



... MICRO-GRAM ...

Volume I, No. 5

February, 1968

CRIME LAB CHEMIST'S SEMINAR NO. 2

Held February 5-9 in Washington, D.C., sixteen analysts attended lectures and laboratory workshops on analytical methods of hallucinogenic, stimulant and depressant drugs. Lab sessions included use of chromatography, infrared and ultra-violet spectroscopy, crystalline tests and extraction processes.

The class had two guest lectures. Dr. Vincent Guinn, Gulf General Atomic talked on "Application of Activation Analysis to Criminalists". Dr. Leo R. Goldbaum, Toxicologist, AFIP, Washington, d.c., discussed the detection of drugs in biological materials. Both talks were well received by the students and visitors.

DRUG USE, BOSTON AREA

Louis Bornstein, Senior Analyst, Division of Food and Drugs, Department of Public Health, Commonwealth of Massachusetts, writes that LSD has been found on chewing gum and chocolate filled candies in the Boston area and some LSD has been in the form of blue and purple tablets.

Cannabis and DMT are being found to some degree.

Numerous amounts of white, double scored tablets containing amphetamines are being found.

AMPHETAMINE TABLETS

BDAC has been encountering quite a few amphetamines also. Most of these have been 10 mg dl-amphetamine sulfate, double-scored tablets. About half have been identified as from the same source, probably a single punch tableting machine. Several others are identical to an authentic sample we have from a U.S. firm. If you wish a ballistics exam of your tablets, send them in (preferably about 20 tablets).

CAUTION: Use of this publication should be restricted to forensic analysts or others having a legitimate need for this material.

HOW TO REQUEST A LABORATORY EXAMINATION

1. Make all requests by letter in duplicate to:

U.S. Food and Drug Administration
Bureau of Drug Abuse Control
Division of Investigations -330
200 "C" Street, S.W.
Washington, D.C. 20204

2. Send evidence by registered mail or REA Express, "Special Handling".
3. Furnish names of subjects, if known, and your case number.
4. Give the nature of the violation.
5. List the evidence
6. State type of examinations desired.
7. Give the basic facts of the case insofar as they pertain to the laboratory examination. Indicate what type of drug is suspected.
8. Reference previous correspondence, if any.
9. Include a statement indicating that the evidence has not been and will not be examined by another expert in the same scientific field.
10. State the disposition of the evidence. If no disposition is given, the remaining portion will be sent back to the contributor by registered mail.

CLANDESTINE DRUG MANUFACTURING

As of January 1, 1968, BDAC Agents had broken up 42 clandestine drug manufacturing operations. Thirty-six of these were making hallucinogenic drugs. The production potential of these facilities staggers the imagination. Add this to our accountability program, and you can see that we have not only eliminated a lot of "rotten barns," but have fixed many others. This makes a lot more efficient use of money and manpower, than chasing the horses after they get out on the street.

DON'T MISS A THING

One of our Field Offices recently sent us a small Parr tablet press that they had picked up in a hippie joint. The press looked clean, but we sent it to the laboratory just to be sure. Results: Constarch, sucrose and marihuana.

CARBONA SNIFFING

We have had reports of children sniffing Carbona, a rug and upholstery cleaning fluid. This product is reportedly almost straight carbon tetrachloride, well known for its toxic effects and deaths.

In fact, because of the degree and nature of the hazard involved in the presence or use of carbon tetrachloride in the household, the Commissioner has proposed that it be considered a banned hazardous substance under the Federal Hazardous Substance Act. This would affect the interstate movement of carbon tetrachloride and mixtures containing it (including that used in fire extinguishers). This proposal appears in the Federal Register for February 16, 1968.

CUPCAKES

BDAC Agents have purchased LSD tablets known as "Cupcakes" or "wedges".

A tablet purchased in the Northwest was uncoated; orange outside; pink inside; 4.2 mm diameter; 2.6 mm thick; and contained 163 mcgs. of LSD. It appeared to have been made on a tablet triturate device.

Such devices were used by pharmacists to make tablets prior to modern pharmacy. Hypodermic tablets were usually made in this manner.

Recently such a device was seized, which was used to make a truncated, cone-shaped LSD tablet. The device consisted of a board with a series of holes and another board with a set of pegs corresponding to the holes in the first board. The board with the holes is set on a flat, smooth surface and the holes are filled with a moist drug mixture. When the drug is dry, the board is pushed down over the board of pegs, pushing out the tablets. Tablets made on this device had the largest diameter about 5.2 to 5.7 mm; the smaller diameter about 5.0 to 5.1 mm; and weighed about 70.6 mg. They contained about 184 mcgs. of LSD per tablet.

RITALIN (METHYLPHENIDATE), CIBA

Our Seattle Agent reports widespread use of the drug, particularly by heroin users.

Recently the Microanalytical Laboratory examined a charred mass from a furnace sent to us by a Connecticut Police Department. Along with parts of hypo needles, the mass showed burned paper from apparent sample packets of Cogentin and Ritalin.

QUEEN ANNE'S LACE

We have had reports that teenagers are smoking the leaves of Daucus carota (Wild Carrot). This is widely distributed, and the domestic carrot is the same plant altered by cultivation. A pharmacognosy book states that the ripe fruits are diuretic, stimulant and a menstrual excitant. The 1960 U.S. Dispensatory references a paper reporting extract of wild carrot effective in causing contraction of the uterus. (Merck's Jahresber., 1936, 50, 102). The Dispensatory also states that two liquid bases have been made from the carrot leaves: pyrrolidine, [CH₂-4NH] and daucine, C₁₁H₁₈N₂.

EUROPEAN DRUGS SUBJECT TO ABUSE

Joseph Koles, in FDA's Bureau of Science, formerly was director of a Defense Department crime laboratory in Europe. He has sent us a list of drugs based on laboratory cases in Europe, which were apparently subject to abuse. This abuse was often connected with the consumption of alcohol.

Romilar tablets - Dextromethorphan HBr (Available in U.S.)

Captagon Tablets - 50 mg. (Chemiewerk Homberg) 7-[2-(1"-Methyl-2"-Phenyl-ethylamino)-ethyl]-theophyllin HCl (No U.S. equivalent product)

Kinortine Tablets - (French) Dextroamphetamine Monotartrate and Caffeine

Nubarene (French) - 3-(o-chlorophenyl)-2-methyl-4(3H)-quinazolinone (Mecloqualone-U.S.)

Tablets containing caffeine and ephedrine

METHYLENEDIOXY AMPHETAMINE (MDA)

In October 1967, we received a capsule, pink at one end and clear at the other, filled with 150 mg. of a white crystalline substance.

Dr. Albert R. Sperling, Bureau of Science, analyzed the substance and identified it as 3,4 methylenedioxyamphetamine. This was our first encounter with the compound. Copies of Dr. Sperling's spectra of the free base and the sulfate are attached.

Over the past months, BDAC Agents and police have purchased the powder and capsules in the New York area sold as mescaline. A clandestine "laboratory" was seized in New York, with 3-1/4 pounds of MDA and 25 pounds of 1,2 methylenedioxy-4-(2-nitropropenyl) benzene, a precursor one step removed from MDA.

Attached is determination and identification data on MDA evidence analyzed by Mr. Richard Fox, an analyst in FDA's New York District.

An infrared spectrogram is also shown in STADTLER STANDARD SPECTRA, Midget Edition, Spectrum No. 20160.

TETRYL WARNING

We are checking out a report that "hippies" in the Minneapolis area have formed an organization for protection against law enforcement officers.

The group, called "The Blue Hand", is reportedly making beads from "Tetrol", which they string on a dynamite fuse.

We believe this may be "Tetryl", also known as Tetralite, Trinitrophenyl-methylnitramine, and Nitramine. Tetryl is said to be more sensitive to shock and friction than TNT, and the explosive hazard is severe, when shocked or exposed to heat or flame. It is commonly used as a booster charge for high explosive shells.

"Tetryl", (N-Methyl-N-2,4,6-Tetranitroaniline) occurs as yellow monoclinic crystals; molecular weight 287.15; density 1.57 at 19°C.

Melting Point 130°C.
Boiling Point 187°C (explodes)
Solubility:
 Water: Insoluble
 Alcohol: 0.422 at 18°C.
 Ether: Very
 Benzene: Soluble
 Acetic Acid: Soluble

We are obtaining analytical data. If you obtain any suspect material, contact us by wire for any needed assistance.

Tetryl can be compressed into pellets or beads. Handle suspect material with caution. Do not mistake for pills or love beads.

LABORATORY INFORMATION BULLETIN

Bureau of Science
February 5, 1968

No. 672
Hallucinogenic Drugs

DETERMINATION AND IDENTIFICATION OF α -METHYL - 3,4-
METHYLENE DIOXYPHENETHYLAMINE (MDA)

RICHARD FOX
NEW YORK DISTRICT

The district laboratories were informed of a new hallucinogen α -Methyl-3, 4- Methyleneoxyphenethylamine (MDA) by a TWX from Washington, dated October 13, 1967. Since that time, New York District has received two samples of this drug through BDAC. The following rapid procedure is used for the analysis and identification of MDA.

Physical Properties

Free Base - Clear, tan liquid

Molecular Weight 179.22

Refractive Index n_D^{20} 1.5407 (lit. Value)

Boiling Point 149-150°C/14mm (lit. Value)

Hydrochloride - White powder

Melting Point 183 - 185°C

Sulfate - White powder

Decomposition upon heating

Ultraviolet Absorption Spectrum

3.54 mg of free base in 100.0 ml of 0.1N H_2SO_4

Maxima at 286 mu and 234 mu

Minima at 256 mu and 220 mu

Absorbance at 286 mu ca. 0.70

Infrared Absorption Spectrum

Free base run neat between two NaCl plates

Photocopies of U.V. and I.R. spectra are submitted.

Analysis

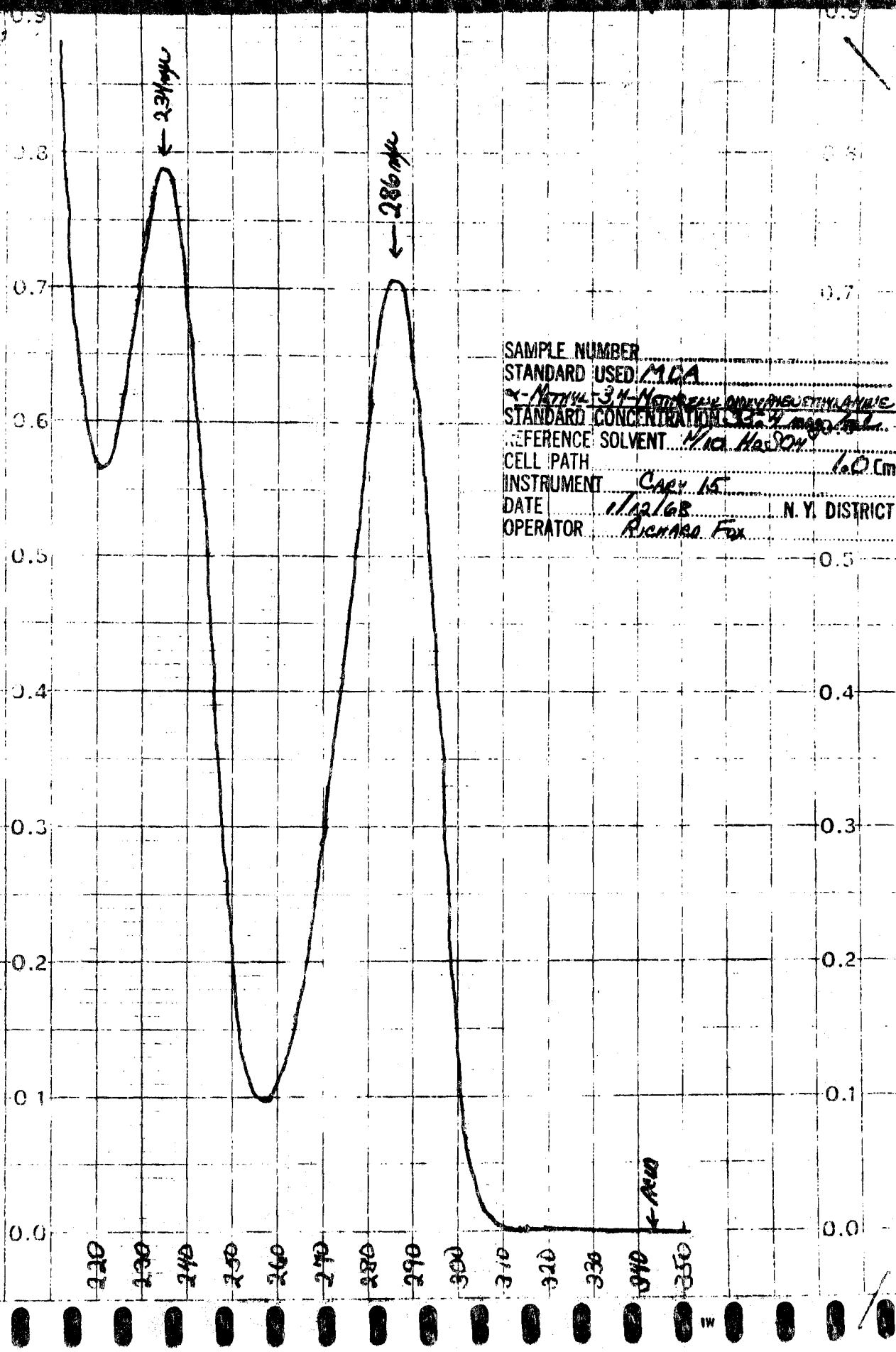
Accurately weigh about 20 mg of sample (equivalent to ca. 3 mg. of MDA). Transfer to a 125 ml. separatory funnel containing 15 ml. of distilled water. Render the solution basic to litmus paper with NaHCO_3 . Extract with four 25 ml. portions of CHCl_3 . Evaporate the combined CHCl_3 extracts just to dryness under a current of air. This leaves a liquid residue of the free base. Transfer residue to a 100ml. volumetric flask with 0.1 N H_2SO_4 and make to volume with 0.1N H_2SO_4 .

Prepare an MDA standard solution containing ca. 30 mcg/ml of free base in 0.1N H_2SO_4 (Free base, sulfate salt, or chloride salt may be used).

Scan the sample and standard solutions against a 0.1N H_2SO_4 blank, in 1.0 cm cells from 350 μ - 210 μ , on a recording spectrophotometer. MDA exhibits maxima at 286 μ and 234 μ . We prefer the 286 μ peak for the determination.

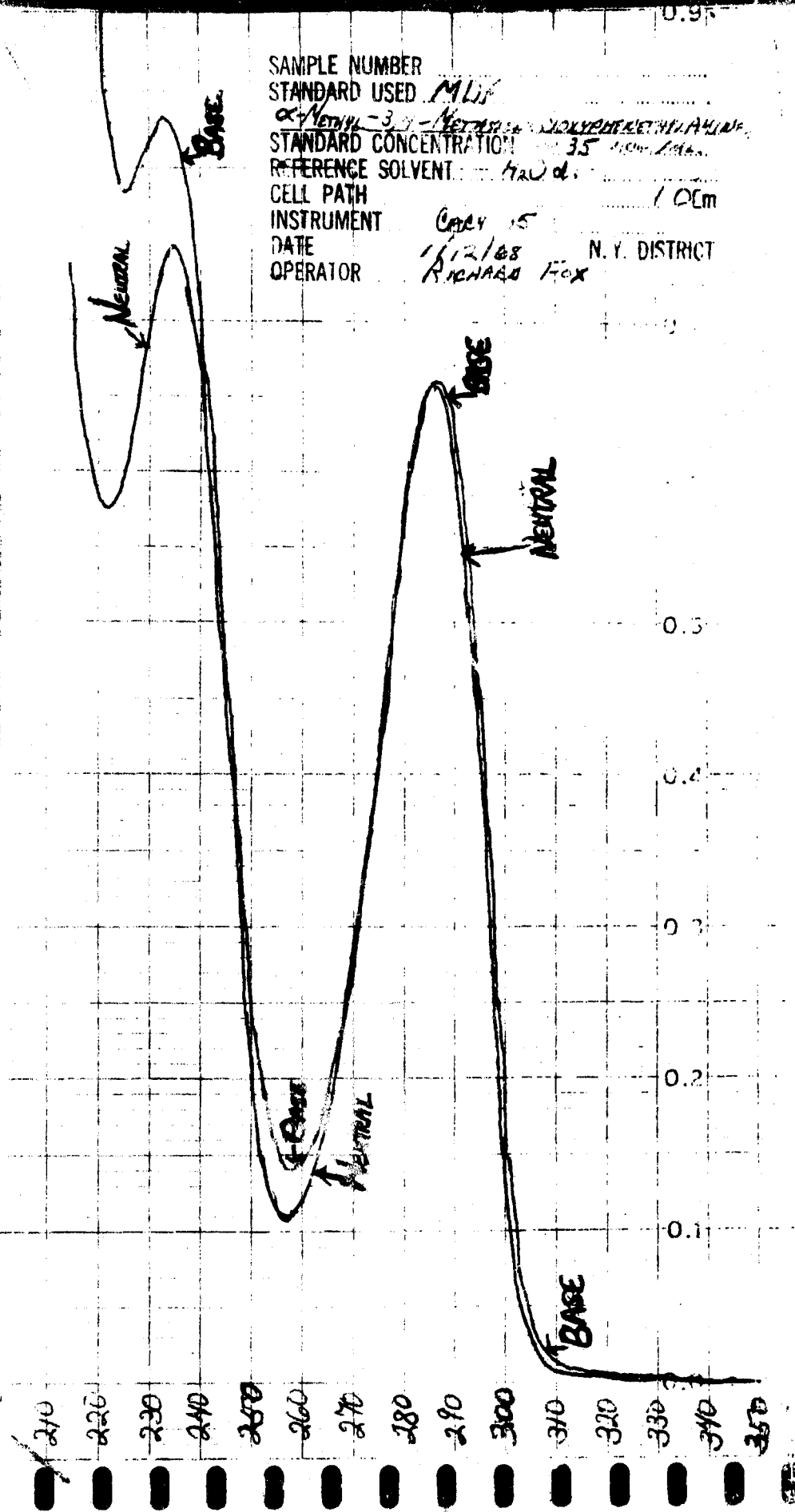
Identification - IR Spectroscopy

Place a few drops of the free base, obtained as in the above procedure, between two NaCl plates and scan on a suitable recording IR spectrophotometer. Compare the resulting spectrum with that of a standard run in the same manner.

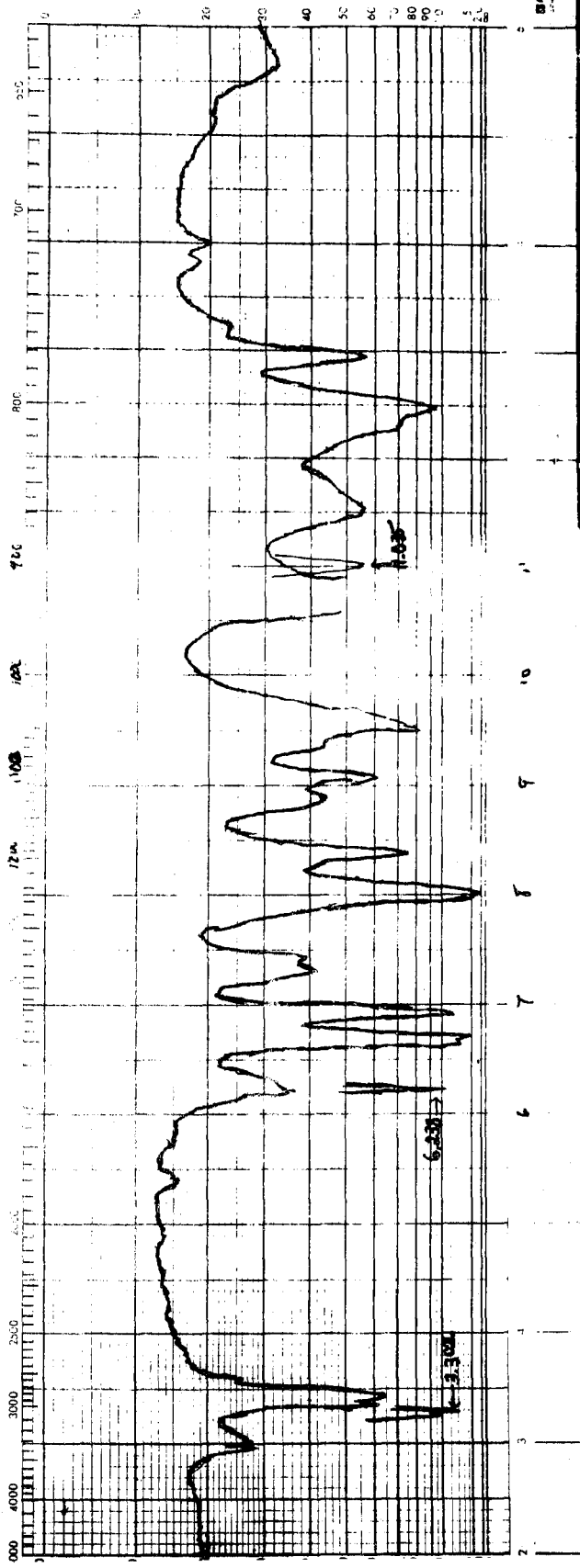


SAMPLE NUMBER
 STANDARD USED: MCA
~~34-NITROBENZENE~~ 34-NITROBENZENE
 STANDARD CONCENTRATION: 0.2 mg/ml
 REFERENCE SOLVENT: MCA HAS 80%
 CELL PATH: 1.0 CM
 INSTRUMENT: CARY 15
 DATE: 11/21/68 N. Y. DISTRICT
 OPERATOR: RICHARD FOX

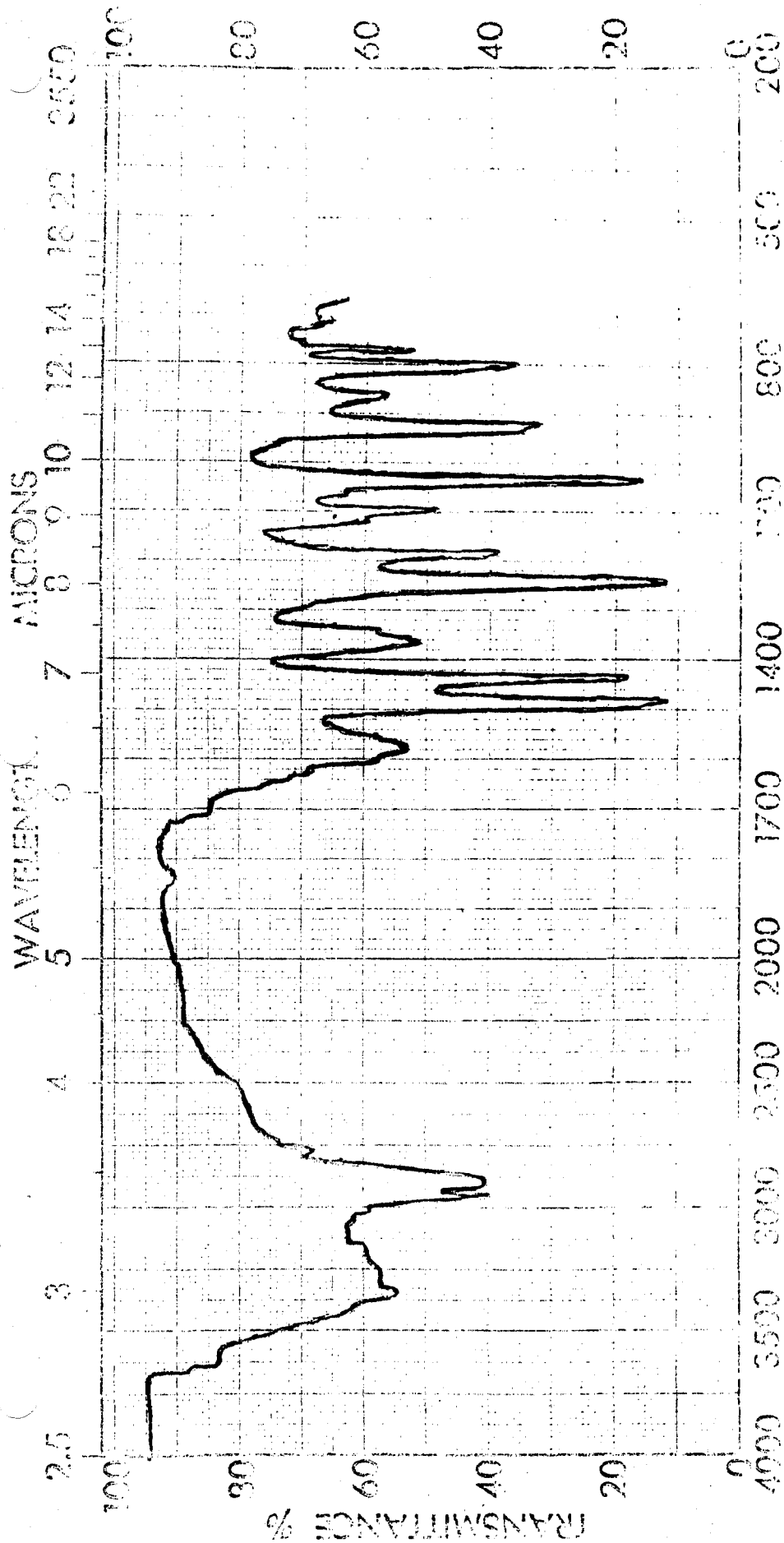
SAMPLE NUMBER
 STANDARD USED *MLJ*
 α -NETHYL-3-(2-NETHYL-2-HYDROXYETHYL)AMINE
 STANDARD CONCENTRATION *35 mg/ml*
 REFERENCE SOLVENT: *EtOH*
 CELL PATH *1 cm*
 INSTRUMENT *Cary 15*
 DATE *1/12/68* N. Y. DISTRICT
 OPERATOR *RICHARD FOX*



SPECTRUM NO. _____
 DATE 1/1/69
 SAMPLE MDA (free base)
 SOURCE _____
 STRUCTURE CC1=CC=C(C=C1)C(=O)O
 PATH _____
 SOLVENT _____
 CONCENTRATION _____
 PHASE NEAT BETWEEN TWO SILICA
 NAME STANDARD HYDROLYZED FILM
 ANALYST RICHARD FOX
 WAVELENGTH _____
 SCATTERED _____
 SPECTROPHOTOMETER _____

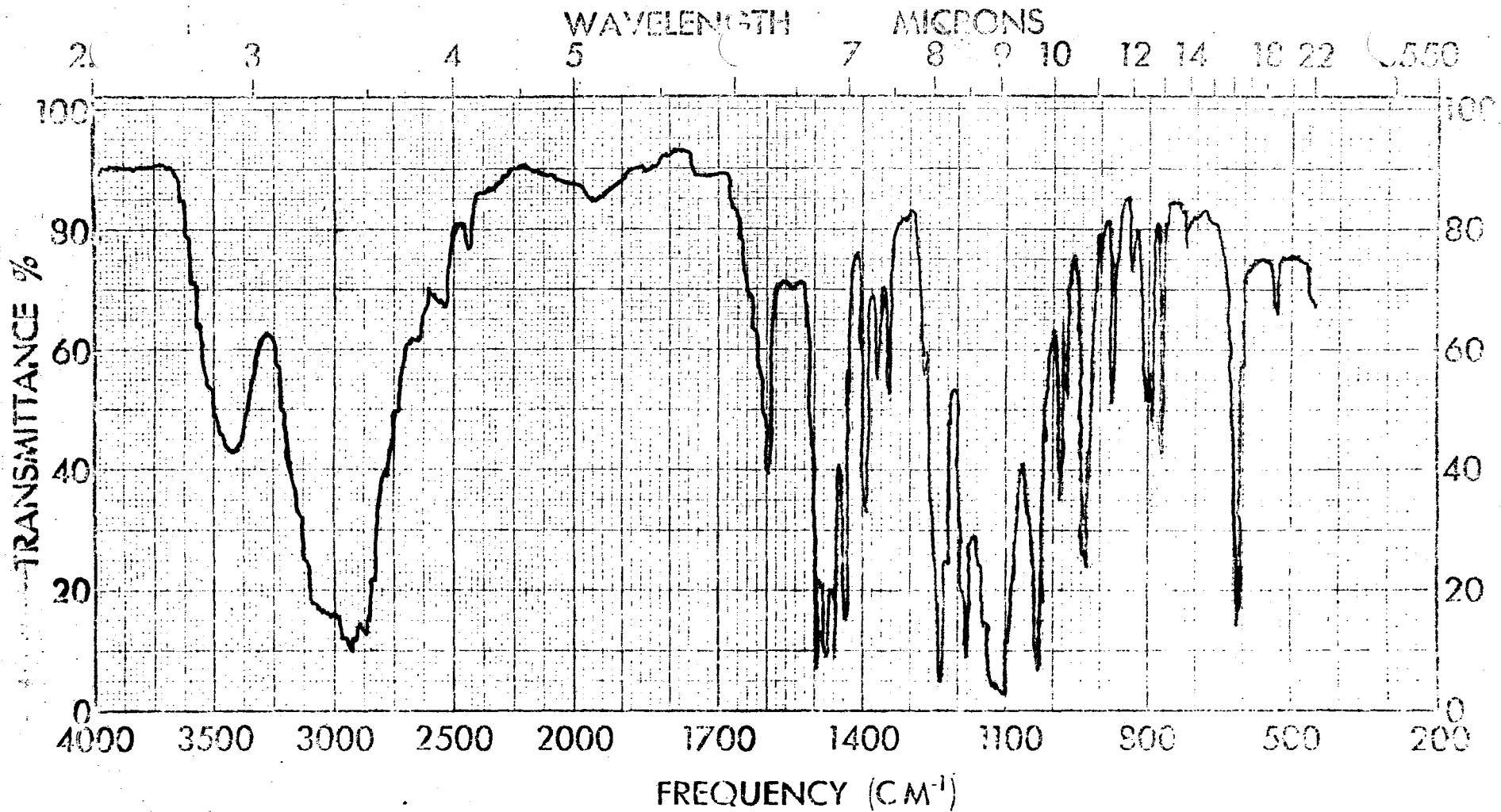


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 100 N. Main St.
 Boston, Mass. 02109



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SPECTRUM NO.	ORIGIN	LEGEND P-E 621	REMARKS
SAMPLE MDA FREE BASE	<chem>CN(C)Cc1ccc(OC)c(OC)c1</chem> CH ₂ -O CH ₃ -O PHASE	1. LIQUID FILM BETWEEN SALT PLATES 2. DATE	3,4 METHYLENEDIAMINE AMPHETAMINE
	PREPARED BY		
	ANALYZED BY	CONRAD PALBERT	SPELWIG



SPECTRUM NO.	ORIGIN	LEGEND P-E 621	REMARKS
SAMPLE MDA Sulfate		1.	3,4-METHYLENE DIOXYAMPHETAMINE SULFATE
	PURITY	2.	
	PHASE KBr	DATE 10-17-67	

TEST TO DISTINGUISH METHEDRINE FROM OTHER AMPHETAMINES

Frank J. Feeney
Alcohol and Tobacco Tax Laboratory
IRS, Treasury Department, Washington, D.C.

Reagents: (A) To a 1% solution of Sodium Nitroprusside add 10% by volume of acetaldehyde. (This reagent should be refrigerated and made fresh at least monthly).

(B) 2% Solution Sodium Carbonate

Procedure: Place one or two milligrams of the amphetamine powder or tablet (use one or two drops of liquid) into a spot plate depression. Add one drop of (A) followed by two drops of (B). An immediate deep blue color indicates the presence of methamphetamine. Benzedrine, Dexedrine or other primary amines yield a slow pink to cherry red color. This test presupposes that the unknown substance has been positively identified as an amphetamine.

Reference: Spot Tests Volume II, Fritz Feigl, Elsevier 1954