# MICROGRAM

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

Washington, D. C. 20537
Office of Science and Drug Abuse Prevention
Vol. II, No.3 Laboratory Operations Division September, 1969

BNDD Forensic Chemist Seminars are tentatively planned to be held in Washington, D. C., as follows:

December 1-5, 1969
February 9-13, 1970
April 27 - May 1, 1970

For more information and application forms, write:

Bureau of Narcotics and Dangerous Drugs
Washington, D. C. 20537

ATTN: Chief, Special Programs Division (TRNS)

Association of Official Analytical Chemists, 83rd Annual Meeting will be held October 13, 1969, at the Marriott Motor Hotel, Twin Bridges, Washington, D. C.

About 1500 chemists, microbiologists, physicists, and their administrators are expected to attend, representing Federal, State, Provincial and local government agencies, universities and industries throughout North America. Over 230 papers will be given on new techniques, methods and instrumentation for the analysis of drugs and other commodities.

Dr. Daniel Banes, Director, Division of Pharmaceutical Sciences, U. S. Food and Drug Administration, will present the AOAC Presidential Address, October 13, at the opening session. Other speakers will be Dr. Henry M. Fales, National Heart Institute, who will talk on Spectrometry and Dr. D. P. Schwartz, U. S. Department of Agriculture, who will discuss methods for isolation and characterization of constituents of natural products. Dr. Herbert L. Ley, Jr., Commissioner of Food and Drug Administration, will be a featured speaker, and the Harvey W. Wiley Award will be given to Dr. C. O. Willits, retired from the U. S. Department of Agriculture, for his outstanding contribution to analytical chemistry. The registration fee for the meeting will be $3.00. For additional information contact:

Association of Official Analytical Chemists
Box 540
Benjamin Franklin Station
Washington, D. C. 20044

Mr. Frederick M. Garfield, Assistant Director, Office of Science and Drug Abuse Prevention, BNDD will be one of eleven scientists to receive the 1969 "Fellow of the AOAC Award" to be presented at the AOAC upcoming annual meeting October 13, 1969. The "Fellow of the AOAC Award" was established as an honorary award in 1961 to recognize long and notable work for the association. Awardees must have performed major service as Associate Referee, General Referee, committeeman, or officer for a period of ten years or more.

5th International Meeting for Forensic Sciences, was held June 5-11, 1969, in Toronto, Ontario, Canada. Several hundred delegates from almost forty countries heard a wide variety of papers presented in criminalistics, jurisprudence, pathology, psychiatry, questioned documents and toxicology. They were hospitably received by the City, Province and Canadian officials and citizens. The meeting was organized by the International Association of Forensic Toxicologists and the Third International Meeting in Questioned Documents. The program was well planned and executed by outgoing President D. M. Lucas and his hard working committees.

The next meeting chaired by the new president, Thomas K. Marshall, M.D., Belfast, Northern Ireland, will be held in Belfast in 1972.

Chief Chemist Jerry Nelson, BNDD Chicago Regional Laboratory, is serving as an advisor to the Illinois Law Enforcement Commission which is considering a new forensic laboratory system for the State of Illinois as well as a number of other items concerned with criminalistics.

SELECTED REFERENCES:

Davila, D. and Supek, Z. J. Pharm. Pharmac., "5-Hydroxy-indole compounds in the perfusates from frog head", 21, 53-54 (1969) (Letter to the editor).

Phillips, G. F. and Mesley, R. J. J. Pharm. Pharmac. "Examination of the hallucinogen 2,5-dimethoxy-4-methylamphetamine," 21, 9-17 (1969).

Laboratory Management "New Role for the Clinical Laboratory: Monitoring the 'Mainliner' " August, 1969, pages 30 and 31.

Nakamura, G. R. and Meuron, H. J. Anal. Chem. "Assay for Heroin in Illicit Preparations Using Partition Chromatography," 41, 8 (July, 1969).

Aldrichimica Acta, Vol. 2, No. 2, 1969, contains information about a reducing agent developed by a Czechoslovakian scientist. Aldrich Chemical Company, Milwaukee, Wisconsin plans to market the compound, sodium dihydro-bis-(2-methoxyethoxy)-aluminate, which can be substituted for lithium aluminum hydride in synthesizing drugs. The new chemical is said to act like lithium aluminum hydride, however, it is soluble in many organic solvents, is quite stable to air oxidation, reacts calmly with water and does not ignite spontaneously.

Superintendent of Documents has a list, "Selected Publications Relating to... Narcotics and Dangerous Drugs". The list includes twenty-eight publications varying in price from $.05 for "Narcotics, Some Questions and Answers" to a $4.00 book, "Ethnopharmacological Search for Psychoactive Drugs, 1967." The list and the publications are available from the Superintendent of Documents, Government Printing Office, Washington, D. C. 20402 .

New Book:

E. G. C. Clarke, Editor: Isolation and Identification of Drugs in Pharmaceuticals, Body Fluids and Postmortem Material. The Pharmaceutical Press, London.

Almost 900 pages, containing four parts: Part 1, Analytical Techniques; Part 2, Analytical and Toxicological Data: Monographs; Part 3, Indices to Analytical Data; and Part 4, Appendices, the book, according to the dust jacket, "... is a practical manual and data book for those who are faced with the problem of identifying an unknown drug which may be present in a pharmaceutical product, in a specimen of tissue or body fluid from a living patient, or in postmortem material... it includes rapid screening methods for the hospital biochemist working in circumstances where speed may make the difference between life and death, and precise techniques for the forensic scientist whose findings must stand up to cross-examination in a court of law; simple methods for the chemist working under primitive conditions in the field, and sophisticated procedures for the research worker with full instrumentation at his disposal."

Price: $39.00. Rittenhouse Book Store Inc., 1706 Rittenshouse Square, Philadelphia, Pennsylvania 19103.

BNDD Laboratories have analyzed a wide variety of substances. These include:

STP
Amphetamine
Methamphetamine
Barbiturates
Amphetamine-Barbiturate Combinations
Ritalin
LSD
Marihuana
Heroin
Cocaine
Phencyclidine
Mescaline
Diethyltryptamine
Dimethyltryptamine
Heroin-Cocaine Combinations
JB-336
JB-318
Methapyrilene
Darvon
Thorazine
MDA
Librium
Doriden
Morning Glory Seeds
Meprobamate
Hawaiian Baby Wood Rose
Psilocybin
Peyote
Benactyzine
Valium
Dimenhydrinate
Talwin
Asthmador
Catnip
Mephentermine

LSD Tablets, known as "Barrels", have been analyzed from Virginia, New Jersey, Minnesota, Michigan, Texas, Pennsylvania, New York, Kentucky, and Puerto Rico. All contained dextrose and corn starch, and were pink and white, green and white, or blue and white. The tablets were round, flat and unscored. They were about 5 millimeters in diameter, 4 millimeters thick and weighed about 90 milligrams. LSD potency: 90 to 115 micrograms.

LSD Tablets, called "Peace Tablets" and "Peace Pills", not to be confused with Phencyclidine HCl "Peace Pills", have been analyzed from both coasts and midwestern United States. The tablets are crudely made and are embossed with the so-called "peace" symbol, which resembles a bird's footprint inside of a circle. The tablets contained a large amount of spray-dried skimmed milk and a small amount of dicalcium phosphate. The tablets were round, flat, unbeveled, unscored, light green, rose pink, white or blue in color, 6.5 millimeters in diameter and ranged from 3.3 to 3.9 millimeters in thickness. Tablet weight varied from 137--146 milligrams. Potency ranged from 90 to 114 micrograms.

Methapyrilene is still appearing with heroin in our Chicago laboratory. Several exhibits also contained quinine, however, quinine has not been appearing in recent evidence. Methapyrilene also was found in yellow capsules alleged to have contained a barbiturate.

Hawaiian Baby Wood Rose was identified by our New York laboratory in bulk powder alleged to have contained LSD.

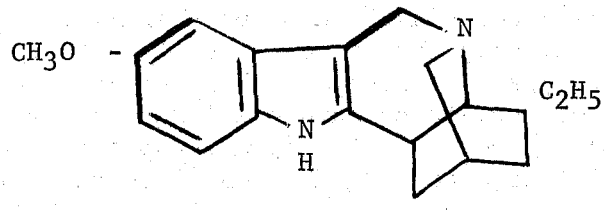
STP "Pumpkin seed" Tablets have been analyzed in our laboratories. These are about 16 millimeters long, about 7.2 millimeters wide, about 4.2 millimeters thick at center, with an edge thickness of about 2 millimeters. The well-made tablets contained approximately 4.8 milligrams of STP per tablet. If coated, they would have the same general shape as Mead Johnson Laboratories Natalins Basic tablets. The "pumpkin seeds" are yellow, about the same shade as Merck, Sharp & Dohme Aldomet tablets. (See the Product Identification Section, page 16, Physicians' Desk Reference, 1969.)

Apparently the "East Village Other", New York City, alluded to these tablets in the July 23, 1969, issue. An item about rising prices due to an alleged scarcity of marihuana included the statement: "... Magic pumpkins seeds are in the area. Also, much acid and reds."

LSD "Sunshine" Tablets appeared in two sizes at a "rock" festival in Texas. Tablets of both sizes were orange in color. One size was round, flat, unscored, unbeveled, about 3.9 millimeters in diameter, and 3.2 millimeters thick, with and average tablet weight of 0.0418 grams. They contained 256 micrograms of LSD. The other size was also flat, compressed tablets, about 6.4 millimeters in diameter and about 3.4 millimeters thick. Average tablet weight was 0.1073 grams. These contained 255 micrograms of LSD. The tablets were poorly made. The August 29 - September 4, 1969, Berkeley Barb states:"...fine orange tablets of acid are around ..."

CORRECTIONS:

Vol. I, No. 2, page 11 (page 3, old page number): Incorrect structure of Ibogaine as noted by Alexander T. Shulgin, Ph.D., Lafayette, California. According to Usdin, Earl and Efron, Daniel H. Psychotropic Drugs and Related Compounds, Public Health Service, U. S. Department of Health, Education and Welfare, the structure should be:




IBOGAINE

Our faces are red department: Front page Vol. II, No. 2, next to last sentence in paragraph three must be disowned. We really did not invent a new instrument. We just got so enthusiastic about our new laboratories that gremlins crept into our typewriter.

Analytical methods in Microgram do not have official status. There has been no attempt to validate these procedures under all conditions, therefore, they should serve only as guides. Publication approved by Bureau of the Budget, April 8, 1969.

Letters to the Editor:

Re: Microgram, II, 2, 51-52:

"...We disagree with the statement "If there is no fluorescence there is no LSD present", made in the final sentence of the second paragraph of the above paper.

The method indicates that 30 ml of chloroform is examined under uv light. Assuming a potency of 150 mcg LSD/tablet, the concentration of the solution would be 10 - 15 mcg LSD/ml. We have found that a concentration of 20 mcg/ml gives no fluorescence under long wave uv light. Therefore, if the analysis were stopped due to the absence of fluorescence, erroneous negative results would be obtained.

/s/ Anthony Romano, Jr.

/s/ Roger Canaff

BNDD New York Regional Laboratory

"... The sentence in question should read "there is probably no LSD present." An absence of fluorescence does not definitely preclude the presence of LSD, as it is sometimes difficult to detect in some samples.

Tests in our laboratory show that LSD in chloroform does fluoresce at the 20 mcg/ml level.

Two ml. volumes of chloroform solutions of LSD base were prepared containing concentrations of 20 mcg/ml., 10 mcg/ml., 5 mcg/ml., and 2 mcg/ml. Fluorescence under long wave ultraviolet light was noted in all solutions. Results were checked and confirmed by Mr. T. Tomczak and Mr. J. Moore of the Washington Regional Laboratory."

/s/ Albert R. Sperling, Ph.D.
BNDD Special Testing & Research Laboratory
Washington, D. C.

## "Cannabis indica" or "Cannabis sativa"?

By: Ferris H. Van Sickle, Forensic Chemist
Chicago Regional Laboratory, BNDD

Many federal, state, county and city chemists in various parts of the country have in recent years been pressed into emergency service to provide microscopic and chemical identification of marihuana for local law enforcement agencies.

It is not unusual to find air and water pollution chemists, health department chemists, industrial hygiene chemists and even in a few instances dairy chemists and bacteriologists who have acquired expertise in Marihuana identification and now qualify as expert witnesses in their respective local courts. They are almost always given support and training by qualified city, state and federal forensic chemists.

In some courts trouble begins when the newly qualified marihuana expert is asked to differentiate between "Cannabis indica" and Cannabis sativa." State laws in some areas of the country are incorrect on this. They treat the two as being different species of marihuana. A current example of this is to quote in part from a "Special Memorandum #70, November 15, 1968" issued to State law enforcement officers by the Wisconsin State Attorney General's office:

> "Wisconsin Statute sec. 161.275 (1) prohibits, among other things the sale of marijuana for smoking or beverage purposes. Section 161.275 (3) prohibits, among other things, the use of marijuana for such purposes. No statute, however, defines the word "marijuana."
>
> Technically, there are two species of marijuana, Cannabis sativa L and Cannabis indica. Only one of these species, however, comes within the statutory definition of marijuana. Section 161.01 (14) lists Cannabis as a narcotic drug. But sec. 161.01 (13) defines Cannabis only as Cannabis sativa L. Thus for the purposes of sec. 161.275 only Cannabis sativa L is considered marijuana.
>
> The second species of marijuana is Cannabis indica. This is not, however, considered to be narcotic for the purposes of the marijuana statute. Cannabis indica is classified as a poison in sec. 151.10 and its sale or delivery (except in special cases) is punishable under sec. 151.12."

Actually there is only one species of marihuana, Cannabis sativa, of which Cannabis indica is only a variety. There are other varieties - Cannabis americana, nebraska, minnesota, etc., all agronomic varieties depending on where it is grown.

There are many references which chemists can use when asked the "indica" question in court - to name several:

(1) "Marihuana, its identification," U. S. Treasury Dept., Bureau of Narcotics, United States Government Printing Office, Washington, D. C. 1948, quote page 2, in part "Cannabis indica, Cannabis americana, and other terms are rather loosely used to designate such agronomic variations. When seed produced in one place is planted in another where different soil and climatic conditions prevail the plants will resemble those from which the seed was harvested. If, however, such plants be cultivated in the new locality for several generations the characteristics of the local variety appear and the plants can no longer be differentiated. From this fact, among others, it is definitely established that all are agronomic modifications of the plant Cannabis sativa, of which only one true botanical variety is recognized."

(2) "The Dispensatory of the United States of America, Centennial (22nd) Edition, Philadelphia and London, J. B. Lippincott Co. Copyright 1937, p.275 on Cannabis," the hemp plant of India has been considered by some as a distinct species, and named Cannabis indica, but the most observant botanists, up on comparing it with our cultivated plant, have been unable to discover any specific difference. It is now, therefore, regarded merely as a geographic variety."

(3) United Nations "Bulletin On Narcotics" Vol II No. 4 Oct. 1950, "Cannabis" by Dr. R. J. Bouquet starting on page 14, and quoting from p.20: "To sum up, there is only one species of hemp (Cannabis sativa), of which Cannabis indica is only a variety. Both varieties have textile fibres and oleaginous seeds, and are capable of providing inebriant resin if the circumstances of habitat and climate provide the necessary conditions."

(4) "Marihuana - A review of the Literature For Analytical Chemists" by A. E. Hodapp, chemist, U. S. Customs Laboratory, New Orleans, La. 1959 - he refers to the Encyclopedia Britannica 1950, Vol. II, "there is only one species of true hemp (Cannabis sativa); however agronomic modification due to differences in soil and climate conditions are known under such names as Cannabis indica, Cannabis americana, Cannabis africana, etc. However, when these modifications are planted in

another region, they cannot be distinguished from the native variety after a few generations."

(5) Marihuana, America's New Drug Problem, by R. P. Walton Lippincott, 1938 p.41 "The genus Cannabis is generally regarded by botanists as monotypic, and the one species Cannabis sativa is now to include the half dozen forms which have been described under different names and which are cultivated for different purposes."

(6) "Forensic Aspects of Cystolith Hairs of Cannabis and other plants." by George R. Nakamura, Alcohol and Tobacco Tax Laboratory, Internal Revenue Service, Box 36075, San Francisco, California 94102. Journal of the Association of Official Analytical Chemists Vol. 52, p.9, 1969 "although this difference in cystolith hair structure was observed between Humulus lupulus and Humulus japonica, the latter originating in China and Japan, the cystolith hairs of Cannabis showed no significant structural differences with respect to geographic origin or whether it is called Cannabis indica, Cannabis americanus or Cannabis sativa. Specimens from Europe and Asia were compared with those collected in America."

(7) "The New Britton and Brown Illustrated Flora of the Northeastern United States and Adjacent Canada" by Henry A. Gleason and published by The New York Botanical Garden, 1952 Vol. II, p. 54, Cannabis L., Hemp. A monotypic species of Asia ---- Cannabis sativa L.

(8) "The Standard Cyclopedia of Horticulture" by L. H. Bailey, published by the Macmillan Co., New York, 1942, Vol. I p. 657. Cannabis sativa "Only one species, but various forms have received specific names."

(9) "A Dictionary of Terms in Pharmacognosy" published by Charles C. Thomas, Springfield, Ill., 1955, p.38. "Cannabis: monotypic genus. Cannabis sativa: the accepted botanical name. Cannabis indica: botanical synonym for Cannabis sativa.

(10) Webster's New International Dictionary of the English Language, 2nd Edition, Unabridged. G. & C. Merriam Company, publishers, Springfield, Mass., under Cannabis: "(1) Bot. A genus of herbs, the type of the family Cannabinaceae, having as the only known species Cannabis sativa the hemp. (2). Pharm. The dried flowering

spikes of the pistillate plants of the hemp. The variety obtained in India is called also Cannabis indica."

(11) "Standardized Plant Names" by Harlan P. Kelsey and Wm. A. Dayton. 2nd Edition published by J. Horace McFarland Co., Harrisburg, Pa., 1942 for The American Joint Committee on Horticultural Nomenclature. p. 20 "Cannabis sativa (gigantia; indica.)"

(12) The Merck Index, Eighth Edition, published by Merck & Co., Inc. Rahway, N. J., U.S.A. p. 201 "Cannabis. Indian Hemp; Indian cannabis; marihuana; hashish; bhang; ganja; charas; kif; hasach. Dried flowering tops of pistillate plants of Cannabis sativa L. var. indica Auth)"

References by both chemists and botanists are cited; the latter being considered the most authoritative. The list could be extended ad infinitum. The author is indebted to the following for constructive comments and additional references: Dr. George R. Nakamura, now a chemist with the Department of Justice, Bureau of Narcotics and Dangerous Drugs, San Francisco, California; Mr. William Butler and Mr. Joseph E. Koles, both chemists on the laboratory staff of the Bureau of Narcotics and Dangerous Drugs, Washington, D. C.

# AN ANALYTICAL METHOD FOR MARIHUANA RESIDUES

David T. Chia
Chemist
U. S. Customs Laboratory
San Francisco, California

Residue in the pipe bowls and stems that are suspected to contain marihuana is usually a mixture of ash, tar, and semicharred vegetable matter. Often, unless such residue is first subjected to a thorough clean-up procedure, the ensuing results of Duquenois-Levine test and/or TLC are rendered inconclusive. This laboratory has found the following clean-up procedure to be rapid and effective.

## Micro-Disposable-Chromatographic Column:

Insert a small wad of fiber glass into a 15 cm long and 1.5 mm. innner diameter disposable pipette (approx. 2¢ per piece) and fill it 2/3 ways with acid alumina. (Certified alumina, acid, Brockman activity 1, 80/200 mesh. Fisher Scientific) Gently tap the column to pack the solid layer. A batch of 20 or more can be made in a matter of minutes and stored.

## PROCEDURE:

Wash the residue from the suspected pipe bowl and stem with small portions of ethyl acetate and send the entire wash solution through the chromatographic column. Collect the filtrate and evaporate a portion of it. To this residue, proceed with Duquenois-Levine Test. If it[1] is positive and no microscopically identifiable marihuana is found, apply the second portion of filtrate on silica gel G thin-layer chromatographic plate along with known reference hashish extract. Develop the plate with Hexane: Diethylether (4:1) and detect with Fast Blue Dye.[2]

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1/ Nakamura, G. JAOAC. Vol. 52, No. 1, 5-19, 1969
2/ Parker, K.D., Wright, J. A. Hine, Halpern. Bulletin on Narcotics Vol. XX, No. 4, 9-14, 1968

# X-RAY CRYSTALLOGRAPHIC DATA OF SOME HALLUCINOGENS

by Victor A. Folen, Forensic Chemist
Special Testing and Research Laboratory
Bureau of Narcotics and Dangerous Drugs

X-Ray powder diffraction data are presented for hallucinogenic substances, most of which have been subject to abuse. The data tabulated here cannot be found in The Organic Index To The Powder Diffraction File.*

The diffraction patterns were obtained on a Norelco Wide-Angle Diffractometer, equipped with a scintillation counter, using nickel-filtered copper radiation.

Interplanar Spacing was based on $\lambda(CuK\alpha,)=1.5405$ Å.

TABLE I. X-Ray Diffraction Patterns of Hallucinogenic Substances

(d=Interplanar Spacing; I/I,=Relative Intensity)

* Am. Soc. Testing Materials, "ORGANIC INDEX TO THE POWDER DIFFRACTION FILE," Smith, J. V., Ed., Philadelphia, Pa., 1967.

| Benactyzine Hydrochloride (JB 313, DMZ) | | Benactyzine Hydrochloride (Cont.) | | Benactyzine Hydrochloride (Cont.) | |
|---|---|---|---|---|---|
| dÅ | $I/I_1$ | dÅ | $I/I_1$ | dÅ | $I/I_1$ |
| 15.9 | 36 | 3.89 | 3 | 2.582 | 5 |
| 8.72 | 100 | 3.84 | 5 | 2.552 | 2 |
| 7.79 | 14 | 3.77 | 66 | 2.518 | 4 |
| 7.58 | 20 | 3.66 | 56 | 2.500 | 3 |
| 7.04 | 10 | 3.60 | 6 | 2.478 | 2 |
| 6.68 | 33 | 3.57 | 10 | 2.400 | 3 |
| 6.20 | 3 | 3.51 | 21 | 2.340 | 3 |
| 5.77 | 48 | 3.43 | 10 | 2.302 | 3 |
| 5.54 | 69 | 3.34 | 22 | 2.180 | 3 |
| 5.29 | 25 | 3.32 | 19 | 2.110 | 3 |
| 5.04 | 2 | 3.21 | 8 | 1.898 | 2 |
| 4.87 | 2 | 3.10 | 13 | 1.815 | 1 |
| 4.45 | 6 | 3.00 | 16 | 1.751 | 2 |
| 4.42 | 6 | 2.885 | 3 | | |
| 4.32 | 16 | 2.848 | 4 | | |
| 4.27 | 9 | 2.743 | 13 | | |
| 4.18 | 1 | 2.662 | 5 | | |
| 4.08 | 3 | 2.640 | 3 | | |
| 3.96 | 5 | 2.608 | 6 | | |

| Diethyltryptamine (DET) | |
|---|---|
| dÅ | $I/I_1$ |
| 8.75 | 69 |
| 7.80 | 47 |
| 7.18 | 19 |
| 6.19 | 27 |
| 5.54 | 69 |
| 5.27 | 81 |
| 4.48 | 31 |
| 4.67 | 78 |
| 4.37 | 47 |
| 4.25 | 43 |
| 4.00 | 58 |
| 3.90 | 15 |
| 3.81 | 7 |
| 3.56 | 100 |

| Diethyltryptamine (DET) | |
|---|---|
| dÅ | $I/I_1$ |
| 3.42 | 36 |
| 3.35 | 6 |
| 3.24 | 22 |
| 3.19 | 6 |
| 3.09 | 3 |
| 2.945 | 15 |
| 2.898 | 9 |
| 2.754 | 6 |
| 2.718 | 5 |
| 2.652 | 4 |
| 2.328 | 4 |
| 2.265 | 8 |
| 2.213 | 2 |

| Dimethyltryptamine (DMT) | |
|---|---|
| dÅ | $I/I_1$ |
| 9.58 | 33 |
| 9.06 | 16 |
| 8.05 | 19 |
| 7.44 | 36 |
| 7.02 | 26 |
| 6.57 | 53 |
| 6.33 | 93 |
| 6.14 | 53 |
| 5.78 | 20 |
| 5.58 | 63 |
| 5.22 | 40 |
| 5.12 | 28 |
| 4.78 | 40 |
| 4.72 | 54 |
| 4.56 | 74 |
| 4.33 | 100 |
| 4.18 | 80 |
| 4.05 | 35 |
| 3.95 | 16 |
| 3.84 | 36 |
| 3.76 | 16 |
| 3.69 | 23 |

| Dimethyltryptamine (Cont.) | |
|---|---|
| dÅ | $I/I_I$ |
| 3.55 | 29 |
| 3.41 | 16 |
| 3.32 | 14 |
| 3.26 | 13 |
| 3.15 | 13 |
| 2.990 | 8 |
| 2.890 | 6 |
| 2.735 | 10 |
| 2.625 | 5 |
| 2.584 | 4 |

| N-Ethyl-3-Piperidyl Benzilate Hydrochloride (JB 318) | |
|---|---|
| dÅ | $I/I_1$ |
| 11.5 | 39 |
| 9.40 | 15 |
| 9.08 | 19 |
| 8.50 | 20 |
| 7.82 | 100 |
| 6.68 | 26 |
| 6.40 | 20 |
| 5.74 | 14 |
| 5.33 | 40 |
| 5.26 | 66 |
| 5.13 | 57 |
| 4.99 | 57 |
| 4.26 | 45 |
| 4.08 | 12 |
| 3.91 | 66 |

| N-Ethyl-3-Piperidyl Benzilate Hydrochloride (Cont.) | |
|---|---|
| dÅ | $I/I_1$ |
| 3.69 | 68 |
| 3.59 | 43 |
| 3.52 | 51 |
| 3.39 | 22 |
| 3.08 | 9 |
| 3.01 | 9 |
| 2.873 | 22 |
| 2.835 | 9 |
| 2.782 | 8 |
| 2.732 | 14 |
| 2.468 | 7 |
| 2.360 | 8 |
| 1.995 | 3 |
| 1.940 | 5 |
| 1.845 | 3 |

Lysergic Acid Diethylamide Tartrate (LSD)

| dÅ | $I/I_1$ |
|---|---|
| 16.9 | 12 |
| 10.8 | 49 |
| 9.44 | 44 |
| 8.43 | 32 |
| 5.80 | 100 |
| 5.71 | 61 |
| 5.50 | 10 |
| 5.17 | 30 |
| 5.04 | 13 |
| 4.78 | 30 |
| 4.56 | 14 |
| 4.39 | 24 |
| 4.12 | 14 |
| 4.03 | 31 |
| 3.87 | 10 |
| 3.79 | 11 |
| 3.69 | 13 |
| 3.60 | 18 |
| 3.38 | 16 |
| 3.29 | 5 |
| 3.21 | 5 |
| 3.14 | 7 |
| 2.900 | 6 |
| 2.810 | 4 |
| 2.740 | 4 |

Isolysergic Acid Diethylamide (Iso-LSD)

| dÅ | $I/I_1$ |
|---|---|
| 11.0 | 21 |
| 6.90 | 100 |
| 5.44 | 46 |
| 5.25 | 22 |
| 5.02 | 15 |
| 4.67 | 3 |
| 4.42 | 41 |
| 4.22 | 12 |
| 4.08 | 12 |
| 3.97 | 10 |
| 3.79 | 20 |
| 3.69 | 18 |
| 3.62 | 10 |
| 3.32 | 5 |
| 3.28 | 10 |
| 3.22 | 8 |
| 3.11 | 4 |
| 3.04 | 2 |
| 2.925 | 2 |
| 2.780 | 1 |
| 2.600 | 1 |
| 2.455 | 2 |
| 2.305 | 2 |
| 2.095 | 2 |
| 1.965 | 2 |

| 3,4-Methylenedioxyamphetamine Hydrochloride (MDA) | | 3,4-Methylenedioxyamphetamine Hydrochloride (Cont.) | | 3,4-Methylenedioxyamphetamine Hydrochloride (Cont.) | |
|---|---|---|---|---|---|
| dÅ | $I/I_1$ | dÅ | $I/I_1$ | dÅ | $I/I_1$ |
| 12.8 | 7 | 3.57 | 24 | 2.385 | 3 |
| 12.4 | 45 | 3.33 | 7 | 2.363 | 6 |
| 6.55 | 7 | 3.27 | 94 | 2.333 | 6 |
| 5.34 | 53 | 3.17 | 14 | 2.221 | 34 |
| 5.22 | 10 | 3.13 | 8 | 2.193 | 4 |
| 5.03 | 6 | 3.08 | 34 | 2.160 | 12 |
| 4.85 | 6 | 3.03 | 19 | 2.083 | 3 |
| 4.74 | 1 | 3.01 | 17 | 2.062 | 4 |
| 4.60 | 26 | 2.927 | 84 | 2.014 | 3 |
| 4.53 | 18 | 2.890 | 4 | 1.954 | 4 |
| 4.45 | 4 | 2.858 | 5 | 1.928 | 6 |
| 4.19 | 21 | 2.825 | 15 | 1.888 | 7 |
| 4.15 | 20 | 2.664 | 6 | 1.867 | 3 |
| 4.09 | 38 | 2.623 | 5 | 1.820 | 3 |
| 4.02 | 100 | 2.590 | 11 | 1.794 | 4 |
| 3.86 | 88 | 2.550 | 10 | 1.779 | 6 |
| 3.79 | 81 | 2.470 | 4 | 1.754 | 5 |
| 3.74 | 25 | 2.420 | 5 | 1.729 | 4 |
| | | | | 1.707 | 6 |

4-Methyl-2,5-dimethoxy-amphetamine Hydrochloride (STP)

| dÅ | $I/I_1$ |
|---|---|
| 10.2 | 100 |
| 6.77 | 32 |
| 6.28 | 7 |
| 5.85 | 4 |
| 5.68 | 15 |
| 5.41 | 15 |
| 5.07 | 35 |
| 4.77 | 4 |
| 4.44 | 6 |
| 4.39 | 4 |
| 4.28 | 7 |
| 3.97 | 11 |
| 3.83 | 11 |
| 3.69 | 5 |
| 3.57 | 31 |
| 3.48 | 8 |
| 3.36 | 80 |

4-Methyl-2,5-dimethoxy-amphetamine Hydrochloride (Cont.)

| dÅ | $I/I_1$ |
|---|---|
| 3.33 | 28 |
| 3.10 | 7 |
| 2.915 | 13 |
| 2.879 | 5 |
| 2.739 | 6 |
| 2.587 | 4 |
| 2.560 | 5 |
| 2.442 | 5 |
| 2.400 | 4 |
| 2.313 | 5 |
| 2.235 | 7 |
| 2.189 | 3 |
| 2.150 | 6 |
| 2.010 | 2 |
| 1.947 | 2 |
| 1.820 | 4 |
| 1.745 | 3 |

1-Methyl-3-Piperidyl α-(2-Thienyl) Mandelate Hydrochloride and 1-Ethyl-3-Piperidyl α-(2-Thienyl) Mandelate Hydrochloride (JB 344)

| dÅ | $I/I_1$ |
|---|---|
| 10.5 | 27 |
| 8.23 | 25 |
| 7.38 | 45 |
| 6.95 | 23 |
| 6.74 | 39 |
| 6.12 | 43 |
| 5.87 | 100 |
| 5.72 | 72 |
| 5.39 | 77 |
| 5.14 | 42 |
| 4.64 | 52 |
| 4.48 | 45 |
| 4.34 | 22 |
| 4.28 | 12 |
| 4.24 | 15 |
| 4.17 | 31 |
| 4.11 | 58 |
| 3.98 | 22 |
| 3.65 | 13 |

1-Methyl-3-Piperidyl α-(2-Thienyl) Mandelate Hydrochloride and 1-Ethyl-3-Piperidyl α-(2-Thienyl) Mandelate Hydrochloride (Cont.)

| dÅ | $I/I_1$ |
|---|---|
| 3.61 | 15 |
| 3.54 | 13 |
| 3.37 | 67 |
| 3.31 | 47 |
| 3.28 | 31 |
| 3.13 | 13 |
| 3.08 | 29 |
| 2.985 | 12 |
| 2.865 | 9 |
| 2.708 | 52 |
| 2.602 | 11 |
| 2.588 | 9 |
| 2.568 | 14 |
| 2.420 | 7 |
| 2.402 | 9 |
| 2.358 | 5 |
| 2.315 | 7 |
| 2.248 | 16 |

# NOTES ON NARCOTIC AND DANGEROUS DRUG ANALYSIS

Compiled by Roger G. Fuelster
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Because of its unique status as the first "on-line" BNDD laboratory, Chicago Regional Laboratory has accumulated some helpful hints which are useful, but not worthy of being written up as a full method. These hints are being passed on for what they are worth; and are by no means original--in fact, many are "borrowed" outright.

## Heroin:

1. Direct UV quantitative method (IRS Manual, 1967 Revision, p. 58, Method "d")--New factors for the simultaneous equations have been calculated:

   Heroin hydrochloride monohydrate, mg/100ml.
   $24.42A_{278} - 5.74\ A_{313} - 0.05\ A_{348}$

   Methapyrilene hydrochloride, mg/100ml.
   $3.87\ A_{313} - 3.09\ A_{348}$

   Quinine hydrochloride dihydrate, mg/100ml.
   $7.72\ A_{348} - 0.340\ A_{313}$

2. Very pure Heroin hydrochloride may be extracted from approximately 3N HCl with $CHCl_3$. A very nice IR results. Extraction of the standard in the same manner is necessary for perfect agreement. Adulterants do not interfere.

## Brown Heroin:

1. Quantitate by GLC on 1% SE-30, approximately 200°, and 50 ml/min. Use ammoniacal $CHCl_3$ as the solvent at approximately 2 mg/ml. Add the solvent to the sample just before injection to prevent hydrolysis.

2. The extraction and IR given above for Heroin work very well, even though the residue is brown.

## Cocaine:

1. The hydrolysis method (IRS Manual, 1967 Revision, p. 81, Method "c") has been modified for better results:

Dilute sample to 100 ml 0.03N NaOH in MeOH. Take 10 ml (or other appropriate aliquot depending on concentration) through hydrolysis using 10 ml 0.3N NaOH MeOH. Dilute to volume with MeOH and read.

The new equations are:

Cocaine hydrochloride, mg/100ml final dilution.
$2.87\ A_{226} - 0.99\ A_{292}$

Procaine hydrochloride, mg/100ml final dilution.
$1.36\ A_{292}$

Tetracaine is seldom encountered. Note the shift in the Procaine maximum.

IR Standards:

When a standard disk is made, extra care is taken to produce a good disk with a "perfect" curve. A copy of the curve is filed, and the disk retained for future use in a small vial with silica gel in the bottom. Much time, effort, and expensive reference material are thus saved. Disks 5 months old have not deteriorated. Watch sulfate salts though--they are known to exchange with KBr and become bromide salts. As a rule, it is better to use the chloride in the first place.

General Alkaloid Extraction:

Place sample and 25 ml 1N $H_2SO_4$ in separatory funnel. Wash with $CHCl_3$ until clean and discard washings. Make basic with 5N NaOH and extract alkaloid with $CHCl_3$, filtering through a cotton plug in stem. Add MeOH and several drops Conc. HCl and evaporate to dryness, then dry at 105°C. If a crystalline solid is not obtained, wash the residue with pet. ether and re-dry. If a crystalline residue is not obtained after this treatment--look for another extraction method.

# Identification of N,N-Dipropyltryptamine (DPT)

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Dipropyltryptamine hydrochloride was synthezied in our laboratory by the method of Brimblecombe.[1] While the hydrochloride salt is crystalline the free base appears to be an oil.

Appearance

Hydrochloride - slightly off white powder

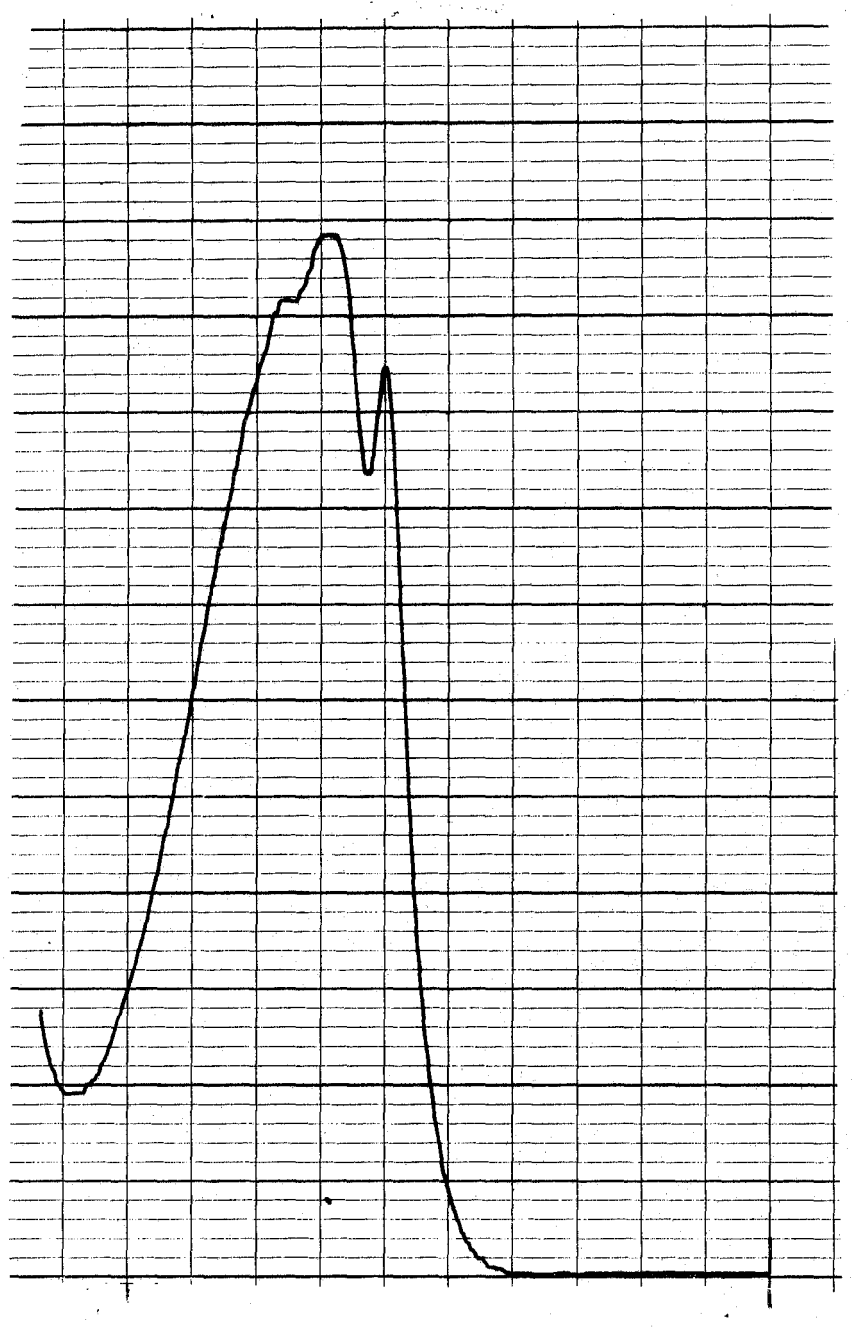
Melting point 178°

Ultraviolet absorption

1 mg in 40 ml of ethanol exhibits maxima at 274, 282 and 290 mu. and a minimum at 242 mu. The spectrum is similar to that of DMT and DET.

The ultraviolet and the infrared spectra of the free base and hydrochloride are presented.

1/ Brimblecombe, R., Downing, D., Green, D., and Hunt, R., British Journal of Pharmacology 23, 43, 1964.




N,N, DIPROPYLTRYPTAMINE IN ETHANOL

A. Sperling
April 28, 1969