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. 11 December, 1971

Seasons Greetings

The Chicago Regional Laboratory has recently received an influx of exhibits containing LSD adsorbed on ion exchange resin beads. While these exhibits will give a positive p-Dimethyl aminobenzaldehyde test, simple solvent extractions will not release the LSD for thin layer chromatography, yielding negative results. Following is a description of dosage forms found to date, characteristics of the beads, and method of extraction for TLC.

Dosage Forms:

1. Maroon, round, flat with domed center, tablets 4.7mm x 2.6mm to 2.8mm thick. (Also contains PCP)

2. Yellow, round, flat tablets, 4.8mm x 3.1mm thick.

3. Brown, round, flat, tablets, 6.5mm x 3.2mm thick.

4. Orange, round, biconvex, tablets, 6.4mm x 5.3mm thick.

5. Clear, colorless, No. 2, capsules of brown powder (cocoa).

6. Orange-brown powder.

Areas Found:

Detroit, Cincinnati, Peoria, Rock Island, Davenport, and Milwaukee.

Street Names:

"Orange-Tang-Mesc"and"Chocolate-Mesc"

Ion Exchange Beads:

Deep brown-red spheres, about 0.06mm to 0.1mm diameter (about 150 to 250 mesh). Infra-red spectra is similar to that of Mallinckrodt Amberlite Ion Exchange Resin IR-120 H. C. P. H+ form, a sulfonated polystyrene. Beads are easily observed with a wide-field microscope. All samples should be routinely screened for these beads.

Extraction

A finely ground portion is mixed with about 100 mg of $NaHCO_3$ and about 2ml water. Heat on steam bath for 10 minutes and add Celite 545 to obtain a fluffy mixture. Place this mixture above a mixture of Celite 545 and about 2ml 2% citric acid, in a 25 x 250mm chromatographic tube. Elute with about 140ml $CHCL_3$ (water saturated). The eluate may be evaporated to a small volume for TLC or to dryness and taken up in MeOH for Fluorescent quantitation.

Because of the minimal number of tablets available for testing, all of the parameters have not been optimized, but preliminary recoveries appear to be good.

Phencyclidine in combination with chocolate powder is being found in Florida. This mixture is being found in red capsules.

Lidocaine being sold as cocaine is still being encountered. Lactose now appears to be the most common diluent in this mixture.

Heroin HCl 31.7% and caffeine 68.3% are being found in combination on the East Coast. This mixture occurs as a cream colored powder.

Cocaine and methamphetamine mixtures have been received by the New Jersey State Police Laboratory. The material occurs as a brown powder packaged in aluminum foil.

The Northwest Indiana Criminal Toxicology Laboratory reports that the following compounds and mixtures are being encountered in their area.

Phencyclidine in unmarked pink capsules being sold as THC. These capsules resemble those also used in the same area for heroin.

LSD as a light tan flecked powder in foil packets and plastic bags, being sold as mescaline.

Caffeine, cocaine and heroin in pink capsules, being sold as heroin.

An Interesting Encounter:

The New York BNDD Regional Laboratory recently encountered three exhibits purported to be pure heroin. However, upon examination, the exhibits were found to contain both heroin and acetyl codeine in the following proportions:

81.8%	heroin	12%	acetyl codeine
87%	heroin	11.2%	acetyl codeine
67%	heroin	4.4%	acetyl codeine

We feel that this particular combination is most likely attributable to an error or to a flaw in the manufacturing process.

If you encounter any samples having the above proportions, please let us know.

MEETINGS

1972 INTERNATIONAL MEETING SITE CHANGED TO EDINBURGH, SCOTLAND Due to the persisting civil disturbance in Northern Ireland, Dr. Thomas K. Marshall, President of the Sixth International Meeting of Forensic Sciences, has announced a move of the meeting site from Belfast to Edinburgh, Scotland. The meeting dates remain the same -- September 21-26, 1972. For further information, write to:

The Secretariat
Sixth International Meeting of Forensic Sciences
Institute of Pathology
Grosvenor Road
Belfast, BT 12 6BL
Northern Ireland

BNDD Forensic Chemists Seminars for the coming fiscal year are planned as follows:

January 31 - February 4, 1972
April 3 - 7, 1972
June 12 - 16, 1972

All sessions will be held at the BNDD National Training Institute, Washington, D.C. For more information and application forms, write to:

Assistant Director for Training
National Training Institute
Police Training Division
Bureau of Narcotics and Dangerous Drugs
1405 Eye Street, N.W.
Washington, D.C. 20537

Annual Meeting of the American Academy of Forensic Sciences, Atlanta, Georgia, March 1-4, 1972. Contact:

Secretary James Weston, M.D.
44 Medical Drive
Salt Lake City, Utah 84113

Or

General Program Chairman
Michael M. Baden, M.D.
Office of the Chief Medical Examiner
520 First Avenue
New York, New York 10016
Telephone: (212) 684-1600

California Association of Criminalists, Semi-annual seminar, May 18-20, 1972. Pierpont Inn, Ventura, California. For further information, contact:

Forrest Letterly
Ventura County Sheriff's Office
501 Poli Street
Ventura, California 93001

BNDD LABORATORY NOTES

DATE 11-19-71 -147-

NO. 24

DRUG TYPE Narcotic

METHODOLOGY TLC

SEPARATION OF HEROIN AND ACETYLCODEINE IN ANALYSIS OF NARCOTICS PARAPHERNALIA

BY

PETER DELMAR
WASHINGTON REGIONAL LABORATORY
BUREAU OF NARCOTICS AND DANGEROUS DRUGS

PROBLEM

The Washington Regional Laboratory has recognized for sometime that the commonly used TLC systems such as those listed in IRS publication 341 and the "T" series mentioned in Clarke's Isolation and Identification of Drugs cannot distinquish between heroin and acetylcodeine. In the case of paraphernalia where the small amount of material present precludes identification by color or microcrystalline tests, GLC has been used. This has the double disadvantage of being more time consuming and increasing the demand on instrument time.

Nakamura[1] has published a method utilizing the "A2" solvent system reported by Cochin and Daly[2]. This method requires 20x20cm plates and is rather time consuming. In the Washington Laboratory, where a great amount of our work involves narcotics and narcotics paraphernalia, it was felt that a faster system using the smaller 10cm plates would be more valuable. With this in mind, a system was developed which can rapidly separate heroin and acetylcodeine.

APPARATUS

1. TLC Plates - Pre-scored 10cm plates coated with a 250 micron layer of Silica Gel GF (available from Analtech Inc. of Newark, Delaware) The plates do not require activation before use.
2. Any standard TLC tank can be used.

REAGENTS

1. Solvent Systems - (A) N-butyl ether, diethyl ether and diethylamine 45:45:10. The solvent can be used immediately after mixing.

(B) Ammonia saturated chloroform, methanol (18:1)

2. Iodoplatinate spray as prepared in IRS, Publication #341.

METHOD

After allowing the solvent tank to equilibrate for 5 minutes, the plate is developed for approximately 10 min using solvent system (A). The plate is dried briefly under an air stream to remove the bulk of the butyl ether. 10 minutes in the oven at 100° completes the drying process.

RESULTS AND DISCUSSION

The following typical RF's can be obtained:

Cocaine	.63-
Acetylcodeine	.46
Heroin	.39
Quinidine	.29
Monoacetyl morphine	.27
Quinine	.20-
Morphine	.05-

As can be seen, solvent system (A) can also separate quinine and quinidine. Should the sample contain quinine, monoacetyl morphine and quinidine, identification becomes difficult. In this case, solvent system (B) can easily separate monoacetyl morphine and quinidine.

These systems have been in use on routine samples for 2 months. The majority of heroin samples analyzed have been found to contain trace amounts of acetylcodeine and in one sample, an amount sufficient to distort quantitation by U.V."Cookers"containing quinine, monoacetylmorphine, heroin, acetylcodeine and cocaine have been successfully chromatographed.

REFERENCES

1. Nakamura, G.R., J.A.O.A.C., Vol. 49, No 5, Pg. 108, 1966.

2. Cochin, J., and Daly, J.W., Experientia, 18, Pg. 294-295, 1962.

BNDD LABORATORY NOTES

DATE 11-29-71
NO. 25
DRUG TYPE Stimulant
METHODOLOGY

QUANTITATION OF AMPHETAMINE IN RESIN COMPLEXES

Jeffrey M. Weber, Forensic Chemist
New York Regional Laboratory

Abstract

On several occasions this laboratory has received samples of capsules containing amphetamine type compounds in resin complexes. Difficulties have been encountered in quantitating such formulations. The following extraction procedure is a modification of the usual double extraction procedure and yields a clean extract of amphetamine suitable for qualitative and quantitative analysis.

Method

Apparatus -

Mechanical shaker
UV Spectrophotometer

Reagents -

1N HCl, Conc. HCl
Ethyl ether (anhydrous)
50% NaOH, 1N NaOH
0.1N H_2SO_4
$CHCl_3$ (reagent grade)

Procedure

Combine the contents of a representative number of capsules, and grind and mix to form a composite. Transfer a weighed portion of sample powder into a 125 ml. separatory funnel containing 50 mls. 1N HCl. Place separatory funnel on mechanical shaker (do not use an ultrasonic bath) for 1 hour. Extract with 10 mls. ether and quantitatively elute the aqueous layer into a second 125 ml. separatory funnel. Make basic with 50% NaOH. Extract with 3 x 10 ml. portions $CHCl_3$. Add 1 drop concentrated HCl to combined extracts and evaporate to dryness. Quantitatively transfer the residue to a volumetric flask using 0.1N H_2SO_4. Read the absorbance of the solution at 257 mμ and compare to standard amphetamine.

In the case of Biphetamine T the resin also contains Methaqualone. The sample powder is extracted as above. The residue (which is a combination of Methaqualone and Amphetamine HCl) is transferred quantitatively with the aid of 25 mls. 0.1N H_2SO_4 into a separatory funnel. The methaqualone is extracted from the acid solution with 3 x 10 mls. portions of $CHCl_3$. The acid is then made basic with 1N NaOH and the amphetamine is extracted with 3 x 10 mls. portions of $CHCl_3$. A drop of conc. HCl is added to each extract and both evaporated to dryness. The amphetamine is dissolved in a definite volume of 0.1N H_2SO_4 and the absorbance of the solution measured at 257 mμ on a UV spectrophotometer. The methaqualone is also dissolved in 0.1N H_2SO_4 and the absorbance measured at 234 mμ.

The final acidic solutions containing amphetamine and methaqualone can be re-extracted to obtain sufficient quantity of each drug for identification by IR or TLC.

Results

Recoveries of 95% - 105% have been obtained using this method on such preparations as Biphetamine and Biphetamine T (Strassenburgh).

FEDERAL REGISTER
Area Code 202 Phone 962-8626

Title 21—FOOD AND DRUGS

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

PART 308—SCHEDULES OF CONTROLLED SUBSTANCES

Removal of Exceptions From Amphetamine and Methamphetamine Combination Products

A notice was published in the FEDERAL REGISTER of September 21, 1971 (36 F.R. 17849), proposing the removal of exceptions from amphetamine and methamphetamine combination products. These exceptions, from application of certain provisions of the Controlled Substances Act (21 U.S.C. 801 et seq.) and the Controlled Substances Import and Export Act (21 U.S.C. 951 et seq.), were granted under the Drug Abuse Control Amendments of 1965 and were continued under the Comprehensive Drug Abuse Prevention and Control Act of 1970 (Public Law 91-513) for administrative purposes.

All interested persons were given 30 days after publication to submit their objections to and comments on the proposal. No objections were received. The Narcotic and Drug Control Division of the State Board of Health of South Carolina, commented specifically on the product Edrisal, noting that the formula of the drug does not preclude a potential for abuse, that similar drugs (Edrisal with Codeine and Daprisal) are listed in Schedule II, and that as the stringent controls of Schedule II are applied to other stimulants, abuse of Edrisal is likely to increase.

After careful consideration of the comments submitted, in view of the fact that no objections were received, and based upon the investigations of the Bureau of Narcotics and Dangerous Drugs, the Director finds that no compound, mixture, or preparation containing any quantity of amphetamine (or its salts, optical isomers, or salts of its optical isomers) or methamphetamine (or its salts, isomers, or salts of its isomers) and one or more active medicinal ingredients not having a stimulant effect on the central nervous system, contains such ingredients in such combinations, quantity, proportion, or concentration as to vitiate the potential for abuse of the amphetamine or methamphetamine substances.

Therefore, under the authority vested in the Attorney General by section 202 (d) of the Comprehensive Drug Abuse Prevention and Control Act of 1970 (21 U.S.C. 812(d)), and redelegated to the Director, Bureau of Narcotics and Dangerous Drugs by § 0.100 of Title 28 of the Code of Federal Regulations, the Director hereby orders that:

1. Section 308.13(b)(1) of Title 21 of the Code of Federal Regulations be deleted and replaced with a new paragraph to read:

§ 308.13 Schedule III.

* * * * *

(b) * * *

(1) Those compounds, mixtures, or preparations in dosage unit form containing any stimulant substances which compounds, mixtures, or preparations were listed on August 25, 1971, as excepted compounds under § 308.32, and any other drug of the quantitative composition shown in that list for those drugs or which is the same except that it contains a lesser quantity of controlled substances.

* * * * *

§ 308.32 [Amended]

2. Section 308.32(b) of Title 21 of the Code of Federal Regulations be amended by deleting the following drugs:

Trade name or other designation	Composition	Manufacturer or supplier
Edrisal	Tablet: Amphetamine sulfate 2.5 mg.; asprin, 162 mg.; phenacetin 162mg.	Smith Kline & French Laboratories.
* * *		
Genegesic Capsules	Capsule: Methamphetamine hydrochloride, 1.2 mg.; chlorpheniramine maleate, 3.8 mg.; phenacetin, 120.0 mg.; salicylamide, 180.0 mg.; caffeine, 30.0 mg.; ascorbic acid, 50.0 mg.	General Pharmaceutical Products Inc.
* * *		
Hovizyme	Tablet: Methamphetamine hydrochloride, 0.5 mg.; conjugated estrogens-equine, 0.125 mg.; methyl testosterone, 1.25 mg.; amylase, 10.0 mg.; protease, 5.0 mg.; cellulase, 2.0 mg.; nicotinyl alcohol tartrate, 7.5 mg.; dehydrocholic acid, 50.0 mg.; ascorbic acid, 50.0 mg.; ferrous fumarate, 6.0 mg.	Ayerst Laboratories.
* * *		
Mediatric	Tablet of capsule: Methamphetamine hydrochloride, 1 mg.; conjugated estrogens-equine, 0.25 mg.; methyl testosterone, 2.5 mg.	Do.
Mediatric Liquid	Solution (15 cc.): Methamphetamine hydrochloride, 1 mg.; conjugated estrogens-equine, 0.25 mg.; methyl testosterone, 2.5 mg.	Do.
* * *		
Special Formula 711	Tablet: d-Amphetamine sulfate, 2.5mg.; mephenesin, 500 mg.; salicylamine, 300 mg.	Detroit First Aid Co.
* * *		
Thora-Dex No. 1	Tablet: Dextroamphetamine sulfate, 2 mg., chlorpromazine hydrochloride, 10 mg.	Smith Kline & French Laboratories.
Thora-Dex No. 2	Tablet: Dextroamphetamine sulfate, 5 mg., chlorpromazine hydrochloride, 25 mg.	Do.
* * *		

In his order published July 7, 1971, in the FEDERAL REGISTER (36 F.R. 12736), the Director recognized that certain combination products containing amphetamine or methamphetamine exempted under the Drug Abuse Control Amendments of 1965 were not expressly excepted under § 308.32. The Director stated that, as a matter of policy, those substances would be deemed excepted under § 308.32 pending further action by the Bureau. This order applies to those substances as well.

The effect of this order is to subject all compounds, mixtures, or preparations containing amphetamine or methamphetamine, except those now listed in Schedule II, to all of the requirements of sections 305, 307, and 309 of the Controlled Substances Act (relating to labeling, recordkeeping, and prescription requirements for controlled substances), sections 1002, 1003, and 1004 of the Controlled Substances Import and Export Act (relating to importation, exportation, transshipment) and § 301.74(d) of Title 21 of the Code of Federal Regulations (relating to sampling of controlled substances).

This order does not transfer any formerly excepted compounds, mixtures, or preparations containing amphetamine or methamphetamine from Schedule III to Schedule II.

The requirements imposed upon the formerly excepted amphetamine and methamphetamine combination products by virtue of the removal of the excepted status shall become effective as follows:

1. *Labeling.* All labels on commercial containers of, and all labeling of, the above formerly excepted stimulant compounds, which are packaged on and after May 1, 1972, shall comply with the requirements of 21 CFR Part 302.

2. *Records and inventories.* Every registrant who is required to keep records under § 304.03 of Title 21 of the Code of Federal Regulations, and who is manufacturing, distributing or dispensing any of the above formerly excepted stimulant compounds, shall take an inventory of all stocks on hand on January 3, 1972, and thereafter shall keep all required records regarding these compounds.

3. *Prescriptions.* All prescriptions for the above formerly excepted stimulant compounds shall comply with 21 CFR Part 306, as applied to substances listed in Schedule III, on and after January 1, 1972.

4. *Importation and exportation.* All importation, exportation, transshipment, and in-transit shipment of the above formerly excepted stimulant compounds shall comply with the requirements of 21 CFR Part 312 on and after January 1, 1972.

5. *Security.* All of the above formerly excepted stimulant compounds shall be manufactured, stored, distributed and shipped in compliance with §§ 301.71–76 of Title 21 of the Code of Federal Regulations on and after January 1, 1972.

This order is effective on the date of its publication in the FEDERAL REGISTER (11–6–71).

Dated: November 1, 1971.

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

[FR Doc.71–16208 Filed 11–5–71;8:45 am]

FEDERAL REGISTER
Area Code 202 Phone 962-8626

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

PART 308—SCHEDULES OF CONTROLLED SUBSTANCES

Phenmetrazine and Its Salts and Methylphenidate; Amphetamine and Methamphetamine Combination Products

A final order was published in the FEDERAL REGISTER of October 28, 1971 (36 F.R. 20686) rescheduling phenmetrazine and its salts and methylphenidate from schedule III to schedule II. Paragraph 3 of that order should read as follows: "3. Section 308.13(b) or Title 21 of the Code of Federal Regulations be revised to read:"

A final order was published in the FEDERAL REGISTER of November 6, 1971 (36 F.R. 21336) removing from the excepted category amphetamine and methamphetamine combination products. The requirements imposed by this order as they pertain to records and inventories and security shall become effective as follows, rather than on the dates shown in the order of November 6, 1971 (36 F.R. 21336).

2. *Records and inventories.* Every registrant who is required to keep records under § 304.03 of Title 21 of the Code of Federal Regulations, and who is manufacturing, distributing, or dispensing any of the above formerly excepted stimulant compounds, shall take an inventory of all stocks on hand on January 1, 1972, and thereafter shall keep all required records regarding these compounds.

5. *Security.* All of the above formerly excepted stimulant compounds shall be manufactured, stored, distributed and shipped in compliance with §§ 301.71–76, of Title 21 of the Code of Federal Regulations on and after March 1, 1972.

These changes are effective on the date of their publication in the FEDERAL REGISTER (11–20–71).

Dated: November 16, 1971.

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

[FR Doc.71–17010 Filed 11–19–71;8:51 am]