



# MICROGRAM

Laboratory Division  
Office of Scientific Support

BUREAU OF NARCOTICS & DANGEROUS DRUGS / U.S. DEPARTMENT OF JUSTICE / WASHINGTON, D.C. 20537

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Vol. V, No. 6

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LSD-Strychnine is being encountered in upstate New York. The combination is found in red, size "00" capsules containing about one gram of a chocolate colored material. Fred W. Grant, Ph.D., Marcy State Hospital, Marcy, New York, reports that the capsules contain as much as 1% (10 mg.) strychnine and that severe toxic reactions have resulted from their ingestion.

LSD mixed with finely ground rose hips has been reported by the New Jersey State Police Laboratory. These specimens, occurring as a brown material, have been encountered either wrapped in aluminum foil or placed in No. 2, clear gelatin capsules.

54% cocaine HCl, 19% procaine HCl, 15.4% benzocaine and 11.1% lactose mixture is reported by the New Orleans, Louisiana, U.S. Customs Laboratory. This is the first time this particular combination has been brought to our attention. The exhibit is believed to be from Columbia, South America.

PCP sold as LSD was analyzed by the Law Enforcement Assistance Council Laboratory, Cape Girardeau, Missouri. The exhibit consisted of light blue (turquoise) colored tablets, flat on both sides and approximately 3 millimeters in diameter. This is the first documented case of PCP in the Southeast Missouri area.

Alleged cocaine exhibit received from the Chicago area was found by the BNDD Special Testing and Research Laboratory to be a mixture of lidocaine sulfate and lactose monohydrate. This is the first time the sulfate salt of lidocaine has been reported. All available references report only the use of the base or the hydrochloride salt in medicinal preparations.

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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 re-

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Cocaine HCl (47.1%) and ephedrine HCl (1.9%) in combination is reported by the BNDD San Francisco Laboratory. The mixture occurs as a white powder in green plastic bags. This is the first BNDD encounter with this combination.

A soft black material recently received from the Scientific Laboratory of the New York State Police was found to be a mixture of marihuana, dextrose and a hard, brittle, black material resembling powdered coal.

### M E E T I N G S

Sixth International Meeting of Forensic Sciences, Edinburgh, Scotland. September 21 - 26, 1972. For further information, write to:

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

California Association of Criminalists - Fall, 1972, 40th. Semi-Annual Seminar, October 19 - 21, 1972. Mansion Inn, Sacramento, California. For further information, contact:

Alan Gilmore  
Sacramento County District Attorney  
Criminalistics Laboratory  
4400 "V" Street  
Sacramento, California 95817

# BNDD LABORATORY NOTES

DATE

May 4, 1972

-60-

NO. 38

DRUG TYPE 3,4 Methylendioxyamphetamine (MDM)

METHODOLOGY Infrared & Ultraviolet Spectrophotometry, Gas Chromatography and Color Tests

## IDENTIFICATION OF 3,4 METHYLENEDIOXYMETHAMPHETAMINE

by

TERRY R. GASTON  
Forensic Chemist

and

GREGORY T. RASMUSSEN  
Forensic Chemist  
CHICAGO REGIONAL LABORATORY, BNDD

Within the past month, the Chicago Regional Laboratory has encountered several exhibits containing 3,4 methylenedioxyamphetamine (MDM), the methamphetamine homologue of MDA. The drug thus far has been encountered only as the hydrochloride salt.

The MDM is similar to MDA in many respects. The two compounds are not distinguished by the commonly used color tests. However, a good indication of whether or not the methamphetamine moiety is present can be obtained with the sodium nitroprusside/sodium carbonate test for secondary amines. Likewise, the ultraviolet spectra of MDA and MDM are quite similar although the MDM apparently has somewhat lower molar absorptivities, as would be expected. One of the best ways to distinguish between the two compounds is by the infrared spectra of their hydrochloride salts. (It is interesting to observe the similarities between MDM HCl and Methamphetamine HCl in the region from  $3200\text{cm}^{-1}$  to  $2400\text{cm}^{-1}$ .) MDM and MDA can also be distinguished by gas chromatography.

### Color tests for MDM HCl

Marquis	Brown→Black→Purple
Mecke	Green→Blue
Froedhe	Brown→Purple→Brown
Zernick	Yellow
Conc. H <sub>2</sub> SO <sub>4</sub>	→Magenta
NaNitroprusside/Na <sub>2</sub> CO <sub>2</sub>	Blue

(continued)

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IDENTIFICATION OF 3,4 METHYLENEDIOXYMETHAMPHETAMINE - continued

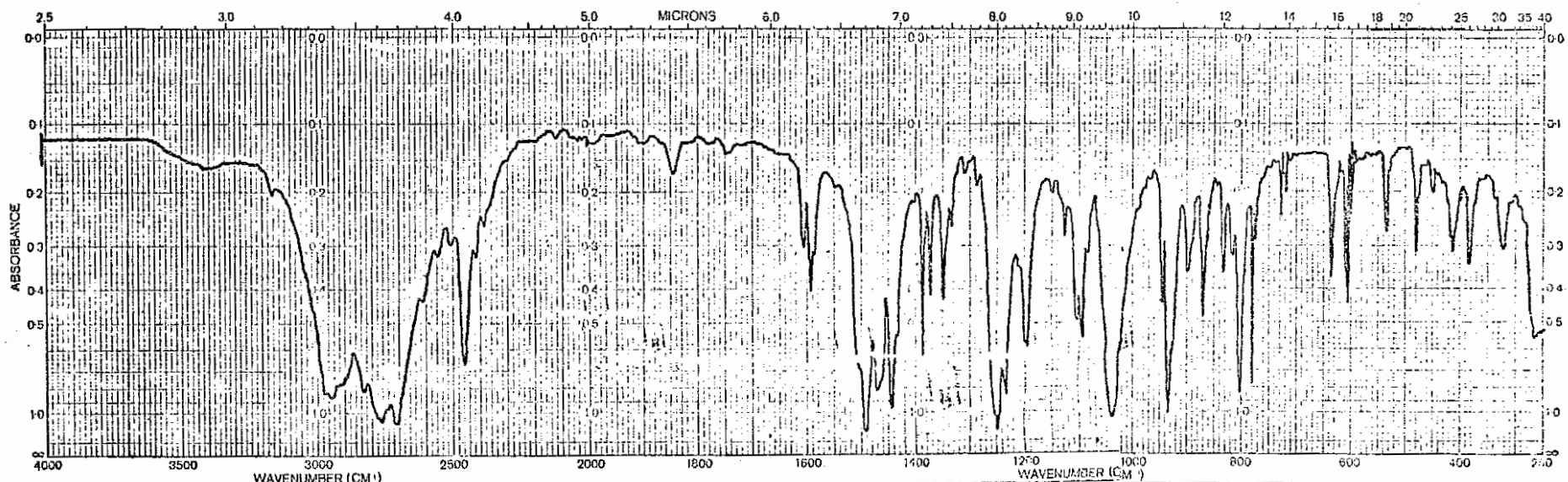
Ultraviolet Spectrum in 0.1N H<sub>2</sub>SO<sub>4</sub> vs. 0.1N H<sub>2</sub>SO<sub>4</sub>

Maxima	284nm 233nm	Estimated A <sub>1%</sub> <sub>1cm</sub>	164
		Estimated A <sub>1%</sub> <sub>1cm</sub>	156
Minima	255nm 220nm		

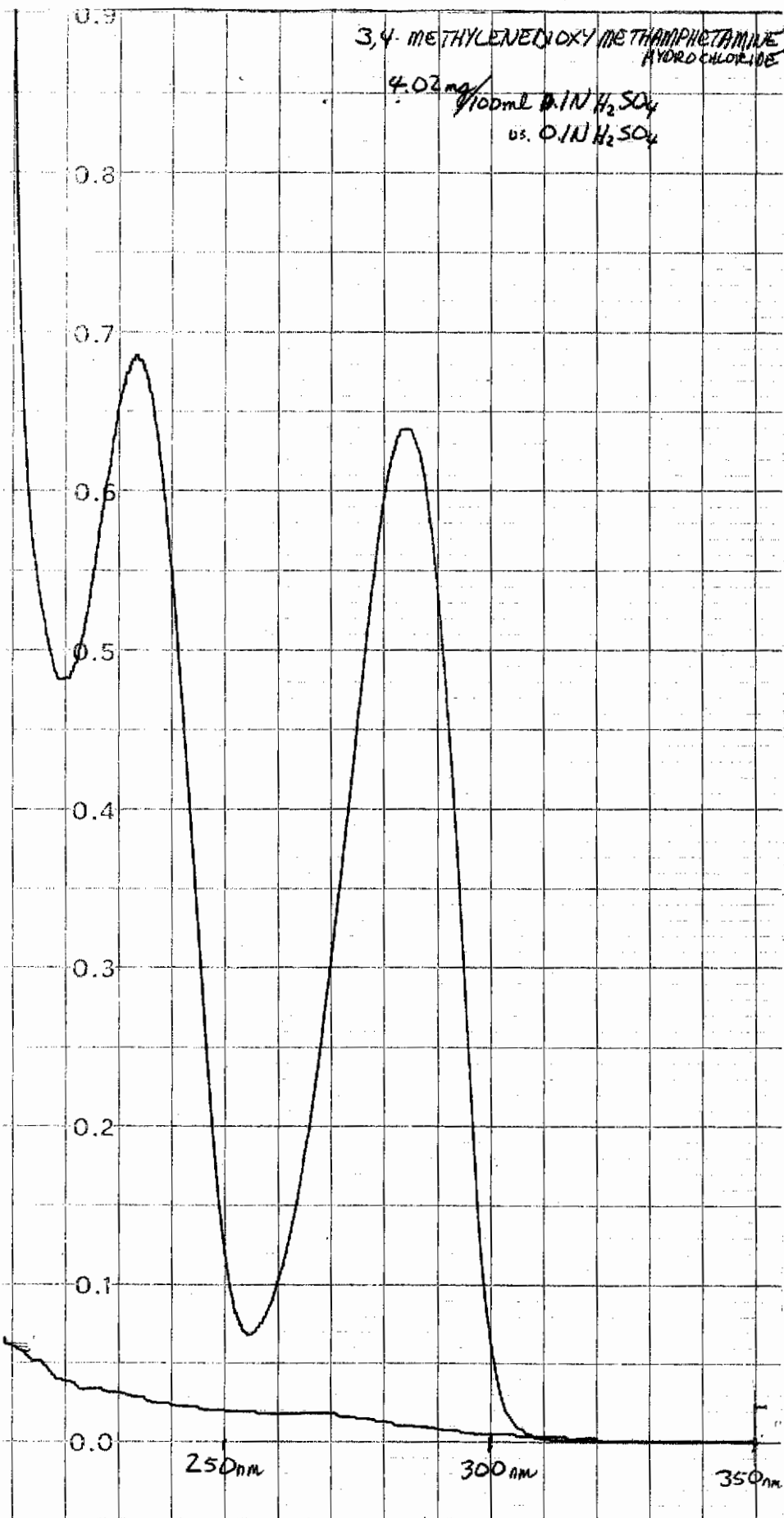
Melting Point - MDM HCl - 150-151°C

Gas Chromatography with Flame Ionization Detector, N<sub>2</sub> carrier

Column Conditions	Retention MDM/Retention MDA
2' 3% OV-17 at 160°C, carrier 40ml/min	1.18
6' 1% SE-30 at 160°C, carrier 40ml/min	1.21



SAMPLE	3,4-DIETHYLENEOXY-2,6-DIMETHYLPYRIDINE HYDROCHLORIDE	SOLVENT	KBr	REMARKS	SCAN SPEED	F	OPERATOR	J.R.
		CONCENTRATION			SLIT	N	DATE	5/2/72
ORIGIN		CELL PATH			PERKIN-ELMER		REF. No.	
		REFERENCE	AIR		PART No. 457-9133			



# BNDD LABORATORY NOTES

DATE February 22, 1972

NO. 35

-64-

DRUG TYPE Mixture

METHODOLOGY

Quantitation of Desbutal Gradumets  
Jeffrey M. Weber  
Michael Tsougros  
New York Regional Laboratory

## Abstract

Difficulty in extracting the active ingredients from long-release preparations is not an uncommon problem. Desbutal Gradumets (Abbott) is such a preparation consisting of a mixture of d-Methamphetamine Hydrochloride and Pentobarbital Sodium.

In the following procedure, separate portions of sample powder are individually treated and extracted for each of the active ingredients.

## Method

### Apparatus:

Mechanical Shaker  
UV Spectrophotometer

### Reagents:

1N HCl, Conc. HCl  
0.1N, 50% NaOH  
0.1N H<sub>2</sub>SO<sub>4</sub>  
CHCl<sub>3</sub> (reagent grade)  
Ethyl Ether (anhydrous)

## Procedure

Methamphetamine - Combine the contents of a representative number of tablets, grind thoroughly (tablets have a thin glossy coating, and are hard with a tough cohesive mixture. They are therefore, not readily powdered.) 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 a mechanical shaker (do not use an ultrasonic bath) for one 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 20 ml. portions CHCl<sub>3</sub>. Add 1 drop concentrated HCl to combined extracts and evaporate to dryness. Quantitatively transfer the residue to a volumetric flask using 0.1N H<sub>2</sub>SO<sub>4</sub>. Read the absorbance

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of the solution at 257 m $\mu$  and compare to standard Methamphetamine. The final acidic solution can be re-extracted to retrieve sufficient quantity of the active ingredient for identification by infrared spectrophotometry.

#### Pentobarbital

Transfer a weighed portion of sample powder into a 100 ml. volumetric flask containing 50 ml. 0.1N NaOH. Place flask on a mechanical shaker (do not use ultrasonic bath) for one hour. Dilute to volume with 0.1N NaOH. Filter a portion of the solution and pipet an aliquot into another volumetric flask so that the final concentration is approximately 1 mg. per 100 mls. of 0.1N NaOH. Read the absorbance of the solution at 240 m $\mu$  and compare against a standard solution of pentobarbital sodium. The basic stock solution may then be extracted to obtain sufficient quantity for identification by infrared spectrophotometry.

#### Results

Recoveries of 94-98% were observed using this method on four batches of Desbutal Gradumets.



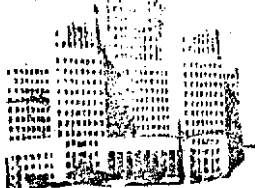


DEPARTMENT OF POLICE



# City of Buffalo

New York



FRANK N. FELICETTA  
COMMISSIONER

## SCIENTIFIC CRIME DETECTION LABORATORY ROBERT E. PERRIGO, DIRECTOR

### A New Form of Methamphetamine

Angelo M. Fatta, Ph.D.

Several cases were encountered recently where a pale yellow crystalline substance was submitted to our laboratory for analysis. In each case the material was being sold as methamphetamine, and appeared to be identical in its appearance and chemical reactions. In one specific case, the material was linked to a near-fatal overdose at one of the local colleges. It is purported that this material has Toronto, Canada as its source.

#### PHYSICAL PROPERTIES:

Light yellow to yellow crystals that are highly dichroic under polarized light. A weak offensive odor. Melting point: 163-166°C with sublimation at 80° and some darkening at 145°. A solution of 1.0 gram in 10 ml distilled water has a pH of 4.53. The compound is very soluble in water and methanol, partially soluble in acetone and ethanol, and insoluble in ether and chloroform.

#### COLOR SPOT TESTS:

Addition of Marquis' or Froehde's reagent to the solid material gave rise to vigorous formation of dense, brown-red choking fumes. Addition of water following the Marquis reagent gave rise to no fluorescence under UV light.

Treatment of the solid with 2.0% sodium carbonate solution followed by sodium nitroprusside-acetaldehyde solution produced a cloudy, dark blue suspension.

Addition of concentrated nitric acid to the solid caused

precipitation of dark, lustrous plates.

No results were obtained with cobalt thiocyanate or Dille-Koppanyi reagents.

#### MICROCRYSTALLINE TESTS:

Phosphoric acid and gold chloride solution produced a dark brown, opaque colloid when added to the solid and heated. No crystals were formed within two hours. A reagent composed of one gram of gold chloride in 100 ml of 67% phosphoric acid gave the characteristic crystals associated with methamphetamine when added to the solid. Some colloid also formed.

#### UV SPECTRUM:

The UV spectrum of the material in water shows an intense peak at 226nm with shoulders at 267, 262, and 256nm. To further resolve the spectrum, a sample was dissolved in water, made basic with  $\text{NaHCO}_3$  and extracted into ether. The ether layer was filtered and allowed to evaporate. The residue was taken up in 0.1N HCl. The absorption spectrum of this solution showed the peak at 226nm much reduced in intensity, with well resolved shoulders at 276, 262, 257, and 249nm. These spectra are shown in Figures 1 and 2.

#### IR SPECTRUM:

The IR spectrum of the solid in a KBr disk contains the peaks indicative of an amphetamine. Several intense bands at 1550, 1205, and  $985\text{ cm}^{-1}$  also appear which do not occur in the standard spectrum.

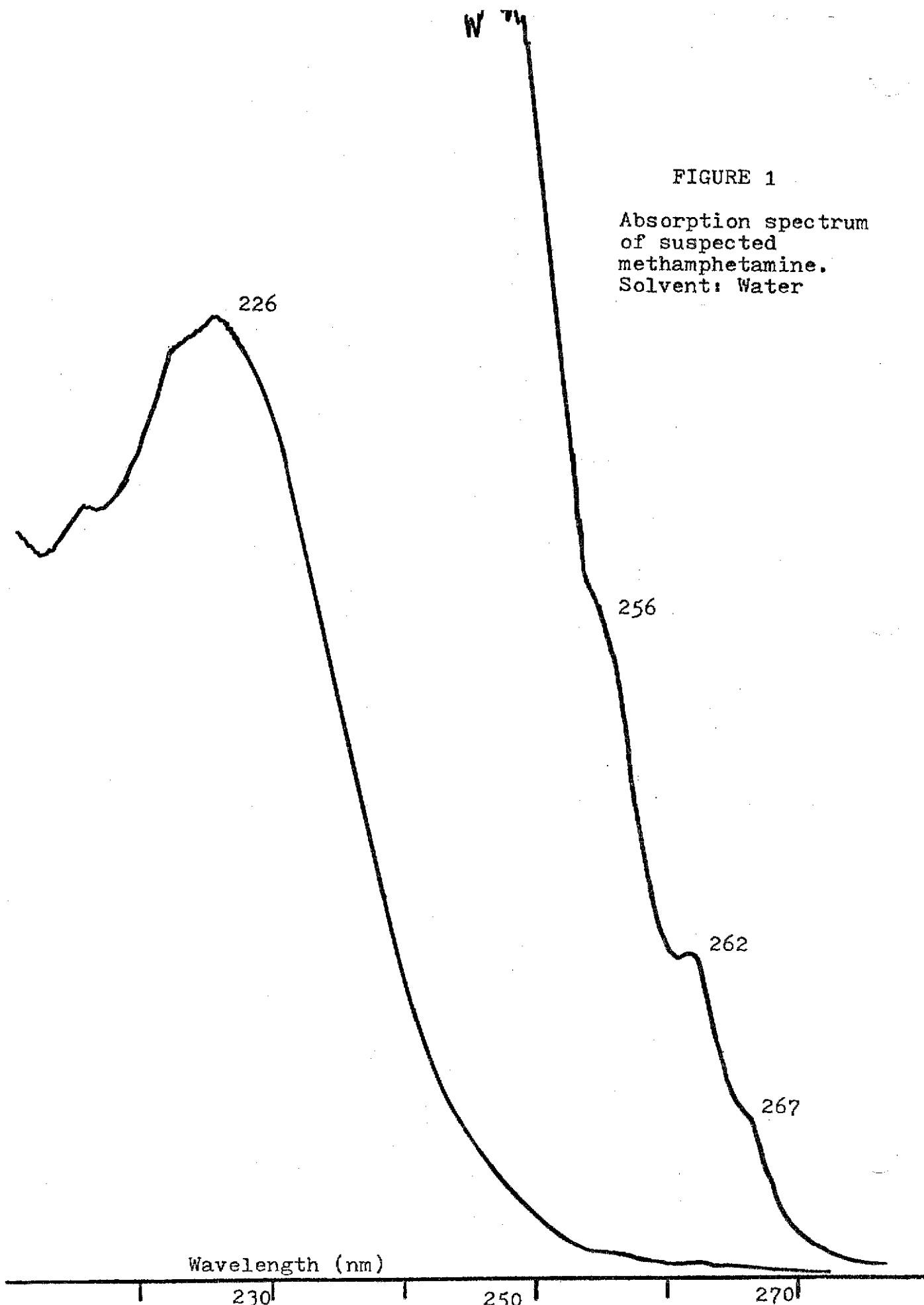
#### DISCUSSION:

In the absence of definitive elemental analysis, the material has been identified as methamphetamine hydroiodide. The yellowish color is attributed to the oxidation of  $\text{I}^-$  to  $\text{I}_2$  and the subsequent formation of the highly colored species  $\text{I}_3^-$  in the solid state. It is this species that is responsible for the intense UV peak at 226nm which masks some of the characteristic amphetamine absorptions. An aqueous solution of  $\text{I}_2$  and KI shows an absorption at 225nm.

The dark lustrous plates formed upon addition of  $\text{HNO}_3$  have been identified as  $\text{I}_2$  by the absorption spectrum in chloroform. The dense choking fumes produced by addition of Marquis or Froehde reagent are thought to be a mixture of iodine vapor, water vapor and HI gas. (This particular reaction can be a source of confusion to agents in the field using field test kits.) Finally, discrepancies between the IR spectrum of the substance and the spectrum of methamphetamine·HCl can be ascribed to distortions in the crystal lattice by the large iodide ion, these distortions giving rise to a perturbation of the selection rules consistent with the molecular point group thereby giving intensity to otherwise weak or forbidden bands.

FIGURE 1

Absorption spectrum  
of suspected  
methamphetamine.  
Solvent: Water



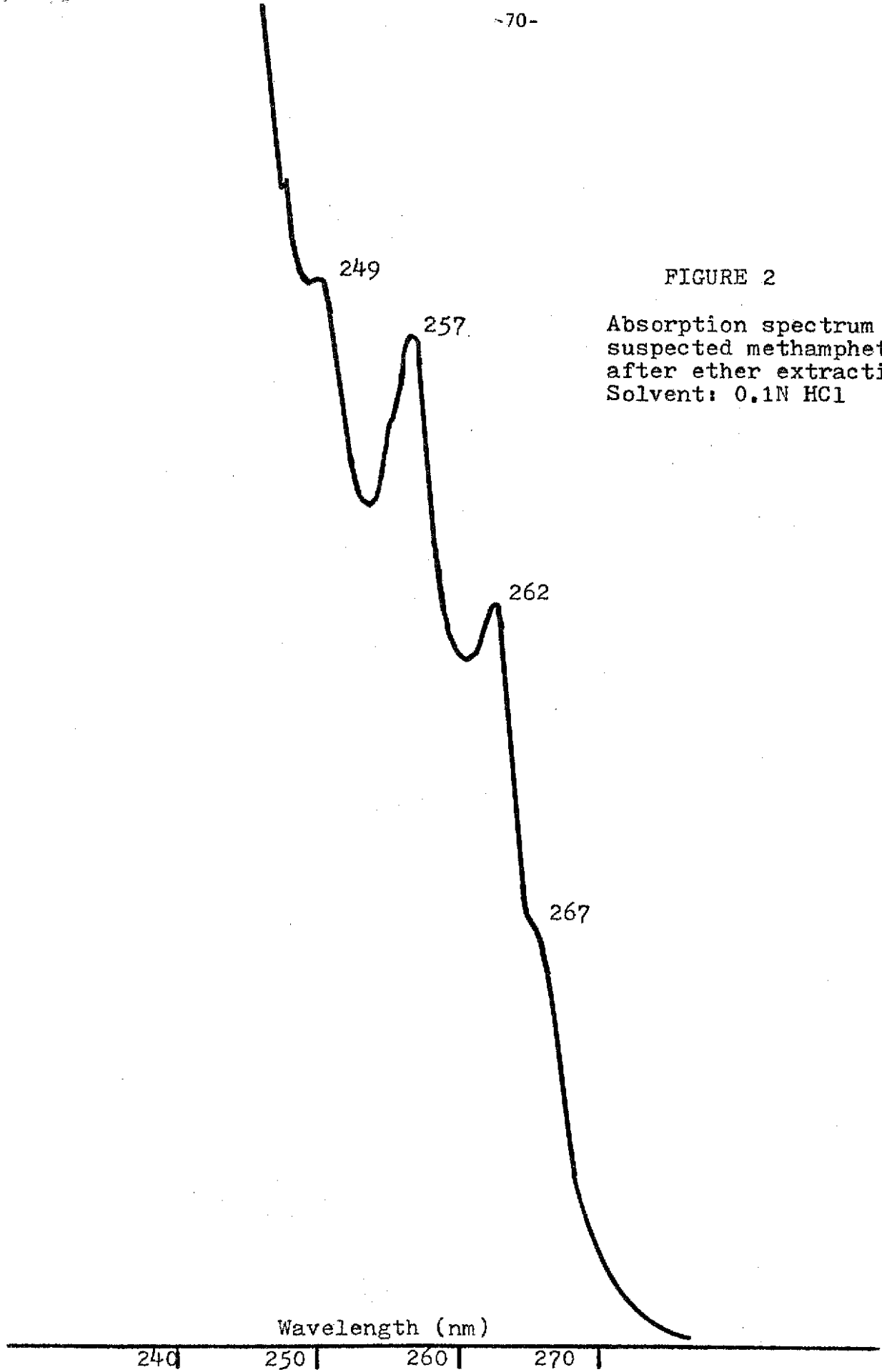


FIGURE 2

Absorption spectrum of  
suspected methamphetamine  
after ether extraction.  
Solvent: 0.1N HCl