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
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 vendor 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 hospitalization. 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 Pongsrithong, 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


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 ($E_{1\%}$) 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°C.

Inlet Temperature: 250°C.

Detector Temperature: 280°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°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
  Drug Control Assistance Unit
  Seattle Regional Laboratory
  Washington State Patrol
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 chroma­
tography 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.

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
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 CHCl₃. PCP will not extract into the CHCl₃ layer under these conditions. The citric acid extract is then made alkaline with sodium bicarbonate and extracted with 2-10 ml portions of CHCl₃.

The combined CHCl₃ extracts are gently brought to dryness on a steam bath. The cooled residue is dissolved in 1 ml of alcohol free spectrograde CHCl₃ 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 CHCl₃ being careful to collect all portions of CHCl₃ 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 CHCl₃. During the washes with spectrograde CHCl₃ colors not removed in the extraction procedure will also be retained by the alumina. Following the 2 ml portion of spectrograde CHCl₃, begin elution of the LSD with spectrograde CHCl₃ 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 CHCl₃ 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 CHCl₃ 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

**DRUG PATTERNS AS TO SAMPLES SUBMITTED BY LOCAL POLICE DEPARTMENTS**

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