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. III, No. 7 November, 1970

President Nixon visited BNDD headquarters on October 27, 1970 to sign the Controlled Substances Act into law (Public Law 91-513). The President was accompanied by Attorney General John Mitchell; Chief Counsel to the President, John Dean; and other officials from the White House and the Department of Justice.

BNDD's thanks to the many state and local officials who furnished information on their laboratory's methamphetamine workload. The amount and kinds of drugs analyzed in local laboratories are valuable intelligence to us. Therefore, we would appreciate copies of analytical workload reports.

BNDD laboratories analyzed 19,731 exhibits during the first fiscal year's operation. Work for other federal agencies and state and local agencies accounted for 14,412 analyses. This was during a period when personnel were being hired, equipment was being acquired, and the enforcement philosophy of the new Bureau was being formed. (One analysis is considered to be all of the examinations or determinations necessary to identify one drug exhibit.)

Analyses included the following:

Heroin	4986
Marihuana	4112
LSD	1854
Amphetamine	729
Barbiturates	611
Cocaine	573
Methamphetamine	418
Methadone	309
Phencyclidine HCl	219
STP	108

Analyses of non-controlled drugs included the following:

Propoxyphene HCl	110
Chlordiazepoxide HCl	80
Diazepam	49
Meprobamate	20
Hawaiian Baby Wood Rose	12
Pentazocine	7
Thioridazine	3

BNDD Special Testing and Research Laboratory is identifying new LSD tablet presses at the rate of ten presses each month. This is partly due to cooperating local police departments who take the trouble to send us material for ballistics examination. In all cases, a report is made to the submitting agency. From these submissions, in the case of clandestinely made tablets, we can determine those from a common source; an indication of the extent of distribution and output of the press; and names and addresses of those involved in the distribution. This information, of course, opens the door to a wealth of additional information. In the case of legitimately manufactured tablets, if we have an authentic sample, the laboratory can determine the manufacturer and the product. Accumulation of this information leads to a measure of the amount and extent of diversion of commercial products.

MDA (3,4-methylenedioxyamphetamine) tablets have been encountered in our Washington Regional Laboratory. These are the first tablets of the compound reported in BNDD. They contained from 96 to 108 milligrams MDA.

LSD-Benactyzine tablets have also been seen for the first time in BNDD's Washington Regional Laboratory. The tablets are round, biconvex, uncoated, unscored, unmarked, and peach color. Average weight is 325 milligrams. They have a diameter of 11.0 millimeters and a depth of 4.3 millimeters. The tablets contained 84 micrograms of LSD and 1.3 milligrams of benactyzine (calculated as the HCl). Excipients were spray dried milk and dolomite.

Cocaine adulterated with 19.8% caffeine has been encountered for the first time by the BNDD Washington Regional Laboratory. The evidence also contained dextrose, boric acid, and lactose.

Material sold as "pure heroin" was identified by the BNDD San Francisco Regional Laboratory as l-narcotine. The evidence submitted by an Alaskan law enforcement agency was a dark brown granular material containing 6.8% l-narcotine by weight. L-narcotine, also known as noscapine, is a naturally occurring opium alkaloid. It is removed from opium after the morphine and codeine are removed, and occurs in amounts varying from 1 to 12 percent, depending upon where the opium poppies

were grown. It has also been reportedly found in Rauwolfia heterophylla. Noscapine is a non-addicting antitussive drug equivalent on a weight to weight basis to codeine, yet lacks the adverse central or gastrointestinal effects of either codeine or morphine.

"Red Rock" may now be purple, brown, or black, we hear. All of the materials apparently contain the same constituents regardless of color. The material is said to be either "snorted," chewed, or mixed with marihuana and smoked.

Barbiturate - "red devils," "Mexican reds," or "red lillies" abuse has become popular among teenagers in the Northwest, according to the local press. The director of a clinic reportedly feels that the popularity of the drug has become widespread in the past six months. One official is said to feel that the increased abuse stemmed from a rock festival held in July. The number of drug injuries are unknown, but include an eleven-year-old boy hospitalized four days after taking a candy coated barbiturate tablet. NOTE: One scientific authority reportedly found strychnine in red-coated secobarbital tablets.

MEETINGS

American Academy of Forensic Sciences annual meeting, Phoenix, Arizona, February 21-26, 1971. Meeting with the Academy are the British Academy of Forensic Sciences, the Canadian Society of Forensic Sciences, the National Association of Medical Examiners, the National Association of Firearm and Tool Mark Examiners, and the new Forensic Sciences Committee of the American Society for Testing and Materials.

"Forensic Sciences and the Environment" is the general theme of this year's meeting.

Monday's topic is "Forensic Sciences in a Closed World Ecosystem." Speakers are: President Edwin Conrad, Professor Francis Camps from England, Dr. Mark M. Luckens, Dr. Charles G. Wilber, Dr. Emanuel Tanay, Dr. Seymour Pollack, Dr. William G. Eckert, and Attorney Robert G. Begam.

"Forensic Sciences and the Environment of the Young Adult" is the title of the Wednesday session. Speakers: Douglas Paxton, Captain Joseph P. McNally, Ralph E. Turner, Professor Albert Picchioni, Dr. Irwin N. Perr, Dr. Charles S. Petty, Dr. Thomas Marshall of Belfast, Ireland (who will discuss "Belfast Rioting"), Hon. Earl D. Morton, and Oliver Shroeder, Jr. A panel discussion will follow.

The plenary session on Friday is entitled, "The Forensic Sciences and the Social Order: Judicial and Administrative Reforms: Directives for the Future." Speakers: Attorney Andre A. Moenssens, Professor

Fred Spies, Attorney Henry Rothblatt, Dr. Douglas Goldman, Ordway Hilton, Joseph D. Nicol, and Arthur J. Bilek.

Section meetings will be conducted on Tuesday and Thursday mornings. Principle topics for the Jurisprudence Section are "Environmental Control," "Alcoholism," and "Drug Addiction." "Autopsy Rationale" and "Penurious Toxicology" are the subjects in the Toxicology Section. The Questioned Documents, Pathology & Biology, General, and Psychiatry sections will also have full programs. The theme for the Criminalistics Section is "Narcotics and Drug Analyses and Methodology."

Besides reports of the Methods Committee, the Criminalistics Section will hear papers on "Physical Evidence Input to the Criminalistic Laboratory," "Systems Analysts Look at the Crime Laboratory," "Trends in Criminalistics Research," "Soil Samples: Their Examination, Comparison, and Evidential Value," "Thin Layer Chromatography Identification of LSD," "An Approach to Automated Drug Identification," Comparative TLC of Cannabis Resin and Plant Material," and "The Forensic Application of Combined Gas Chromatography - Mass Spectrometry for the Screening and Identification of Drugs and Narcotics."

A special symposium on Thursday afternoon will discuss "The Battered-Child Syndrome." Professor Francis Camps and Dr. C. Henry Kempe, University of Colorado, are two of the participants.

Evening programs are also being planned. The Toxicology Section has three scheduled evening sessions. Monday evening, June Jones will moderate an open discussion on the subject of "Toxicology Registry of Non-Fatal Overdosage." This will be followed by a presentation by Leo Dal Cortivo on "Spectrofluorimetry: Its Significance to Analytical Toxicology." On Tuesday evening, Dr. Robert Forney will moderate "Toxicologists in Training." On Wednesday evening, the Section will discuss "G.C. - M.S. Is This a Practical Tool for Toxicologists?"

On Wednesday evening, the Jurisprudence Section and Psychiatry Section will jointly discuss "Critical Evaluation of Psychiatric Evidence." Participants will include: Hon. Robert Kingsley, Associate Justice of the California Court of Appeal, and Professor Henry Weihofen, University of New Mexico Law School.

The Criminalistics Section will hold a workshop on "Narcotics and Drug Analyses and Methodology" on Wednesday evening. Topics will include "The Forensic Science Laboratory and Drugs of Abuse," "Extraction Procedure for the Quantitative Analysis of Marihuana," "The Determination of Hallucinogenic Drugs by Gas Chromatography, Specifically 'PCP' and 'JB' Compounds," "Thin Layer Chromatography of Lysergic Acid Derivatives," "Spectrophotometric Assay for Heroin amd Methamphetamine in Illicit Separations," "A Rapid Method for the Determination of Excipient Sugars in Illicit Heroin Samples by Gas Chromatography," and "Microcrystal Tests for Some Drugs of Abuse."

On Thursday, the Criminalistics Section will hear papers on the "Diffrentiation of Microgram Quantities of Acrylic and Monacrylic Fibers Using Pyrolysis Gas-Liquid Chromatography," "Sexing Human Hair by Use of Sex Chromatin Bodies," "Comparative Electromagnetic Radiation Studies in Criminalistics," "A Forensic Application of the Chronograph," "Identification of Arson Accelerants by Gas Chromatographic Patterns Produced by a Digital Log Electrometer," "Some Recent Applications of Nuclear Analysis Techniques to Typical Forensic Evidence," "New Activation Analysis Studies of Hair - Based Upon Very Short-Lived Induced Activities," "A Study of Public and Official Perception of Laws Dealing With Alcoholically Impaired Drivers - Interim Observations," and "Photographic Evidence and the Assassination of President Kennedy."

Two special sessions of the Academy are scheduled for Monday and Tuesday evenings. On Monday evening, Dr. Charles S. Petty will conduct a discussion on various pathology problems. On Tuesday evening, Attorney Melvin Belli will be leader of a session on "Controversial Medico-Legal Scientific Subjects."

The noted author, Attorney John P. Frank of Phoenix will be the principal speaker at the Annual Banquet.

Other events include a special Wednesday tour for ladies, the Academy luncheon, a fellowship hour, and a barbecue. Advanced registration is $50.00. Forms are available from Arthur H. Schatz, J.D., Secretary-Treasurer, American Academy of Forensic Sciences, 750 Main Street, Suite 1000, Hartford, Connecticut 06103. Registration after January 20, 1971, or at the door will be $60.00. Prompt registration is advised in order to make early reservations for accomodations.

BNDD Forensic Chemist Seminars are planned for:

February 8 - 12, 1971
April 12 - 16, 1971
June 14 - 18, 1971

All sessions are held at BNDD Headquarters, Washington, D. C. and are free of charge. Participants must furnish their own transportation, meals, accomodations, and incidental expenses.

The course is intended primarily for forensic chemists analyzing drugs in crime laboratories. Applications may be obtained from:

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

Microgram is primarily intended for the chemist in the crime laboratory concerned with drug analysis although we have printed material of interest to other disciplines. As we have said in the past, methods in Microgram are not official nor have they been formally validated. And we say again, publication in Microgram does not preclude later publication in a professional journal. Bearing the foregoing in mind, we invite material for publication in Microgram--it is not confined to BNDD chemists.

1. Analytical methods.
2. Comments on any of the methods previously published. Do they work? If they do not, what is wrong and what will correct the method?
3. Material for the glossary, preferably submitted on 3"x5" cards. (We plan to print the glossary as a separate document eventually.)
4. Notice of meetings.

Fire Protection Guide on Hazardous Materials published by the National Fire Protection Association, 60 Batterymarch Street, Boston, Mass. 02110, has been revised and the third edition is now for sale at $5.50 per copy. This paperback book is a quick guide to hazardous chemicals found in the laboratory. It is also convenient to take along when police officers request help with a clandestine laboratory seizure.

BNDD LABORATORY NOTES

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DATE November 4, 1970

NO. 11

DRUG TYPE Hallucinogen

METHODOLOGY

DETERMINATION OF LSD IN ILLICIT PREPARATIONS BY FLUORESCENCE SPECTROSCOPY

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Methods and conditions for obtaining the fluorescence of lysergic acid diethylamide (LSD) have been reported by Axelrod,[1] Udenfriend,[2] and others.[3-6] The fluorescence technique is especially suited to the determination of LSD because of the extremely high sensitivity of the method. This study was conducted to determine the feasibility of routine quantitative analysis of LSD preparations encountered in a forensic science laboratory.

LSD solutions appear to decompose under the influence of UV light, following apparent first order kinetics (Figure 1). A typical breakdown pattern in methanol showed that about half of the LSD had decomposed in about 44 minutes. The amount decomposed during the elapsed time for a typical scan was about 2-4%. Removal of the cuvette from the light source for ten minutes failed to bring the fluorescence back to its original intensity as reported by Axelrod.[1] By decreasing the amount of exciting light (narrowing the excitation bandpass) the reaction, as expected, slows considerably to the extent that breakdown is negligible during the scan time.

Studies were also conducted in 0.004 N HCl and distilled water (Figure 2). The dilute acid medium promoted rapid breakdown; meanwhile, water proved to be the most stable medium. Despite this fact, methanol was considered to be the most suitable medium for directly assaying LSD preparations, fluorimetrically, for the following reasons:

1. Since the Raman peak is considerably smaller with methanol than with aqueous solutions, interference from the Raman peak, as an additive effect, is less likely to occur in methanol that in aqueous media, when operating at high sensitivities.

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* Presently located in Laboratory Operations Division, Washington, D.C.

2. Methanol is superior to aqueous media for direct extraction of LSD. Also, the solutions scanned on the fluorimeter can be recollected and evaporated to dryness quite easily from methanol for further work with no need for extractions.

3. The absorptivity of LSD in methanol is about 10% greater than in dilute acid. Since fluorescent activity is proportional to absorptivity, a slight enhancement of sensitivity is obtained in methanol.

EXPERIMENTAL

Apparatus:

A Perkin-Elmer Hitachi Model MPF-2A spectrophotofluorimeter was used with the following parameters:

Excitation Wavelength	318 mu
Emission Wavelength	400 mu
Excitation Slit	6 mu
Emission Slit	10 mu
Sensitivity	Variable

Procedure:

Accurately weigh a portion of the ground sample powder into a volumetric flask. Add a few mls. of methanol to the flask and agitate the solution by swirling or, mechanically. Dilute to volume with methanol. Filter a portion of this solution into a glass-stoppered flask discarding the first few mls. of filtrate. Prepare a standard solution of LSD in methanol in which the final concentration will be between 0.025 - 0.1 mcg./ml. Use the final standard solution to adjust the instrument at the proper sensitivity. After properly setting the instrument, discard the solution. Check to see if the solution will provide a spectrum that will remain on scale by manually setting the instrument at wavelengths of maximum excitation and emission. If the recorder pen goes off scale, quantitatively dilute a portion of the filtrate until the pen remains on scale.

After recording the fluorescence and excitation spectra of the sample, refill the cuvette with a fresh portion of standard solution and record its spectra. Use the fluorescence maximum, at about 400 mu, for quantitating the concentration of LSD in the illicit sample preparation.

The relationship of fluorescence vs. concentration for LSD is linear from 0.025 - 0.1 mcg./ml. At concentration levels above 0.1 mcg./ml., the relationship ceases to be linear. Opening both bandpasses to 10 mu

and lowering the sample sensitivity to 2 extends the upper limit of linearity to 0.2 mcg./ml.

The simple procedure described above has been successfully employed in this laboratory to analyze even highly colored samples, where the usual extraction procedures failed to remove the color, rendering them difficult to assay by UV or colorimetric procedures. The enhancement of sensitivity permits accurate, rapid analysis at levels as low as a few tenths of a microgram of LSD.

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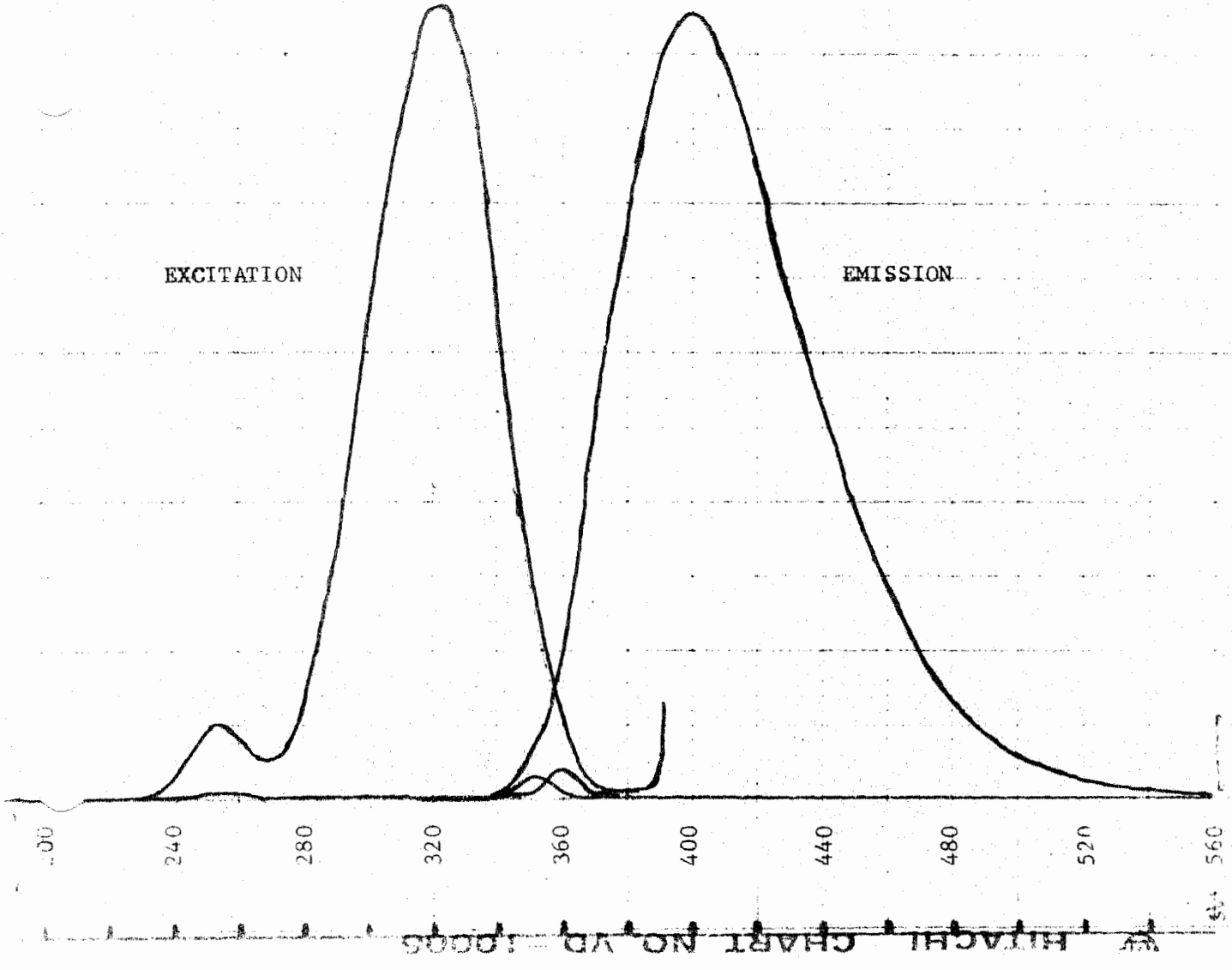


Figure 1

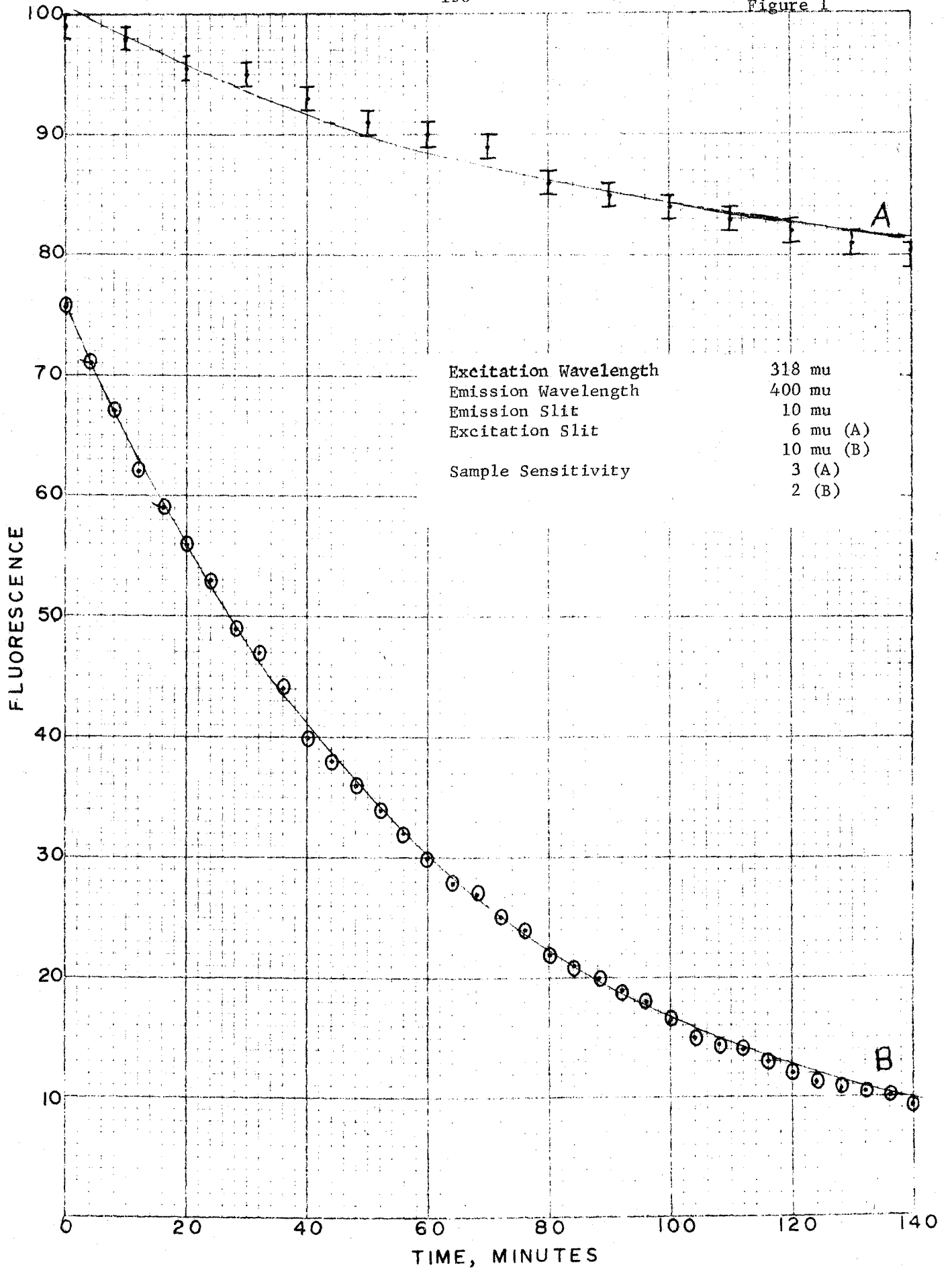
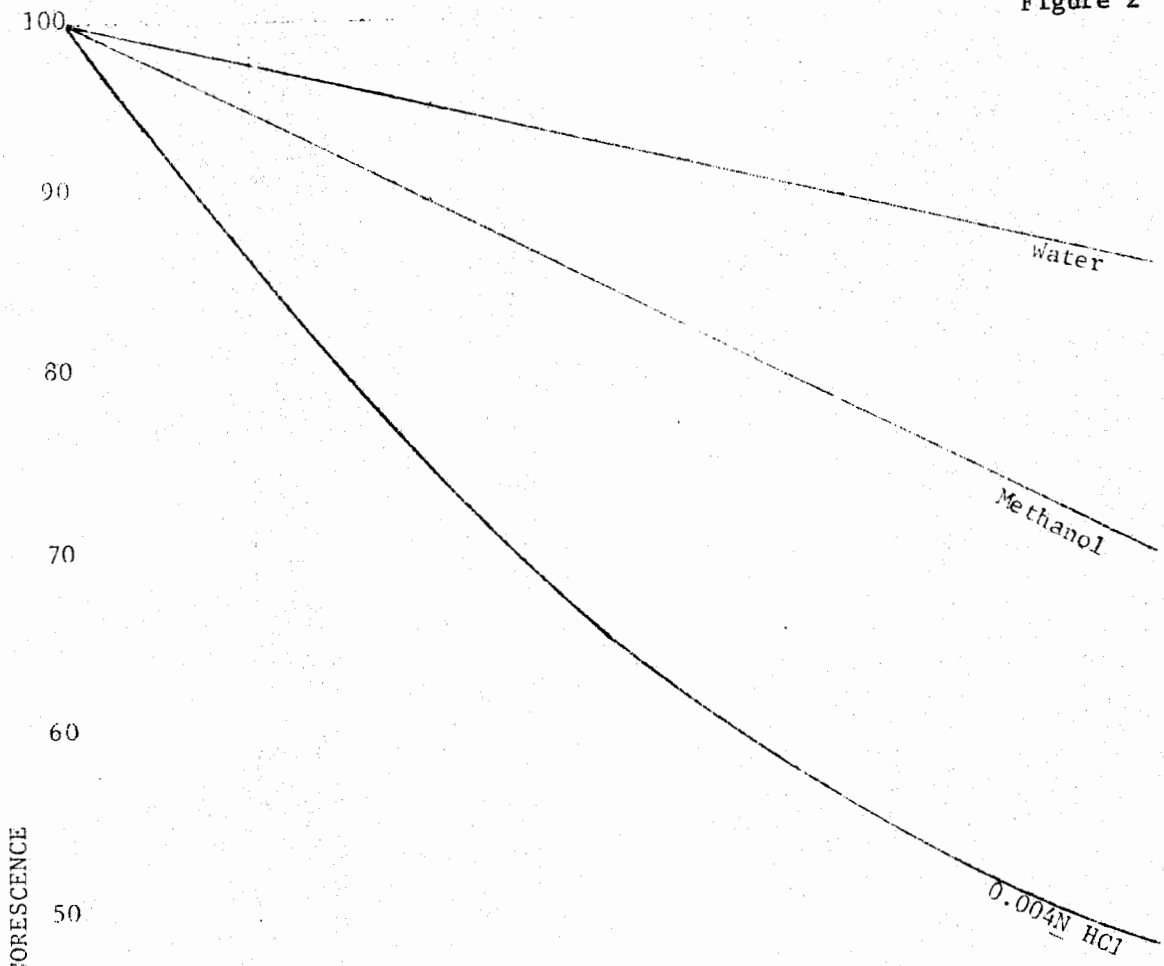


Figure 2



Solution	.004N HCl	MeOH	H_2O
Emission mu (max.)	428	400	424
Excitation mu (max.)	324	318	322
Emission Slit	10 mu	10 mu	10 mu
Excitation Slit	6 mu	6 mu	6 mu