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. IV, No. 3 March, 1971

ALPHA-METHYLTRYPTAMINE was recently found in a clandestine laboratory seized in Illinois. Approximately 3 pounds of the powder and one pint of liquid was on hand. The drug is not under federal control.

PEYOTE buttons were recently encountered in New England.

HAWAIIAN BABY WOOD ROSE and the seeds of its cousin, MORNING GLORY, were recently identified in an exhibit from the Midwest.

2,5-DIMETHOXYAMPHETAMINE is being encountered by all BNDD laboratories. It has been obtained as suspected STP, heroin, MMDA and mescaline, and has been seen in capsules, plastic bags, glassine envelopes and a red tablet. It is not under federal control.

MDA (3,4-METHYLENEDIOXYAMPHETAMINE) as the phosphate was encountered for the first time in the New York region. The drug was found when a clandestine laboratory was seized. From a review of the chemist's notes, he had been trying various ways to increase the yield of his product.

PCP-LSD combinations are being seen by all BNDD laboratories. The mixture is being found in capsules and in tablets of various colors. LSD in the tablets ranges from about 280 to 480 micrograms, and the PCP ranges from about 3 to 5 milligrams. The capsules have a wide potency range.

PCP with procaine has been analyzed by the San Francisco laboratory.

LSD gelatin flakes are still being seen in various parts of the United States. The flakes were recently seen in Australia in material sent from the U. S. They were first encountered in England in the possession of a U. S. citizen.

The flakes are usually 3 or 4 millimeters square, and are called "Clear Lights" or "Window Glass." Round flakes have also been encountered, known as "Contact Lens." Analysis shows approximately 140 to 180 micrograms LSD.

HEROIN-METHAPYRILENE is still seen in the Midwest. The potency varies. Recent analyses, for example, show 8% heroin with 9.1% methapyrilene, and 4% heroin with 1.6% methapyrilene. The mixtures usually contain quinine and lactose, but may contain procaine, mannitol, starch or other material.

Methapyrilene is an antihistamine, which, as a side effect, causes drowsiness. Because of this, it is promoted under various trade names as an over-the-counter product intended to induce sleep.

LSD on saccharin tablets was recently seen in the Midwest. They contained 80 micrograms of LSD.

WHAT IS IT? THC is still being promoted on the street, but is always found to be some other drug--usually PCP (Phencyclidine HCl). Suspected cocaine has also been found to be PCP. Suspected heroin has been identified as MDA, suspected hashish as opium, suspected amphetamine as mescaline, suspected mescaline as LSD, suspected STP as 2,5-dimethoxyamphetamine, and of course, suspected marihuana can be almost any plant material.

"Legal Hash" advertised in an underground newsletter was examined in the BNDD New York Regional Laboratory, and was found to be catnip (Nepeta cataria L).

PCP (Phencyclidine) with piperonal was found in a small, white capsule submitted to the BNDD San Francisco Regional Laboratory by a local police department. Piperonal is not a precursor to PCP, but is used in the synthesis of MDA (3,4-methylenedioxyamphetamine).

Heroin in cigarettes was encountered by the BNDD Dallas Regional Laboratory in an exhibit from local Texas police department. A portion of tobacco had been removed, a quantity of heroin added and the cigarette paper twisted closed. Quantitation of one cigarette showed 63.6 milligrams heroin.

Hydroxychloroquine sulfate has been encountered by the BNDD New York Regional Laboratory. The material was purported to be quinine to be used for cutting.

LSD on lozenges has appeared in the West. The lozenges were "Lakerol" yellow pastilles made in Sweden and contained 58.3 micrograms of LSD each. The LSD apparently was placed on the outer surface of the lozenges.

MEETINGS

Sixth International Meeting of Forensic Sciences, Belfast, 1972 will be held September 21-26, at the Queen's University, Belfast, Northern Ireland. Additional information can be obtained by writing to:

The Secretariat
Sixth International Meeting on Forensic Sciences
Institute of Pathology
Grosvenor Road
Belfast, BT12 6BL
Nortern Ireland

NOTICE

Copies of the flyer on the last page of this issue have been distributed in the Far East by U. S. Air Force, Office of Special Investigations. If any are encountered, we would appreciate all available information.

TRAINING

The Institute for Advanced Analytical Chemistry, Georgetown University, is offering the following two-week short courses:

Forensic Optical Microscopy
Instructor - Dr. Mary Willard
Dates: May 17 - 28, 1971

Applications of Gas-Liquid, Thin Layer and Gel Permeation Chromatography in Forensic Analysis
Dates: June 21 - July 2, 1971

Fluorescence and Phosphorescence Techniques in Forensic Analysis
Dates: July 12 - 23, 1971

For further information, contact:

Director
Institute for Advanced Analytical Chemistry
Georgetown University
Washington, D. C. 20007

or call: (202) 625-4431

The estimated tuition is $450.

SELECTED REFERENCE

Coumbis, Richard J.; Fulton, Charles C.; Calise, Joseph P.; and Rodriguez, Cary; The Necessity of Elution and Identification of Drugs Indicated by Thin-Layer Chromatography, Journal of Chromatography, Vol. 54, No. 2, (January 20, 1971), p. 245-250.

BNDD LABORATORY NOTES

DATE February 16, 1971

NO. 13

DRUG TYPE Cocaine and Caffeine Mixture

METHODOLOGY Spectrophotometric and Gas Chromatographic

The Gas Chromatographic and Spectrophotometric Analysis of Cocaine and Caffeine Mixtures

Chemist James M. Moore
Washington Regional Laboratory

BACKGROUND

The Washington Regional Laboratory recently received a large quantity of suspected cocaine that was sold on the illicit market. Preliminary solubility, color and thin-layer chromatographic tests indicated the presence of cocaine diluted with a sugar. However, gas chromatographic analysis revealed a peak eluting before and in addition to cocaine. Further tests identified this component to be caffeine. The results of the chromatographic and spectrophotometric procedures were compared to a gravimetric method. Additionally, methodology is suggested for a smaller sample consisting of cocaine, caffeine, procaine and a sugar.

SPECTROPHOTOMETRIC PROCEDURE

Sample Preparation - Into a 100 ml. beaker place 2 gm. of diatomaceous earth (Celite 545, acid-washed, Johns-Manville, New York, N.Y.) and then add 1 ml. of 0.5N H_2SO_4. Mix with spatula until fluffy. Transfer this mixture to a glass chromatographic column (250 mm. long by 22 mm. i.d., with one end constricted to a stem) which contains a piece of glass wool in its stem. Tamp the mixture in the column until packed moderately. Into another 100-ml. beaker weigh accurately a portion of powdered sample equivalent to 5-20 mg. of cocaine. Add 2 ml. of 0.5N H_2SO_4 to the sample and swirl the beaker to dissolve the cocaine and caffeine. To this solution add 3 gm. of diatomaceous earth and mix until fluffy. Transfer this mixture quantitatively to the chromatographic column and pack moderately. Place a plug of glass wool on top of packing. Elute the caffeine from the column into a 100 ml. volumetric flask with 100 ml. of water-washed chloroform. Save this flask for the caffeine analysis. Elute the cocaine from the column into a 250-ml. separatory funnel by first passing a sufficient volume of ammonia-saturated chloroform through the column to neutralize the acid layer followed by 100 ml. of water-washed chloroform. Add 25.0 ml. of 0.5N H_2SO_4 saturated with chloroform to the separatory funnel and shake the funnel vigorously for one minute. Allow the layers to separate and save the upper aqueous layer for the cocaine analysis.

GRAVIMETRIC ANALYSIS

Weigh accurately about 1 gm. of powdered sample into a 150 ml. beaker. Add about 25 ml. of chloroform and warm gently on a steam bath to dissolve the cocaine and caffeine. Quantitatively filter the chloroform solution through filter paper into a 100-ml.-volumetric flask. Wash beaker with several small portions of chloroform, transferring each through the filter paper into the flask. Dilute the flask to volume with chloroform. Pipette 75.0 ml. of the chloroform solution into a previously tared 100 ml. beaker. Gently evaporate the chloroform solution to dryness on a steam bath under a current of air. Dry residue at about 50°C. for 1/2 hr. Weigh the dried residue and calculate the percentage of chloroform soluble materials.

QUANTITATIVE RESULTS

See Table 1 for the results using the three procedures.

IDENTIFICATION

(A) Caffeine

(1) Microcrystalline - Observe crystals formed by reacting powder with a drop of gold bromide in dilute hydrochloric acid (1).

(2) Thin-layer chromatography - Mix portion of powdered sample with chloroform and apply to a silica gel g plate containing a fluorescent indicator. Use solvent systems given in Internal Revenue Service, "Methods of Analysis" Rev. 6-67, pp. 92-94. Observe the caffeine spots on the developed plate under short wave ultraviolet light (caffeine cannot be visualized using iodoplatinate spray used for cocaine).

(3) Infrared spectroscopy - Evaporate a portion of the chloroform eluant obtained in the spectrophotometric procedure to dryness. Obtain the infrared spectrum of 1-2 mg. of the caffeine residue mixed with 200 mg. of KBr.

(B) Cocaine

(1) Microcrystalline - a positive test could not be obtained on the dry powder using gold chloride (caffeine evidently interfered with the test).

(2) Thin-layer chromatography - Proceed as with caffeine except visualize the cocaine spots using iodoplatinate spray.

Caffeine Analysis - Dilute, if necessary, the eluant containing caffeine with chloroform to a final concentration of about 0.015 mg/ml. Prepare a standard caffeine solution in chloroform of equal concentration. Scan both sample and standard solutions in the ultraviolet between 300 and 230 mu using the maximum at about 275 mu for quantitative purposes.

Cocaine Analysis - If necessary dilute the acid aqueous layer in the separatory funnel to a final cocaine concentration of about 0.20 mg/ml. with 0.5N H_2SO_4 that has been saturated with chloroform. Prepare a cocaine standard of equal concentration and in a similar solvent. Scan the sample and standard solutions in the ultraviolet between 350 and 250 mu using the maximum at about 275 mu for quantitation. If desired the residual chloroform in the acid solution can be evaporated and both the sample and standard solutions can be diluted with 0.5N H_2SO_4 to a final concentration of about 0.01 mg/ml. At this concentration the maximum about 230 mu is used for quantitation.

GAS CHROMATOGRAPHIC PROCEDURE

Sample Preparation - Weigh a portion of powdered sample equivalent to about 10 mg. of cocaine into a 25 ml. volumetric flask. Pipette 5.0 ml. of n-eicosane[1] internal standard solution (1.0 mg/ml. in chloroform) into the flask. Dilute to volume with chloroform. Prepare a mixed standard in chloroform consisting of cocaine, caffeine and n-eicosane having concentrations of 0.40, 0.30, and 0.20 mg/ml. respectively.

G.L.C. Quantitation - Inject the sample and standard solutions into the gas chromatograph operating under the following parameters:

(a) Instrument - Perkin Elmer 900
(b) Column - A 6 foot by 1/4 inch i.d. glass column packed with 3% OV-1 on Chromosorb WHP (80-100 mesh)
(c) Temperatures - Column 210°C; Injector and detector 275°C
(d) Carrier Gas - Nitrogen, at a flow rate of about 50 ml/minute
(e) Detector - A flame ionization detector with air and hydrogen flow rates to the detector of about 300 and 30 ml/minute, respectively
(f) Recorder - A 1-mv recorder operated at a chart speed of 1/2 inch/minute
(g) Sensitivity - 10^{-9} AFS

See Figure 1 for a typical chromatogram. The time marked on the x-axis is in minutes and the y-axis is 10^{-9} AFS.

[1]Obtained from Applied Science Laboratories, State College, Penna.

(3) Infrared spectroscopy - Make the 0.5N H_2SO_4 solution obtained in the spectrophotometric procedure basic with stronger ammonia water and extract the cocaine free base with chloroform. Evaporate the chloroform gently to dryness on a steam bath under a current of air. Obtain the infrared spectrum of 2 mg. of the cocaine free base mixed with 200 mg. of KBr.

DISCUSSION

Mixtures such as the ones encountered in this sample should be subjected to chromatographic analysis prior to quantitative analysis of the active component. In the larger of these samples gas chromatography and thin-layer chromatography using short-wave and iodoplatinate visualization were used to detect the caffeine in addition to the suspected cocaine. Using these screening results a separation scheme for a quantitative analysis was developed.

Chromatographic screening tests on the smaller sample revealed procaine in addition to cocaine and caffeine. In the gas chromatographic screening test procaine elutes between caffeine and cocaine with a retention value similar to that of the internal standard n-eicosane used in the gas chromatographic analysis of the cocaine-caffeine mixture. In the thin-layer chromatographic screening test procaine is visualized with iodoplatinate spray as is cocaine. To quantitate this three component mixture gas chromatography or column chromatography may be conveniently used. In the gas chromatographic assay cocaine, caffeine and procaine all elute within 5 minutes of injection with base line resolution. The parameters for this assay are given within the body of this paper. An internal standard other than n-eicosane is needed because of the similarity of its retention time to procaine. The column chromatographic method given within the body of this paper can be modified to accomodate the three component mixture. This consists of the incorporation of a 0.1N HNO_3 column (2) or a 2N HCL column (3) into the procedure to retain the procaine.

Figure 1.

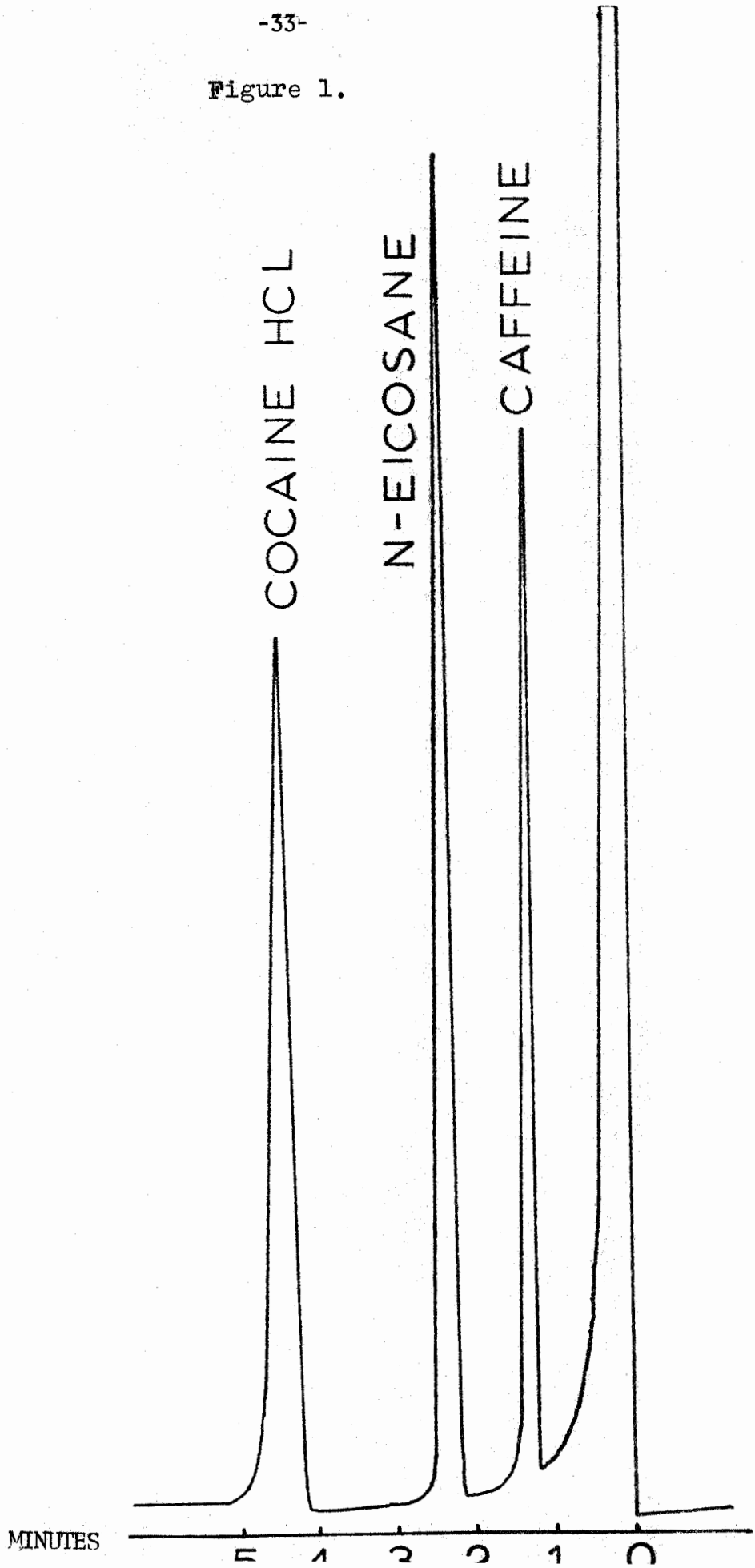


Table 1. Cocaine and Caffeine Assay Results

Method	%Cocaine Found	%Caffeine found	Total % Found
Ultraviolet	21.0	20.9	41.9
G.L.C.	20.8	19.2	40.0
Gravimetric	....	....	42.7

References

(1) Association of Official Analytical Chemists, X, p. 602 (1965).

(2) "The Analysis of Cocaine in Illicit Preparations," by James M. Moore, Microgram, Vol. 111, No. 3, May, 1970, p. 89 - 96.

(3) "Ion Pairing Chromatographic Separation and Determination of Cocaine," by Roger F. Canaff, Microgram, Vol. III, No. 4, June, 1970, p. 121 - 122.

BNDD LABORATORY NOTES

-35-

DATE March 12, 1971

NO. 14

DRUG TYPE Narcotic

METHODOLOGY Column Chromatography and Spectrophotometry

QUANTITATIVE ESTIMATION OF METHADONE IN ORANGE JUICE ("TANG")

Thaddeus E. Tomczak
Forensic Chemist
Washington Regional Laboratory
Bureau of Narcotics and Dangerous Drugs

Problem

In some "Methadone Maintenance Programs," methadone is supplied to the participants in an orange flavored solution ("Tang") at a nominal concentration of 5 mg. methadone hydrochloride per cc. of "juice." On occasion, police agencies have obtained samples of such methadone involving fatal overdoses. Immiscible solvent extraction invariably result in serious emulsions, so a column chromatographic method was attempted with apparent success.

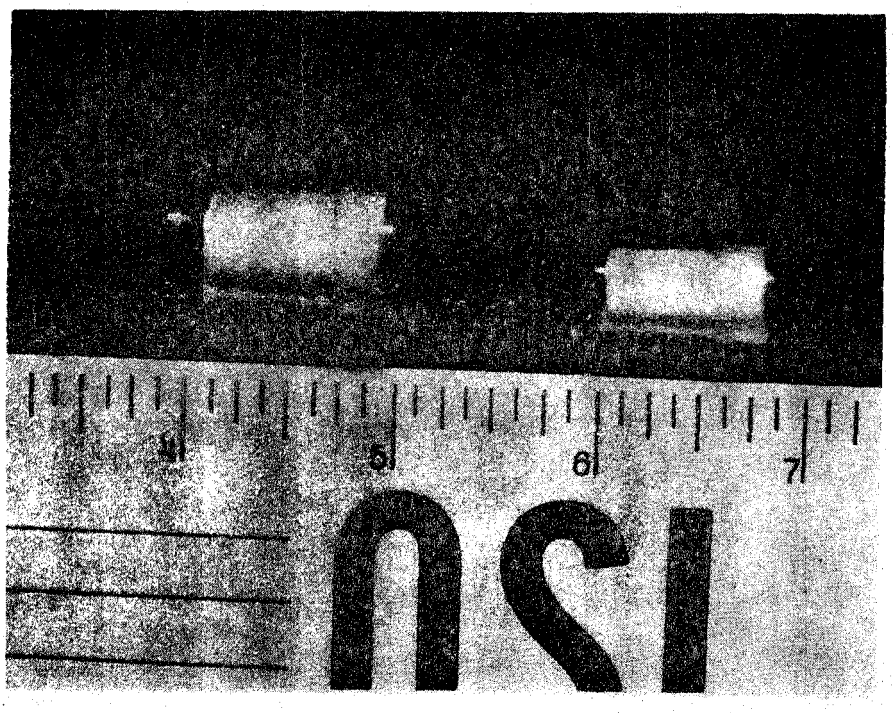
Procedure

Neutralize a 2 ml. sample of methadone in orange juice with "excess" $NaHCO_3$ (no effervescense on addition of dry $NaHCO_3$). Mix resulting mixture with 4 gm. Celite and pack in chromatographic column. Elute methadone base with 50-60 ml. "water washed" $CHCl_3$ so the eluate passes through a second column consisting of 1 ml. 1N-HCl on two grams of Celite.

Quantitative Estimation

The effluent of the second column was collected in a separatory funnel containing about 10 ml. 0.5N-H_2SO_4 and successively extracted with 5 ml. portions 0.5N-H_2SO_4 until the acid extract volume was 25 ml. The U.V. spectrum of this solution was "run" using the absorbance maximum at about 292 nm. for quantitation.

Spectra obtained were excellent, but no recovery studies were done. Our analysis on two separate bottles from the same "clinic" showed 6.58 mg./ml. and 6.45 mg./ml.



HEROIN

HEROIN IS COMMONLY SOLD IN THE REPUBLIC OF VIETNAM IN CLEAR PLASTIC CONTAINERS SIMILAR TO THOSE REPRESENTED ABOVE. THE PLASTIC CONTAINERS ARE USUALLY REFERRED TO AS "CAPS" OR "BARRELS". THE CONTAINERS COME IN TWO SIZES, LARGE CONTAINERS 1 1/8 inches LONG BY 5/8 inches IN DIAMETER, AND SMALL CONTAINERS 7/8 inches LONG BY 7/16 inches IN DIAMETER. THE CONTENTS OF THE CONTAINERS IS A WHITE POWDERY SUBSTANCE, SIMILAR IN NATURE TO FLOUR OR BAKING POWDER.

IF YOU SHOULD FIND A CONTAINER MATCHING THE DESCRIPTION ABOVE, NOTIFY THE PROPER LAW ENFORCEMENT AUTHORITIES AT ONCE.

REMEMBER "HEROIN CAN KILL"