixed together.

PROCEDURE

Prepare a standard l-amphetamine solution of approximately 0.2 mg/ml. An excellent test for amphetamine can be obtained with as little as 10 micrograms of amphetamine.

Perform the volatility test for d-amphetamine in the usual manner.<sup>1</sup> After the d-amphetamine-gold chloride crystals are observed note the presence or absence of any dl-amphetamine-gold chloride crystals. Remove the cover slip and add a fresh drop of gold chloride solution. Add one drop of the standard l-amphetamine solution to the slide and replace the cover slip. The formation of large quantities of dl-amphetamine-gold chloride crystals confirms the presence of d-amphetamine.

1. A.O.A.C., Methods, 10, 32.288, p. 597.

BUREAU OF NARCOTICS AND DANGEROUS DRUGS / U.S. DEPARTMENT OF JUSTICE

BND-115 (9/69)



# BNDD LABORATORY NOTES

DATE June 9, 1970

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NO. 6

DRUG TYPE Glutethimide

METHODOLOGY Infra-Red Spectrophotometry

## RAPID IDENTIFICATION OF GLUTETHIMIDE (DORIDEN) TABLETS

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Chicago Regional Laboratory  
Bureau of Narcotics & Dangerous Drugs

### OBJECTIVE

A rapid direct infra-red identification of the principal drug in tablet and capsule formulations is sought.

### BACKGROUND

Existing methods for qualitative analysis of drug dosage forms calls for selective solvent extraction and isolation of the drug from the excipient and binding material. In some cases, where the tablet weight is close to the weight of the drug ingredient, direct infra-red examination of tablet scrapings or powder from the capsule can be tried. This works well for example on Roche's Noludar 300 (Methyprylon) and fails on the tablet inserts of propoxyphene HCl (Darvon) in Lilly's Darvon compound capsules. It was decided to try the technique on another commonly encountered drug, namely glutethimide (Doriden).

### APPARATUS

Infra-red Spectrophotometer

### PROCEDURE

Simply scrape off the tablet or remove some of the powder from the capsule and make directly into a KBr disk.

### RESULTS AND DISCUSSION

The procedure described actually gave infra-red curves that were sharper in detail than glutethimide extracted with chloroform from the tablet material. Apparently the crystalline structure is altered by the chloroform extraction.

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BND-115 (9/69)

DATE 7/13/70

NO. 7

DRUG TYPE Hallucinogens

METHODOLOGY Column Chromatography and Spectrophotometry

CHROMATOGRAPHIC SEPARATION & DETERMINATION OF PHENCYCLIDINE  
HYDROCHLORIDE (PCP) IN THE PRESENCE OF LSD

Paul DeZan  
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New York Regional Laboratory  
Bureau of Narcotics & Dangerous Drugs

BACKGROUND

Capsules have appeared on the illicit market, identified as "Product IV", and consisting of a mixture of Phencyclidine Hydrochloride (PCP) and Lysergic Acid Diethylamide (LSD). The average capsule dosage was found to be 200 mcgs. of LSD and 2.7 mgs. of PCP.

The LSD content was determined by spectrophotofluorimetry, according to the procedure described by Canaff and DeZan.<sup>(1)</sup>

PCP was separated from LSD by column chromatography utilizing a 5% Sulfamic Acid column and quantitated by UV spectrophotometry.

QUANTITATION

Transfer and accurately weighed portion of the sample powder (equivalent to the contents of one capsule) into a beaker. Triturate the powder with 3 x 5 ml. portions of chloroform filtering each extract into another beaker. Evaporate the combined chloroform extracts to dryness. To the residue add 2 mls. of water and stir vigorously. Make solution alkaline with a few crystals of sodium carbonate and add 3 grams of acid washed Celite 545. Mix thoroughly and quantitatively transfer into a chromatographic column containing a pledget of glass wool. On top of the packed column place a 2.1 cm. circle of filter paper (Whatman #540) and a small pledget of glass wool. This column is designated as Column I.

Prepare a second chromatographic column consisting of 2 mls. of 5% Sulfamic Acid and 3 grams of acid washed Celite 545. Place filter paper and glass wool on top of the packed column. This column is designated as Column II.

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Mount the columns so that the effluent from Column I flows into Column II. Elute Column I with 50 mls. of water-saturated ether allowing the eluant to flow into Column II. After rinsing the tip of Column I discard the column and the ether extract. Elute Column II with 75 mls. of water-saturated chloroform. The PCP will come through the column with the eluant and the LSD will remain trapped on the column. Rinse the tip of the column with chloroform and evaporate the collected effluent to dryness. Dissolve the residue with 5.0 mls. of 0.1N HCl. Scan the solution on a spectrophotometer and compare against a standard solution of PCP using the 262 m $\mu$  peak for quantitation.

#### QUALITATIVE ANALYSIS

Transfer the acid sample solution into a 30 ml. separatory funnel and extract with 3 x 10 mls. of chloroform. Evaporate the combined extracts to dryness. To help crystallize the residue, add 2 mls. of anhydrous ether, swirl and decant. Prepare a KBr disc of the dry residue. Obtain the infrared spectrum and compare to that of a standard curve for Phencyclidine Hydrochloride.

TLC is used to specifically identify LSD. Although PCP did not interfere in the TLC procedure, the free base form did cause a methanolic solution of the extracted two drugs to become turbid. This definitely interfered with the polarimetric measurement for d-LSD. A set of columns were devised so that the PCP could be retained and the LSD liberated. This consisted of a basic column (identical to Column I of quantitative procedure) and a 2% citric acid column (prepared as described for Column II). Elute the basic column with 30 mls. of water-saturated ether, allowing the effluent to enter the citric acid column. Elute this column with sufficient water-saturated chloroform (75-90 mls.) to insure complete removal of the first fluorescing band off the column. The residue obtained, after evaporation of the chloroform solution should be sufficiently clean for polarimetric analysis.

#### DISCUSSION

PCP, in the presence of LSD, has been successfully separated and determined by column chromatography and UV spectrophotometry.

LSD is quantitatively determined by diluting a separate sample portion with methanol and recording the fluorescence spectrum of the final solution. The presence of PCP did not interfere with the spectrophotofluorimetric analysis.

Circular filter paper (Whatman #540, 2.1 cms.) has been placed on top of the packed column in order: (1) to prevent disruption of the column and thus allow for maximum concentration and an even distribution of the drugs at the top of the column, and (2) to decrease the rapid rate of flow of the eluant so that the problem of column stripping is minimized.

REFERENCES

1. "Determination of LSD in Illicit Samples by Fluorescence Spectroscopy",  
by R. Canaff and P. DeZan, pending publication in Microgram.

DATE July 29, 1970

NO. 9

DRUG TYPE Depressant Drugs

METHODOLOGY

EXTRACTION AND CLEAN-UP OF MEPROBAMATE FROM  
TABLET MATERIALS AND OTHER DRUGS

Roger G. Fuelster  
Bureau of Narcotics and Dangerous Drugs  
Chicago Regional Laboratory

PROBLEM:

Meprobamate alone or in combination with other drugs is difficult to obtain in pure form for infra-red determination.

METHOD:

- A. Apparatus: 3 - 60ml separatory funnels
- B. Reagents: 1N NaOH  
1N H<sub>2</sub>SO<sub>4</sub>  
Chloroform - reagent grade

PROCEDURE:

Place sample in separatory funnel and dissolve in 20ml H<sub>2</sub>O and 3ml 1N H<sub>2</sub>SO<sub>4</sub>. Extract with CHCl<sub>3</sub>, collecting extract in second funnel. Wash extract with 10ml H<sub>2</sub>O and 1ml 1N H<sub>2</sub>SO<sub>4</sub> and transfer CHCl<sub>3</sub> to third funnel. Wash extract with 20ml H<sub>2</sub>O and 3ml 1N NaOH. Filter extract through cotton and evaporate to dryness. Dry briefly at 105° and scratch oily residue obtained with a spatula until a crystalline powder forms. Obtain IR spectrum in usual manner.

For quantitative determination, weigh sample - make quantitative extractions and weigh residue obtained.

If other drugs are also present, the basic drugs may be recovered by making the aqueous solution remaining in funnel one basic with 5ml 1N NaOH, and extracting with CHCl<sub>3</sub>. The acid drugs may be recovered by acidifying the aqueous solution remaining in funnel three with 5ml 1N H<sub>2</sub>SO<sub>4</sub> and extracting with CHCl<sub>3</sub>. Filter all extracts through cotton. Add MeOH and several drops HCl to the extract containing the basic drugs. Evaporate extracts to dryness and dry at 105°C. Identify by IR or other convenient method.

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