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

Washington, D. C. 20537
Office of Science and Drug Abuse Prevention
Vol.III,No.1 Laboratory Operations Division Jan.-Feb.,1970

Notice to law enforcement agencies in the states of Arizona, Colorado, New Mexico, Utah and Wyoming using the BNDD laboratory. Send all drug evidence to our Dallas Regional Laboratory instead of to the San Francisco laboratory. Mr. James H. Kluckhohn is Chief Chemist of the Dallas laboratory. The address and telephone number are:

Dallas Regional Laboratory
Bureau of Narcotics and Dangerous Drugs
Room 1023
1114 Commerce Street
Dallas, Texas 75202
214 749-3188 or 3160

This change was made to prevent possible future delays in analyses and competition for chemists' time in court. The increasing workload in our San Francisco Regional Laboratory makes this change desirable, and we believe it will be of mutual benefit.

Pentazocine, according to the BNDD Special Testing and Research Laboratory, has an ultraviolet absorption spectrum in 0.1 N Hydrochloric Acid with a maximum at 278. In 0.1 N Sodium Hydroxide, the maxima are at 238 and 297. It appears that the values have been reversed in Isolation and Identification of Drugs, edited by E.G.C. Clarke, The Pharmaceutical Press, London, England.

15% of the marihuana examinations by BNDD laboratories have been negative. The plant material identified has included alfalfa, hay, tobacco, catnip, stramonium, indian hemp, Mate', and a member of the St. John's Wort family.

Lysergic Acid Diethylamide (LSD)-Phencyclidine HCl (PCP) combination capsules continue to be encountered. They have contained white, pink, or yellow powder. LSD content has been approximately 100 micrograms, with only a trace of PCP. It is interesting that the "Berkeley Barb", October 3-9, printed an alleged interview with a "dope-dealer" who was bemoaning the poor quality of LSD and other drugs on the street. He claimed that many of them were combinations of LSD, STP, "Speed" or PCP.

LSD in coca was recently analyzed. The 450 grams seized contained about 94 milligrams of LSD, or the equivalent to 100 micrograms in 0.5 grams of powder.

From "Drug Intelligence," No. 1, November, 1969, the newsletter from the State of Washington Board of Pharmacy:

'"Crank" is another term being used for Methamphetamine crystals.

"TMA" found usually as a small red capsule similar to seconal capsules, reported to contain THC, Mescaline and LSD. Probably contains just LSD...

"Greendomes", small green tablets with a dome on one side. Contain weak LSD and is selling for $3.00 to $5.00 on the street for one hit.

A white tablet has been appearing in Seattle with double scoring which makes it appear to be a dl-amphetamine tablet (Benzedrine) has been identified as a vitamin tablet. It appears that the dealer purchased large white vitamin tablets with a thin ½ scoring mark. He "filed" two deep grooves and beveled the edges so that they are easily mistaken for an amphetamine tablet.'

Canadian Society of Forensic Science Journal, pages 106-107, carried a "Message from the President." In part, he said:

"An acknowledgment of accomplishments should always be tempered with a warning about becoming too complacent. No matter how well the forensic scientist has developed a procedure he should never be completely satisfied with it. There was a time when detection of the presence or absence of a foreign substance was regarded as pretty sensational. In most areas this has been replaced by quantitative analysis with its subsequent advantages. However, there are areas where analytical techniques have improved to the point where ultra micro amounts of material may be detected. Sometimes the amount of material detected with our sophisticated equipment is below the levels which would be of practical significance and therefore the analyst must present the results in a quantitative manner. Furthermore, failure to employ standards of known composition, modern quality control, and proven analytical techniques, will only result in ultimate discredit to an analyst and perhaps an entire laboratory.

With the ever increasing use of forensic sciences in our courts, every effort must be made to ensure that only the very best possible results are made available to those charged with the responsibility of making decisions in our courts. Always strive towards perfection."

Ether, is a well known fire hazard. A greater hazard exists in peroxide formation. An apparently empty bottle of ether, after evaporation of the liquid, or a partially filled bottle, may contain enough explosive peroxide to seriously injure personnel or cause loss of life. Peroxide formation is not limited to previously opened bottles. It may occur in sealed bottles, although the hazard is not nearly as great.

With the increasing number of clandestine laboratory seizures, this problem requires immediate attention, since peroxide may be detonated merely by placing the container on the table.

Containers known to have been full, or partially full, and are now empty, should be treated as explosive charges. Careful addition of water to the bottle will lessen the potential hazard. Addition of a piece of copper wire to partially full bottles of ether should be standard procedure immediately upon storage of ether. The copper wire will retard peroxide formation and water prevents detonation.

Laboratory stocks of ether should be dated upon receipt, and the oldest container should be used first.

Boston Laboratory, Division of Food and Drugs, Massachusetts Department of Public Health, has sent us their fiscal year report. For fiscal year 1964-1965, 834 samples were analyzed. In fiscal year 1968-1969, 21,995 samples were analyzed for several agencies. The report cannot be reprinted in its entirety, however, the most significant analyses are:

Material Analyzed	Number of Samples	Number of Drugs Analyzed
Green Herbs	5,624	5,245 cannabis
Brown Material	446	353 cannabis
Black Material	10	4 opium
Residue	4,721	1,930 cannabis
		682 opium derivatives
		233 amphetamine
Sugar Cubes	8	4 LSD
Paper	1	1 LSD
Candy	10	1 LSD
Powder	6,634	5,235 Heroin
		435 amphetamine
		394 cocaine
Liquid	374	144 acetone
		34 toluol
		30 codeine
Tablets	2,531	289 LSD
		249 amphetamine
		161 barbiturate
Capsules	1,610	370 barbiturate
		245 amphetamine
		108 amphetamine and barbiturate

Amphetamine abuse appears to be of increasing concern.

"U.S. Medicine," Vol. 5, No. 24 (December 15, 1969) carries a front page interview with medical educator Charles Watkins, M.D., New Orleans, about the use of amphetamines and other stimulants by students.

Dr. George D. Lundberg presented a paper at the American Society of Clinical Pathologists, Chicago, Illinois, on amphetamine-induced disease based on 100 admissions to the Los Angeles County-University of Southern California Medical Center.

"U.S. News and World Report" carried portions of testimony before the House Select Committee on Crime, November 18, 1969, San Francisco, California. Under the head, "No Other Drug Has This Wide a Group of Hazards," testimony by Dr. George R. Edison, Director of Student Health Service and Chairman of the Board of Trustees of the Community Drug Crises Center, University of Utah, is reported. Testimony by Dr. Benjamin Sheppard, Executive Director of the Catholic Services Welfare Bureau for the Archdiocese of Florida is published under the quote: "Alarming Increase in Number of 'Speed' Abusers."

The "Berkeley Barb," November 28 - December 4, carried an item pointing out the dangers of amphetamines.

Forensic Chemists' Seminar for state and local chemists will be held April 27 - May 1, 1970. For information and application blanks, write:

Bureau of Narcotics and Dangerous Drugs
Washington, D.C. 20537
Attention: Special Programs Division, TRNS

American Society of Pharmacognosy, consists of professionals interested in the field of botanical drugs. Annual meetings are usually held at one of the large universities. Many important papers on the botanical hallucinogens will be found in the society's quarterly publication Lloydia. Lloydia is also a source for information on alkaloids and other constituents of pharmaceutical interest. Membership is $15.00 per year including a subscription to the journal.

A 250 page supplement to Lloydia, Volume 33, "Alkaloid-Bearing Plants and their Contained Alkaloids," is being published by the society. The cost is $6.00 for nonsubscribers, or $3.00 for subscribers. Checks or money orders should be sent to the American Society of Pharmacognosy Treasurer, Dr. Leonard Worthen, University of Rhode Island, Kingston, Rhode Island 02881, U.S.A.

Membership in the society is $15.00 per year, including subscription to the journal. Application should be sent to:

Rolf S. Westby, Secretary
The American Society of Pharmacognosy
Eli Lilly and Company
740 South Alabama Street
Indianapolis, Indiana 46206

Meetings:

Second World Meeting on Medical Law, Washington, D.C., August 18 - 21, 1970. Contact: R. Dierkens, Dr. Jur., Agrege Law Facility, Secretary General, 5 Apotheekstraat B-9000 Ghent, Belgium.

The Western Conference on Criminal and Civil Problems, Wichita, Kansas, May 14, 15, and 16, 1970. Contact: W. G. Eckert, M.D., Saint Francis Hospital, Wichita, Kansas.

Microgram distribution is guided by two major factors. First, the Bureau of the Budget limits the number of copies that can be printed per year. Second, some of our identification and analytical methods are restricted because the unscrupulous chemist might be able to use them to our disadvantage. Although Microgram is intended primarily for forensic chemists in laboratories of law enforcement agencies, we include all forensic scientists serving law enforcement agencies, because of the need of forensic toxicologists, pathologists and others for the information in Microgram.

THIN LAYER CHROMATOGRAPHIC IDENTIFICATION OF DMT, DET, AND DPT

Albert Sperling Ph.D.
Forensic Chemist
Special Testing and Research Laboratory
Bureau of Narcotics and Dangerous Drugs

Although samples of DMT (N,N-Dimethyltryptamine), DET (N,N-Diethyltryptamine) and DPT (N,N-Dipropyltryptamine) can be identified by their infrared spectra, thin layer chromatography represents a very rapid method of identification. Utilizing silica gel G plates of 250 micron thickness, the following solvent system is used to separate DMT and DET.

Ether	24
Methanol	3
Ammonium hydroxide	0.3

The plates are visualized with a spray prepared as follows:

p-Dimethylaminobenzaldehyde	2 Gm.
Ethanol	50 ml.
Hydrochloric acid	50 ml.

The compounds appear as bluish-purple spots. DMT has an Rf of .4 and DET has an Rf of .6 in this system.

With the suspected appearance of DPT, a modification of this system was needed in order to provide reasonable Rf values for all three compounds.

The following two systems were found to be satisfactory.

System 1

Ether	24
Methanol	1
Ammonium hydroxide	0.2

The Rf values for the compounds in this system are as follows:

DMT	.17
DET	.31
DPT	.78

System 2

Ether	25
Acetone	5
Ammonium Hydroxide	0.2

The Rf values for the compounds in this system are as follows:

DMT	.19
DET	.38
DPT	.78

The plates may be spotted directly after a simple extraction of the sample with benzene or hexane.

ANALYSIS OF LSD BY COLUMN CHROMATOGRAPHY

Albert Sperling Ph.D.
Forensic Chemist
Special Testing and Research Laboratory
Bureau of Narcotics and Dangerous Drugs

Many types of LSD samples cannot be quantitated or identified by a simple shakeout procedure because of interfering substances which may be present. Column chromatography must then be applied to clean up and isolate the sample.

Procedure

Preparation of Columns

Place a pledget of glass wool at the bottom of a chromatographic column 25 mm x 250 mm. Thoroughly mix the appropriate immobile aqueous phase with acid washed celite 545 (Johns Manville Co.) and transfer to the column. Tamp down moderately until a uniform mass in obtained. Place a pledget of glass wool on top of the celite layer.

Preparation of Standard Solution

Accurately weight approximately 300 micrograms of LSD tartrate and dissolve in 3 ml of water.

Make the solution basic by adding sodium carbonate and then extract three times with 3 ml. portions of chloroform into a 10 ml volumetric flask. Bring to volume with chloroform.

Preparation of Sample

Suspend and mix the sample (at least two tablets if available) in a minimum volume of 1% citric acid (approximately 3 ml). Make basic with sodium carbonate and add five to eight grams of celite. Mix thoroughly and transfer to a chromatographic column as described above. This is designated as column I.

Preparation of Column II

Mix 4 grams of celite and 3 ml of 1% citric acid and prepare a column as described above.

Preparation of Column III

Mix 4 Grams of celite and 3 ml of 10% citric acid and prepare a column as described above.

ANALYSIS

Mount the columns in series so that the effluent from column I flows into column II and then into column III. Add 75 ml. of water saturated chloroform to column I, and allow to pass through all the columns. Remove and discard column I. Add 150 ml. of water saturated chloroform to column II and allow to pass through column III. Remove and discard column II. Elute column III with another 50 ml. of water saturated chloroform. Remove column III and extrude the celite into a beaker and then transfer to a separatory funnel with the aid of water. Make the solution basic with sodium carbonate. Filter the residual chloroform layer through glass wool into a beaker. Extract with two more 30 ml. portions of chloroform and filter into the same beaker. Evaporate the chloroform to a convenient volume (usually 10 ml). Transfer to a volumetric* flask, bring to volume, and scan the ultraviolet spectrum of the sample. Determine the absorbance at about 308 mu and compare it with that of the standard. Calculate the quantity of LSD present by the formula:

$$\frac{A_u \times C_s \times .78 \times V}{A_s \times T} = \text{micrograms of LSD free base}$$

* Ideal concentration is 20 micrograms per ml.

where A_u is the absorbance of the unknown solution

A_S is the absorbance of the standard solution

C_S is the concentration of LSD tartrate standard solution in micrograms per ml.

.78 is a factor converting the weight of LSD tartrate to the equivalent weight of LSD free base

V is volume in ml. of the unknown solution

T is the number of tablets or capsules used

Quantitation may also be done colorimetrically. (1)

Thin Layer Chromatography

The chloroform solution is evaporated to about 1 ml. and 10 microliters of standard and sample are spotted on silica gel G 250 micron plates. Two different plates are spotted each using a different solvent system.

System I

Chloroform	9
Methanol	1

System 2

Chloroform (saturated with ammonium hydroxide(	18
Methanol	1

LSD has an Rf value of about .6 in these two systems. The plates are viewed under long wave ultraviolet light and LSD exhibits a blue-purple fluorescence. The plates are then visualized by spraying with p-dimethylaminobenzaldehyde, and LSD appears as a blue-purple spot. This spray reagent is prepared as follows:

p-dimethylaminobenzaldehyde	2 Gm.
ethanol	50 ml.
concentrated hydrochloric acid	50 ml.

It is necessary that both of these systems be used in order to obtain a positive identification of LSD. If an infra-red identification is desired, the chloroform solution is evaporated to dryness and a potassium bromide pellet is prepared. At least 200 micrograms is needed to prepare a satisfactory micro pellet.

REFERENCE

1. Alexander, T., Microgram, Vol. I, No. 2

FEDERAL REGISTER

P. 19654 - 19655

Chapter II—Bureau of Narcotics and Dangerous Drugs, Department of Justice

PART 320—DEPRESSANT AND STIMULANT DRUGS; DEFINITIONS, PROCEDURAL AND INTERPRETATIVE REGULATIONS

Requirements for Exportation of Controlled Substances

On November 7, 1969, a notice was published in the FEDERAL REGISTER (34 F.R. 18042) stating that the Director, Bureau of Narcotics and Dangerous Drugs, pursuant to the provisions of section 701 of the Food, Drug, and Cosmetic Act, 52 Stat. 1055, as amended (21 U.S.C. 371); and under the authority vested in the Attorney General by Reorganization Plan No. 1 of 1968 (33 F.R. 5611) and redelegated to the Director, Bureau of Narcotics and Dangerous Drugs by § 0.200 of Title 28 of the Code of Federal Regulations, proposed to establish defi- [illegible]ures for the exportation of controlled dangerous substances to allow the efficient enforcement of the provisions of section 801(d) of the Act (21 U.S.C. 381).

After reviewing the comments received on the proposed regulations it is concluded that the regulations should be adopted in the form set forth below. Therefore, pursuant to the authority cited in the preceding paragraph, Part 320 of Title 21 of the Code of Federal Regulations is amended by adding to the existing sections the following new section:

§ 320.20 Exportation.

(a) The provisions of section 801(d) of the act (21 U.S.C. 381 (d)), provide that a drug intended for export shall not be deemed to be adulterated or misbranded but that if such an article is sold or offered for sale in domestic commerce, it is not exempt from control. The provisions of Part 370 of Title 15 of the Code of Federal Regulations (15 CFR 370.2), contain the following definition: "*U.S. Exporter*. That person who, as the principal party in interest in the export transaction, has the power and responsibility for determining and controlling the sending of the commodities and technical data out of the United States." Therefore, such exporter, who may be the manufacturer, compounder, processor, wholesaler, or distributor of the controlled substances will be deemed the "U.S. Exporter" under 15 CFR 370.2, and must comply with one of the following alternative procedures in order to insure that controlled substances "intended for export" are in fact exported:

(1) The "U.S. Exporter" will execute the Shipper's Export Declaration, Form 7525–V, if required by 15 CFR 379.1–379.13, and have the controlled substances delivered to a bonded carrier for delivery to the port or border for shipment directly to the consignee in the foreign country without shipping the substances to a "forwarding agent." A copy of the invoice describing the controlled substance must be attached to each copy of the Shipper's Export Declaration and these documents must accompany the shipment. Form 7525–V may be obtained at a cost of $1 per 100 from any local Customs or Department of Commerce Field Office, and assistance in the execution of such Forms is also available at such offices. A "U.S. Exporter" may not, under any circumstances, physically release a shipment of controlled substances to anyone, including the consignee, at his agent, within the United States.

(2) The "U.S. Exporter" may ship the controlled substances to a "forwarding agent" as defined in 15 CFR 379.4(f), who will execute the required Shipper's Export Declaration and further act as an exporting agent for the principal. When a "forwarding agent" is utilized, a copy of the invoice describing the controlled substance must be attached to each copy of the Shipper's Export Declaration and these documents must accompany the shipment. A "forwarding agent" may not, under any circumstances, release a shipment of controlled substances to anyone, including the consignee, or his agent, within the United States. The "forwarding agent" must either deliver the controlled substances to the port or border, or deliver the controlled substances to a bonded carrier approved by the principal for delivery to the port or border.

(b) In the event that controlled substances intended for export by a "U.S. Exporter" or a "forwarding agent" are introduced or delivered into domestic commerce before they are exported, such introduction or delivery shall be considered a domestic sale, delivery, or other disposition of a controlled substance under 21 U.S.C. 360a, and a prohibited act under 21 U.S.C. 331(q)(2).

Any person who will be adversely affected by the regulations may at any time within 30 days from the date of its publication in the FEDERAL REGISTER file with the Chief Counsel, Bureau of Narcotics and Dangerous Drugs, Department of Justice, Room 613, 1405 I Street NW., Washington, D.C. 20537, written objections thereto. Objections shall show wherein the person filing will be adversely affected by the regulations and specify with particularity the provisions deemed objectionable and the grounds for objections. If a hearing is requested, the objections must state the issues for the hearing, and such objections must be supported by grounds legally sufficient to justify the relief sought. Objections may be accompanied by a memorandum or brief in support thereof. All documents shall be filed in six copies.

Effective date. The regulations shall become effective 31 days from the date of publication in the FEDERAL REGISTER, except as to any provision that may be stayed by the filing of proper objections. Notice of the filing of objections or lack thereof will be announced by publicati[illegible] in the FEDERAL REGISTER.

Dated: December 8, 1969.

JOHN E. INGERSOLL,
Director, Bureau of Narcotics and Dangerous Drugs.

[F.R. Doc. 69–14780; Filed, Dec. 12, 1969; 8:45 a.m.]

FEDERAL REGISTER

P. 19660

DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

Food and Drug Administration

[21 CFR Part 3]

TETRAHYDROCANNABINOLS

Investigational-Use Conditions for Hallucinogenic Drugs; Statements of General Policy or Interpretation

Section 3.47 of the regulations under the Federal Food, Drug, and Cosmetic Act provides that no person may sell, deliver, or otherwise dispose of certain named hallucinogenic drugs for clinical testing in man, for tests in vitro or in laboratory research animals, or for clinical investigations in animals until a proposal for such studies has had advance approval by the Commissioner of Food and Drugs. The named hallucinogenic drugs are not generally recognized as safe and effective for any medical purpose but are limited to investigational use. They have been extensively distributed through illegal channels for misuse that has been associated with very serious adverse effects including serious mental changes and psychotic manifestations.

The Director of the Bureau of Narcotics and Dangerous Drugs has requested the Commissioner of Food and Drugs to amend § 3.47 to add to the listing of drugs therein certain tetrahydrocannabinols. Under section 201(v) of the act, these tetrahydrocannabinols were listed previously in § 320.3(c)(3) as having a potential for abuse because of their hallucinogenic effect.

To justify the proposed additional control, the Director, Bureau of Narcotics and Dangerous Drugs, advised that "as the tetrahydrocannabinols are the main active ingredients in marihuana, we rely upon the universal acceptance that there is extensive illicit distribution of marihuana for misuse and that this misuse has been associated with serious adverse effects."

A policy of the Food and Drug Administration is to cooperate fully with the Bureau of Narcotics and Dangerous Drugs in its efforts to control drug abuse. Therefore, pursuant to the provisions of the act (secs. 505, 511(b), 701(a), 52 Stat. 1052, as amended, 1055; 70 Stat. 229; 21 U.S.C. 355, 360a(b), 371(a)) and under authority delegated to the Commissioner (21 CFR 2.120), it is proposed that § 3.47 be amended to add the tetrahydrocannabinols by revising the introductory text of paragraph (a) to read as follows:

§ 3.47 Investigational-use conditions for certain hallucinogenic drugs.

(a) No person may sell, deliver, or otherwise dispose of bufotenine (5-hydroxy-*N*-dimethyltryptamine) and its salts, DET (*N,N*-diethyltryptamine) and its salts, DMT (dimethyltryptamine), DOM (4-methyl-2,5-dimethoxyamphetamine; 4-methyl-2,5-dimethoxy-α-methylphenethylamine; and "STP"), ibogaine (7-ethyl-6,6α,7,8,9,10,12,13-octahydro-2-methoxy-6,9-methano-5H-pyrido [1′,2′:1,2] azepino [4,5-b] indole) and its salts, LSD (LSD-25; *d*-lysergic acid diethylamide), mescaline and its salts, psilocybin (psilocibin), psilocyn (psilocin), and tetrahydrocannabinols (that is, the synthetic equivalents of the substances contained in, or in the resinous extractives of, Cannabis species plants and/or synthetic substances, derivatives, and their isomers with similar chemical structure and pharmacological activity; such as Δ^1 *cis*- or *trans*-tetrahydrocannabinol and their optical isomers, Δ^6 *cis*- or *trans*-tetrahydrocannabinol and their optical isomers, and $\Delta^{3,4}$ tetrahydrocannabinol and its optical isomers (since nomenclature of these substances is not internationally standardized, compounds of these structures, regardless of numerical designation of atomic positions, are covered)):

* * * * *

Any interested person may, within 30 days from the date of publication of this notice in the FEDERAL REGISTER, file with the Hearing Clerk, Department of Health, Education, and Welfare, Room 5440, 330 Independence Avenue SW., Washington, D.C. 20201, written comments (preferably in quintuplicate) regarding this proposal. Comments may be accompanied by a memorandum or brief in support thereof.

Dated: November 10, 1969.

HERBERT L. LEY, Jr.,
Commissioner of Food and Drugs.

[F.R. Doc. 69-14805; Filed, Dec. 12, 1969; 8:47 a.m.]

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