

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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July, 1971

Cocaine free base and cocaine hydrochloride have both recently been encountered in the same exhibits. The exhibits were from Illinois and from a foreign country.

Secobarbital capsules and tablets are being seen from an unknown manufacturing source. If any resembling the following description are encountered, we would appreciate available information and samples if possible.

The capsules are orange or orange-red, No. 3 size, standard shape, hard gelatin and contain 74 to 100 milligrams secobarbital sodium per capsule. They also contain a large amount of gelatinized starch fragments and a small amount of talc.

The tablets are white, flat, round, unscored and contain about 90 milligrams secobarbital sodium per tablet. They were made on a multiple punch tabletting machine equipped with 1/4 inch diameter punches.

Samples should be sent to Chief Chemist, Special Testing and Research Laboratory, Bureau of Narcotics and Dangerous Drugs, Washington, D.C. 20537. Additional instructions on submitting samples for ballistics examination can be found on the front page of Microgram, Vol. III, No. 5, August, 1970.

"DMA" is a mixture of heroin, cocaine and methamphetamine, according to a U.S. Army source in California. This should not be confused with 2,5-dimethoxyamphetamine, which is also called "DMA."

Hashish packed in a cuckoo clock kit from Germany was reported by the St. Louis, Missouri, Police Department Laboratory. According to the report, the hashish had several wrappings. The inner cloth wrappings had dark spots on one side, forming a uniform

Analytical methods in **Microgram** do not have official status. Use of funds for printing this publication approved by the Bureau of the Budget, April 8, 1969. **CAUTION:** Use of this publication is restricted to forensic scientists serving law enforcement agencies.

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pattern and was stuck to the hashish. Of the four edges of the wrapper, one was folded with no seam, two were made sewn with grey thread, and one appeared hand sewn with white thread. The wrapper had the appearance of a pocket turned inside out and was made of heavy material such as that in pockets in work clothes or uniforms. The hashish sole was convex to the side with the spots, suggesting that the packet was dried on a curved surface, such as a large drum similar to a commercial photo drier.

The clock kit package had pictures of three cuckoo clocks in the upper right corner, "1910" was under one, "1920" under the middle one and "1930" under the clock on the right. In the lower right corner was "KUCKUCK-UHREN-BAUKASTEN Do-it-yourself-CUCKOO-CLOCK." The background had a scene of a clock vender on a mountain trail. Price tag on the kit states: "452 12/154281/\$5.50."

STP (4-methyl-2,5-dimethoxyamphetamine) HCl tablets were analyzed by the BNDD Chicago Regional Laboratory in a submission from local police in Illinois. The tablets were pink-brown, a color similar to that of light cocoa. They were 6.5 millimeters in diameter and about 3.0 millimeters thick. Quantity was insufficient to determine potency.

Patchouly oil, it is rumored, causes a false positive reaction with the modified Duquenois test. Researchers in the BNDD Special Testing and Research Laboratory explored the matter and found that the substance does not give a modified Duquenois reaction. There is no transfer of a pink or violet color to the chloroform layer when the second extraction of the test is performed.

They feel, also, that an experienced analyst should not report a false positive using the Duquenois-Negm, or direct test. This conclusion is based on the following:

1. The color forms immediately on the addition of the hydrochloric acid. In the normal test, the color forms slowly and there is a succession of color changes before the final purple color develops.
2. The color which forms is not the one normally associated with the test. It is a bright magenta color which, on standing for a considerable time, changes to a dark purple that still does not match the purple of the normal test.

LSD in perfume and in solutions containing perfume have been encountered by The Department of Customs & Excise laboratories in Australia. The

solutions contained from 1490 micrograms per milliliter up to 21,120 micrograms per milliliter in a solution having a strong perfume odor. Tablets have also been encountered impregnated with the perfume. The tablets contained 49 micrograms LSD per tablet. One exhibit consisting of the perfume base was tentatively identified as "Shantung," manufactured by Polack & Schartz, Holland.

If you have seen any LSD solutions having an odor of perfume, notify Frank E. Peters, Ph.D., Commonwealth Analyst, Department of Customs & Excise, Canberra, A.C.T. 2600, Australia.

Nitrous oxide ("Laughing gas") is reportedly being abused in Maryland by university students. According to press reports, the students are using Whipmaster Chargers, prepared by British Oxygen Co. Ltd, London. The cartridges are used in whipped cream dispensers such as those found in soda fountains and restaurants. Because of the possible damage or death caused by lack of oxygen to the brain, if the gas is inhaled over a prolonged period, the State of Maryland is formulating new regulations. "Laughing gas" has been abused at various times for the past 200 years.

Annual report from the Boston Laboratory, Division of Food and Drugs, Massachusetts Department of Public Health is of interest, but it is too lengthy to print in full. One page is attached. Almost 40,000 analyses were done for 17 agencies, which submitted a wide variety of drugs. BNDD welcomes these reports, and they are read with interest by both laboratory and enforcement personnel.

California Toxicology Service, Inc., 2333 W. 3rd Street, Los Angeles, California 90057, should be added to the list of drug reference standard sources. Standards are reportedly available in 1, 2, and 3 milligram quantities in 6 milliliter screw cap vials. The purchaser adds ethanol or other solvent to get desired concentration.

The listing of these firms is for information only, of course, and does not imply any endorsement by the federal government.

Louisiana Association of Forensic Science had its third meeting on July 17 in Baton Rouge. The new organization, consisting of forensic scientists in the state, plans about four meetings a year.

Strychnine has been rumored, in recent months, to be in drugs, in some cases resulting in hospitalizations. The material from these episodes

analyzed by BNDD have not shown strychnine. Strychnine was also alleged to be in drugs at the May Day demonstration in Washington, D.C., but none was found in material submitted to the BNDD Washington Regional Laboratory. As usual, all of the suspected mescaline was either LSD or LSD-PCP, and suspected THC was found to be either PCP or LSD.

Secobarbital is known as "Dry Liquor" according to a June 21 press report from Thailand. Dr. Komol Pengsrihong, Under-Secretary of Public Health, has warned of the dangers of taking the drug without supervision by a physician. Dr. Komol told the press that anyone selling the drug without a prescription would be prosecuted.

MEETINGS

Midwestern Forensic Analysts' Seminar, October 7-9, 1971, Chicago. Drug identification problems, firearms residue determination, 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.

Association of Official Analytical Chemists, October 11-14, 1971, Marriott Motor Hotel, Twin Bridges, Washington, D.C. On Thursday, October 14, a symposium on the forensic sciences will be held, with the keynote address: "The Prosecutor and Forensic Scientist." About 35 firms will exhibit laboratory equipment and supplies. Registration fee: \$3.00.

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 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.

SELECTED REFERENCES

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Gelehrter, Thomas D., M.D., "Lysergic Acid Diethylamide (LSD) and Ex-strophy of the Bladder," The Journal of Pediatrics, Vol. 77, No. 6 (December, 1970).

"Symposium on Analytical Methods for Forensic Science," Journal of the Association of Official Analytical Chemists, Vol. 54, No. 4 (July, 1971).

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SEPARATION OF MIXTURES OF PROCAINE, COCAINE,
AND PHENCYCLIDINE USING GAS CHROMATOGRAPHY

BY: Robert M. Dews*

Because of the large number of samples received by this laboratory containing mixtures of procaine and cocaine, and procaine and phencyclidine (PCP), a rapid method of detecting the presence of these compounds was needed. Spot tests and UV methods were unacceptable for mixtures of these compounds because of the similarity of the reaction of cobalt thiocyanate with procaine and cocaine, and the fact that the UV spectra of mixtures of procaine and cocaine is inconclusive for the presence of either one, and the absorptivity ($E: \frac{1\%}{1\text{cm}}$) value of PCP is so low that it would be missed entirely in a mixture with procaine or cocaine. Using the described method of GC separation, PCP, procaine, and cocaine can be clearly separated.

APPARATUS

Beckman GC-45 gas chromatograph with flame ionization detector with 6 ft x 2 mm glass column packed with 3% OV-17 on Chromsorb WHP, 100-120 mesh.

1 microliter Hamilton syringe

Separatory funnels, 60 ml

Vacuum oven

Evaporating dishes

REAGENTS

0.1 N Sodium Hydroxide

Chloroform (Baker Instra analyzed grade)

Methanol (Baker Instra analyzed grade)

Standard solutions of PCP, procaine, and cocaine in methanol, approximately 1 milligram per milliliter.

GC PARAMETERS

Column Temperature: 230^o C.

Inlet Temperature: 250^o C.

Detector Temperature: 280^o C.

Chart Speed: 0.5 in/min

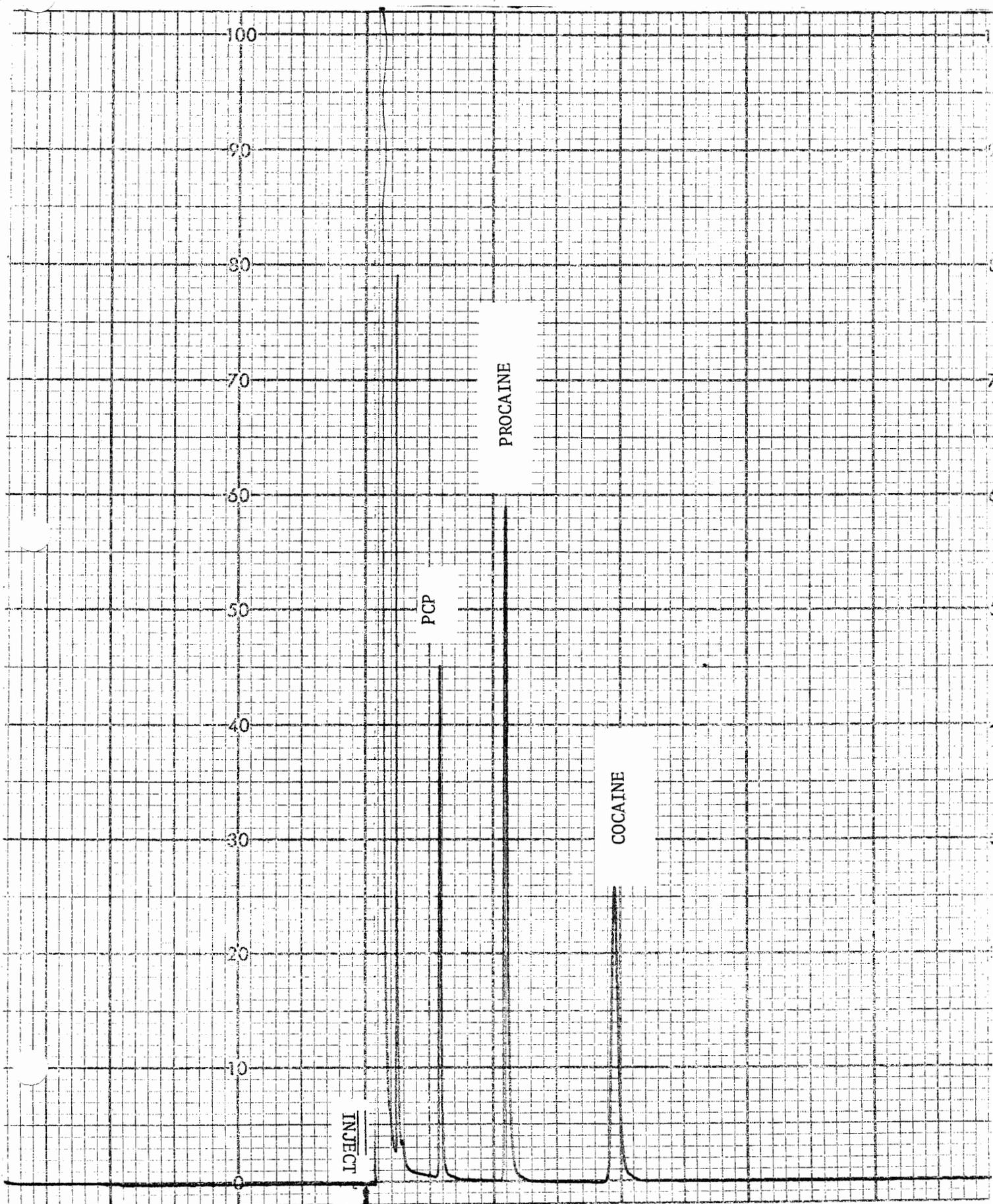
Flame ionization detector is used with Hydrogen flow rate of 40 cc/min and an Air flow rate of 285 cc/min.

Helium is used as the carrier gas with a flow rate of 30 cc/min at a head pressure of 40 PSIG.

PROCEDURE

A sample of the suspected powder is dissolved in approximately 10 ml of 0.1 N NaOH to give an approximate concentration of 1 mg/ml. This NaOH solution is then extracted with approximately 25 ml CHCl₃. After completely separating, the CHCl₃ layer is transferred to an evaporating dish and taken to dryness in a vacuum oven at 30^o C. The residue obtained after taking to complete dryness is then taken up in approximately 10 ml of methanol. This methanol now contains, as the free base, any PCP, procaine, or cocaine that may have been present in the original sample. A 0.5 microliter sample of this solution is then injected on the GC under the parameters given. The resulting chromatogram is then compared with that of a solution containing the three compounds.

* Robert M. Dews, Criminalist
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Washington State Patrol



BNDD LABORATORY NOTES



DATE April 26, 1971
NO. 15
DRUG TYPE
METHODOLOGY

SEPARATION OF LSD AND PCP BY MICRO ALUMINA COLUMN CHROMATOGRAPHY

by:

Buddy Goldston
Michael D. Miller
Dallas Regional Laboratory
Bureau of Narcotics & Dangerous Drugs

In recent months Dallas Regional Laboratory has received for analysis tablets containing LSD in combination with Phencyclidine HCl. In a continuing demonstration of the versatility of John D. Wittwer's micro alumina column technique (1), we will describe a procedure that incorporates alumina column chromatography to separate LSD and PCP in pure form for instrumental modes of analysis.

APPARATUS

1. Disposable pipet having an I.D. of 5mm and a total length of about 15 cm.
2. Portable U.V. light.

REAGENTS

1. Spectrograde chloroform that is alcohol free.
2. Spectrograde chloroform that contains 3/4% Ethanol as a preservative.
3. Aluminum Oxide - chromatographic (Merck or Alcoa).

PROCEDURE

A representative portion of sample powder is weighed and transferred to a dry 50 ml beaker. Add 2 ml of 0.1N HCl followed by gentle warming on a steam bath for no more than 1 minute. Cool and make the residue slightly alkaline with sodium bicarbonate and add 3 grams of Celite 545. Prepare a chromatographic column and elute the LSD and PCP with 75 ml water-washed ether into a 125 ml separatory funnel containing 10 ml 8% citric acid. PCP is water insoluble in ether and will extract with the LSD into

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the citric acid layer upon shaking the funnel. Repeat the citric acid extraction combining both fractions in a second separatory funnel. Color additives will normally remain in the ether layer but in the event some color does transfer it may be necessary to wash the citric acid extract with a 1-10 ml portion of CHCl_3 . PCP will not extract into the CHCl_3 layer under these conditions. The citric acid extract is then made alkaline with sodium bicarbonate and extracted with 2-10 ml portions of CHCl_3 .

The combined CHCl_3 extracts are gently brought to dryness on a steam bath. The cooled residue is dissolved in 1 ml of alcohol free spectrograde CHCl_3 and applied to the micro-alumina column previously described by Mr. Wittwer in Microgram. The column is then washed with an additional 2 ml of alcohol free spectrograde CHCl_3 being careful to collect all portions of CHCl_3 eluting from the micro-column. The LSD, when viewed under UV light, will remain as a stationary blue fluorescent band at the top of the column during the washes with alcohol free spectrograde CHCl_3 . During the washes with spectrograde CHCl_3 colors not removed in the extraction procedure will also be retained by the alumina. Following the 2 ml portion of spectrograde CHCl_3 , begin elution of the LSD with spectrograde CHCl_3 containing approximately 3/4% Ethanol. The progress of the LSD band down the column may be monitored with a portable UV lamp. The portion of eluate collected before the LSD band will be designated Fraction I and will contain the PCP as the free base in good analytical yield. We have found that 3/4% Ethanolic spectrograde CHCl_3 as a second chromatographic solvent system, in addition to initiating the elution of LSD, will also insure complete recovery of PCP from the column. Fraction II or the portion of eluate collected during the elution of the blue fluorescent band will contain LSD in a state sufficiently pure for ultra-violet, infrared, and polarimetric analysis. Fraction III or all eluate collected after the elution of the blue band will normally contain Iso-LSD, but occasionally it is found in Fraction II. In this situation it may be necessary to re-chromatograph Fraction II by the method of Pioch (2) if Iso-LSD is present to the extent that it distorts the infrared spectra of the normal form.

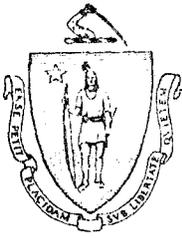
Compactness of the micro-column, activity of the alumina, length of the column, and Ethanol content of the CHCl_3 are all factors pertinent to the chromatographic separation of LSD from Iso-LSD on this column.

RESULTS AND DISCUSSION

The application of alumina chromatography is by no means restricted to the specific separation of LSD and PCP. This methodology can be extended to a variety of other nitrogenous bases where separation from LSD is desired.

REFERENCES

- (1) MICROGRAM, Vol. 2, No. 2, page 49, June 1969.
- (2) Pioch, R.P., Chemical Abstracts, Vol. 50, 1956, page 10,803.



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DRUG PATTERNS AS TO SAMPLES SUBMITTED BY LOCAL POLICE DEPARTMENTS

	<u>1967-1968</u>	<u>1968-1969</u>	<u>1969-1970</u>	<u>1970-1971</u>
GREEN HERB				
Cannabis	4,552	5,245	6,470	9,945
BROWN MATERIAL				
Cannabis	81	353	949	1,004
POWDER				
Heroin	1,151	5,235	9,832	12,138
Cocaine	152	394	377	539
LSD	4	8	8	26
Amphetamine (speed)	429	435	464	436
Barbiturate	13	38	10	11
RESIDUE				
Opium Derivative	148	682	557	993
Cannabis	610	1,390	1,994	3,459
Heroin	172	60	316	1,495
Amphetamine	181	233	199	106
TABLETS				
LSD	34	289	543	446
Dolophine	19	56	180	226
Amphetamine	178	249	251	350
Barbiturate	97	161	169	307
Amphetamine & Barbiturate	69	121	224	316
Doriden	28	47	49	79
CAPSULES				
LSD	32	72	31	65
Amphetamine	160	245	217	336
Barbiturate	214	370	451	900
Amphetamine & Barbiturate	28	72	69	95