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

Vol. III, No. 4 June, 1970

STANLEY P. SOBOL, has been selected as Chief Chemist of the BNDD Special Testing and Research Laboratory in Washington, D.C.

As Chief Chemist of the headquarters laboratory, Sobol will supervise a research group which identifies and analyzes new drugs of abuse and develops methods for their analysis. Sobol also will head a group which identifies unusual substances and is responsible for the National Authentic Drug Library used for the detection of counterfeit drugs. This collection is also used to identify the source of tablets seized from illicit channels.

Sobol has been working as a forensic chemist in the Bureau's Laboratory Operations Division, where he assisted in the development and operation of the Bureau's new laboratory system. He has been compiling the Bureau's drug analytical manual, and, among other duties, has been acting as the research coordinator for the laboratory system. Prior to joining the Bureau of Narcotics and Dangerous Drugs, Sobol served as a chemist with the Food and Drug Administration.

"False Marihuana" was identified as Henna (Lawsonia inermis) in a sample recently submitted to the BNDD Special Testing and Research Laboratory by the National Police of Germany. Reportedly this substance is being used as "Fake Pot" in England, Holland and Germany.

Henna is relatively nontoxic, but reportedly may cause irritation of the skin in sensitive individuals. Since ancient times, it has been used for dyeing hair and nails. It is usually obtained from North Africa or India. It contains about 1% lawsone, (2-hydroxy-1, 4-naphthoquinone), and is one of the natural occurring napthoquinones. (see Merck Index, 8th Ed., and U.S. Dispensatory, 26th Ed.)

"Zoom" or "Super Grass" - In California, PCP on parsley is called "Super Grass". In the Washington, D.C. metropolitan area, PCP on parsley reportedly is called "Zoom" The preparation purportedly is being smoked.

Peace Symbol on LSD impregnated papers have been encountered by the Washington Regional Laboratory for the first time. LSD is apparently dropped on the tan, coarse paper, and the "peace" symbol is drawn over each spot. The papers contain four spots, each spot containing 174 mcg. LSD.

LSD on Gelatin Flakes were found in possession of a U.S. citizen arrested in London. Allegedly, the 10,000 gelatin flakes had been smuggled from the United States. Each flake contained 120 micrograms LSD, and was enclosed in foil or paper packets and plastic boxes.

Analysis by the BNDD Special Testing and Research Laboratory confirmed the flakes to be gelatin having opaque dotted areas. Flake size was approximately 3 to 6 millimeters, in approximate squares, 0.38 millimeter thick. The average weight of 100 flakes was 106.5 milligrams. This is the first time that LSD in this form has been seen by the BNDD Special Testing and Research Laboratory.

London's LSD problem is thought to originate from LSD manufactured in the United States.

Since March of this year, an increasing number of reports have been received that several Americans have been arrested in London in possession of large quantities of LSD tablets. From the surrounding evidence, it was apparent that they were major traffickers.

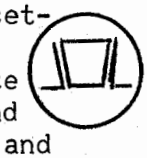
Arrangements have been made for Scotland Yard to submit tablet evidence to the BNDD Special Testing and Research Laboratory for "ballistics" examination, to identify the sources of the drug.

LSD triangular shaped tablets were encountered for the first time by the Special Testing and Research Laboratory. In May, tablets were submitted by BNDD agents from the Jacksonville, Florida area, and identical tablets were also received from the Dallas, Texas, Police Department. The Dallas exhibit was from a lot of 4,000 tablets. Both exhibits consisted of tablets made on the same single set of punches. From examination of the tablets it appears that the punches used to compress the tablets were of poor quality.

Heroin with maltose as a diluent was recently found in exhibits submitted to the BNDD San Francisco Regional Laboratory. In some instances lactose and/or procaine was also present. The exhibits were from the Seattle area.

"Vivarin" tablets, which are yellow, single scored tablets, have been submitted on several occasions, as alleged amphetamine, to the BNDD Special Testing and Research Laboratory. "Vivarin" is an over-the-counter stimulant reportedly distributed by the J.B. Williams Company Inc., and contains 200 mg of caffeine alkaloid and 150 mg of dextrose. Microscopic examination has shown that the product also contains corn starch and a cellulose derivative. The tablets are round, biconvex, single scored, and have no imprint. They are 11.2 mm diameter, 5.7 mm thick, 3.7 mm thick at edge. Average weight --580 mg.

"Comeback" tablets. Alleged amphetamine tablets recently submitted to the BNDD Special Testing and Research Laboratory have been called by the name "Comeback". Those submitted contained caffeine only, instead of acetaminophen, salicylamide, and caffeine alkaloid found in legitimate "Comeback," which is distributed by Thayer Laboratories. Legitimate "Comeback" tablets are round, biconvex, unscored, orange-colored and imprinted as shown. They are 11.2 mm in diameter, 4.9 mm thick, and have an edge thickness of 2.6 mm. Average weight is 470 mg.



1-Phenylcyclohexylethylamine (N-Ethyl-1-Phenylcyclohexylamine) was recently seized in a clandestine laboratory. Laboratory notes and copy of Patent #3,097,136 dated July 9, 1963, also seized, contained the synthesis method. The patent, issued to Parke Davis and Co., is for a class of compounds which include phenlyclidine HCl (PCP), and alleges the compounds to produce a depressant-like effect on the central nervous system of mammals.
(Refer also to Chem. Abstracts Volume 59, 1963, Col 138 b)

Clandestine methamphetamine laboratory exploded, causing suspects to flee their motel room. Investigation disclosed that methamphetamine was being manufactured in the bathroom of the motel unit. There was no fire from the explosion, and it was theorized by the agents and a BNDD chemist at the scene, that the reaction flask exploded from pressure alone. Rubber tubing from the flask was attached to an aspirator placed on the shower outlet and from there to a shower drain. The tubing apparently became blocked causing the explosion.

2,5 -Dimethoxyamphetamine has been encountered in clear, No. 0, gelatin capsules by the BNDD Dallas Regional Laboratory. The compound has an ultraviolet spectrum very similar to 4-methyl -2,5-dimethoxyamphetamine (STP, DOM). Microchemical tests on the powder with Marquis and Mecke's Reagent also indicated the possibility of STP. Further testing in the Special Testing and Research Laboratory revealed that the 2,5 -dimethoxyamphetamine was present as the hydrobromide salt.

Nitrous Oxide, N_2O,(Laughing Gas) - Thefts of nitrous oxide cylinders have been reported periodically over recent years. Allegedly, the gas produces a "kick." Remington's Practice of Pharmacy, 13th ed., states, in part, "Nitrous oxide is the weakest but probably the safest inhalation general anesthetic. .....Inasmuch as high concentrations of Nitrous Oxide are required, little room is left in the mixture for oxygen. This may result in serious anoxia and tissue damage, especially to the central nervous system."

GLOSSARY

B.

BHANG	Marihuana
BIG BLOKE	Cocaine
BIG JOHN	Police
BIG MAN	Supplier
BINDLE	Small package of narcotics (usually a one ounce package)
BINGLER	Seller of narcotics
BINGO	To inject drugs
BIZ	Equipment for injecting drugs
BLANKS	Poor quality merchandise
BLASTED	Under the influence of drugs
BLOW	Miss the vein in injecting
BLOW	Leave a place
BLOW A POT	Use marihuana
BLOW A STICK	Smoke marihuana
BLOCK CHARLIE OR SNOW	Sniff cocaine
BLOW HORSE	Sniff heroin
BLUE BIRDS	Amytal (amobarbital sodium)
BLUE DEVILS	Amytal (amobarbital sodium)
BLUE VELVET	Pyribenzamine (an antihistamine) which, when injected produces a marked state of euphoria
BLUES	Amytal (amobarbital sodium)
BOBO BUSH	Marihuana
BOOST	Rob
BOMBIDO	Injectable amphetamines

BOXED	In jail
BREAD	Money
BROKER	Dope peddler to addicts
BULL	Police
BURNED	Received phony narcotics
BURNED OUT	Sclerotic blood vessels from too many injections
BUSTED	Arrested
BUY	Make a purchase of drugs
BUZZ	Moderate euphoric reactions to drugs
BUZZ-WAGON	Automobile

(To be continued)

The Northern Illinois Police Crime Laboratory, Highland Park, has furnished us with a list of narcotics and dangerous drug exhibits analyzed during 1969:

DRUG		AMOUNT
Amobarbital	Tablets/Capsules	7
Amphetamine	Tablets/Capsules	111½
Chlorpheniramine Maleate	Tablets/Capsules	3
DMT	Tablets/Capsules	1
Ergonovine	Tablets/Capsules	8
Heroin	Grams	.05
Indomethacin	Tablets/Capsules	9
LSD	Tablets/Capsules	90½
Librium	Tablets/Capsules	1
Marihuana	Grams	2,527.87
	Grams (Seeds)	8.18
	Cigarettes	15
	Plants	5
MDT	Tablets/Capsules	3

Mesantoin	Grams	.10
Methamphetamine	Tablets/Capsules	1,046
Paregoric	Ounce	1
PCP	Tablets/Capsules	5
Pentobarbital	Tablets/Capsules	11
Phenobarbital	Tablets/Capsules	49½
Preludin	Tablets/Capsules	2
Propoxyphene Hydrochloride	Tablets/Capsules	19
Secobarbital Sodium	Tablets/Capsules	2
STP	Tablets/Capsules	21
Tetracycline	Tablets/Capsules	2
Thyrar	Tablets/Capsules	9

SELECTED REFERENCES:

Pharmacy Times, "The Leading 200 Drugs in 1969", 36,29-33, (April, 1970)

Farnsworth, N.R., Journal of the American Pharmaceutical Association, "Pharmacognosy and Chemistry of Cannabis Sativa", NS9, 8, (August, 1969)

Phillips, G.G. and Gardiner, J. J. Phar. Pharmac. "The Chromatographic Identification of Psychotropic Drugs", 21, 793-807, (1969)

Fujimoto, J.M. and Wang, R.I.H. Toxicology and Applied Pharmacology, "A Method of Identifying Narcotic Analgesics in Human Urine after Therapeutic Doses", 16. 186-193, (1970)

Harden, M.R. and Rasmussen, R.R., The Journal of Medicinal Chemistry, "Synthesis of Compounds with Potential Central Nervous System Stimulant Activity. II. 5-Spiro-Substituted 2-Amino-2-oxazolines", 13, (1970)

MEETINGS:

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

Canadian Society of Forensic Science, 18th Annual Meeting, Banff, Alberta, Canada, September 23-25, 1970. Advance registration forms available from Mr. Anthony, Chief Crown Prosecutor, Municipal Courts Building, Edmonton, Alberta, Canada.

American Academy of Clinical Toxicology, annual meeting in San Francisco, October 24-26, 1970. Meeting will include two one-day symposiums. One, on the "Clinical Toxicology of Substances of Abuse" (other than narcotics), the other on "Legal Aspects of Clinical Toxicology." Address: P.O. Box 2565, Houston, Texas 77001

1971 American Academy of Forensic Sciences Meeting will be held jointly with the:

British Academy of Forensic Sciences
Canadian Society of Forensic Sciences
National Association of Medical Examiners

The meeting will be held in Phoenix, Arizona. Contact:

Robert J. Joling, A.B., J.D.
612 Kenosha National Bank Building
625 57th Street
Kenosha, Wisconsin 53140

CORRECTION: The alpha-Methyltryptamine item in the last issue (Vol.III, No.3 May, 1970) was in error. We suggest that you paste the following corrected item over the previous entry:

alpha-Methyltryptamine 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.); Microgram, I, 4, p.42 and I, 6, p. 80. alpha-Methyltryptamine is not a federally controlled drug.

AN INSTRUMENTAL METHOD FOR SEPARATING AND IDENTIFYING THE COMPONENTS IN AMOBARBITAL-SECOBARBITAL CAPSULES

By

Alois Beck, B.S.,**
William Jensen, M.S.**
Ronald Backer, Ph.D.,**
Richard Barnett, M.S.,**

A previously described method for the separation and identification of cannabinoids of marihuana[1] has been applied to the analysis of amobarbital-secobarbital capsules. While many combination drugs can be separated by solvent extraction because of differences in their basicity and acidity, the combination in these capsules does not lend itself to this type of separation. Although this paper is concerned with the amobarbital and secobarbital combination, the technique can be applied to other combination drugs which have similar acidic or basic properties. The three step procedure includes solvent extraction, gas chromatography and infrared spectrophotometry.

Equipment: 1 separatory funnel (30 ml or less), 1 beaker (5 ml), 1 small funnel, 2 disposable glass pipettes, 1 syringe (100 ul), Whatman #1 filter paper, cotton, KBr powder (IR), Polystyrene or sheets 1/16" thick for preparing KBr disc according to Shumaker.[2]

Instruments used:

1. Gas Chromatograph-Varian Aerograph, Model 1860 equipped with a flame ionization detector and using nitrogen as the carrier gas. The column used was 10' x 1/4" stainless steel, packing with 3% Se-30 on Chromosorb W-AW-DMCS. The instrument was fitted with a 10:1 effluent splitter. The column temperature was 200°, the detector temperature 250° and nitrogen flow was 40 ml/min.

**Milwaukee Health Department, Bureau of Laboratories, Milwaukee, Wisconsin 53202

[1]Backer, R., Jensen, W., Beck, A., Barnett, R. A Simple Method for the Infrared Identification of the Cannabinoids of Marijuana Resolved by Gas Chromatography, J. For. Sci. In press.

[2]Shumaker, W. H., Foamed Plastic in Press Disc Technique for Spectrophotometry, Chem. Analyst., 50,22 (1961).

2. Infrared Spectrophotometer-Perkin-Elmer Model 457

Procedure:

1. Solvent Extraction: Take up 50-100 mg of the sample in 2 ml of 0.1 N-NaOH. Filter through Whatman No. 1 paper into a separatory funnel. Extract with 2 ml of chloroform and discard the extract. Acidify the solution with 1.0 N-HCl and extract with 2 ml of chloroform. The chloroform extract is evaporated to dryness under a stream of air.

2. Gas Chromatography: Prepare the two glass disposable pipettes as follows: A cotton pledget is pushed into the pipette as illustrated (Fig. 1). The tip of the pipette is then pushed into some KBr powder to a depth of about 2.5 mm. This is about 10 mg. The KBr is then gently tamped back to the cotton. The residue from the extraction procedure is taken up with 100 ul chloroform. Ten microliters are injected into the gas chromatograph. The first pipette is inserted into the effluent port as the pen begins tracing the first peak (amobarbital). It is removed as soon as the pen begins its downward travel. The second pipette is inserted into the effluent port as the pen approaches the top of the second peak (secobarbital). It can be removed after the pen has completed its downward travel.

3. Infrared Spectrophotometry: Two discs are cut from the polystyrene or polyurethane sheets. A small opening is cut in the center of the disc. The disc is placed on a steel plate. The KBr from the first pipette is emptied into the disc opening and another steel plate is placed on top of the disc. This is now pressed in a hydraulic press at a high enough pressure (10,000-15,000 psi) to give a transparent window. The pressed disc is placed in a disc holder and the spectrum is run on the infrared spectrophotometer. The procedure is repeated for the KBr in the second pipette.

Note 1 The retention time of the barbiturates were determined using standards obtained from Eli Lilly & Company. The following times were recorded under the conditions of this experiment: Amobarbital, 2 minutes 18 seconds; Secobarbital, 2 minutes 58 seconds.

Note 2 Collection directly on potassium bromide packed in a disposable pipette has some inherent losses due to effluent escape around the exit port, incomplete condensation, and inaccurate timing of the insertion and removal of the pipette. Any change in the split-ratio due to increased resistance was not measured. Interferences in the infrared spectrum from gas chromatograph column bleed was not a problem.

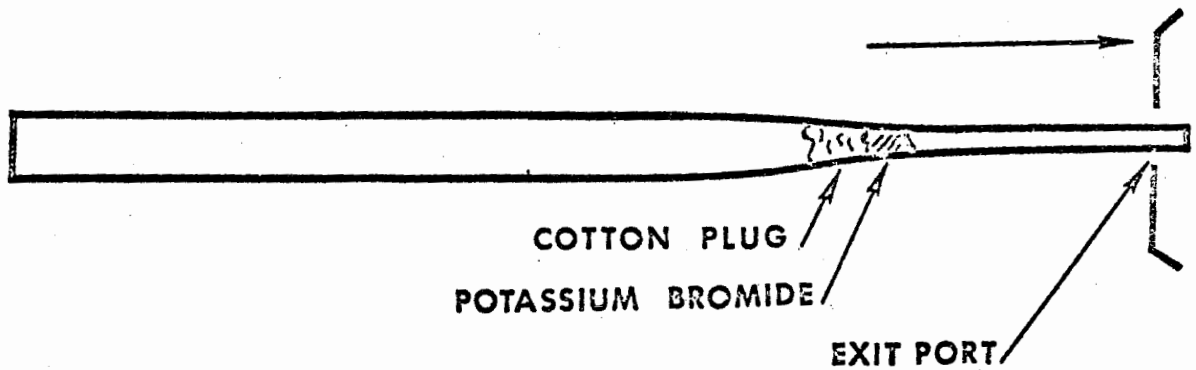
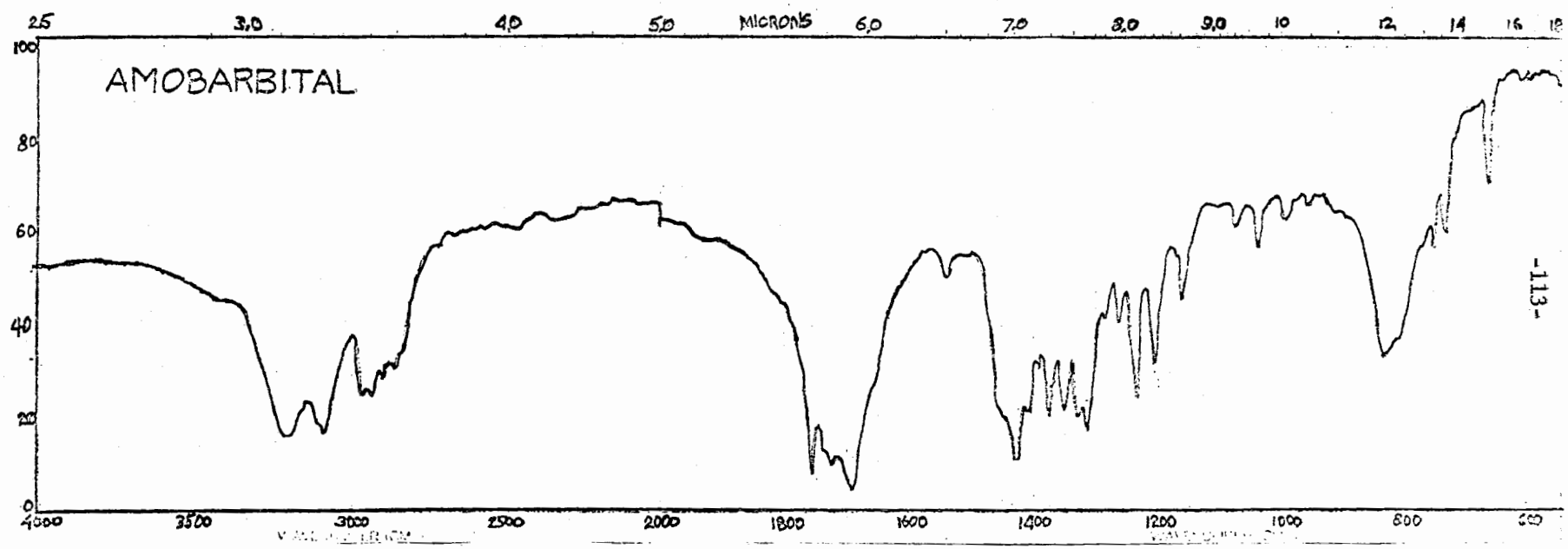
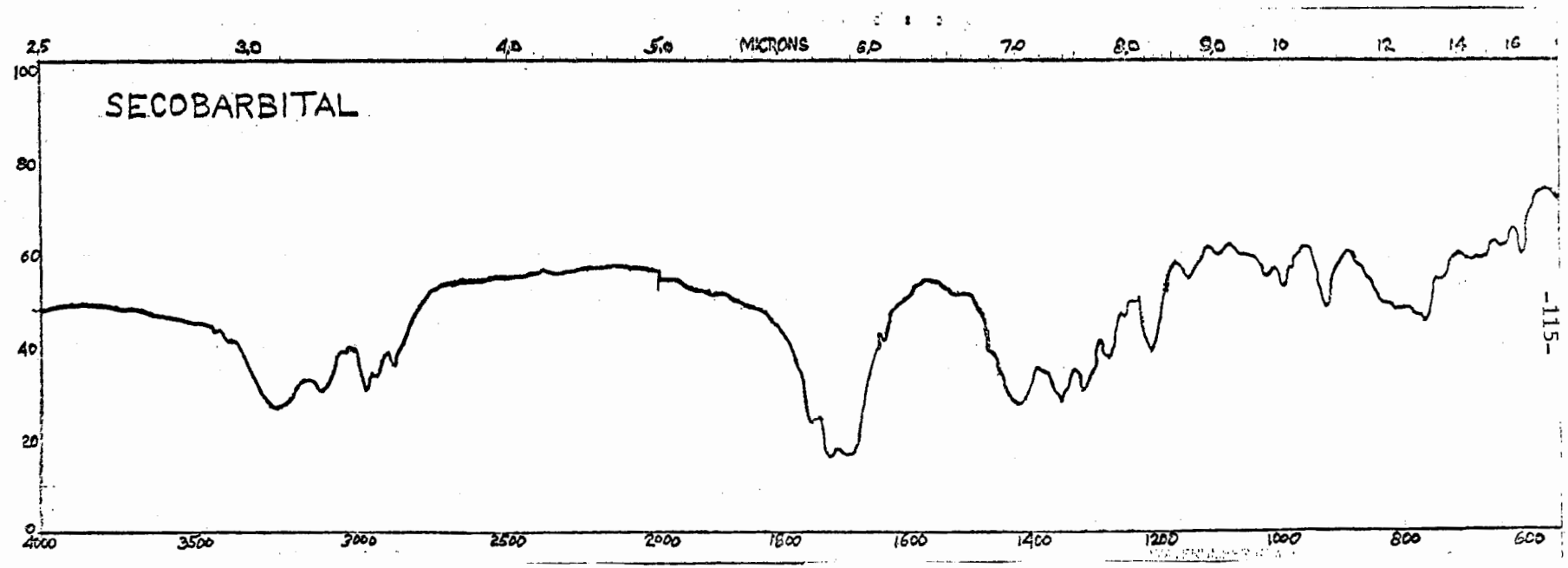


Figure 1





QUALITATIVE AND QUANTITATIVE DETERMINATION OF BENACTYZINE HYDROCHLORIDE (DMZ), JB 336, AND JB 318 BY GAS CHROMATOGRAPHY

By

James P. Done, Forensic Chemist
Chicago Regional Laboratory, BNDD

Several reagents, e.g., sulfuric acid and Marquis Reagent, employed in spot tests for Benactyzine, JB 336 and JB 318 do not distinguish between these compounds, and therefore, the spot tests obtained with these reagents are not entirely qualitative.

The infrared spectra of Benactyzine, JB 336 and JB 318 are very similar. In order that a suitable spectrum can be obtained, a considerable amount of the drug must be available and usually a thorough and vigorous extraction procedure must be employed.

In this laboratory samples of JB JB 336 have been received which consisted of as little as two tablets of 8 mg., JB 336 each. Therefore, a technique for qualitative and quantitative determination of these compounds was sought.

A convenient and rapid gas chromatographic method for the qualitative and quantitative determination of these drugs in low dosage was developed. Benactyzine, JB 336, and JB 318 may be analyzed as the hydrochloride salts, and therefore, extraction procedures can be entirely elminated.

Method:

Column: 6 ft. x 4 mm i.d. glass column packed with 1% SE-30 on Supelcoport 80/100.

Director: Flame ionization detector.

Instrument: Parameters:

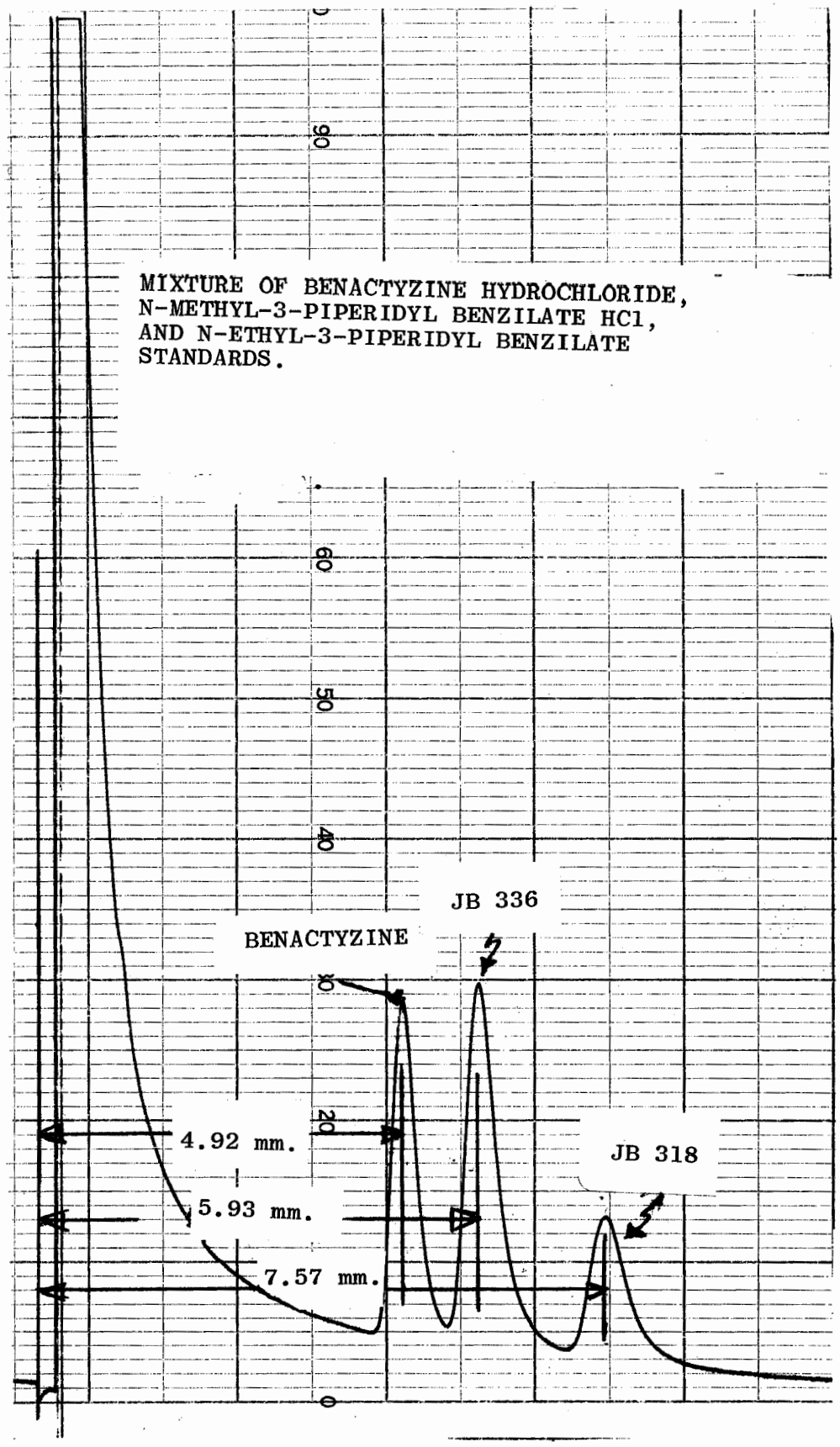
Column Temperature	170°C
Injector Temperature	280°C
Detector Temperature	260°C
Attenuation:	100/2

Carrier gas: Nitrogen Flow rate: 80 ml/min.
Chart Speed: 1/2 inch/minute

Procedure: Transfer the weight of finely ground powder equivalent to the weight of one dosage form (usual dose is 2 to 10 mg) to a glass stoppered 10 ml. volumetric flask. Add about 5 ml of ethanol, shake for 1 minute, and then make to volume with ethanol. Inject appropriate volume onto chromotographic column to insure measurable peak areas.

Results:

Compound	Peak area/mcg Injected	Retention Time
Benactyzine HCl	146.4 mm^2	4.92 min.
JB 336	146.6 mm^2	5.93 min.
JB 318	166.7 mm^2	7.57 min.



MIXTURE OF BENACTYZINE HYDROCHLORIDE, N-METHYL-3-PIPERIDYL BENZILATE HCl, AND N-ETHYL-3-PIPERIDYL BENZILATE STANDARDS.

DATE May 12, 1970
NO. 1
DRUG TYPE Narcotic
METHODOLOGY Column Chromatography and Spectrophotometry

ION PAIRING CHROMATOGRAPHIC SEPARATION AND DETERMINATION OF COCAINE

Roger F. Canaff
Forensic Chemist
New York Regional Laboratory
Bureau of Narcotics & Dangerous Drugs

BACKGROUND

Cocaine is usually found as the hydrochloride and adulterated with procaine hydrochloride or tetracaine hydrochloride and sugar. Cocaine hydrochloride is soluble in chloroform and the hydrochloride salts of procaine and tetracaine are not as soluble. Consequently, cocaine can be separated using a Celite column with a suitable strength of hydrochloric acid as immobile solvent. Since cocaine hydrolyzes rapidly in the presence of strong acid, it is advisable to perform the analysis as rapidly as possible. The cocaine hydrochloride is collected in a volumetric flask and a portion is evaporated on the steam bath and rediluted with dilute sulfuric acid to a concentration of about 15 mcg. per ml. The UV spectrum is scanned, and the peak at 233 mμ used for comparison.

PROCEDURE

Quantitative Analysis:

Accurately weigh a portion of the sample equivalent to about 15 mg. cocaine hydrochloride. Transfer quantitatively to a column consisting of 3 grams acid washed Celite 545 and 2 milliliters of 2N hydrochloric acid with small portions of water-washed chloroform, allowing the washings to flow into a 100 ml. volumetric flask. Elute to volume with water-washed chloroform. Withdraw a 10 ml. aliquot and evaporate to dryness. Transfer residue to a 100 ml. volumetric flask with 0.1N sulfuric acid. Dilute to volume with 0.1N sulfuric acid. Immediately scan UV spectrum from 320 - 210 mμ, using the maximum at about 233 mμ for quantitative analysis. Compare with a freshly prepared standard solution of cocaine hydrochloride in 0.1N sulfuric acid.

Qualitative Analysis:

Pass remainder of chloroform extracts through a 2 inch sodium sulfate column. Evaporate to dryness and scan IR spectrum using Nujol mull or KBr disc technique.

Recovery

mg. Cocaine HCl added	mg. Cocaine HCl found	% Recovered
13.5	13.5	100.1
15.7	15.3	97.5

DATE May 12, 1970
NO. 2
DRUG TYPE Hallucinogen
METHODOLOGY Fluorimetry

Fluorimetric Determination of STP and MDA

Roger F. Canaff
Forensic Chemist
New York Regional Laboratory
Bureau of Narcotics & Dangerous Drugs

BACKGROUND

Because of its extreme sensitivity, fluorescence spectroscopy can be used to advantage by the forensic chemist to determine many substances without prior separation or cleanup. Two such substances are 4-methyl-2,5-dimethoxy-α-methyl phenethylamine (STP) and α-methyl-3,4-methylenedioxy phenethylamine (MDA). In addition, the differences in the fluorescent characteristics (excitation and emission peak wavelengths) provide a means of distinguishing them. The fluorescence-concentration relationship was found to be linear from 0.25-1.0 mcg/ml. for both compounds. Above the upper concentration limit, the relationship ceased to be linear.

PROCEDURE

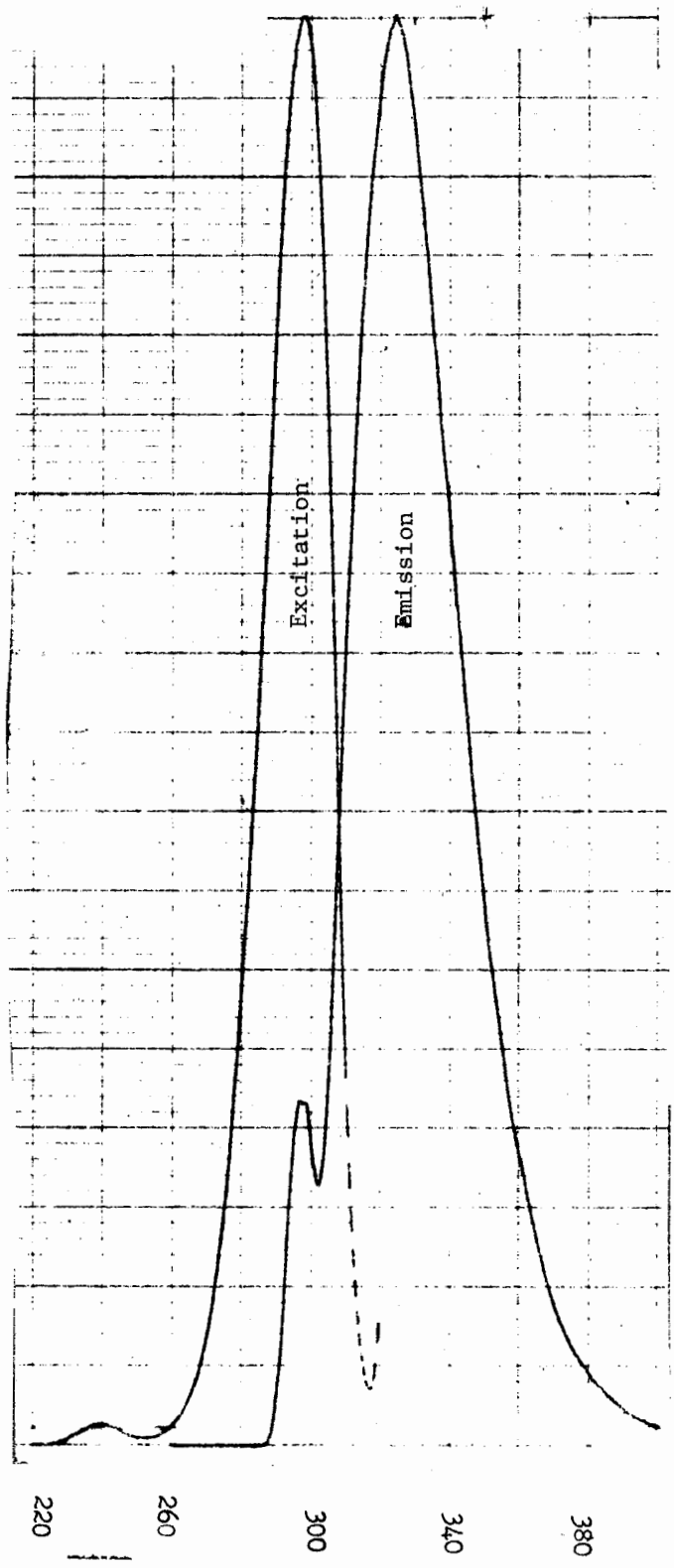
Accurately weigh a portion of sample equivalent to 1 mg. STP or MDA into a 100 ml. volumetric flask. Add about 50 ml. methanol and shake mechanically or place in ultrasonic bath for ten minutes. Dilute to volume with methanol. Filter a portion or allow to settle and draw off 1.0 ml. to a 25 ml. volumetric flask. Dilute to volume with methanol. Dial the following instrumental parameters:

	STP	MDA
Excitation Slit	6 mμ	6 mμ
Emission Slit	6 mμ	6 mμ
Sample Sensitivity	3	3
Excitation Wavelength	298 mμ	292 mμ

Set the instrument to a fluorescence of about half scale deflection (50) for the appropriate standard at a concentration of 0.4 mcg/ml. using the

"Sample Adjust" control. Scan the fluorescence excitation and emission spectra of sample and standard and compare the fluorescence at the wavelength of maximum emission: 324 mμ for STP and 322 mμ for MDA.

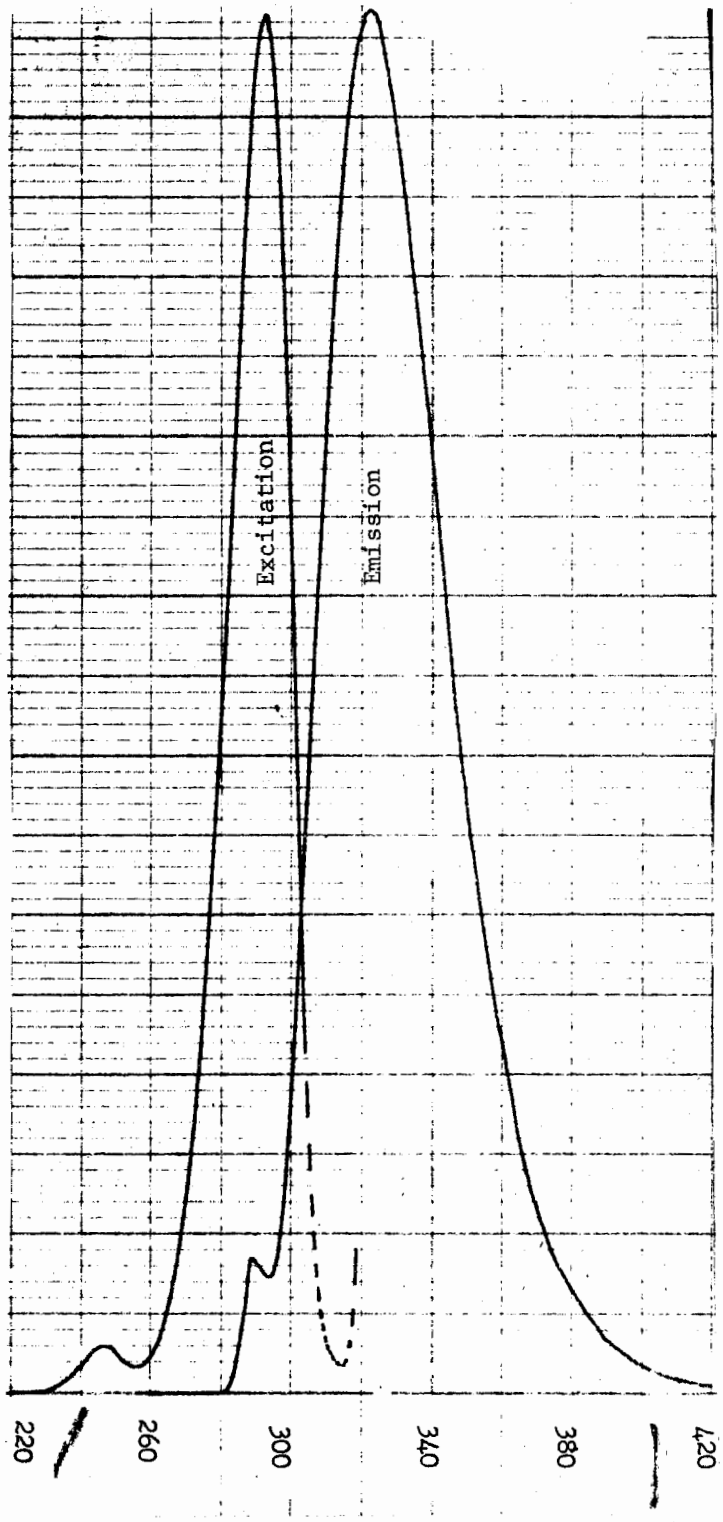
Fluorescence spectra for STP and MDA are shown in Figure 1,2.



STP HCl

0.96 mcg/ml in MeOH
Slits 6mu/6mu
S. Sens. 3
Filter 290

Fig. 1



MDA HCl

0.90 mcg/ml in MeOH
Slits 6mu/6mu
S. Sens. 3
Filter 290

Fig. 2

DATE June 5, 1970
NO. 3
DRUG TYPE Stimulant and Depressant Drugs
METHODOLOGY

A QUANTITATIVE SEPARATION OF METHAMPHETAMINE AND BARBITURATE FROM TABLETS INCORPORATING VITAMIN MIXTURES, BY COLUMN PARTITION CHROMATOGRAPHY

Stanley Schreiber
Forensic Chemist
Dallas Regional Laboratory
Bureau of Narcotics & Dangerous Drugs

PROBLEM

On several occasions this laboratory has received samples of tablets containing mixtures of Methamphetamine, Barbiturates, and Vitamins. Prior to this, the analyst resorted to an extraction of some type for the recovery of the Barbiturate and finally distillation of Methamphetamine. As a result, in many cases, the recoveries were low and much time was wasted. The following column partition method proved to be a fast and clean quantitative separation of both Methamphetamine and the Barbiturate.

METHOD

Apparatus:

a. Two glass partition columns.

Reagents:

a. 1N NaOH
4N HCl
0.1N HCl
1M $NaHCO_3$ (freshly prepared)
NH_4OH (concentrated and 1+ 24)
Chloroform (water-washed)
Celite "545" - acid-washed

PROCEDURE

a. Column I

Top Layer: Sample equivalent of approximately 5 mg Methamphetamine Hydrochloride mixed with 3 gms acid-washed Celite and 2 ml 1N $NaHCO_3$.

Bottom Layer: 3 gms acid-washed Celite mixed with 2 ml 4N HCl.

b. Column II

3 gms acid-washed Celite mixed with 2 ml 0.1N HCl.

BARBITURATE

Place Column I over Column II and elute both with 200 ml water-washed chloroform. Eluate from Column II will contain the Barbiturate. Methamphetamine will pass through the 4N HCl layer and be trapped onto the 0.1N HCl Column (II) and the coloring material and vitamins will remain on the sodium bicarbonate and 4N HCl layers of Column I. Evaporate the eluate containing Barbiturate to 100.0 ml and take a suitable aliquot to dryness on a steam bath. Dissolve and dilute the residue with 1+24 NH_4OH and record the U.V. spectra from 300 to 220 mu with maximum absorbance at 240 mu. The remaining chloroform solution may be evaporated to dryness and the residue used to prepare the KBr disc for infrared identification or for optical crystallographic identification of the Barbiturate.

METHAMPHETAMINE

Elute Methamphetamine from Column II with 2 ml of Ammonium Hydroxide followed by 125 ml of water-washed chloroform into a 150 ml beaker. Evaporate the eluate on a steam bath to approximately 80 ml. (To rid the eluate of Ammonia) and transfer quantitatively to a 125 ml separatory funnel containing 10.0 ml of 0.1N HCl. Extract Methamphetamine into the HCl layer by shaking. Record the U.V. spectra of the acid layer (using a reference of chloroform saturated 0.1N HCl) from 300 to 220 mu with maximum absorbance at 257 mu. Transfer the solution employed in U.V. quantitation to a 125 ml separatory funnel and make basic with sodium hydroxide. Extract Methamphetamine with 2-5 ml portions of chloroform; add 2 drops of concentrated HCl to the chloroform extract and evaporate to dryness on a steam bath. The residue is placed in an oven preset at 100°C for 5 minutes. The dried

Methamphetamine salt may then be identified by infrared spectrophotometry and microcrystalline tests. Microcrystalline tests may also be made directly from the tablet mixture.

RESULTS

Recoveries of 95-98% were observed using this method on tablets such as Obedrin-LA manufactured by S.E. Massengill Company, Bristol, Tennessee, and also more recently a 135,000 tablet sample manufactured by Morton Pharmaceuticals containing Methamphetamine Hydrochloride and Vitamins.

REFERENCE

Doyle, T.D., Levine, J., Anal. Chem., Vol. 39, No. 11 (1967); JAOAC, Vol. 51, No. 1 (1968).

DATE June 5, 1970
NO. 4
DRUG TYPE Stimulant Drugs
METHODOLOGY

A QUANTITATIVE SEPARATION OF METHAMPHETAMINE AND d-dl AMPHETAMINE FROM TABLET MATERIALS BY COLUMN-PARTITION CHROMATOGRAPHY

Stanley Schreiber
Forensic Chemist
Dallas Regional Laboratory
Bureau of Narcotics & Dangerous Drugs

PROBLEM

Tablets (and capsules) containing both Methamphetamine and d+dl Amphetamine have previously been assayed and reported as total amphetamine. By use of this method of separation, however, the analyst can easily separate and identify Methamphetamine from d+dl Amphetamine.

METHOD

A. Apparatus: 3 glass partition columns.

B. Reagents:
1N NaOH
4N HCl
0.1N HCl
1M $NaHCO_3$ (freshly prepared)
NH_4OH
Chloroform (water-washed)
Celite "545" (acid-washed)

PROCEDURE

Column I

Sample equivalent to 5 mg Methamphetamine salt and 5 mg d+dl Amphetamine salt (total of 10 mg of Amphetamines) mixed with 3 gms acid-washed Celite and 2 ml 1M $NaHCO_3$.

Column II

3 gms acid-washed Celite mixed with 2 ml 4N HCl.

COLUMN III

3 gms acid-washed Celite mixed with 2 ml 0.1N HCl.

Place Column II over Column III and Column I over Column II. Elute the 3 columns with approximately 200-250 ml of water-washed chloroform. Discard the eluate. The d+dl Amphetamine is trapped onto the 4N HCl Column (II) and the Methamphetamine passes through the 4N HCl Column and is trapped onto the 0.1N HCl Column (III). Elute the d+dl Amphetamine from Column II and Methamphetamine from Column III with 2 ml of Ammonium Hydroxide followed by 125 ml of water-washed chloroform into separate 150 ml beakers. Evaporate each of the eluates on a steam bath to approximately 80 ml (to rid the eluates of ammonia) and transfer each quantitatively to 125 ml separatory funnels containing 10.0 ml of 0.1N HCl. Extract the Amphetamines (in each funnel) into the HCl layer by shaking. Record the U.V. spectra of the acid layers (using a reference of chloroform saturated 0.1N HCl) from 300 to 220 mu with maximum absorbance at 257 mu. Transfer the solutions employed in the U.V. quantitation to separate 125 ml separatory funnels and make each basic with sodium hydroxide. Extract each with 2-5 ml portions of chloroform; add 2 drops of concentrated HCl to each chloroform extract and evaporate to dryness on a steam bath. The residues are placed in an oven preset at 100°C for 5 minutes. The dried Amphetamine salts may then be identified by Microcrystalline tests for Methamphetamine and d+dl Amphetamine.

RESULTS

Recoveries of 89-94% for Methamphetamine and 96-99% for d+dl Amphetamine were observed using this method on tablets such as Amphaplex tablets manufactured by Palmedico, Inc., Columbia, South Carolina.

REFERENCE

Doyle, T.D., Levine, J ., Anal. Chem., Vol. 39, No. 11 (1967); JAOAC, Vol. 51, No. 1 (1968).

FEDERAL REGISTER

Title 21—FOOD AND DRUGS

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

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

Meprobamate; End of Stay of Effective Date of Order Listing Drug as Subject to Control

In the matter of listing meprobamate as a drug subject to control under the Drug Abuse Control Amendments of 1965:

By an order published in the FEDERAL REGISTER on March 1, 1968 (33 F.R. 3635), the Commissioner of Food and Drugs stayed the effective date of the final order published in the FEDERAL REGISTER on December 6, 1967 (32 F.R. 17473), listing meprobamate as a drug subject to control under the Federal Food, Drug, and Cosmetic Act, as amended by the Drug Abuse Control Amendments of 1965. This stay was requested by Wallace Laboratories, a division of Carter-Wallace, Inc., Cranbury, N.J., pending their petitioning, pursuant to section 701(f) of the Federal Food, Drug, and Cosmetic Act, for judicial review in the U.S. Court of Appeals and review of petition, if any, for writ of certiorari to the U.S. Supreme Court.

On November 4, 1969, the U.S. Court of Appeals for the Fourth Circuit confirmed the order of December 6, 1967 (32 F.R. 17473), of the Commissioner of Food and Drugs. On June 1, 1970, the U.S. Supreme Court denied the petition for writ of certiorari.

Therefore it is ordered, That the stay of effectiveness granted by the order of March 1, 1968 (33 F.R. 3635), on the listing of meprobamate in § 320.3(c)(1) (formerly 166.3(c)(1)) as a drug subject to control under the Drug Abuse Control Amendments of 1965, be ended.

Effective date. This order shall become effective on July 6, 1970.

Dated: June 2, 1970.

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

[F.R. Doc. 70-7038; Filed, June 5, 1970; 8:47 a.m.]