

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. 1

January, 1971

Can LSD be detected in biological samples? A simple question and the answer according to a recent Government report (see below) is just as simple - "Yes". Medical, legal, and police authorities understandably are puzzled, therefore, when their analysts tell them it is not possible. The apparent contradiction is perhaps more easily explained if we present an analogy. "Can one drive a car at 200 m.p.h.?" The unqualified answer is "Yes", but the insertion of one word, "usually", would make the answer "No".

To return to the LSD problem, the answer is "Yes" when a large dose has been ingested, the analytical sample is adequate and taken at the right time, when the analyst has the necessary equipment and experience, and, perhaps most important of all, if he knows before starting his analysis that LSD is the suspected poison. (All too often he has everything to seek and yet in reality nothing to find!) Another vital factor is the criteria used to judge success. One man's proof is another man's probability. The scientist who has personally administered LSD and subsequently obtains a plasma extract with a fluorescence greater than the previous biological blank may well be entitled to attribute this increase to LSD - the forensic toxicologist can not.

This statement of the obvious was prompted by a review of some of the contributions received in the past year which judged purely on the facts reported are often conflicting. This conflict can usually be resolved when additional data is requested. Thus, it is important in a positive case to give the methods of identification, in a negative one, the amount of material analyzed and the sensitivity of the method employed so that the reader can assess the significance of the report. Alha's criticism of members' case notes (Bulletin 1968, Vol. 5, No. 1) unfortunately is often justified and whilst I defended this brevity, I agree with your President's opinion that if we claim to be a scientific society, we must be more professional in our reporting.

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.

The preceding is an editorial reprinted from the Bulletin Of The International Association Of Forensic Toxicologists, Vol. 7, No. 3, 1970, by permission of the editor, Mr. John V. Jackson, Metropolitan Police Laboratory, 2 Richbell Place, London W C 1, UK. The report referred to in the editorial is: "The Amphetamines and Lysergic Acid Diethylamide (LSD)" Report by the Advisory Committee on Drug Dependence, paragraph 93, page 31. HMSO publication, 1970, London."

LSD gelatin squares, known as "Clear Lights," have been seen in Australia where they sell for about \$10.00 each. From the description, they sound like the gelatin flakes previously encountered in England and in the United States. (See Microgram, Vol. III, No. 6, [October, 1970], p. 173)

Pronestyl Capsules (procainamide hydrochloride) manufactured by E. R. Squibb and Sons, are being emptied and the powder sold as a narcotic, according to a midwestern state police laboratory. A sample was given to the BNDD Chicago Regional Laboratory and the presence of procainamide hydrochloride was confirmed.

Methylenedioxyamphetamine hydrochloride was identified in an exhibit by the BNDD Special Testing and Research Laboratory. The methylenedioxy linkage appears to be in the 3,4-positions. The exhibit was furnished by the Chicago Police Department Crime Laboratory.

Dramamine (dimenhydrinate) and barbiturates are being abused in Alaska according to a report received in the BNDD San Francisco Regional Laboratory. According to an Alaskan source, young people are taking ten Dramamine tablets in combination with one barbiturate tablet.

Vitride Reducing Agent, sodium bis (2-methoxyethoxy) aluminum hydride, is now available through Eastman Organic Chemicals. The compound was developed by Dr. Jaroslav Vit, a Czechoslovakian scientist. According to the Eastman Organic Chemical Bulletin, Volume 42, No. 3, 1970, "Vitride . . . is as strong as lithium aluminum hydride, and . . . is more versatile, more convenient, and much safer" (See Microgram, Vol. II, No. 3, [September, 1970], p. 60)

Flat toothpicks containing 5 micrograms LSD per toothpick have been encountered by a New Mexico police department.

Index for Microgram 1970 (Volume 3, Nos. 1-8) will be included with a future issue.

SELECTED REFERENCES:

McGlothlin, William; Jamison, Kay; and Rosenblatt, Steven, Marihuana and the Use of Other Drugs, Nature, Vol. 228, (December 19, 1970), p. 1227-1229.

"What the Fire Service Should Know About Clandestine Drug Laboratories," Fire Command! magazine, Vol. 37, No. 11 (November, 1970). Not a scientific paper, but an article alerting fire protection personnel to the dangers of chemicals used to make drugs. Shows the need of teamwork among law enforcement officers, chemists, and firemen. Reprints available from National Fire Protection Association, 60 Batterymarch Street, Boston, Mass. 02110. Cost is \$0.50 each.

MEETINGS:

1971 National Symposium On The Forensic Sciences. The meeting sponsored by the Forensic Science Society (South Australian Branch) will be held in Adelaide, South Australia, February 10-13, 1971. Copies of the program and registration forms can be obtained from:

The Forensic Science Society (S.A. Branch)
Box 194, G.P.O.
Adelaide, South Australia 5001

American Academy of Forensic Sciences annual meeting, Phoenix, Arizona, February 21-26, 1971. Registration forms are available from Arthur H. Schatz, J.D., Secretary-Treasurer, American Academy of Forensic Sciences, 750 Main Street, Suite 1000, Hartford, Connecticut 06130. Registration after January 20, 1971 or at the door is \$60.00. (See also Microgram, Vol. III, No. 7, November, 1970).

Sixth International Meeting of Forensic Sciences, Belfast, 1972 will be held September 21-26, at the Queen's University, Belfast, Northern Ireland.

A mailing list is being prepared and those who would like to receive further information when published should write to:

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

Forensic Chemists' Seminars for state and local chemists will be held

April 12-16, 1971
June 14-18, 1971

For information and application blanks, write:

Special Training Division
National Training Institute
Bureau of Narcotics and Dangerous Drugs
Washington, D. C. 20537

DRUG STANDARDS, or suitable compounds for references, are available from local chemical supply houses or from the following and other firms:

K & K Laboratories, Inc.
121 Express Street
Engineers Hill
Plainview, New York 11803

Eastman Kodak Co.
Eastman Organic Chemicals
Rochester, New York 14650

Aldrich Chemical Company, Inc.
10 Ridgedale Avenue
P. O. Box AA
Cedar Knolls, New Jersey 07927

Koch-Light Laboratories Ltd
Colnbrook Buckinghamshire
England

S. B. Penick & Co.
100 Church Street
New York, New York 10008

U.S.P. Reference Standards are available at \$20.00 each from:

U.S.P. Reference Standards
4630 Montgomery Avenue
Bethesda, Maryland 20014

National Formulary Reference Standards are available from:

N. F. Reference Standards
American Pharmaceutical Association
2215 Constitution Avenue, N. W.
Washington, D. C. 20037

A price list is available on request.

BNDD LABORATORY NOTES



DATE December 23, 1970

-5-

NO. 11

DRUG TYPE Hallucinogen

METHODOLOGY Spectrophotometric

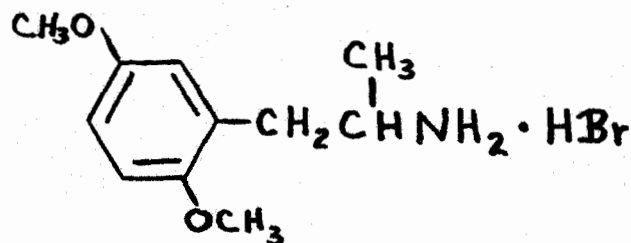
ANALYSIS AND IDENTIFICATION OF 2,5-DIMETHOXYAMPHETAMINE

Paul DeZan
Forensic Chemist
New York Regional Laboratory
Bureau of Narcotics & Dangerous Drugs

BACKGROUND

2,5-Dimethoxyamphetamine is a compound which is very similar to STP (2,5-Dimethoxy-4-methyl-amphetamine). It is appearing in the illicit market throughout the country. At present, the dosage preparation appears to be in the form of No. 0 capsules filled with the pure substance. Average net contents of the capsules were 200 mgs. which assayed 98.6% 2,5-Dimethoxyamphetamine Hydrobromide.

Recently, a large quantity of the pure powder was seized by BNDD agents. Analysis by Special Testing and Research Laboratory, in Washington, revealed the material to consist of a mixture of the Hydrobromide and Hydrochloride salt of 2,5-Dimethoxyamphetamine.



$C_{11}H_{17}NO_2 \cdot HBr$

Mol. wt. = 275.03

2,5-Dimethoxyamphetamine Hydrobromide

BUREAU OF NARCOTICS AND DANGEROUS DRUGS / U.S. DEPARTMENT OF JUSTICE

PHYSICAL PROPERTIES:

a. Hydrobromide:

Description - a fine, white, crystalline powder.
Solubility - soluble in chloroform and water;
insoluble in ether.
Melting Point - 129° - 131°C

b. Hydrochloride:

Description - a fine, white, crystalline powder.
Solubility - soluble in chloroform and water;
insoluble in ether.
Melting Point - 107.5-111.5°C - found
111.5-112.5°C - literature value (2)

c. Free Base:

Description - a colorless, odorless liquid. The
liquid appears to solidify upon standing
in the form of a carbonate salt, because
of absorption of carbon dioxide from the
atmosphere.
Solubility - not determined
Boiling Point - 137° - 140°C - literature value (1)

QUALITATIVE TESTS:

A. Color Tests

Although 2,5-dimethoxyamphetamine is very similar to
STP, the following color reagents will differentiate the two
compounds.

<u>Color Reagent</u>	<u>2,5-Dimethoxyamphetamine</u>	<u>STP</u>
Marquis Reagent	light yellowish green	colorless → faint yellow
Conc. Nitric Acid	lemon yellow → orange	green flashes → lemon yellow
Mecke's Reagent	dark brown → reddish brown	dark green & yellow

(All color reactions occur within one minute after the addition of the
color reagent)

B. Microcrystalline Tests:

Reagent - saturated aqueous picric acid.

A few crystals of the sample powder are added directly to a drop of the reagent on a glass slide. With 2,5-dimethoxyamphetamine, precipitation will occur, but will remain amorphous. Orange droplets can be seen but no crystal formation occurs. With STP you will get yellow-orange blades, plates and needles.

QUANTITATIVE ANALYSIS:

- A. UV Spectrophotometry - Since dosage preparations contain no diluents, a weighed portion of the capsule powder can be diluted directly with 0.1N HCl in a volumetric flask. The final sample dilution should contain approximately 4 mgs. per 100 mls. This solution is scanned on a UV spectrophotometer and compared against a standard solution of similar concentration, utilizing the 288 m μ peak for quantitation.

2,5-dimethoxyamphetamine and STP provide very similar UV spectra. A slight difference occurs at the minimum of each spectra. For 2,5-dimethoxyamphetamine the minimum occurs at 250 m μ ; for STP, the minima occurs at 254 m μ .

- B. Spectrophotofluorimetry - if interferences occur in the UV spectrophotometric measurement the chemist still has a chance to quantitate his sample by further diluting the prepared UV sample solution to a concentration of approximately 0.3-1.0 mcg/ml. This solution can be scanned on a spectrophotofluorimeter and compared against a standard solution of similar concentration.

The instrumental parameters are as follows:

Fluorescence maxima	-	324 m μ
Excitation maxima	-	297 m μ
Slits (Fluo./Excit.)	-	9/9
Filter	-	310
Sensitivity		3

IDENTIFICATION BY IR:

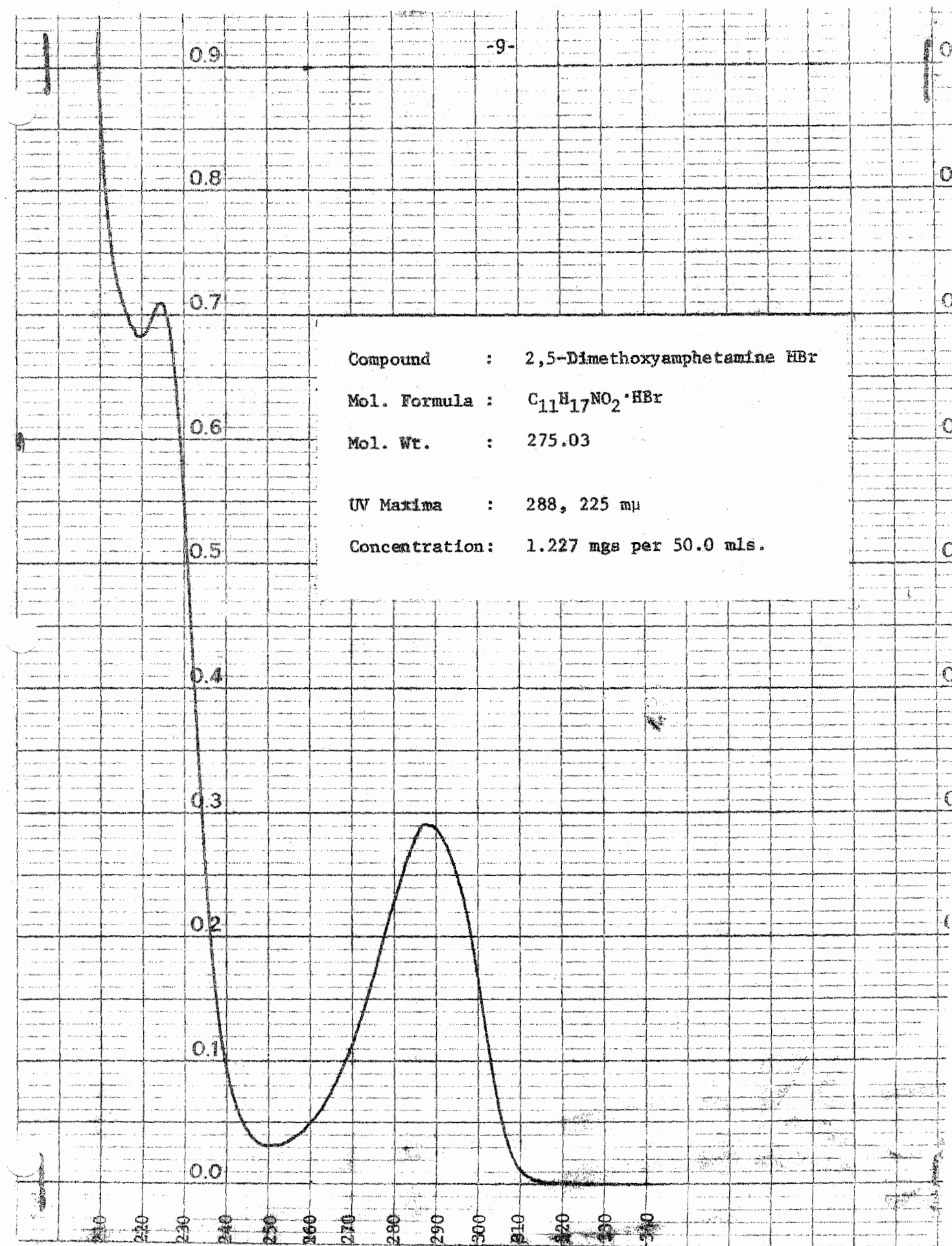
A KBr disc was prepared with a portion of the capsule powder. The infrared spectrum was compared to that of a standard curve for 2,5-Dimethoxyamphetamine Hydrobromide.

ACKNOWLEDGEMENT

I wish to acknowledge the assistance of Mr. Stanley Sobol, Chief Chemist, Special Testing and Research Laboratory, BNDD, and his staff for their efforts in identifying the salt of 2,5-dimethoxyamphetamine and in particular, Albert Sperling, Ph.D., whose advice assisted in identifying the compound.

REFERENCES

1. JACS 62, 161 (1940).
2. J. Med. Chem. 13, 26 (1970).



-OT-

30 70
40 60
50 50
60 40
70 30
80 20
90 10
100

220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540

Compound: 2,5-Dimethoxyamphetamine HBr

Conc: 0.3 mcg./ml.

Solvent: 0.1N HCl

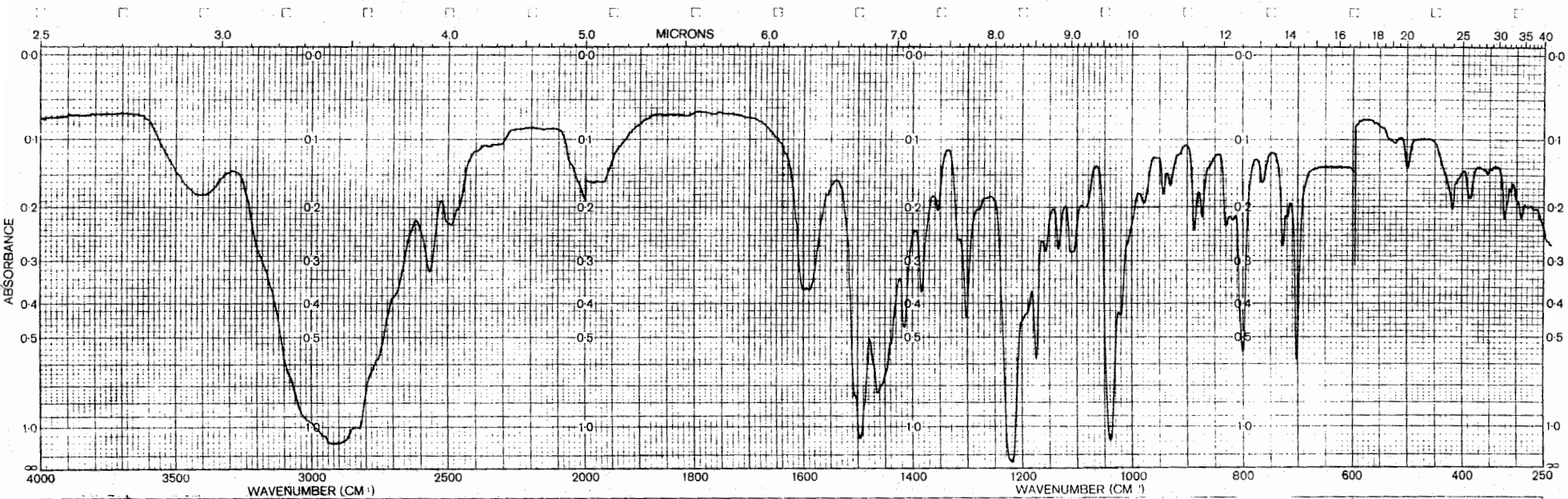
Emission: 324 m μ

Excitation: 297

Slits (Em/Ex): 9/9

Sensitivity: 3

Filter: 310



SAMPLE 2,5-DIMETHOXYAMPHETAMINE HCl ORIGIN	SOLVENT KBR CONCENTRATION ± 1% CELL PATH REFERENCE AIR	REMARKS USED MINIPRESS	SCAN SPEED F N <small>RECORDING CHARTS</small>	OPERATOR P. DEZAN DATE REF No
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