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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Philip V. Porto has been appointed Chief Chemist of BNDD's New York Regional Laboratory. He was previously Supervisory Chemist in the same laboratory.

Mr. Porto was born in New York City, where he attended public schools and graduated from Stuyvesant High School. He attended Harvard University under the Navy V-12 program, then earned a B.S. in Chemistry after a year at Long Island University, Brooklyn, New York. He later did post graduate work at the Polytechnic Institute of Brooklyn, Brooklyn, New York.

After several years experience as a medical research assistant at Mt. Sinai and Delafield hospitals in New York, Mr. Porto became an organic chemist with the U.S. government's General Services Administration. He later transferred to the Internal Revenue Service, Alcohol & Tobacco Tax Division, where he spent the next sixteen years. He was a Hearing Officer for the Internal Revenue Service, and was Chief Chemist in the A.T.F.'s New York laboratory when he transferred to BNDD in 1969. In the latter years with A.T.F., Mr. Porto was on the adjunct faculty of Nassau Community College, Nassau, Long Island, where he taught chemistry.

Mr. Porto resides on Long Island with his wife, Grace. They have two children. One is a junior in college, the other is a nurse.

Saccharin tablets purported to be LSD were analyzed by BNDD's New York laboratory. The small tablets had a white base covered with a sparse orange coating. Saccharin was found, but no LSD was detected. The tablets may have been intended to pass as LSD "Sunshine" tablets.

Caffeine tablets apparently intended to imitate "Obedrin-L.A." tablets manufactured by S.E. Massengill Company, Bristol, Tennessee, frequently appear in BNDD laboratories. These have been seen with varying frequency since 1968. They are not counterfeit, and caffeine is not a controlled substance.

LSD "Orange Mushroom" tablets are being encountered. The tablets are 8.07-8.08 mm diameter, 6.11-6.17 mm maximum thickness and 2.2 mm thick at the edge. One face is strongly convex - "high domed" - with a flat area approximately 3.0 mm in diameter at the top of the dome. The other face is flat with a centered, stem-like, projecting, truncated cone. The cone is approximately 4.5 mm diameter at the base, 3.5 mm diameter at the apex, and is 2.0 mm long. The bottom of the stem has a round, shallow depression at the center. The flat portion of the face surrounding the "stem" is about 1.8 mm wide and has 14 prominent ridges running radially from the base of the "stem" to the edge of the tablet.

The tablets, made with a single pair of punches, contained a large amount of amorphous proteinaceous material, moderate amounts of sodium bicarbonate and dextrose hydrate, and small amounts of sucrose and corn starch. The tablets contain about 40 micrograms of LSD.

"Antilog," a brown mixture containing Hawaiian Baby Wood Rose, peyote and mescaline reportedly appeared in California. None has been encountered by BNDD laboratories.

Heroin reportedly is mailed into the United States on the back of Polaroid photographs. Servicemen are said to place heroin on the back of the photo, then cement an extra back over it. As a result, the heroin is sandwiched between the two backs of the photograph.

Heroin was encountered by the BNDD Washington laboratory attached to the instruction tag for tropical boots. The tag with its heroin was mailed to the United States by a serviceman in the Far East.

Mixture of cocaine free base and cocaine HCl has been reported by the BNDD San Francisco laboratory. Two exhibits of the mixture were analyzed. Both were white powders and both contained sodium bicarbonate as a diluent. One exhibit was in foil and the other was in a white plastic container. A mixture of cocaine free base and cocaine HCl have been previously reported in exhibits from Illinois and from a foreign country (Microgram, Vol. IV, No. 6, July, 1971).

"Transmission GO" is reportedly being sniffed by teenagers in South Carolina. The product is a fluid intended to stop leaks in automatic transmissions of automobiles and is manufactured by GO Products, P.O. Box 16182, Jacksonville, Florida. It is a red liquid marketed in an eight ounce bottle. The fluid is said to contain toluene.

Propoxyphene is the most commonly abused drug in the Wilwaukee, Wisconsin area, according to a letter to the editor in JAMA, 216, No. 12, June 21, 1971.

Heroin in glassine envelopes bearing the outline of a black five-pointed star are being seen. The envelopes are sealed with either red or clear cellophane tape. We would like to have the facts on any encounters with these packages.

alpha-L-Acetylmethadol suppresses heroin withdrawal symptoms two or three times as long as methadone, according to a recent report. [JAMA, 216, 1303 (1971)]

Potentilla gracilis, also known as cinquefoil, silver weed, wild tansy and silver feather, was recently being sold in Montana as a legal marihuana substitute. The plant, abundant in Utah, Idaho and Western Montana, grows in most states. It has no known hallucinogenic properties.

MEETINGS

Association of Official Analytical Chemists will meet October 11-14, 1971, in the Mariott Motor Hotel, Twin Bridges, Washington, D.C. Registration: $3.00

On Thursday, October 14, a symposium on the forensic sciences will be held. The keynote address, "The Prosecutor and the Forensic Scientist," will be delivered by the Honorable Thomas Flannery, U.S. Attorney, Washington, D.C.

This address will be followed by:

> "Recent Advances in Forensic Activation Analysis," Vincent P. Guinn, Ph.D., University of California, Irving, California
>
> "The Use of NMR and Mass Spectral Data in the Forensic Laboratory," Stanley P. Sobol, Chief Chemist, Special Testing & Research Laboratory, Bureau of Narcotics & Dangerous Drugs, Washington, D.C.
>
> "The Law Enforcement Standards Laboratory," Robert Mills, Law Enforcement Standards Laboratory, National Bureau of Standards, U.S. Department of Commerce, Washington, D.C.

"Thin-Layer Chromatography of Marihuana," James M. Parker, Assistant Director, Pittsburgh & Allegheny County Crime Laboratory, Pittsburgh, Pennsylvania.

"A Fully Automated Fluorimetric Method For the Determination of Morphine in Human Biological Material," Stanley Morgenstern, Musa Sansur and Anthony Buccafuri, Technicon Corporation, Tarrytown, New York.

Midwestern Forensic Analysts' Seminar, October 7-9, 1971, Chicago. Drug identification problems, firearms residue determinations, firearms range determinations, new blood identification techniques, glass, paint, and fiber identification techniques and "state of the art" in the above topics will be discussed. Contact: Robert A. Boese, Criminalistics Division, Chicago Police Department, 1121 South State Street, Chicago, Illinois 60605.

California Association of Criminalists, Semi-Annual Seminar, October 21-23, 1971, Hyatt House, Burlingame, California. Contact: Paul M. Dougherty, Chief Criminalist, San Mateo County Sheriff's Office, Hall of Justice and Records, Redwood City, California 94063. The association has about 140 members from various parts of the United States and represents most of the forensic sciences.

Annual Meeting of the American Academy of Clinical Toxicology, October 21-23, 1971, Marriott Motel, City Avenue, Philadelphia, Pennsylvania. Contact: Program Chairman, American Academy of Clinical Toxicology, P.O. Box 2565, Houston, Texas 77001.

Annual Meeting of the American Academy of Forensic Sciences, Atlanta, Georgia, February 29-March 4, 1972. Contact: Secretary James Weston, 44 Medical Drive, Salt Lake City, Utah 84113.

The 6th International Meeting of Forensic Sciences, Queen's University, Belfast, Northern Ireland, September 21-26, 1972. Contact: The Secretary, The 6th International Meeting of Forensic Sciences, Institute of Pathology, Grosvenor Road, Belfast, Northern Ireland.

ENCLOSURE

"Schedule II Amphetamine And Methamphetamine Drugs Commonly Prescribed," a leaflet prepared by BNDD, is being mailed with this issue.

BNDD LABORATORY NOTES

DATE March 4, 1971
NO. 17
DRUG TYPE D^9-Tetrahydrocannabinol
METHODOLOGY Gas Liquid Chromatography

IDENTIFICATION OF D^9-TETRAHYDROCANNABINOL BY P-VALUES

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INTRODUCTION

The p-value identification is a method based on the distribution of the drug residue between two immiscible phases, has been found applicable at micrograms and other levels as well. Each drug has a characteristic distribution ratio, which in residue analysis has been found practically independent of drug concentrations or other excipient materials such as tablet binders, plant residues, starches, dyes, or other adulterants. It is a convenient method, easy to carry out, and rapid. (1)

An aliquot of an upper phase containing a given amount of D^9-THC is analyzed by gas liquid chromatography. To this same aliquot, a second aliquot of equal volume but different lower phase is added. The two phases are shaken then allowed to separate. The upper phase is again determined by gas liquid chromatography. A standard D^9-THC solution is treated similarly and the p-values of the standard and sample compared for identity.

The ratio of the second analysis to the first is the p-value, i.e., the amount of THC in the upper phase (second analysis) divided by the total amount of THC (first analysis). One analysis must be run to determine the amount of THC present. Only one other (on the aliquot extracted with lower phase) is needed to determine the p-value. Solvent equilibration is necessary.

Each drug has an individual response factor to a flame ionization detector. The response factor and the distribution ratio of the drug between two immiscible solvents characterize the observed p-values, hence detection of another compound with a similar retention time to the drug of interest is possible because the observed p-values are different than those ordinarily obtained.

(1) Beroza, M. and Bowman, M.C., Journal Analytical Chemistry 37; 291 (1965)

APPARATUS: Gas Liquid Chromatograph

Instrumental Parameters of Gas Liquid Chromatography
Detector —— Flame Ionization
Detector Temperature —— 300°C
Injection Port Temperature ——300°C
Column Temperature ——225°C
Column —— Glass, 6ft.,4mmID containing 2% OV-17 on 60/80M
gas chrom Q conditioned 280°C for 16 hours
Carrier Gas ——Nitrogen at a flow rate of 60 ml/minute
Recorder Chart Speed ——1/2" per minute

CALIBRATION: Gas Liquid Chromatograph

Adjust air, hydrogen,nitrogen flow rates,and sensitivity so that 5 ug. of injected standard D^9-THC yield about 1/2 chart scale deflection.

STANDARDS: Preparation

Std. D^9_8-THC = 1.0 mg/ml
Std. D^8-THC = 0.05 mg/ml
Std. CBD* = 0.2 mg/ml
Std. CBN** = 0.12 mg/ml
Internal standard methadone HCl 0.25 mg/ml in $CHCl_3$

*Trans-cannabinidiol
**Cannabinol

METHOD

Select a representative portion of the marihuana sample and finely grind it to pass a 40 mesh sieve. Mix the ground portion and weigh 0.5 gram to 1.0 gram of sample into a 60 ml. conical flask. Add 20 ml. of petroleum ether and heat for several minutes using a warm water bath. Decant the solution through filter paper, passing the filtrate through a 1/2 x 1" column of anhydrous sodium sulfate into a 150 ml. beaker. Repeat the extractions twice combining each extraction.

Evaporate extracts to dryness and dissolve the residue in the upper phase selected for the p-value determination. Transfer solution to a 10 ml. volumetric flask and dilute to volume. Take a 4 ml. aliquot,evaporate to dryness and dissolve residue in 4 ml. of the internal standard solution. Determine D^9-THC by gas liquid chromatography and express response in terms of area under the D^9-THC curve per ul of sample injected. Take a second 4 ml. aliquot of upper phase and transfer to a 15 ml glass stoppered conical test tube. Add 4 ml. of a lower phase, stopper and shake for one minute. Remove a 3 ml. aliquot of upper phase, evaporate to dryness and dissolve residue with 3 ml. of internal standard solution. Again determine D^9-THC

by gas liquid chromatography expressing response in terms of area per ul injected. Calculate the p-value and compare this value against a known standard D^9-THC p-Value treated similarly or previously determined from standard marihuana plant extracts.

CALCULATION

$$\text{p-Value} = \frac{D^9\text{-THC content in upper phase (second analysis)}}{D^9\text{-THC content in upper phase (first analysis)}}$$

RESULTS AND DISCUSSION

The three systems, see tables 1,2,and 3, show D^9-THC to have a characteristic distribution between two immiscible solvents. This distribution is practically independent of D^9-THC concentrations, and of the amount of plant residue present in a sample extraction.

The amount of water extracted from the plant material with petroleum ether, room temperature, solvent purity, and non linearity of D^9-THC response to the flame ionization detector, effect the observed p-values. Water interference is reduced by passing the petroleum ether extract through ananhydrous sodium sulfate column.

Reagent grade solvents are usually pure enough to use. However, if large deviations occur in p-values, then solvents should be suspected.

Room temperature effect equilibration of solvents, in turn effecting p-values. To reduce equilibria variations of the solvents,because of temperature, the system should be shaken together while solvents are at temperatures between 22 to 25°C. The equilibrated phases should be separated and stored in separate closed containers.

Aliquots are measures out with pipettes and should be greater than one ml. One ml. aliquots are not always reliable. Greater volume aliquots may be used for extractions, then concentrated to smaller volumes for determinations of D^9-THC content if necessary.

To prevent errors in p-value calculation because D^9-THC response may be non-linear at high concentrations to the flame ionization detector, instrumental conditions are set up so that 0.8 µg. to 8.0 µg. D^9-THC response vs. concentrations curve is linear. Samples of D^9- are diluted to yield concentrations in order that 4 to 8 ul. of injected solutions into a gas liquid chromatography contains 0.8 to 8.0 µg. of D^9-THC.

The use of an internal standard aids in improving precision of results. However, if a large error is encountered in observed p-values, then the response of the internal standard should be checked. A larger than normal peak indicates a compound of similar retention time in the sample to that of the internal standard causing an error in calculating D^9-THC response.

Deviation between standard and sample p-values or from the average of tabulated results should not be more than $\pm$ 0.05. If the deviation is greater than $\pm$ 0.05, then the analyst should determine the cause of the deviation.

The unusual p-values may be due to another compound of similar retention time, or to a compound having a different flame ionization response factor. In any case, the p-value would indicate if the peak used for D^9-THC identity was actually due to the D^9-THC in the marihuana sample. Observed p-values of sample D^9-THC similar to known values or similar to p-values obtained from a standard help to confirm identity of D^9-THC by gas liquid chromatography.

Table One

p-values of D^9-THC

Equilibrated Petroleum Ether, upper phase and Acetonitrile (lower phase)

Standards

conc. of D^9-THC	p-values (1)	(2)
0.6 mg/ml	0.22	0.21
0.8 mg/ml	0.19	0.20
1.0 mg/ml	0.20	0.17

Average (6) 0.20

Marihuana Samples (D^9-THC Concentrations Different)

No.	p-value	No.	p-value
1	0.23	7	0.19
2	0.22	8	0.22
3	0.21	9	0.23
4	0.18	10	0.17
5	0.18	11	0.21
6	0.17	12	0.20

Average (12) 0.20

Table Two

p-values of D^9-THC

Equilibrated 100 ml of 2, 2, 4-Trimethylpentane, (upper phase) and 100 ml of 80% aqueous Formamide, (lower phase)

Standards

conc. of D^9-THC	p-values (1)	p-values (2)
0.6 mg/ml	0.90	0.89
1.0 mg/ml	0.92	0.88
1.5 mg/ml	0.88	0.89

Average (6) 0.89

Marihuana Samples (D^9-THC Concentrations Different)

No.	p-values	No.	p-values
1	0.90	7	0.87
2	0.91	8	0.90
3	0.89	9	0.89
4	0.88	10	0.86
5	0.90	11	0.88
6	0.92	12	0.90

Average (12) 0.89

Table Three

p-values of D^9-THC

Equilibrated 100 ml of Hexane (upper phase), and 100 ml of 80% aqueous Dimethylsulfoxide (lower phase)

Standards

conc. of D^9-THC	p-values (1)	p-values (2)
0.2 mg/ml	0.58	0.58
0.8 mg/ml	0.62	0.64
1.5 mg/ml	0.57	0.62

Average (6) 0.60

Marihuana Samples (D^9-THC Concentrations Different)

No.	p-values	No.	p-values
1	0.58	7	0.60
2	0.58	8	0.61
3	0.56	9	0.56
4	0.60	10	0.57
5	0.64	11	0.62
6	0.62	12	0.63

Average (12) 0.60