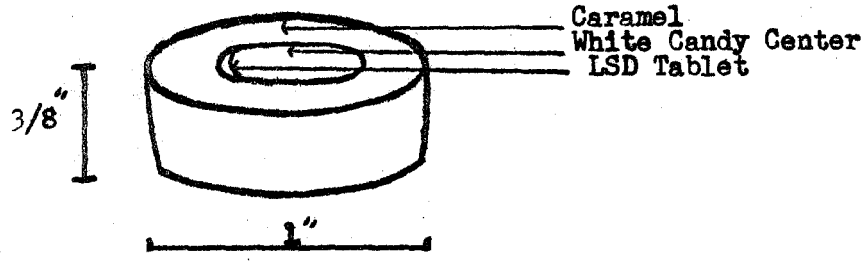
MICROGRAM

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. 10 November, 1971

LSD tablets in candy is reported by the Northern Illinois Police Laboratory. The candy is in the shape of a circular disc approximately one inch in diameter and 3/8 inch thick with an outside ring of caramel and a white creme center. The LSD, in tablet form, is placed between the caramel and the creme center. These candies come wrapped in cellophane, five to a cardboard tray, four trays to a carton and are sometimes referred to as "Bull's Eyes."



Lidocaine 35.9% and cocaine 1.7% in combination is reported by the Chicago Regional Laboratory. This mixture, a brown powder, was found in colorless number 5 and pink number 4, hard gelatin capsules. This is the first encounter with Lidocaine containing cocaine. (See also Microgram, Vol. IV, No. 9, October, 1971)

Amobarbital, secobarbital and quinine mixtures were recently received by the New Jersey State Police Laboratory. The exhibits were received as a brown powder packaged in aluminum foil.

Heroin (3.5%) and phenylpropanolamine HCl (41.3%) has been encountered on the East Coast. This is the first time we have found these substances in combination.

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
Special 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

SELECTED REFERENCES

McGlothlin, W. H., Arnold, D. O., "LSD Revisited," Arch. Gen. Psychiat., Vol. 24, Jan., 1971.

Nureo, D. N., Lerner, M., "The Feasibility of Locating Addicts in the Community," International Journal of the Addictions, 6 (1): 51-62, 1971.

Curry, A. S., "Advances in Forensic Toxicology," The Chemical Rubber Co., 18901 Cranwood Parkway, Cleveland, Ohio 44128. Price: $32.00.

BNDD LABORATORY NOTES

DATE July 20, 1971

NO. 23

DRUG TYPE Quantitation of Amphetamine in Resin Complexes

METHODOLOGY

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Products such as Strasenburgh's "Biphetamine" present a problem in quantitative analysis. The resin complex is difficult to break up unless lengthy refluxes and distillations are resorted to. The following procedure gave satisfactory results on two different lots of Biphetamine.

Prepare a composite sample in the usual manner, grinding the resin beads and thoroughly mixing with the rest of the contents. Weigh accurately a portion of the composite equivalent to approximately 20 mg. of amphetamine base into a 50 ml. volumetric flask. Place a small funnel into the neck of the flask to serve as a condenser. Add 25 ml. of 1N ammonium chloride in 70% methanol and heat the flask for 40 minutes on a steam bath, swirling occasionally to keep material from clumping. Cool flask, and dilute to volume with 70% methanol. Filter, and determine UV absorbance in usual manner, using a standard solution of approximately 25 mg. amphetamine sulfate in 50 ml. 70% methanol.

RAPID QUALITATIVE GLC IDENTIFICATION OF AMPHETAMINE AND VARIOUS HALLUCINOGENS

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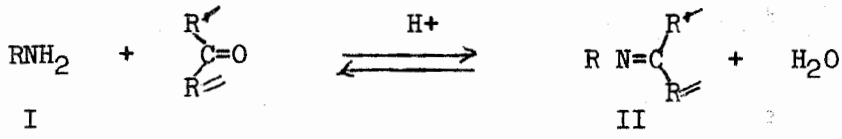
San Diego, California

Primary amines (I) condense rapidly with ketones forming imines (II), commonly known as Schiff bases. (1)



I II

Under proper reaction conditions an equilibrium mixture of I and II can be attained providing a useful vehicle for GLC analysis of I. Two parameters are obtained for identification from a single injection.

Amphetamine and various hallucinogens have been identified and screened through Schiff base formation using acetone as the carbonyl component. The rapidity and ease of this procedure make it of value in a forensic laboratory.

EXPERIMENTAL

A Microtec MT 220 with FID was used. The samples were run on a 1/4" (4') 2% OV 17 (on 80/100 Gas Chrom Q) column at 115°C (amphetamine) and 185°C (hallucinogens). Inlet temperature: 300°C. Detector: 300°C. Carrier Flow: 65 cc/min.

The following procedure is used routinely for known high dosage tablets. Methanol is the solvent of choice for a convenient reaction rate when the acid salts are extracted directly. Acetone gives Schiff bases with convenient retention times relative to the parent amine.

> One white double scored amphetamine tablet (0.06 g) was crushed in a vial containing methanol (1/2 ml). Acetone (1/2 ml) was added and the resulting solution filtered. The reaction mixture was injected at once but is stable for several days.

When low concentrations necessitate extraction of the amine from a basic solution, the following procedure is effective.

> One drop of an amine extract, which had been washed and concentrated, was added to a 1% solution of acetic acid in ethyl acetate (30 drops) followed by addition of acetone (10 drops). The resulting solution was injected at once but is stable for a day or two.

The following data was obtained for amphetamine, two substances isomeric with amphetamine, and four hallucinogens.

Retention Time (minutes)

	Amine	Schiff Base
Amphetamine[a]	1.3	2.4
2-(p-tolyl)-ethylamine[a]	2.3	6.0
3-phenyl-1-propylamine[a]	2.5	6.4
2,5-dimethoxyamphetamine (DMA)[b]	1.1	1.5
3,4-methylenedioxyamphetamine (MDA)[b]	0.79	1.1
3,4,5-trimethoxyphenethylamine (Mescaline)[b]	2.4	4.5
4-methyl-2,5-dimethoxyamphetamine (STP)[b]	1.3	1.7

[a]Column 115°C
[b]Column 185°C

(1) W. J. A. Vanden Heuvel, W. L. Gardiner, and E. C. Horning, Anal. Chem., 36, 1550 (1964).

SEPARATING DRUG MIXTURES BY PREPARATIVE THIN-LAYER CHROMATOGRAPHY (TLC)

by R. Martin Smith, Ph.D
and
Frank C. Dolejsi, Jr.
Wisconsin Department of Justice/Crime Laboratory Bureau
Madison, Wisconsin

Illicit drugs are often obtained in mixtures which are difficult to examine by means of hard spectra (such as infrared or NMR), so that more roundabout methods have been employed in the past in the analyses of these materials. We have found that in many cases single drugs can be separated from recalcitrant excipients or from naturally occurring mixtures of drugs by means of preparative thin-layer chromatography. This method is relatively fast and inexpensive, and for us has given perfectly reproducible spectra.

We commonly use two different kinds of thin-layer plates, depending upon the volume of crude sample and the characteristics of the particular drug mixture. In cases where the volume of crude sample is relatively small and where a single compound is being separated from materials with relatively dissimilar R_f values, commercially prepared 20 x 20 cm Silica gel GF 254 plates (thickness 200 microns, available from Analtech, Inc., Newark, Delaware) are convenient. For cases where a single compound is being separated from materials with very similar R_f values, we spread our own plates to a thickness of 0.5 mm using 20 x 20 cm glass plates, a commercial spreader, and Silica gel GF 254 (Acc. to Stahl). These materials are available from Brinkman Instruments, Inc., Westbury, N.Y., or similar firms. The commercial plates are stored in their original containers, whereas prepared plates are dried to room temperature for 3-5 hours, then stored in an oven at 110° until used.

EXPERIMENTAL

Δ^9-TETRAHYDROCANNABINOL FROM MARIJUANA AND HASHISH. The extraction procedure used for these materials depends entirely on the form and strength of the sample. In general 0.5 to 1.0 gm is sufficient; less material is needed with particularly strong hashish samples, while considerably more may be needed with abnormally weak marijuana samples. In any case, it is advisable to examine the sample by analytical TLC before attempting preparative TLC on it; such an examination not only indicates the strength of the sample, but also indicates the amount of Δ^9-THC relative to other components. If a sample is strong in other components but rather weak in Δ^9-THC, our experience has indicated that extra care in streaking the plates is needed to separate this particular constituent from the others.

Crude vegetable material or resin is placed in a test tube, covered with 5-10 ml of chloroform (reagent grade) and allowed to stand for about ½ hr. The resulting extract is filtered through glass wool to remove most of

the solid material, and the filtered matter is washed with a second 5-10 ml portion of chloroform. The filtrate is reduced in volume to 1-2 ml by blowing a gentle stream of air across it.

The extract is taken up into a small pipette (1-2 ml is sufficient) and streaked on to a 20 x 20 cm plate (thickness 0.5 mm) so that the material to be purified occupies only a thin straight line (width less than 0.5 cm) about 1.5 cm from the bottom of the plate. The plate is developed in reagent grade benzene for approximately 0.5 hr. or until the solvent front is about 1 cm from the top of the plate. The plate is then removed from the tank and air-dried.

Visualization of the plate is accomplished by partially covering the plate to be visualized with a second clean glass plate as shown in figure 1. The remaining exposed portion of the plate (which represents about 10% of the area of the plate) is sprayed with Duquenois reagent (made from 10 gm vanillin and 5 cc acetaldehyde diluted to 500 cc with ethanol, then diluted to 1 liter with concentrated hydrochloric acid) until a series of colors appears. Four bands are commonly observed at intermediate R_f values: one or two bands near R_f 0.5 - 0.6 and two bands near R_f 0.3 - 0.4. The second of the upper two bands is Δ^9-THC (see figure 2).

The remainder of the plate is then visualized under ultraviolet light to make certain that the desired band of material crosses the plate in essentially a straight line. The desired band is outlined with a pencil and scraped from the plate with a spatula or razor blade. Do not include that portion of the plate which has been sprayed with the Duquenois reagent. The material from the plate is placed in a vial and covered with 2-3 ml of spectrograde chloroform, then filtered through a small amount of sodium sulfate. The filtered material is washed a second time with 2-3 ml of chloroform, the filtrates are combined, and the solvent is removed under a gentle stream of air. The resulting oil is transferred to salt plates for infrared analysis. The spectrum obtained in this manner is virtually identical with a spectrum of Δ^9-THC previously published in this publication[1] (see figure 3).

MESCALINE FROM PEYOTE. About 0.5 - 1.0 gm of plant material (one peyote button or less) is crushed and mixed with several ml of water. Enough conc. NaOH is added to make the solution distinctly basic, and the resulting mixture is extracted with two 10 ml portions of chloroform. The extract is reduced in volume to about 1 ml under a stream of air, taken up into a small pipette, and streaked on to a 20 x 20 cm plate (thickness 200 microns) so that the material to be purified occupies a thin straight line near the bottom of the TLC plate.

The plate is developed in Davidow's solution (ethyl acetate/methanol/ammonium hydroxide - 85:10:5) until the solvent front reaches the top of the plate (about 1 hr.). After drying, the plate is visualized by spraying with iodoplatinate solution (made by dissolving 2 gm of chloroplatinic acid in 200 ml water, then adding 20 g potassium iodide and diluting to

1 liter with water), and the lower streak is marked (R_f approximately 0.2; it is wise to run standard material under similar conditions to judge how your own system works). The plate is subjected to ammonia vapor to remove the iodoplatinate, and the marked area is scraped off the plate. This material is dissolved in a few ml of water which is made basic with ammonium hydroxide and a few drops of NaOH. Extraction with 10 ml of chloroform, evaporation to near dryness, addition of a few drops of conc. HCl, and further evaporation to complete dryness leads to a solid suitable for infrared analysis. The spectrum obtained in this manner is identical with that of mescaline hydrochloride obtained from the Aldrich Chemical Co., Milwaukee, Wis. (figure 4).

SEPARATION OF AMPHETAMINE MIXTURES. Mixtures of amphetamines, or mixtures containing an amphetamine, may be separated in a manner completely analagous to that just described for mescaline. Use enough material to yield about 5-10 mg of the amphetamine, about 1 tablet or capsule. The developed plate may be visualized by either iodoplatinate spray or ultraviolet light. In either case standard material should be examined by analytical TLC to determine the R_f value of the material being purified. This method is also useful for purifying amphetamine preparations containing basic chloroform soluble dyes or excipients.

SEPARATION OF BARBITURATE MIXTURES. One tablet or capsule is crushed, mixed with a few ml of water made acid with a few drops of conc. HCl, and extracted twice with 5 ml of chloroform. The extract is reduced in volume to about 1 ml and streaked on to a 20 x 20 cm plate (thickness 200 microns). The plate is developed in a 1:1 mixture of isopropylether/chloroform for at least 2 hrs. (the solvent front will reach the top of the plate long before this time) and visualized by ultraviolet light (the desired bands will absorb against the fluorescent background). Standard materials should be examined to determine the R_f values of the materials being purified by this method. The desired bands are scraped from the plate, separately mixed with 5-10 ml of acidified water, and each extracted with two 10 ml portions of chloroform. Evaporation of the solvent leads to solids suitable for infrared analysis. This method is particularly useful for mixtures of amobarb/secobarb, phenobarb/secobarb, and phenobarb/other acid extractable compounds.

References

1) A. R. Sperling, Microgram, Vol. II, No. 2 Pg. 37 (1969)

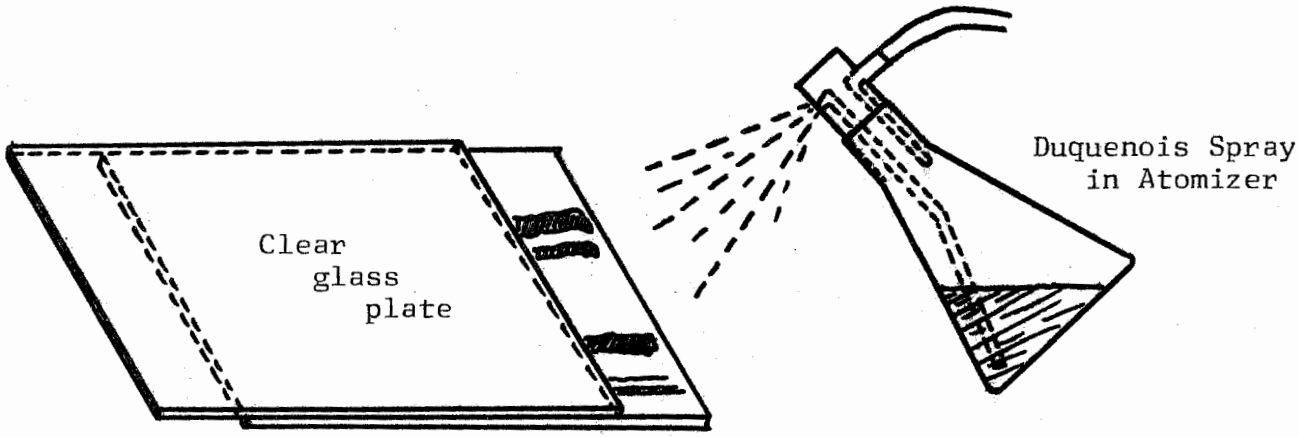


FIGURE 1. VISUALIZING THE DEVELOPED MARIJUANA PLATE.

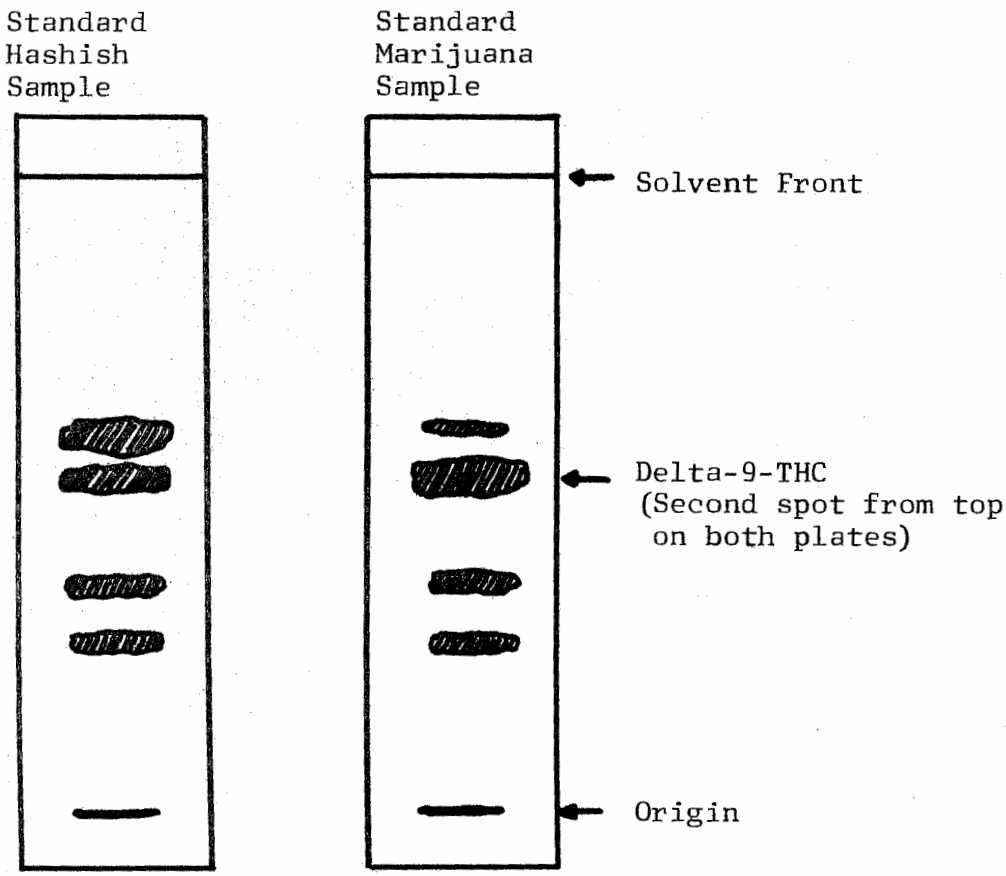


FIGURE 2. LOCATION OF SPOTS ON THE VISUALIZED MARIJUANA PLATE.

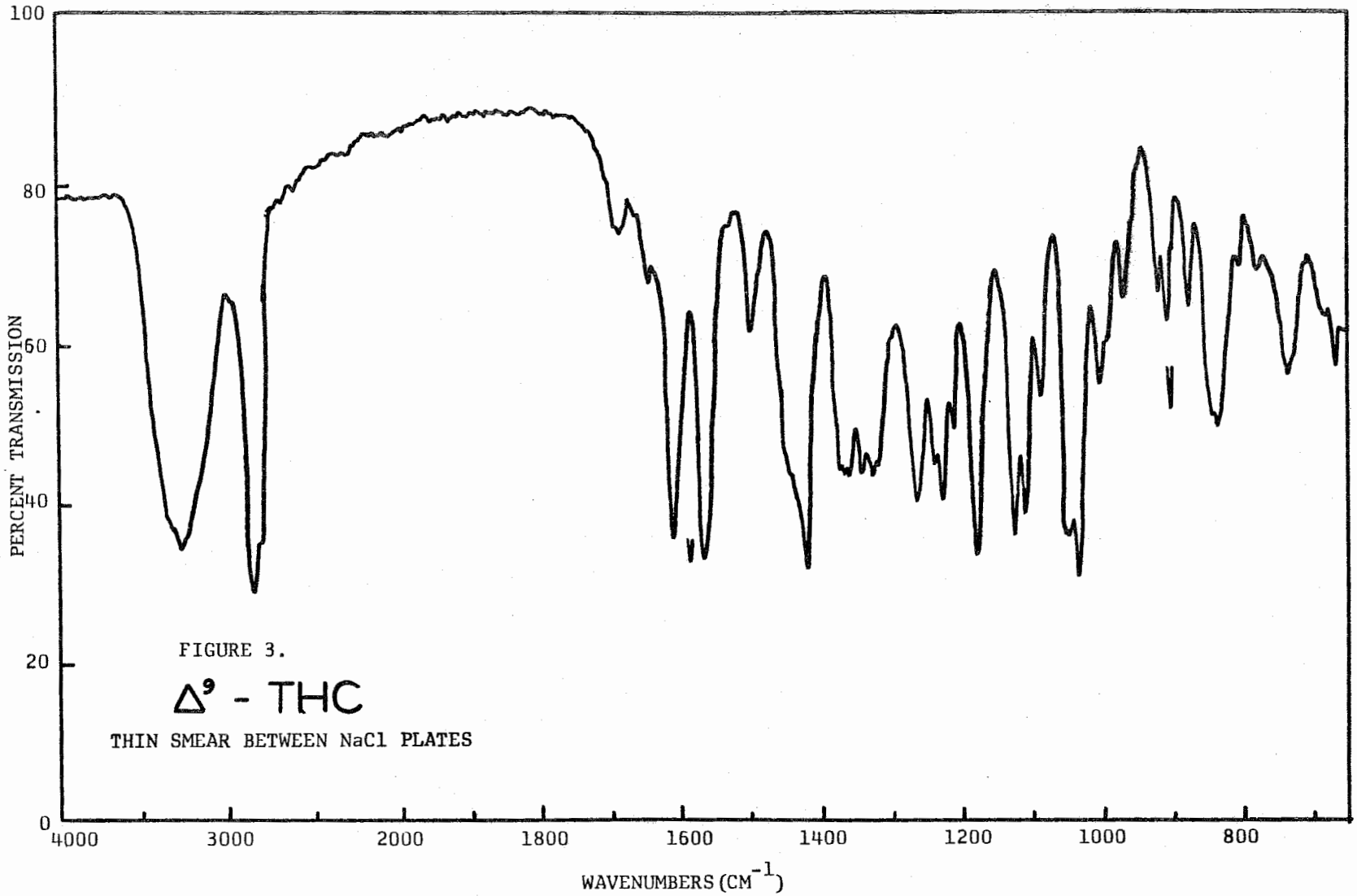


FIGURE 3.

Δ^9 - THC

THIN SMEAR BETWEEN NaCl PLATES

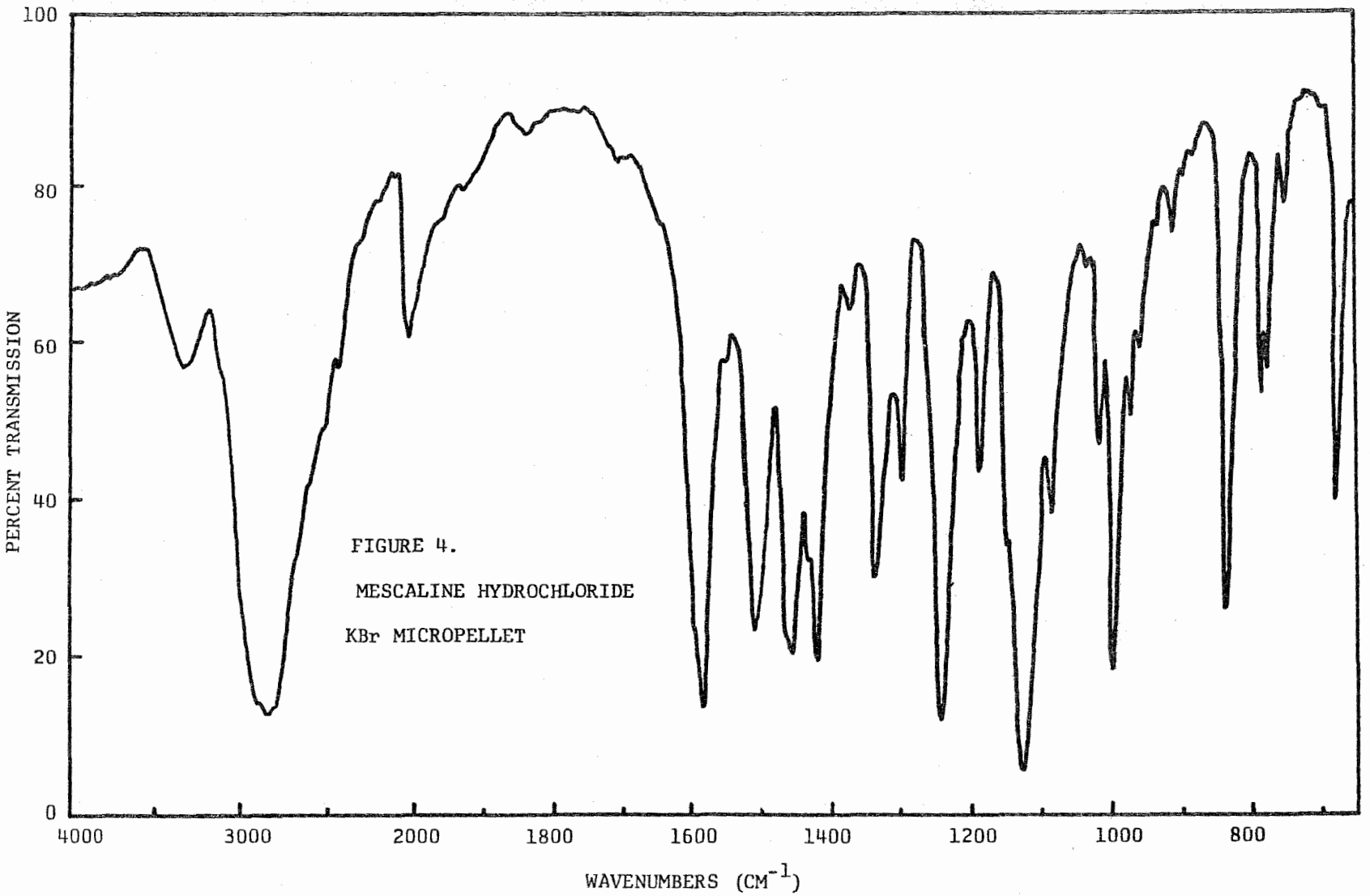


FIGURE 4.

MESCALINE HYDROCHLORIDE

KBr MICROPELLET