Journal of Forensic Sciences, Vol 15, No 1, pp. 51-64 (1970)

Clandestine Drug Laboratories

John W. Gunn, Jr., B.S., Donald W. Johnson, B.A., and William P. Butler, B.S.

During the past three years agents of the Bureau of Narcotics and Dangerous Drugs have seized 95 illicit laboratories. Many others have been seized by state and local police. These laboratories had capabilities of producing drugs worth up to \$3,000,000 per day on the illicit market. They ranged in size from extensive, well-equipped sophisticated installations to very crude operations carried on in bathrooms, basements, garages, abandoned warehouses—anywhere a manufacturing operation could be established without detection (Figs. 1-3). Quality controls were non-

purity or safety of the products produced. Tables I and II indicate the states where the laboratories were located and the types of drugs involved.

Congress recognized, in the Drug Abuse Control Amendments (DACA) of 1965, the need to control the illicit manufacture, compounding, or processing of depressant, stimulant and hallucinogenic drugs. Along with these controls, this legislation also prohibits the illegal sale, delivery or other disposition of drugs in these three classes.

When the law was passed in 1965, manufacture and sale were regarded as misdemeanors punishable by up to one year in jail or \$1,000 fine, or both. Congress in 1968 through Public Law 90-639, known as the Staggers-Dodd Bill, increased the penalties

for trafficking in dangerous drugs to felonies subject to imprisonment up to five years, a fine up to \$10,000, or both.

Under the original legislation there was no penalty for possession if the drugs were intended for personal use, for use by members of the household, or for administration to animals belonging to the household. The manner in which the drug was acquired did not affect the legality of its possession.

Under the Staggers-Dodd amendments, the manner in which the drug is acquired does affect the legality of its possession. The amendment permits a person to possess only depressant or stimulant drugs obtained directly from a practitioner who is licensed by law to prescribe, or those drugs obtained pursuant to a valid prescription.

Illegal possession for personal use is now punishable as a misdemeanor, with imprisonment of up to one year or a \$1,000 fine, or both. Possession with intent to sell is a felony, with penalties equivalent to those for illegal manufacture or distribution.

The law permits persons who use depressant or stimulant drugs in research, teaching, or chemical analysis to possess these

TABLE I
Clandestine Laboratories and States Where Located

State	1966	1967	1968	Total
California		10	16	26
Colorado		1	1	2
Connecticut			2	2
District of Columbia			1	1
Georgia	1	1		2
Illinois	1		4	2 5
Kentucky			2	2
Maryland		1	2	3
Massachusetts	2	1 5	$egin{array}{c} 2 \\ 2 \\ 3 \\ 2 \end{array}$	10
Michigan			2	2
New Jersey		1		1
New York	2	7	6	15
Oregon			1	1
Pennsylvania		2	5	7
Rhode Island			2	2
Tennessee		1		1
Texas		$\frac{1}{7}$	2	9
Virginia			1	1
Washington			1	1
West Virginia			1	1
Wisconsin		1		1
Total	6	37	52	95

TABLE II

Clandestine Laboratories by Drug Involved and State Where Located

	Hallucino-	.	Methamphet-	
State	gens	Stimulants	amine	Total
California	12	2	12	26
Colorado	2			2
Connecticut	2			2
District of Columbia	1			1
Georgia	1	1 *		2
Illinois	4	2		6
Kentucky	2			$egin{array}{c} eta \ 2 \ 3 \end{array}$
Maryland	2		1	3
Massachusetts	10			10
Michigan	2			2
New Jersey	1			1
New York	13		3	16
Oregon			1	1
Pennsylvania	6	2	1	9
Rhode Island	1		1	2
Tennessee	1			1
Texas	9 **			9
Virginia	1			1
Washington			1	1
West Virginia	1			1
Wisconsin	1		1	2
Total	72	7	21	100 ***

*This laboratory was involved in manufacturing counterfeit McNeil

Syndrox ® tablets.

**** The total figure is larger than the number of clandestine laboratories seized because some laboratories were manufacturing more than one substance.

drugs or to manufacture them for this purpose. If manufacture

is involved, the law requires the person to have registered with

stances is encountered, the proof of legal possession rests with the defendant and not with the government as it did before. A problem can arise with clandestine laboratories, when the operation is just getting off the ground and no illegal drug has been produced. The Bureau has been successful in some of these cases by having sufficient intelligence regarding the chemicals and precursors present on the site and the individuals involved in the operations, so that a charge of illegal manufacture of dangerous drugs could be substantiated. Sometimes it is expedient to let the production proceed until an illegal drug has

^{**} Eight of the clandestine laboratories in Texas were involved in manufacturing mescaline.

demnation of depressant, stimulant, hallucinogenic and counterfeit drugs and any equipment used in the illicit manufacture, compounding or processing. Agents are authorized to make seizures in admiralty libel procedures or by executive seizure prior to the institution of libel proceeding. In the latter instances the property must be brought under the jurisdiction of the court as promptly as possible. Thus there are ample mechanisms under the law to proceed against violators, their supplies, drugs and equipment.

There has been considerable discussion about how difficult or

how easy it is to produce dangerous drugs, particularly hallucine-

been produced. At that point a charge of failure to register, as

well as illegal manufacture and possession, can be made. The

Drug Abuse Control Amendments provide for seizure and con-

genic and stimulant drugs, in an illicit laboratory. Some authorities claim that the production of these drugs requires a great deal of skill and experience in organic chemistry, as well as a well-equipped laboratory; however, illicit production of various hallucinogenic and stimulant drugs has been encountered in both crude and sophisticated laboratories, and by amateurs as well as accomplished chemists.

It appears that the illicit production of dangerous drugs has

become an intellectual and professional challenge to many individuals associated with their misuse. Some of the more knowledgeable and experienced chemists have achieved clandestine production which approaches commercial scale.

Where does the illegal manufacturer obtain his knowledge?

Many of the procedures for the synthesis of controlled drugs are

the subject of U. S. patents. Like all patents, these procedures are readily accessible to anyone who desires to obtain them from the Patent Office. Further, during investigation of illicit drug laboratories, individuals were encountered who had made exhaustive searches of the scientific literature for chemical syntheses applicable to the production of hallucinogenic and stimulant drugg. In governal of the illicit laboratories resided there were

lant drugs. In several of the illicit laboratories raided there were found extensive files of literature on drugs which had been cataloged by type of drug, specific reactions used in the synthesis, and pharmacological action of the particular drug. Copies of extensive review articles on psychotropic drugs were also filed. Many of these clandestine manufacturers are as well aware

as any graduate student of chemistry how to use scientific literature as a resource. The great advances made in copying machines have also assisted the illegal operator in obtaining the scientific information that he needs.

For the entrepreneur who is too busy to do his own scientific literature search, many of the methods have been prepared into booklets by other enterprising persons. These booklets sell for \$1.50 to \$2.00. One such publication has the title "Turn on Book," subtitled "Synthesis and Extraction of Organic Psychedelics." There is a question as to whether or not copyrights have been violated in the reprinting of legitimate scientific journal



Fig. 1—Hallucinogenic drug laboratory. A drug from this disorderly laboratory might not only be unsanitary but might also contain a toxic chemical contaminant.

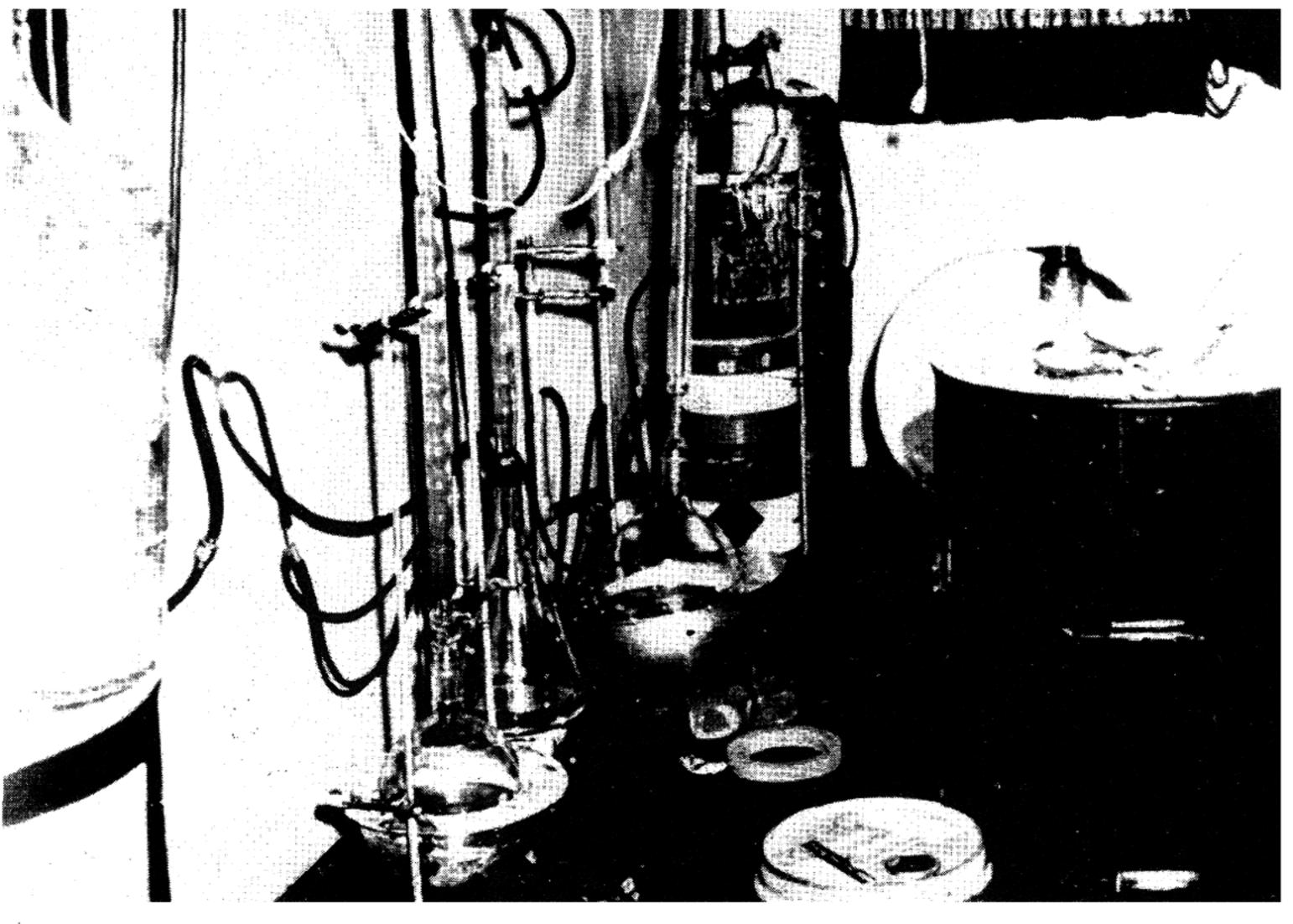


Fig. 2—Methamphetamine laboratory. Some chemicals purchased in drums and 5-gallon cans indicate size of production.

articles in such books. In addition to these books, psychedelic information centers have operated throughout the country which have printed newsletters about the latest drugs, and, in many cases, included "recipes" for their synthesis or manufacture. Much of this material has been "plagiarized" and has been sold separately or compiled into books such as that mentioned above.

In addition to amphetamine and methamphetamine, nearly all of the hallucinogenic drugs on the market today have been produced in illicit laboratories. These include LSD, STP, DMT, DET, mescaline, psilocybin, and phencyclidine, also known as PCP or the Peace Pill. Although synthesis of the tetrahydrocannabinols (THC) which are the active principles of marihuana has been attempted by clandestine laboratories, no laboratory that has been successful has been encountered, nor has any of this drug been found on the street. A number of buys represented as THC have been made, but in most cases the drug has turned out to be phencyclidine. Apparently, the process for making THC is too complicated. It may also be that the raw materials, which are also complex substances, are not readily available or are too difficult to produce in the clandestine laboratory.

It is not intended to try here to give an exhaustive review of all the syntheses that could be used for the manufacture of the abused drugs; however some comments on the most commonly encountered syntheses may be of interest.

Lysergic acid: Lysergic acid is a must for the production of LSD (lysergic acid diethylamide). Lysergic acid is controlled under the Drug Abuse Control Amendments making its availability limited. Although the total synthesis of d-lysergic acid has been achieved (1) the only practical source evailable to the illicit manufacturer is the hydrolysis of the pharmaceutically important ergot alkaloids, ergotamine and ergonovine. The method of Jacobs, et al. (2), which employs ergotamine tartrate, is frequently used. One hundred grams of ergotamine tartrate will yield approximately 9 g of d-lysergic acid monohydrate.

Attempts to manufacture lysergic acid by using a submerged culture method, employing Aspergillus or Claviceps, species paspali or clavatus, also have been encountered. Usually the culture becomes contaminated with Mucor plumbeus or one of the other cosmopolitan organisms.

Lysergic acid diethylamide: Conversion of d-lysergic acid to LSD can be accomplished, theoretically by any common procedures for the formation of amides. The procedure described by Garbrecht (3) appears to be most

favored. In this method 9 g of d-lysergic acid monohydrate will have a theoretical yield of 10 g of the tartrate salts of LSD. The actual yield is about 50% of theoretical.

Other methods are those of Pioch (4), Stoll and Hoffman (5), and Bernardi and Patelli (6).

N,N Dimethyltryptamine (DMT): DMT, an hallucinogen which has been called the "Business Man's Special" because of its short duration of action of one hour or less. It is usually synthesized from indole by the method of Specter and Anthony (7). The final steps in the manufacturing procedure are dependent on how the DMT will be sold. For example, if the DMT is to be placed on a carrier such as tobacco, marihuana or parsley leaves, then the tetrahydrofuran (THF) solution of DMT is poured over or dropped on the material to be treated. THF evaporates leaving DMT treated material which can be rolled into cigarettes or sold in packets for use in a pipe.

Pure DMT can be realized by crystallization from the THF solution. A yield of 10-15 g may be obtained from 25 g of indole.

A substitution of diethylamine for dimethylamine in the synthesis will yield approximately the same amount of DET (diethyltryptamine), an hallucinogenic drug also controlled by the Drug Abuse Control Amendments.

Mescaline: Mescaline may be obtained by the extraction of the alkaloid from the peyote cactus, Lophophora williamsii, or by chemical synthesis. Both methods have been encountered. The reduction of 3,4,5 trimethoxyphenylacetonitrile with lithium aluminum hydride, as described by Tsao (8), is a fairly simple procedure which can be accomplished in a crude laboratory. This synthesis will yield approximately 1.2 g of mescaline sulfate for 2 g of 3,4,5 trimethoxyphenylacetonitrile. When 3,4,5 trimethoxyphenylacetonitrile is not available, mescaline can be synthesized from 3,4,5 trimethoxybenzyl chloride; 3,4,5 trimethoxybenzyl alcohol; or 3,4,5 trimethoxybenzolc acid as described by Tsao. The synthesis, however, becomes longer and more complicated.

Psilocybin and Psilocin: Hoffman (9) described the chemical synthesis of psilocybin and psilocin in 1959. During the synthesis of these compounds, DMT is formed first. The further synthesis of psilocybin and psilocin requires considerable additional procedure. This may be the reason why very little of these two hallucinogens has been seen on the illegal market.

Psilocybin can also be produced by biochemical methods using a pure culture of Psilocybe cubensis following the method of Catalfomo (10). There have been attempts to follow this route to produce psilocybin but all have been failures.

Amphetamines and Methamphetamines: Of the 95 laboratories seized, 28 were producing stimulant drugs. Methamphetamine, known as "Meth," "Speed," or "Crystal" is the most popular product.

Phenylacetone (phenyl-2-propanone) is the primary precursor in the synthesis of amphetamine and methamphetamine. The syntheses of am-

phetamine have followed the methods of Hartung (11, 12). In these, hydroxyl amine is reacted with phenylacetone, with subsequent catalytic reduction using palladium black as the catalyst with hydrogen under pressure. Lithium aluminum hydride may be substituted as the reducing agent in these syntheses. By either method 100 g of phenylacetone will yield approximately 130 g of amphetamine as the sulfate.

Methamphetamine may be synthesized by reacting phenylacetone with methylamine, with subsequent catalytic reduction using palladium black and hydrogen under pressure (13). Lithium aluminum hydride has also been substituted in this synthesis as the reducing agent.

Ephedrine reacted with hydrogen iodide or with hydrochloric acid and zinc or tinfoil can also produce methamphetamine.

A list of the key chemical reagents needed for the synthesis of the drugs discussed above, as well as for STP and THC, are listed in Table III. A similar list has been furnished to agents of the Bureau to aid them in recognizing materials which have a potential for being used in the manufacture of drugs.

Most of the illicit operations raided by the agents have been those manufacturing the powdered forms of the drugs. One laboratory had an ampule-filling operation for methamphetamine and several had tablet-punching equipment. Most of the tablets from these laboratories were crude productions; however some were expertly made by persons obviously sophisticated in the art of tablet-making.

Raiding a clandestine laboratory can be a dangerous operation. During one raid, an agent had a corrosive material thrown into his face requiring his hospitalization. Many chemicals, including highly flammable solvents and toxic materials, are likely to be present, therefore it is believed that a chemist should assist the raiding agents. The chemist can be helpful in obtaining the search warrant by describing equipment and chemicals involved, any characteristic or distinctive odors, and possibly he may even identify the drug being produced from these earmarks. After the raid the chemist can be of help in the collection and preservation of evidence, and can eliminate many exhibits of doubtful value. Because of his knowledge he can select the necessary items for subsequent laboratory examination to prove the violation. The Bureau uses chemists in cases involving clandestine laboratories, and their assistance has been invaluable.

TABLE III

Identification of Clandestine Laboratories by Chemicals Present *

Lysergic Acid	
Ergotamine Tartrate Nitrogen Gas Ammonia Gas Potassium Hydroxide	

LSD (d-Lysergic Acid, N,N-
Diethylamide)
Lysergic Acid
Nitrogen Gas
Dimethylformamide Sulfur Trioxide
Acetonitrile
Trifluoroacetic Anhydride
Diethylamine

$\overline{\mathrm{DMT}}$	(N,N-Dimethyltryptamine)
Indole	
Our a 1	1 Chlorida

Oxalyl Chloride Tetrahydrofuran Dimethylamine

Mescaline	(3,4,5	Trimethoxyphen-
ethylamine	<u>)</u>	

3,4,5 Trimethoxyphenylacetonitrile 3,4,5 Trimethoxybenzoic Acid 3,4,5 Trimethoxybenzyl Chloride 3,4,5 Trimethoxybenzyl Alcohol

DET (N,N-Diethyltryptamine)

Indole Oxalyl Chloride Tetrahydrofuran Diethylamine

STP(4-Methyl-2,5-Dimethoxyam-

phetamine)

Phosphorus Oxychloride N-Methylformanilide Dimethylformamide 2,5 Dimethoxytoluene 2,5 Dimethoxy-4-Methylbenzaldehyde Ammonium Acetate

Nitroethane 1-(2,5-Dimethoxy-4-Methylphenyl)-2-Nitropropene

Amphetamine

Phenylacetone (phenyl-2-propanone) Formamide Hydroxyl Amine

Methamphetamine

Phenylacetone (phenyl-2-propanone) Ephedrine Zinc or tinfoil Methylamine

THC (Tetrahydrocannabinols)

Olivetol (5-pentylresorcinol) Citrol (Geraniol) Cinnamic Acid (beta-phenylacrylic acid) Isoprene-3-methyl-1,3-butadiene Verbenol (alpha-pinene) Hexane Ethyl Ether Hexane Malonic Acid Pyridine

^{*} Many common organic solvents (ether, methanol, ethanol, chloroform, etc.) are used in the manufacture of these drugs. Lithium aluminum hydride, a powerful reducing agent, is usually used in the synthesis of mescaline, DMT, DET, STP, amphetamine and methamphetamine.

The laboratory and our forensic chemists are regarded as an arm of enforcement. These specialists can make or break a case. It is essential that the drugs be identified and, in most instances, that a quantitative examination be made. Chemists, trained in modern-day laboratory instrumentation, are absolutely necessary to identify the bizarre substances being abused.

Summaries of two cases involving illegal laboratories may be of interest:

Case 1: A landlord, who took great pride in a home he leased, was irked by his tenant's failure to maintain the grass in front of the place. He went to the door to ask that the grass be kept watered but there was no response to his knock. While looking around, he noted an overpowering odor emanating from the house—as if a "dead body" were inside. He called the police who, instead of a corpse, found a clandestine laboratory. Agents of the Bureau were called in on the case, and they found the laboratory to be a sophisticated facility, with the latest equipment and a wealth of books and reprints from scientific journals. It was operated by a chemist who kept a well-organized laboratory notebook. The entire water supply of the house was converted to laboratory use, which accounted for the failure to water the grass. It took seven truckloads to haul away almost 300 pieces of laboratory glassware and equipment. Drugs seized included amphetamines, LSD, mescaline, MDA, STP and several hundred grams of ergotamine tartrate used in the synthesis of lysergic acid. If this quantity of the drug had been converted to LSD it would have produced over \$3,000,000 worth of LSD at the going price of \$5 a trip. Figure 3 shows the main part of the laboratory.

Case 2: Not many months ago, the Bureau learned that a West Coast firm was placing orders with various companies throughout the country for chemical and laboratory equipment which could be used to manufacture stimulant drugs. Investigation verified the information and showed that the firm was a supply front for a clandestine methamphetamine laboratory. Interstate surveillance disclosed that the individuals involved flew to various areas of the country in order to pick up supplies. Additional investigation, including aerial surveillance, finally located the laboratory in a residence in the mountains. Agents on the ground kept the site under observation and witnessed deliveries of chemicals and equipment. Armed with a search warrant and accompanied by the sheriff, a posse raided the laboratory and arrested four persons. Chemicals, equipment, and about 30 lb of manufactured methamphetamine powder were seized. This was a highly sophisticated laboratory operation. At the time of the raid, the methamphetamine, worth nearly a million dollars on the illicit market, was being processed.

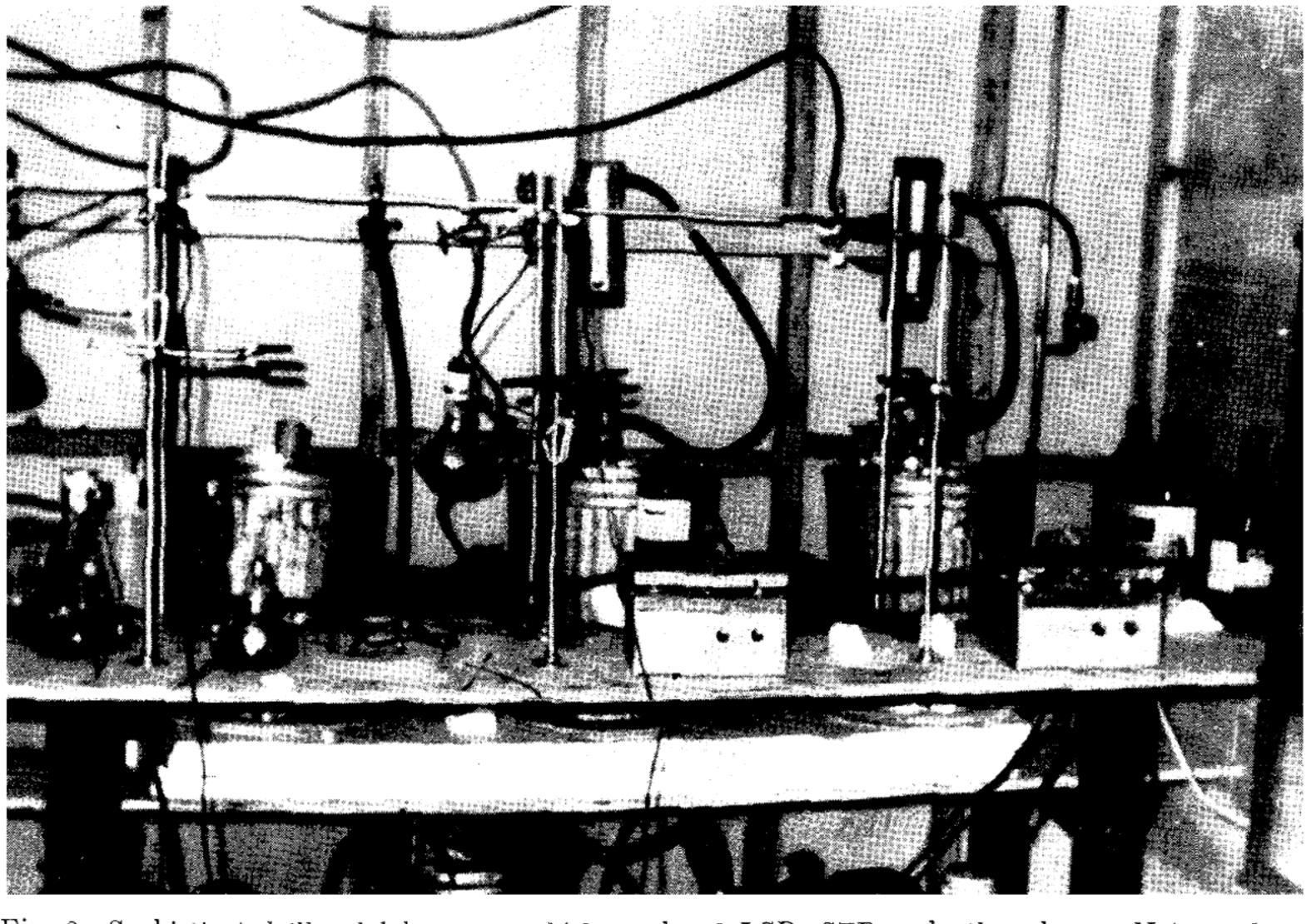


Fig. 3—Sophisticated illegal laboratory which produced LSD, STP and other drugs. Note garbage cans adapted to use as cooling jackets for 22-liter flasks.

Summary

- 1. Clandestine laboratories are a recent innovation of the drug culture.
- 2. The clandestine laboratory may be a small one- or two-man enterprise, or it may be a sophisticated undertaking involving many persons and a substantial investment of money and effort.
- 3. The drugs produced are of unknown quality, which in itself is a danger to users.
- 4. For the most part, there is adequate law at the federal level and in most states to cope with the problem.
- 5. Ferreting out these installations is a challenge to law enforcement.
- 6. The forensic chemist is an important member of the enforcement team in the closing down of clandestine laboratories.
- 7. Raiding a laboratory is hazardous, and it must be carefully planned to protect enforcement personnel and to make the raid at the right time to secure the greatest amount of effective evidence.

REFERENCES

- 1. Kornfeld, E. C., et al. The Total Synthesis of Lysergic Acid and Ergonovine. J. Am. Chem. Soc. 76, 5256 (1954).
- 2. Jacobs, W., and L. Craig. The Ergot Alkaloids. II. The Degradation of Ergotamine with Alkali. Lysergic Acid. J. Biol. Chem. 104, 547 (1934).
- 3. Garbrecht, W. L. Synthesis of Amides of Lysergic Acid. J. Org. Chem. 24, 368 (1959). U. S. Pat. 2,774,763 assigned to Eli Lilly & Co.
- 4. Pioch, R. P. Preparation of Lysergic Acid Amides. U. S. Pat. 2,736,728 assigned to Eli Lilly & Co.
- 5. Stoll, A., and A. Hoffman. d-Lysergic Acid-d-1-Hydroxy-2-Butylamide. Helv. Chim. Acta. 26:944, 1943.
- 6. Bernardi, L., and B. Patelli. Process for the Preparation of Lysergic Acid Amides. U. S. Pat. 3,141,887 assigned to Societa Farmaceuteci Italia.
- 7. Speeter and Anthony. The Action of Oxalyl Chloride on Indoles. A New Approach to Tryptamines. J. Am. Chem. Soc. 76, 6209 (1954).
- 8. Tsao, Makepeace U. A New Synthesis of Mescaline. J. Am. Chem. Soc. 73, 5495 (1951).
- 9. Hoffman, A. Psilocybin and Psilocin. Two Psychotropically Active Principles of Mexican Hallucinogenic Fungus. Helv. Chim. Acta. 42, 1557 (1959).
- 10. Catalfomo, P. The Production of Psilocybin in Submerged Culture by Psilocybe Cubensis. Dissertation Abs. 24, 953, No. 3 (1963).

11. Hartung, W., and J. Munch. Amino Alcohols. VI. The Preparation and Pharmacodynamic Activity of Four Isomeric Phenylpropylamines. J. Am. Chem. Soc. 53, 1875 (1931). 12. Hartung, W. Catalytic Reduction of Nitriles and Oximes. J. Am. Chem. Soc. 50, 3370 (1928). 13. Ogata, A. α and β Aminoalkyl (Aryl) Benzenes and Their Derivatives. J. Pharm. Soc. Japan. 451, 751 (1919). United States Department of Justice Bureau of Narcotics and Dangerous Drugs

Washington, D. C. 20537