
PRACTICAL
LSD
MANUFACTURE
BY·UNCLE FESTER·
HAZE
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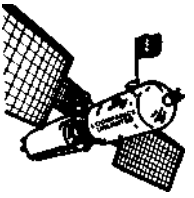
Practical LSD Manufacture

by Uncle Fester



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Practical LSD Manufacture

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Preface

The DBA has recently estimated the total number of clandestine LSD labs operating in the United States at only 100, with most of them located in northern California. This alarmingly low number of labs leaves the supply of LSD in this country at constant peril. Further, the concentration of production in so few hands has left us awash in a mediocre swill comparable to the beer spewed out by the major brewers.

This distressing situation results from the convergence of a series of factors. The botanical sources of lysergic acid are not easily available in large quantities. The actual production of LSD from these botanical sources is a touchy and involved operation. These roadblocks, however, pale in comparison to the most important factor — the inaccessibility of good information to those motivated to put it into action.

I can think of no other area of organic chemistry which, to we common working pot-boilers, is shrouded in as much mystery, or is as thoroughly obfuscated as the production of LSD. The scientific articles dealing with this topic are barely readable by the typical person with an undergraduate degree in chemistry. They assume a level of understanding of the arcane

field of lysergic chemistry not generally possessed by even those skilled in the "cooking arts."

The "underground publications" covering this topic have done little to clean up this situation. They have merely regurgitated the original unintelligible works until they have become like mantras, repeatedly chanted and not understood.

It is here that this book shall break new ground. Rather than presenting this field as a magic act, the sources of lysergic acid raw materials in nature shall be detailed, and their mystery removed. The processes required to isolate this raw material and move it on in pure form to LSD shall be expounded upon. Common threads shall be drawn between the various procedures to show what variations in technique are acceptable, and which produce the disappointing commercial product we are all too often cursed with.

A special added feature of this book will be the result of my own investigations into the production of the most wonderful psychedelic: TMA-2, derived form the roots of the calamus plant. For those unable or unwilling to wade through the difficulties that attend cultivating ergot, or growing crops of morning glories, digging up the roots of this common plant offers a most convenient and low-profile route to an awe-inspiring substance. You will be quite pleased, I'm sure.

Fester

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LSD Production: An Overview

The synthesis of LSD is not a task to be undertaken lightly by the novice wannabe drug chemist. It requires a level of skill roughly double that needed to produce more conventional drugs such as methamphetamine. A person contemplating this task should be well trained prior to beginning the attempt, as learning while "on the job" is likely to lead not only to failure, but also the probable poisoning of the said wannabe drug chemist.

This fact of life is due to both the nature of the product itself, and the involved procedures required to convert ergot, morning glory seeds, or Hawaiian baby woodrose seeds into LSD. The potency of LSD is truly phenomenal — 10,000 doses per gram — and is easily absorbed through the skin. This is how Albert Hofmann, the discoverer of LSD, got his first trip. He was skilled enough that his boo-boo involved a small enough dose that his brain was not fried. Beginner chemists tend to get the stuff they are cooking all over themselves, and would not be so lucky.

Lysergic acid, its precursors, and LSD are all very fragile molecules, and quite prone to destruction by light, air and heat. The common makeshift basement lab set-ups used by most clandestine operators will not do for anyone contemplating LSD synthesis. Real laboratory equipment is needed, such as a distilling kit with ground

glass joints for doing reactions in, and for distilling home synthesized reagents to an acceptable degree of purity. A vacuum desiccator is essential to dry lysergic compounds without burning them. A vacuum pump rather than an aspirator is the only acceptable source of vacuum for this desiccator. One must be prepared to spend about $5000 up front to equip such a lab, but the paybacks are potentially enormous if one avoids detection. See my Third Edition of *Secrets of Methamphetamine Manufacture* for many useful tips on how to obtain chemicals and equipment, set up shop and move the product without getting caught. The wise operator will never pass up the opportunity to use the five-finger-discount method, industry contacts, waste exchanges and the surplus market to stock his or her lab.

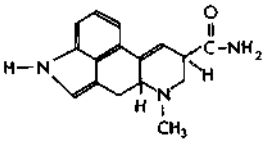
The minimum level of skill I would trust to undertake this task would be at least a full year of college organic chemistry lab, and a few biology courses with lab where the use of chromatography was taught to isolate biological substances from complex mixtures. Sterile culture technique in these biology classes is a real plus if the plan is to cultivate ergot in a rye field. Long gone are the days when a guy like Owsley, with only a little training and a smart wife, could buy pure ergotamine tartarate and all the other chemicals needed to brew legendary acids like White Lightning and Orange Sunshine. Today's operator must be prepared to isolate lysergic acid precursors from materials like ergot, morning glory seeds, or Hawaiian baby woodrose seeds. He must also be ready and able to synthesize in pure form closely watched organic reagents like diethylamine.

There is a constant and unyielding maxim in organic chemistry: GIGO — garbage in, garbage out. If the materials used in an organic synthesis are not pure to a reasonable degree, the result is a complex mixture in which the desired product comprises only a small proportion. Even a seemingly very simple reaction cannot escape this law. Case in point is the hydriodic acid and red phosphorus reduction of ephedrine to methamphetamine. If in this reaction the ephedrine is not fairly free of the fillers and binders found in the stimulant pills from which it is extracted, the result at the end of the reaction is a heavy reduction in the yield of product, and the formation of a most stubborn emulsion from which the desired meth is extracted only with

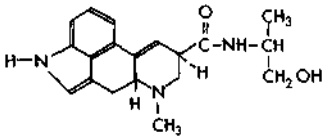
great difficulty. This is the origin of the revolting peanut butter consistency of most meth seen on the market. Similarly, one can only expect success in the production of high-grade LSD if care is taken throughout the procedure to ensure that the materials used meet the requirement of a reasonable degree of purity.

The actual synthesis of LSD is an exquisite combination of farming skills, biology, biochemistry and organic chemistry. In its preferred embodiment, a scheme for the large-scale manufacture of LSD would center around someone playing weekend hobby farmer on an acre or two of land. On this land, our happier-than-most farmer would plant either rye to be infested with the Claviceps fungus to produce a crop of ergot; morning glories for the eventual harvest of their seeds; or, if local weather conditions permit, Hawaiian baby woodrose, also for the harvest of its seeds.

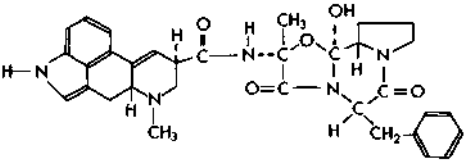
Mother Nature's bounty is then squirreled off to the lab site for the biochemical phase of the process — the isolation of the lysergic alkaloids. Here one or more of a series of alkaloids are freed from the very complex plant matrix and hopefully isolated in a pure form. These alkaloids all have one thing in common — they are amides of lysergic acid. See the structures of the major naturally occurring amides pictured below:



ergine

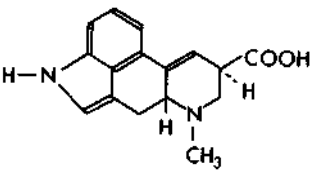


ergonovine

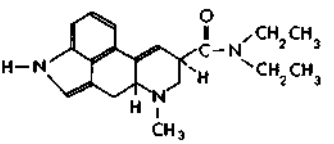


ergotamine

They all contain the lysergic acid molecule shown below:



The lysergic acid molecule is the key to all known methods of LSD production. The common thread that all the synthetic routes to LSD share is that the path they travel starts with the naturally occurring alkaloids, the amide linkage is lopped off to give lysergic acid, and then the lysergic acid is reacted with diethylamine to give LSD shown below:



The nuts and bolts of how this is done will be explained in the succeeding chapters.

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Sources Of The Lysergic Amides

Let me begin this chapter by nuking an oft-chanted mantra, this mantra being the claim that a person can grow ergot fungus in a culture medium and get it to produce lysergic acid amides to feed into LSD production. This claim as seen in *Psychedelic Chemistry* and other publications I read while in college is pure BS. It is truly unfortunate that nature does not cooperate in this manner, since this would obviously be the best way to set up a large-scale production operation, as the logistical complications of crop growth and harvest would then be eliminated.

Let me give a science and literature reading lesson to those who have made these claims. See *Proceedings of the Royal Society of London,* Series B, Volume 155, pages 26 to 54 (1961). Also see US Patent 3,219,545. You will note while reading these articles detailing how to get lysergic amide production in a culture medium that these guys had to scour the globe to find that rare strain of claviceps fungus that will cooperate in this manner. The vast majority of claviceps fungi just will not produce these alkaloids while being cultured. See the following articles to convince yourself of just how futile it is to collect a wild strain of claviceps and try to get it to produce lysergic acid amides in culture: *Ann. Rep. Takeda Res. Lab* Volume 10, page 73 (1951); and *Farmco,* Volume 1, page 1 (1946); also *Arch. Pharm. Berl*. Volume 273, page 348 (1935); also *American Journal of*

Botany, Volume 18, page 50 (1931); also *Journal of the American Pharmacy Association* Volume 40, page 434 (1951); also US patent 2,809,920; also *Canadian Journal of Microbiology*, Volume 3, page 55 (1957), and Volume 4, page 611 (1958) and Volume 6, page 355 (1960); also *Journal of the American Pharmacy Society* Volume 44, page 736 (1955).

With this matter disposed of, it is time to move on to what actually are viable sources of lysergic acid amides for the production of LSD. This is the farming end of the acid business. It is only through raising ergot-infested rye, or growing morning glories and Hawaiian baby woodrose that the required feedstocks of lysergic compounds can be obtained without making a target of oneself. I have for years seen ads in *High Times* offering morning glory seeds and Hawaiian baby woodrose seeds for sale, but these are offered in small amounts at high prices. I would bet my bottom dollar that these outfits, if they are not front operations, will at least report to the heat any large orders they get. To avoid detection, the aspiring LSD manufacturer must be ready to get his hands dirty, and spend some time as a farmer.

The most difficult farming choice, and as luck would have it, the one that gives the purest acid, is to grow a patch of ergot-infested rye. The reason why ergot is superior to growing morning glory seeds or woodrose seeds is that these seeds have a considerable amount of another type of alkaloid in them besides the ones that yield lysergic acid. These other alkaloids are of the clavine type, meaning that they have the lysergic-acid skeleton, but lack the carboxyl grouping. In its place will be a methyl grouping, an alcohol grouping, a methyl alcohol grouping or combinations of the above. These clavine alkaloids will likely be carried all the way through into the product, producing both the GIGO situation during the synthetic operations and a contaminated product when finished. I will present my ideas on how to remove them, but they are best avoided in the first place.

Ergot is the name given to a dark brown to purplish black horn-shaped growth occasionally seen nestled amongst the healthy grains in the head of the rye plant. It is typically in the neighborhood of 10 to 15 mm long, and can reach diameters of about 5 mm. The ergot consists of tightly interwoven hyphae of the fungus *Claviceps*

purpurea, and it grows parasitically upon the rye plant. During the Middle Ages, when ergot infested rye was quite common, great poisoning epidemics called St. Anthony's Fire or *ignis sacer* would break out among the people who ate it. For some reason that escapes me, they never, over the course of hundreds of years, connected this most lamentable malady to eating the ergot infesting their rye. The usual response to an outbreak was to burn a witch or two in the hope that this display of piety would so please God that they would be saved.

A most wonderful book has been written on the topic of ergot, and upon the history of these mass poisoning outbreaks. The book is titled *Ergot and Ergotism* by G. Barger, and it is absolute must reading for anyone seriously contemplating growing ergot. In this book you will find a series of pictures of ergot growing on rye in the wild, and a much more detailed presentation of both the chemistry of ergot and its life cycle than will be given here.

You may well have noticed that outbreaks of ergot poisoning are no longer commonplace. This is mostly because modem farming practices such as plowing, crop rotation, drainage of fields and the use of fungus-resistant seed strains make the present day crop of rye a much less hospitable place for the ergot to grow in than the sloppily run dumps that our peasant ancestors presided over. Yet, the occasional head of ergot is still there to be found in fields of rye, and a field trip to a patch of rye to gather some ergot is the necessary first step of purposely growing your own patch of rye just overrun with ergot. Such field trips are made considerably easier thanks to the fact that wild ergot on a modern farm will be mostly growing around the edges of the field. There is no need to run all over the farmer's rye, and cause him to want to ventilate you for trampling his crop.

When a few dozen heads of wild ergot have been collected, the stage is set for you to begin growing truly worthwhile crops of ergot rather than the pitiful scattered kernel or two found on your typical farm. To get these bountiful yields of ergot, biological skills will be called upon to get an infestation rate in your own crop of rye that far exceeds that seen in even the most slovenly days of Dark Ages serfdom.

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To grow ergot successfully, one must have some knowledge of the life cycle of the Claviceps fungus. The kernel of ergot seen growing on the rye plant is the form this fungus takes to make it through the winter. In the wild state, the ergot falls off of the rye plant when the grain matures, and lays there on top of the dirt until the following spring. Then, when warm weather returns, the kernel of ergot sprouts off a bunch of tiny growths that look for all the world like so many minute mushrooms. In the head of each of these little mushroom growths are millions of spores. These spores are the fungus equivalent of seeds.

When the mushroom growths have reached a length of about 20 mm, they are mature, and the head of the mushroom explodes, sending the millions of spores floating through the air. These spores, either by luck of air currents or by hitching a ride upon insects, find their way into the flower of the rye plants growing nearby. The flower of the rye plant is nothing spectacular. Rye is a grass, and its flowers look like most other grass flowers — just a filamentaceous dab of color scattered over the head of the plant which soon grows into seeds.

Upon being deposited into the flower of the rye plant, the spore germinates and takes over the flower. The fungus then grows by sucking nutrients out of the rye plant, until a new kernel of ergot has been formed to repeat the process again next year.

The biological sciences are made to order to take the hit-and-miss aspect out of the process of rye flower infestation. Instead of the random action of air currents or insects to bring spores into contact with their new home, one may germinate these spores in a sterile culture medium, grow them until they have multiplied a million-fold, then spray them onto the rye plants just as they are blooming to ensure a heavy infestation with ergot. This method has been in use since the 1920s with great success in the commercial production of ergot. See the reference by Hecke (pages 1921-1922) in the back of the *Ergot and Ergotism* book mentioned above for complete experimental details. Yields of ergot using this method average a few hundred pounds per acre. A couple of acres could supply most of the United States with high-grade acid.

To put this plan into action, the few dozen kernels of ergot are kept cool and dry during the winter, then as spring approaches they are made ready to germinate by putting them in the refrigerator for one month to six weeks with the temperature held steady from just above freezing to 3° C. This will make the ergot think that it has gone through winter, and works better than actually freezing the stuff. Without this treatment, the ergot will not germinate to form the mushroom stage of its life cycle.

After our artificial winter has passed for the ergot, we must make it think that it is at home in the dirt. To do this, a terrarium is thoroughly cleaned out with bleach water and several rinses. Then a layer of clean sand about an inch thick is put in the bottom of the terrarium, and the ergot is sprinkled on top of the sand. Finally, a little more sand is sprinkled over the top of the ergot until they are each just covered up. The terrarium is kept at room temperature, with an occasional misting with water to keep the sand moist but not soaking wet.

After about a month in the terrarium, the ergot begins to sprout. In the case of ergot, sprout means to grow a bunch of the little mushrooms mentioned before. They grow towards the light, starting out short and fat, and becoming increasingly thin as they grow. The heads of these mushrooms will be covered with what appear to be warts when they are ripe. Misting with water must be continued during the sprouting of the ergot to keep it growing.

When the mushrooms sprouting from a particular grain of ergot are ripe, they should be harvested. The individual grains will not all sprout or ripen at the same time, so this is a harvest one-grain-at-a-time operation. The ripe grain is carefully scooped out of the sand with a spoon, and the sand is then dilute-bleach-water-misted away to leave the bare grain covered with mushrooms. Care must be taken when handling the sprouted ergot, as rough handling will cause the ripe heads of the mushrooms to explode and spew forth their load of spores.

From this point onward, best results are going to be had using sterile-culture technique. The next objective is to remove the spores from the heads of the mushrooms growing out of the ergot, and put

them into a sterile culture medium made from diluted malt extract, where they will grow for a week or so producing a culture broth loaded with germinated spores which can be sprayed onto the blooming heads of rye, yielding a heavy infection rate of ergot in your patch of rye.

I have some helpful observations to share on the matter of home sterile-culture technique, based upon my own experiences. It has been my observation that keeping one's cultures free from contamination by freeloading wild germs is often considerably more difficult in the kitchen than it is in a biology lab. The typical university lab is supplied with filtered air from the central heating and air conditioning unit. The amount of dust particles and animal dander floating in the air is much smaller than usually seen in the home. This is especially true if your housekeeping is bad, like mine. The threat from wild contamination is most severe if you live in a warm, moist area, like the eastern half of the US in the summer. When doing home cultures, the sterile transfers should be done in an air-conditioned room with an effective air filter.

To begin the sterile culture portion of ergot farming, a series of 2000 ml conical flasks are filled about one inch deep with nutrient broth made by diluting malt extract with 5 volumes of water. Malt extract is found at stores and outlets catering to the home brewer. It comes in cans, and is a very thick liquid. Avoid the crystalline version of malt extract. The tops of the conical flasks are loosely plugged with cotton, and then sterilized in a pressure cooker at 15 Ibs. pressure for a little over 1/2 hour.

When they have cooled down to room temperature they are moved into the room in which the sterile transfers will be done. The spores from the heads of the mushrooms are sterilely transferred into these flasks for growth. This is done by taking a microscope slide cover slip, and while holding it with a tweezers, passing the cover slip through the flame of an alcohol lamp. Then, when the cover slip has cooled down, it is impregnated with spores by holding the cover slip over the head of a mushroom with a sterilized tweezer and lancing the mushroom head with a similarly sterilized needle. Remember that the heads of these mushrooms are ready to explode when ripe. The spore-

impregnated cover slip is then dropped into the conical flask, and the cotton plug replaced. In this manner, a whole series of flasks can be seeded with Claviceps fungus from a single ergot grain.

The spores germinate shortly after landing in the nutrient broth. From there they grow into a slimy film floating on the surface of the broth. The best growth is obtained at a temperature of 25-30° C. This fungus needs oxygen to grow, but a few days of growth in the 2000 ml flask will not exhaust the supply there. Longer periods of incubation would require that some fresh oxygen be supplied to the flasks.

Best results are obtained when the fungus is actively growing when it is sprayed onto the rye plants. This means that the whole ergot sprouting and culturing operation must be timed to coincide with the flowering of the rye plants. In my own state of Wisconsin, the rye comes into bloom in early to mid-June, depending upon the weather. The blooming of rye lasts for about a week, so timing is critical. It is possible to spray a little before the onset of blooming, but spraying too late is mostly a waste of time.

The spraying is a very simple operation. A metal or plastic hand pump sprayer with a capacity of about 3 gallons is filled about half full of water. The contents of one of those conical culture flasks are then put into the sprayer, and mixed around thoroughly by shaking. Then more water is added to fill the sprayer, and the solution is then sprayed onto the crop. This is best done early in the morning, while dew is still on the plants. The aim should be to get a fairly light misting over the entire crop. This can be repeated every day for the week that the rye is in bloom.

From here nature takes over, producing kernels of ergot identical to the ones harvested the year before. There is general agreement that the most potent ergot grows during very hot summers. No farmer has control of the weather, but if there is a choice as to where our ergot farmer sets up shop, it would then be best to choose a state with very hot summers, or at least the southward-facing slope of a hill. It is also generally agreed that the ergot is at its most potent about a week or so before the rye grain are fully ripe. This is when the rye crop should be harvested.

The harvesting of the rye (ergot) crop should not be done with a combine, as these machines pass the grains through a sieve. Most of the ergot would then be lost, as it is much larger than the rye kernels. Rather, the rye plants should be cut down using a hand or mechanical sickle, and they should then be gathered up into shocks as seen in old time pictures or paintings of grain harvesting. Next, the grains should be beaten off the rye plants into a container such as a bushel basket. We are talking about old time farming here! The ergot is then separated from the rye kernels by dumping the bushel basket full of grain into a tank full of saturated salt solution in water. The ergot floats to the top of the salt water, while the rye sinks. The ergot is skimmed off the top of the water, rinsed, and immediately spread out to dry in the sun. The ergot must not be allowed to get moldy, as this ruins its potency.

This procedure is the preferred source for the lysergic acid amides. It is preferable both to growing morning glory seeds and Hawaiian baby woodrose seeds because the alkaloid content of the ergot is about 10 times higher, and also because the ergot has very small amounts of the clavine alkaloids contaminating it. The case can be made that the simplicity of the seed growing operations as compared to growing ergot argues in favor of using that method. My thoughts on this matter are that ergot is needed for really high quality acid, and that if a person wants an easy drug to make, he should check out my recipe for Cat in the third edition of *Secrets Of Methamphetamine Manufacture.*

There is an excellent alternative source of ergot for those living close to the Gulf coast, the Atlantic coast south of New York, and the Pacific Northwest's Puget Sound. In the saltwater marshes along the coast, the marsh grass Spartina is subject to a very heavy infestation with wild ergot. Yields of wild ergot in the range of 150 pounds per acre are pretty common in areas that have been disturbed, such as by ditches or in "spoil areas." (See *Mycologia,* Volume 66, pages 978 to 986 (1974) for full details and pictures.) Harvesting the ergot in this case would probably be best done in a manner similar to that used by Native Americans to harvest wild rice. They simply travel through the

grass in a shallow-draft rowboat, bend the heads of grain into their boats, and beat it off with a stick.

If the choice is made to fuel LSD production using morning glory seeds, one should be aware that not all varieties are created equal. Some types of morning glories contain little or no ergot alkaloids. The best varieties to choose are Heavenly Blues, Pearly Gates or Flying Saucers. The only growing tips I have to share are to give the plants a moderate dose of nitrogen fertilizer when they are young to encourage heavy growth, then switch to organic fertilizers so as not to mess up the plant's hormonal balance during flowering and seed production.

There have been recent reports of a wholly new source of lysergic acid amides. The so called Sleepy Grass *(Stipa robusta)* of the desert areas of the American West is reported to have an alkaloid content approaching that of ergot, and should be a good source of raw material to feed into acid production. See *Discover* magazine, Dec. 92.

Additional Reading On Growing Ergot:

Gulf Res. Rep. 3(1), pages 105-109 (1970), "Observations on Claviceps purpurea on Spartina alterflora." *Canadian Journal of Botany* Vol. 35, pages 315-320 (1957), "Studies on Ergot in Gramineous Hosts." *Pharmacognosy* (1965), pages 321-327. *Agricultural Gazette of New South Wales* Vol. 52, pages 571-581 (1941), "Artificial Production of Ergot." *Pythopathology* Volume 35, pages 353-360 (1945), "The Field Inoculation of Rye With Claviceps purpurea." *American Journal of Botany* Volume 18, pages 50-78 (1931), "The Reactions of Claviceps purpurea to Variations in Environment."

3
Extraction And Isolation Of Lysergic Acid Amides

After the harvest of the crops, the farming phase of acid production is now over. This is a good news/bad news situation for the acid chemist. The good news is that the voluminous pile of crop will in short order be reduced in size to a quantity more conveniently handled in the lab. For example, ergot typically contains from *V** to *¥2%* alkaloids by weight. A 200 pound harvest of ergot will, after extraction, yield *Vi* to a full pound of lysergic acid amides. This quantity is worth several millions of dollars if moved wholesale at a dollar per dose. The yield from a similar amount of morning glory seeds will be reduced by a factor of about 5, but still be substantial. Hawaiian baby woodrose seeds are intermediate between the two.

The bad news takes several forms. A significant amount of solvents will be needed to perform the extraction from the crop. It is at this juncture that the acid chemist will need to employ industrial contacts, theft, or the formation of a front operation to get the several 55-gallon drums of solvents needed to execute the extraction. The aroma that solvents give off also precludes doing this procedure in a residential neighborhood. A shed back on the farm site or a business front setting is much more suitable.

It is also at this phase that the delicate natures of the lysergic molecules express themselves. While they are locked up in ergot or in seeds, these molecules are pretty stable, so long as the crop is kept

cool, dry, and free from mold. Once they are released, they are prey to light, heat, air, and bad chemical handling. A clock begins to tick on the shelf life of your product. Once the extraction is begun, the chemist must consider himself committed to the task, and not allow himself to be distracted by other matters while the product spoils.

There are several alternate procedures for the extraction of the amides from ergot. They all produce roughly similar results. This is fortunate, as it allows the acid chemist to choose the materials used based upon availability rather than being rigidly locked into using a certain set of materials.

The first step in the extraction procedure, regardless of whether ergot or seeds are being extracted, is a thorough grinding. A blender is suitable for this job, and a coffee grinder may work as well if it gives a fine grind. Once the crop has been ground up, it is immediately vulnerable to attack by light and air, so as soon as it is ground it should be wetted with the solvent chosen for use in the next step: defatting.

Defatting is a very important step in the isolation of pure alkaloid. The fats and oils present in the crop must be removed because if they were left in, a tenacious emulsion would form during the extraction of the alkaloid, and you could forget about ever getting even close to a pure amide extract. For all practical purposes, all that would be extracted would be garbage.

Defatting can be done with any one of several very common and easily available solvents. For a 200 pound crop, one can count on using at least one, and possibly two 55 gallon drums of solvent. The defatting can be done with either hexane, petroleum ether (not ethyl ether) mineral spirits or naphtha. The preferred procedure for small scale extractions is to put the ground-up, solvent-soaked crop into a burette, and then keep dripping fresh solvent onto the top of the material until the solvent coming out at the bottom of the burette does not leave a grease stain on filter paper when the solvent dries. This is easily scaled up for our 200 pound crop by replacing the burette with clean pipes about 4 inches in diameter, and about 4 feet long, with suitable valves and filters at the bottom to prevent everything from falling out. (See Figure 1). When all the fats have been removed from

the crop, the best procedure is to evaporate the remaining defatting solvent from the crop under a vacuum. This is not practical for a large crop, so letting the remainder drip out of the bed over a period of a few hours is called for.

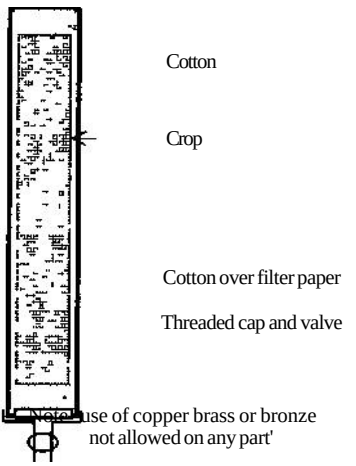


Figure 1 ***Apparatus for large-scale defatting***

With the fats removed, the ergot alkaloids can be extracted from the crop. Note here the word *alkaloid*. This is the key to all variations of the extraction procedure. There is a piperidine nitrogen atom in the lysergic portion of these molecules that possesses basic properties similar to ammonia and amines. This atom allows the lysergic molecules to form salts with acids, and also causes the solubility characteristics of the molecule to change depending upon whether the molecule is in acid or basic solution. It further allows the lysergic amides, including LSD, to form crystals from solution.

The lysergic amides as found in our crop are tied up in the plant material in association with acidic substances. To get the amides to extract out in a solvent, this salt must be free-based. There are two preferred solvent and basing agent combinations. Choice number one is used in the USP procedure. This combination is ammonia as the free-basing agent in a solvent of chloroform. The other preferred combination was used extensively in Europe. This combination used MgO (magnesia) as the basing agent with a solvent of ethyl ether or benzene. There have been comparisons of the two methods, and the European variation gives an extraction that is about 25% more complete than the USP method. It is, however, not nearly as practical

as the USP method for large-scale extractions because it would be necessary to dump the crop out of the extraction pipes, and then grind the solid MgO into an intimate mixture with the crop prior to extraction with ether. The USP method allows the much simpler procedure that follows:

The extraction solvent is made up by adding one-tenth gallon strong ammonia (28% NH_3OH; 56% NHtOH) to nine-tenths gallon methanol. After mixing, this is added to nine gallons of chloroform to give 10 gallons of extraction solvent. The use of methanol is necessary because without it the ammonia does not mix into the chloroform. Instead, it would float on top of the chloroform giving an unhomogenous mixture.

The extraction is done by trickling this extraction solvent into the top of the bed of crop, allowing it to flow downward through the crop, and collecting the extract as it flows out the bottom of the pipe. This extract must be protected from light to prevent its destruction. The extraction of a 200 pound crop requires about 150 gallons of solvent. One can monitor the extraction by catching a little bit of the solvent coming out the bottom of the pipes in a watch glass, and shining a black light upon it in a darkened room. The lysergic amides in the crop fluoresce a bluish color. When this color no longer appears in the extract, the extraction is complete.

Next, the approximately 150 gallons of solvent must be evaporated down to a more convenient amount. If one's crop was not so bountiful as 200 pounds, this is a lot simpler, and can be done in laboratory glassware. For a large crop, a more industrial approach must be taken. The two main precautions to prevent damage to the product are the same in either case. The evaporation must be done with a vacuum, so that the product is not exposed to heating above 40° C (105° F), and the product must not be exposed to light.

To evaporate the large industrial quantity of solvent, a 55-gallon steel drum is filled about two-thirds full of the extraction solvent. On the top of the drum are two threaded openings. Opening number one is secured with the original bung. The other opening is tightly stuffed with a rubber stopper. This rubber stopper has a hole drilled in it, and a section of pipe is put through the hole in the stopper so that it

extends about an inch below the stopper. To this pipe, a line of vacuum tubing is attached, leading to a vacuum pump. This pump should be the typical shop pump that can pull a vacuum of about 21 inches of mercury out of the possible 30 inches. This is enough to greatly speed the evaporation without causing the chloroform to boil. Boiling may raise a head of foam that would carry product along with it, causing great losses.

On a laboratory scale, a stronger vacuum can be used from an aspirator. By using red or yellow darkroom light bulbs for illumination, damage to the product can be kept to a minimum. The stronger vacuum speeds up the process quite a bit. Use boiling chips to prevent bumping.

As the chloroform evaporates away, more of the extraction solvent may be added to either the 55-gallon drum or the distilling flask, depending upon the scale of production. The evaporation is continued until the extraction solvent has been reduced to one-fifteenth its original volume. For the 200-pound crop, the 150 gallons of extraction solvent has been reduced to 10 gallons.

An accessory which may speed up and smooth out this evaporation is a capillary air bubbler. This is made by taking a section of glass tubing, and poking it through a rubber stopper. The end of the glass tubing is then heated to redness in a flame, and pulled into a very fine capillary. The tubing is then stuck into the solution being evaporated, extending nearly to the bottom. The vacuum will pull a fine stream of air bubbles through the solution and aid evaporation.

When the chloroform has been reduced to one-fifteenth of its original volume, it must be diluted with ether. The reason for this is that the next step is extraction of the ergot alkaloids into a tartaric-acid solution, and it has been found that this is very difficult from pure chloroform. When the solution is predominantly ether, the transfer of the alkaloids into the tartaric-acid solution can be done efficiently. For the drum-sized batch, add 30 gallons of ether and two gallons of alcohol. Similarly, for smaller batches add three volumes of ether and a little alcohol.

At this point, an important matter must be addressed. This matter is central snoopervision of chemical transactions. Note the "Love

Letters From The Heat" section at the end of this book concerning the Chemical Diversion Trafficking Act of 1988, and its amendments since then. This federal law requires chemical dealers to "identify their customers, maintain retrievable records, and report suspicious transactions" for a list of chemicals compiled at the end of this book. Ether is on the mandatory snitch list in amounts above 25 gallons, and you can take it to the bank that regular chemical outlets will be following the letter of the law. You can also bet that connections met through the waste exchanges are mostly concerned with getting the stuff off their hands, not kissing up to the DBA. The serious experimenter may wish to try substituting benzene for ether, since it is not now on the mandatory snitch list.

The alkaloids are next extracted out of the ether solution into decimolar (15 grams per liter) tartaric acid in water. The alkaloids form a salt with the tartaric acid that is soluble in water, and leave the extraneous plant compounds in the ether. This extraction should be done four times with a volume of tartaric-acid solution that is one-seventh the volume of the ether solution. For example, with about 40 gallons of ether solution in a drum, extract with about 6 gallons of tartaric acid solution four times. This means a fresh six gallons on each extraction. If a stubborn emulsion forms, the addition of a little alcohol to the mix will break it.

Tartaric acid is the preferred acid for this extraction because the tartaric acid salt of the alkaloids is relatively stable in light. A .2N solution of sulfuric acid can be used instead if precautions are taken to protect the solution from exposure to light. This method may be preferable because it has become a hassle to buy tartaric acid. Recently, at my place of work, I had occasion to order one pound of Rochelle salts (potassium sodium tartarate) from a major chemical supplier. This material was for use in a laboratory scale cyanide copper plating bath, where the Rochelle salt acts as a complexor. To get them to sell me this material, I had to answer a battery of questions, in spite of the fact that the firm at which I work has had a long customer relationship with this major chemical supplier. Less scrutiny of tartaric acid purchases would likely be encountered from a firm which supplies chemicals to the plating industry. To get tartaric

acid from Rochelle salts, just dissolve them in water, and then add hydrochloric acid until the pH of the decimolar solution reaches 2.

The tartaric-acid solution containing the alkaloids should now be free-based, preferably with ammonia. The ammonia should be added slowly with vigorous stirring until the pH of the solution reaches 8 to 8.5. A higher pH must be avoided, since at these pHs racemization to the inactive iso form of lysergic occurs.

The free-based alkaloids can now be extracted out of the water solution into ether. The extraction should be done four times, each time with a volume of ether ^{1}A that of the water solution. The combined ether extracts should be dried over some magnesium sulfate previously wetted with ether to prevent it from absorbing alkaloid during the drying process.

Finally, the ether is evaporated away under a vacuum to yield a residue of fairly pure alkaloids. The alkaloids in this form are very fragile, and must be immediately transferred to a freezer for storage.

4
LSD Directly From The Lysergic Amides — The One-Pot Shot

When the lysergic amides have been extracted in pure form from the crop, work should begin without delay to convert it to LSD. Diligence in this matter is very important because possession of the extracted amides is strong evidence of intent to manufacture LSD. Further, mere possession of lysergic acid or ergine is prohibited as they are federal "controlled substances." The goal must be to get the hot potato out of one's hands and convert it to cash as fast as possible.

There are several possible methods to follow in the conversion of the lysergic amides to LSD. The first two presented in this book are excellent, and highly recommended. The third one is OK. Beyond that, we are talking last resort. In all cases, the overriding factor which must take precedence is ease of availability of the required chemicals. A bottle of trifluoroacetic anhydride in hand beats homemade anhydrous hydrazine in the bush.

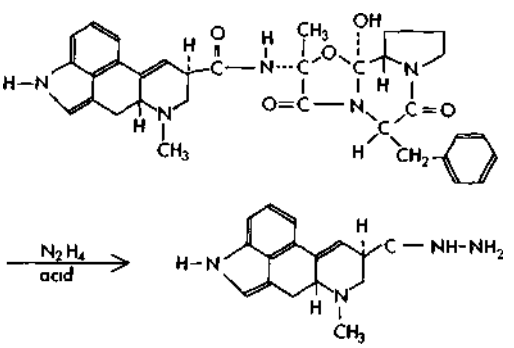
The first LSD manufacture method presented here is what I like to call "the one-pot shot." It can be found in US patent 3,239,530 and US patent 3,085,092, both granted to Albert Hofmann. This method uses anhydrous hydrazine to cleave the ergot amides to produce lysergic acid hydrazide. The hydrazide is then isolated by extraction,

and reacted with acetylacetone (2,4-pentanedione) to form a pyrazole intermediate, which is then reacted with diethylamine to form LSD.

This method at first glance seems complicated, but the actual manipulations involved here are less challenging than proceeding through lysergic acid. Further, the yields are higher with this method than those proceeding through lysergic acid, and there is less formation of the inactive iso-LSD than with other methods. Iso-LSD is not a complete loss since it can be converted to the active LSD, but it is best to avoid its formation in the first place.

This method has a serious drawback. Anhydrous hydrazine is not available off the shelf at your local hardware store, and attempts to procure it through normal channels are likely to catch the attention of those shit-eating dogs at the DBA. I include in this chapter directions for making your own anhydrous hydrazine, but be warned here that failure to use a nitrogen atmosphere during the distillation of anhydrous hydrazine will likely lead to an explosion. On that cheery note, let's begin!

Step One:
Conversion of Ergot Amides
to Lysergic Acid Hvdrazide



The reaction above is illustrated for ergotamine, but the process is just as valid when a mixture of amides is used as extracted from the

crop. Further, the crop amides have been left in the freebase form, so the procedure given in example 5 in US patent 3,239,530 is used. This is superior to trying to make a hydrochloride salt of the amides, as suggested in example 1, because this would expose the active ingredients to loss and destruction during the unnecessary handling.

There are three main precautions to be followed while executing this procedure. Water must be rigorously excluded from the reaction mixture, as hydrazine hydrate will react with the amides to form racemic lysergic acid hydrazide rather than our desired product. To ensure the exclusion of water from the reaction, the glassware should be baked in an electric oven prior to use, and be allowed to cool off in a dessicator. A drying tube should be attached to the top of the condenser used, to prevent humidity in the air from getting in the mix. Naturally, the hydrazine used had better be anhydrous.

Another danger to success is exposure to light. Work should be done under a dim red darkroom bulb. The flask containing the reaction mixture should be wrapped in aluminum foil to exclude light. Procedures such as extractions and filtering should be done as rapidly as possible without causing spills.

Finally, this reaction should be done under a nitrogen atmosphere, as hot hydrazine and oxygen do not get along too well.

In a 500 ml round-bottom flask place a magnetic stirring bar, 10 grams of the ergot amide mixture (dried in a vacuum dessicator to ensure its freedom from water), 50 ml of anhydrous hydrazine, and 10 ml of glacial acetic acid. A condenser equipped with a drying tube is then attached to the flask, and the flask wrapped in a single layer of aluminum foil. The flask is then lowered into a glass dish containing cooking oil heated to 140° C on a magnetic-stirrer hot-plate. When the flask goes into the oil, the heat should be backed off on the hot-plate so that both oil and flask meet each other in the middle at 120° C. Monitor the warming of the contents of the flask by occasional insertion of a thermometer. Stir at moderate speed. In about 10 minutes, the desired temperature range is reached, and some gentle boiling begins. Maintain the temperature of the oil bath at 120-125° C, and heat the batch for 30 minutes.

When 30 minutes heating at 120° C is complete, add 200 ml water to the batch, increase the oil temperature to 140° C, and rig the glassware for simple distillation. Distill off between 200 to 250 ml water, hydrazine hydrate and acetic acid mixture. Then remove the flask from the heated oil, and allow it to cool. Use of an aspirator vacuum to assist the distillation is highly recommended.

When the flask has cooled, add 100 ml of decimolar tartaric-acid solution (1.5 grams tartaric acid in 100 ml water) to the flask, and 100 ml ether. Stopper the flask, and shake vigorously for a few minutes, with frequent breaks to vent off built-up pressure from the flask. If the stirring bar bangs too violently in the flask, remove it with a magnet rather than break the flask.

Pour the contents of the flask into a 250 ml sep funnel, and drain the lower layer (water solution of lysergic acid hydrazide tartarate) into a 250 ml Erlenmeyer flask wrapped in foil. To the ether layer still in the sep funnel, add 50 ml fresh decimolar tartaric-acid solution, and shake. Examine the water layer for the presence of lysergic acid hydrazide with a black light. If there is a significant amount, add this also to the Erlenmeyer flask.

Place the magnetic stirring bar in the Erlenmeyer flask, and stir it moderately. Monitor the pH of the solution with a properly calibrated pH meter, and slowly add .5M (20 grams per liter) sodium hydroxide solution until the pH has risen to the range of 8-8.5. Higher pH will cause racemization. The freebase is then extracted from the water solution with chloroform. Two extractions with 100 ml of chloroform should complete the extraction, but check a third extraction with the black light to ensure that most all of the product lysergic acid hydrazide has been extracted.

The chloroform extracts should be evaporated under a vacuum in a 500 ml flask to yield the product. This is best done by rigging the 500 ml flask for simple distillation, and applying an aspirator vacuum to remove the chloroform. Assume that the yield from this procedure will be about 5 grams of lysergic acid hydrazide if ergot was the crop used. Assume that the yield will be about 7.5 grams if seeds were used. The difference here is due to the fact that in ergot, the amides

are largely composed of substances in which the portion lopped off is about as large as the lysergic acid molecule. Seeds tend to be more conservative as to their building upon the lysergic molecule. A careful weighing on a sensitive scale comparing the weight of the flask before and after would give a more exact number.

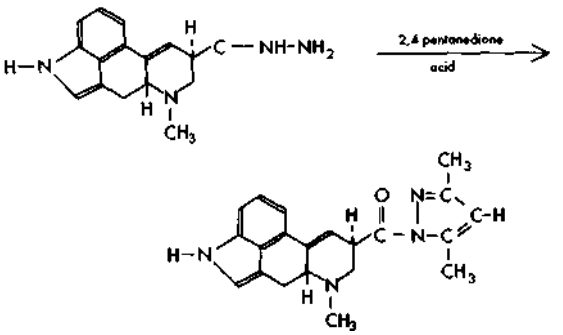
Both of these choices are really very poor, because lysergic acid hydrazide, unlike most other lysergic compounds, crystallizes very well with negligible loss of product. At the hydrazide stage of LSD manufacture, one has a perfect opportunity to get an exceedingly pure product, freed from clavine alkaloids and other garbage compounds carried in from the extraction of the complex plant material.

I refer the reader to US patent 2,090,429 issued to Albert Hofmann and Arthur Stoll, the dynamic duo of lysergic chemistry, dealing with lysergic acid hydrazide. In this patent, they describe in a rather excited state how they were able to produce pure lysergic acid hydrazide from tank scrapings that were otherwise impure junk.

Lysergic acid hydrazide has the following properties: it dissolves easily in acid, but is very difficultly soluble in water, ether, benzene and chloroform. In hot absolute ethanol it is slightly soluble, and is crystallizable in this solvent to yield "beautiful, compact, clear, on six-sided cut-crystal plates that melt with decomposition at 235-240° C."

This is obviously the way to go. The hydrazide should be recrystallized from absolute ethanol, and then dried under a vacuum to remove residual alcohol clinging to the crystals. About 300 ml of hot ethanol is required to dissolve each gram of lysergic acid hydrazide during the crystallization. Upon cooling, a first crop of pure lysergic acid hydrazide is obtained. Then, by boiling away half of the mother liquor and cooling, an additional crop is obtained. This process can be continued as long as the crystals obtained look nice.

Step Two: Lysergic Acid Pyrazole



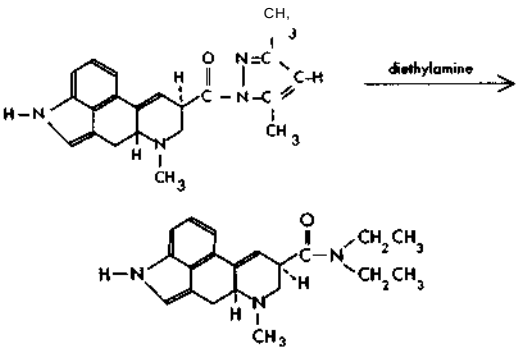
In this reaction, one mole of lysergic acid hydrazide is dissolved in an inert, water-miscible solvent like ethanol. Then an excess of 1-molar hydrochloric acid is added to form a salt with the lysergic acid hydrazide. To this mixture is then added two moles of acetylacetone (2,4-pentanedione), which forms the desired pyrazole. This reaction is not nearly as touchy as the formation of the hydrazide. The presence of traces of moisture from the air poses no problem. 2,4-pentanedione finds use in analytical chemistry as a chelating agent for transition metals, and as such should be available without raising too many red flags. Synthesis of this compound is not hard, and directions for doing so are found in US Patents 2,737,528 and 2,834,811.

To do the reaction, the flask containing the 5 grams of hydrazide is wrapped in a single layer of foil to exclude light. Then a magnetic stirring bar is added, along with 18 ml of ethanol, 18 ml water, 20 ml 1-molar HC1 (made by adding one part 37% HC1 to 11 parts water) and this mixture is stirred for a few minutes. Then 3.5 grams (3.5 ml) of 2,4-pentanedione is added at room temperature, and the stirring continued for an hour or so.

The product is recovered from solution by the slow addition with stirring of 20 ml 1-molar NaOH (40 grams per liter). This neutralization throws the pyrazole out of solution as a solid. The solid is collected by filtration through a Buchner funnel, and rinsed off with

some water. The crystals are then dried under a vacuum, preferably with the temperature elevated to 60° C. Further purification can be done by crystallization. If so desired, dissolve the crystals in chloroform, then add 8-10 volumes of ether to precipitate the product. I do not feel this is necessary if the hydrazide used was reasonably pure, since all the reagents used in the last step are soluble in water. The water rinse should have carried them away. Further, alcohol and 2,4-pentanedione are volatile, and would be removed in the vacuum drying.

Step Three:
LSD



This simple and easy reaction is done as follows: In a flask wrapped in a single layer of foil are placed 1 gram lysergic acid pyrazole, and 30 ml diethylamine. Diethylamine is a definite "do not purchase" item. Easy directions for its synthesis are given in this chapter. The two ingredients are swirled until mixed, then allowed to stand at room temperature for about a day.

The excess diethylamine is then distilled off, and saved for use in future batches. Dimethylpyrazole is a high-boiling-point substance, and easily separated from diethylamine. When most of the diethylamine has been distilled off, a vacuum is applied, and the residue is evaporated to dryness. The evaporation is completed by

warming the flask in boiling water for a few minutes with continued application of vacuum. The residue is almost pure LSD.

Purification and Storage

At this point, the process has yielded LSD freebase. In this state, the substance is quite unstable and not suitable for storage. A judgment as to the purity of the product is therefore needed in quick order, because which method of further processing to use is dependent upon the purity of the product. If there is reason to believe that a significant amount of iso-LSD is mixed in with the product, the following chromatographic separation is called for. The iso-LSD can then be recovered and converted to the active LSD, which greatly increases the value of the product. Iso-LSD can be expected to be formed using the process in this chapter if the additions of sodium hydroxide were not sufficiently slow, and local areas of high pH developed in the solution. Using methods in other chapters proceeding through lysergic acid, a large amount of the iso product can be expected if lysergic acid was made by use of hydrazine hydrate or HOH. Also, some of the natural alkaloids are of the iso form and yield iso-LSD. The procedure for acid production using trifluoroacetic anhydride will always make a lot of the iso product. The best procedure I can recommend is: whatever method has been used, check the product through chromatography for the presence of the iso-LSD. The following procedure is taken from US patent 2,736,728.

3.5 grams of LSD freebase is dissolved in 160 ml of a 3-1 mixture of benzene and chloroform (120 ml benzene, 40 ml chloroform). Next, a chromatography column is constructed from a burette. It must hold about 240 grams of basic alumina (not acidic alumina), so a 100 ml burette is called for. A wad of cotton and filter paper is stuffed down the burette against the stopcock to keep the particles of alumina from flowing out. The 240 grams of basic alumina are then poured into the burette with tapping to assure it is well packed. The alumina should then be wetted with some 3-1 benzene-chloroform.

Now the 160 ml of benzene-chloroform containing the LSD is run slowly into the burette, followed by more benzene-chloroform to develop the chromatogram. As the mixture flows downward through the alumina, two zones that fluoresce blue can be spotted by illumination with a black light. The faster-moving zone contains LSD, while the slower-moving zone is iso-LSD.

When the zone containing LSD reaches the spigot of the burette, it should be collected in a separate flask. About 3000 ml of the 3-1 benzene-chloroform is required to get the LSD moved down the chromatography column, and finally eluted.

The iso-LSD is then flushed from the column by switching the solvent being fed into the top of the column to chloroform. This material is collected in a separate flask, and the solvent removed under a vacuum. The residue is iso-LSD, and should be stored in the freezer until conversion to LSD is undertaken. Directions for this are also given in this chapter.

For the fraction containing the LSD, conversion to LSD tartrate must be done to make it water soluble, improve its keeping characteristics, and to allow crystallization. Tartaric acid has the ability to react with two molecules of LSD. Use, then, of a 50% excess of tartaric acid dictates the use of about 1 gram of tartaric acid to 3 grams of LSD. The three grams of LSD would be expected from a well-done batch out of a total 3.5 LSD/iso-LSD mix.

The crystalline tartrate is made by dissolving one gram of tartaric acid in a few mis of methanol, and adding this acid solution to the benzene-chloroform elute from the chromatography column. Evaporation of the solvent to a low volume under a vacuum gives crystalline LSD tartrate. Crystals are often difficult to obtain. Instead, an oil may result due to the presence of impurities. This is not cause for alarm; the oil is still likely 90%+ pure. It should be bottled up in dark glass, preferably under a nitrogen atmosphere, and kept in a freezer until moved.

If chromatography reveals that one's chosen cooking method produces little of the iso products, then the production of the tartrate salt and crystallization is simplified. The residue obtained at the end

of the batch is dissolved in a minimum amount of methanol. To this is then added tartaric acid. The same amount is added as above: one gram tartaric acid to three grams LSD. Next, ether is slowly added with vigorous stirring until a precipitate begins to form. The stoppered flask is then put in the freezer overnight to complete the precipitation. After filtering or centrifuging to isolate the product, it is transferred to a dark bottle, preferably under nitrogen, and kept in the freezer until moved.

LSD from (so-LSD

Two variations on this procedure will be presented here. The first is the method of Smith and Timmis from *The Journal of the Chemistry Society* Volume 139, H pages 1168-1169 (1936). The other is found in US patent 2,736,728. Both use the action of a strong hydroxide solution to convert iso material into a mixture that contains active and iso material. At equilibrium, the mixture contains about 2/3 active material and 1/3 iso material. These substances are separated by chromatography, and the iso material saved to be added to the batch the next time isomerization is done. In this way, eventually all of the product becomes active material.

Method One

The iso-LSD as eluted from the chromatography column is first evaporated under a vacuum to remove the solvent. The residue is then dissolved in 1-molar alcoholic KOH, and boiled under reflux, preferably with a nitrogen atmosphere, for 30 minutes.

The mixture is next cooled and diluted with 3 volumes of water. It is next acidified with HC1, then made alkaline again with sodium carbonate. The product is now extracted from solution with ether or chloroform. After removal of the solvent, the product can be chromatographed as previously described.

Method Two

The iso-LSD as eluted from the chromatography is first evaporated under a vacuum to remove the solvent. The residue is dissolved in the minimum amount of alcohol, and then one half volume of 4-molar KOH in 100 proof vodka is added. The mixture is allowed to sit at room temperature for a couple of hours, then the alkali is neutralized by adding dry ice. The solvents are next removed under a vacuum, and the residue chromatographed as previously described.

Preparation of Anhydrous Hydrazine

Anhydrous hydrazine can be made from the easily available raw materials: bleach, ammonia, sulfuric acid and potassium hydroxide. This is not a task to be undertaken lightly, as there are dangers inherent in the process. Hydrazine will likely detonate during distillation if the distillation is not done in a nitrogen atmosphere. Also, hydrazine is a vicious poison prone to absorption through the skin or by inhalation of its vapors. It is very corrosive to living tissue, and its burning effects may be delayed. Hydrazine can also be assumed to be a carcinogen. All steps in its preparation must be done with proper ventilation, and protection of the body from spills.

Step One: Hydrazine Sulfate

$$2NH_3 + NaOCl \longrightarrow NH_2NH_2 + H_2O + NaCl$$

$$NH_2NH_2 + H_2SO_4 \longrightarrow NH_2NH_2H_2SO_4$$

Into a 3-quart-capacity glass baking dish (Pyrex) put 750 ml strong ammonia (28% NH_3), 350 ml distilled water, 190 ml 10% gelatine solution, and 700 ml 12.5% bleach. This strength of bleach is

available from pool supply companies and makers of cleaners. The 5.25% strength Clorox will not do here. One must also be aware that traces of iron and copper have a very bad effect upon the yield, so do not dispense with the use of distilled water. The bleach is another possible source of iron. In checking out this reaction, the Pro Chemicals brand of bleach worked fine. I can't vouch for other brands. If all else fails, the bleach can be made from chlorine and NaOH in distilled water. (See *Organic Syntheses Collective* Volume 1, page 309.) The Pro Chemicals brand of bleach analyzed at 10 ppm iron by atomic absorption, and this amount did not interfere with the reaction. One must also check the bleach to make sure it is alkaline, as free chlorine prevents the formation of hydrazine.

When the ingredients have been mixed in the baking dish, it is heated as rapidly as possible until it has been boiled down to one-third of its original volume. Being a wimp and boiling it down too slowly reduces the yield. Take not more than two hours.

The dish is then removed from the heat, and allowed to cool. When the dish nears room temperature, it should be nestled in ice to chill thoroughly. The solution should then be filtered to remove suspended particles from the solution.

The filtered solution is next put in a beaker, and nestled in ice mixed with salt until the temperature of the solution reaches 0° C. When that temperature is reached, 10 ml of concentrated sulfuric acid for each 100 ml of solution is slowly added with constant stirring. If the stirring is not strong, or if the filtering was poorly done, a product contaminated with brown particles results. If done well, hydrazine sulfate precipitates as white crystals. The mixture is allowed to stand in the cold for a few hours to complete the precipitation. The crystals are then filtered by suction, and the crystals rinsed off with cold alcohol. The yield is 25 to 30 grams of hydrazine sulfate.

Step Two: Hydrazine Hydrate

Mix 100 grams dry hydrazine sulfate with 100 grams powdered KOH and place the mixture into a copper and silver retort. Then add 15 ml water, and distill off the hydrazine hydrate formed though a downward-inclined glass condenser. There is little need for heat to be applied at the beginning of the distillation because so much heat is generated in the reaction between the KOH and the sulfate. Later, strong heating is required to distill out the last of the hydrazine hydrate.

This crude product contains water beyond the monohydration of hydrazine. It is purified by fractional distillation. Pure hydrazine hydrate boils at 117° C to 119° C. The forerun contains the excess water. It should be converted back to hydrazine sulfate by addition of sulfuric acid as done in step one. The yield is 10 grams of hydrazine hydrate.

During the fractional distillation, there are some precautions which should be followed. Hydrazine hydrate attacks rubber and cork, so the use of these materials must be avoided in the distillation. It also attacks most kinds of stopcock grease. The distillation is most safely done under nitrogen. Nitrogen should be introduced into the distilling flask, and the system flushed of air for about 15 minutes. Then the rate of nitrogen flow is reduced, and distillation commenced. The product will also attack glass, albeit slowly. It should be stored in 304 or 347 stainless steel. 316 stainless is not acceptable.

Step Three: Anhydrous Hydrazine

100 grams (100 ml) of hydrazine hydrate is mixed with 140 grams powdered sodium hydroxide. The apparatus is thoroughly flushed with nitrogen, then the rate of nitrogen addition to the distilling flask

is slowed, and fractional distillation is commenced through an efficient fractionating column of about 15 theoretical plates. Anhydrous hydrazine distills at 112° C to 114° C. Anhydrous hydrazine is obtained at *99%+* purity.

Another method for producing anhydrous hydrazine exists which gives a higher yield of product, but it uses anhydrous ammonia and more complicated glassware and procedures. See *Journal of the American Chemical Society* Volume 73, page 1619 (1951), and Volume 76, page 3914 (1954). Also see *Hydrazine* by C.C. Clark, *The Chemistry of Hydrazine* by L.F. Audrieth, and *Industrial and Engineering Chemistry* Volume 45, pages 2608 and 2612 (1953). Also see *Inorganic Syntheses* Volume 1, page 90 (1939).

Anhydrous hydrazine can be stored in dark glass bottles under refrigeration for years.

Other variations on the alkali hydroxide dehydration of hydrazine hydrate exist which give higher yields of less-pure hydrazine. See pages 48-54 in the *Chemistry of Hydrazine* mentioned above. It lists many references. Especially interesting is *Journal of the American Chemical Society* Volume 71, pages 1644-47 (1949).

Preparation of Diethylamine

$$NH_3 + CH_3CH_2I \longrightarrow xHI + CH_3CH_2NH_2 + (CH_3CH_2)_2NH + (CH_3CH_2)_3N$$

The reaction which produces diethylamine also yields as by-products ethylamine and triethylamine. The relative amounts of each compound produced depends upon the molar ratio of the two starting materials. Use of only a little ethyl iodide favors the formation of mostly ethylamine. Use of a lot of the ethyl iodide favors the formation of triethylamine. Somewhere in the middle, a roughly even split occurs. This will be done here. See *Journal of the American Chemical Society* Volume 69, pages 836 to 838 (1947).

A section of clean steel pipe *2l/2* to 3 inches in diameter is obtained, and fine threads are cut into each end so that a cap may be screwed onto each end. A really nice touch would be to have all the pieces plated with a half-thousandths-inch of electroless nickel, but the plater may think you are constructing a pipe bomb when he sees the pipe and caps.

The bottom of the pipe is secured by screwing the cap on over threads coated with Teflon tape. Welding may also be used. The pipe is then nestled into a Styrofoam cooler, and is then filled about *Vi* full of rubbing alcohol, and then to this solvent dry ice is added, slowly at first to prevent it from boiling over, then more rapidly. The top of the pipe should be covered to prevent frost from forming inside the pipe as it cools down.

Next, add 175 ml of ethyl iodide to the pipe, and let it cool down. It will not freeze, as its melting point is about 100° below O° C. Then liquid ammonia is added to the pipe. This is best done by inverting a cylinder of liquid ammonia, attaching plastic tubing to the valve, and cracking open the valve to feed the liquid into the pipe. About 525 ml of liquid ammonia is called for. In a 3-inch-diameter pipe, that plus the ethyl iodide will fill it half full. This is not an operation to be done in a residential neighborhood, as the fumes are tremendous. A rural setting with beaucoup ventilation is more proper.

Now secure the top of the pipe by screwing on the cap tightly over Teflon tape. The pipe is now moved into a tub of ice water, and allowed to sit in this ice water for 45 minutes to an hour to warm up to 0° C.

When the pipe has warmed to O° C, it should be shaken to mix the two reactants, and returned to the ice water. This shaking should be repeated a few times at 5-minute intervals. When 30 minutes have passed from the first shaking, the pipe should be returned to the dry ice bath and allowed to cool.

When the pipe has cooled, the cap on the top of the pipe is loosened. Then the pipe is returned to the tub of ice water, and the ammonia is allowed to slowly evaporate away. This will take overnight, and raise great plumes of stink.

After most of the ammonia has evaporated, the contents of the pipe should be emptied into a beaker. The foul substance is a mixture of ammonia, ethlyamine, diethylamine, triethylamine, and the hydriodides thereof. The best route to follow is to cool this mixture in ice, and slowly add with stirring 90 grams of sodium hydroxide dissolved in 100 ml of water. This neutralizes the HI in the mix, yielding the freebases of all.

This mixture should be extracted several times with toluene. Toluene is chosen because it is available at the hardware store, and its boiling point is higher than any of the amines. The extracts should be filtered, and dried over sodium hydroxide pellets.

The toluene extracts should then be transferred to a flask, and the mixture fractionally distilled through an efficient column. Ethylamine distills at 16° C, diethylamine distills at *55*° C, and triethlyamine distills at 89° C. The diethylamine fraction should be collected over a 20-degree range centered on 55° C, and this fraction then redistilled to get the pure product. The yield of diethylamine is about 40 ml. Absolute freedom from water in the product can be assured by letting the crude distillate sit over a few chips of KOH for a few hours prior to the final distillation.

Preparation of Tartaric Acid

My experience with the chemical scrutinizers while ordering a pound of Rochelle salts should serve as a lesson to those embarking upon LSD manufacture. Substances which are useful for this purpose will raise red flags if obtained through normal channels. It must then be the highest priority to avoid these normal channels, or to subvert their scrutiny by preparing yourself those substances with direct use in the synthesis.

The most low-profile method for getting tartaric acid is to follow the procedure given below. It uses cream of tartar from the grocery store and gives good results. See *Chemical Engineering Progress* Volume 43, page 160 (1947). Also *Organic Syntheses Collective* Volume 1 for alternate procedures. I worked out this procedure by

myself in my lab, and it gives good results. That such a simple procedure, using such easily obtained materials, so effectively subverts the feds' control over tartaric acid shows what a bunch of ninnies they really are.

To make tartaric acid suitable for use in making the tartaric salt of LSD, weigh out 10 grams of cream of tartar, and put it into a 100 ml beaker. I used McCormick brand, and it was nicely white and fluffy. Other brands will do, so long as they too are white and fluffy.

To the 10 grams of cream of tartar, add water until the 50 ml mark is reached in the beaker. This produces a milky white suspension. Stir for a while to try to dissolve as much as possible, then add 10 ml 37% lab-grade hydrochloric acid. The mixture of calcium tartarate and potassium hydrogen tartarate that comprises cream of tartar reacts to form tartaric acid, along with KC1 and $CaCl_2$- A clear solution results after about a minute of stirring.

Now the water and excess hydrochloric acid are removed by vacuum evaporation. It is preferable to use a vacuum here, as heating at normal pressure may result in isomerization of the tartaric acid, and the replacement of some of the hydroxyl groupings in tartaric acid with chlorine. Also, hydrochloric acid was used here instead of sulfuric because the reaction is much faster, and the excess HC1 is removed during the evaporation. The solution should be evaporated down to a volume of about 10 ml. It will be yellowish in color, and have crystals of tartaric acid floating around in it, along with KC1 and $CaCl_2$.

Next, add 100 ml of 91% isopropyl alcohol, and dissolve the crystals of tartaric acid. KC1 and CaCh will not dissolve, and should be filtered out. 91% isopropyl alcohol is chosen because it is available at the drugstore, is not too good a solvent for tartaric acid for crystallization, and is less likely to form esters with tartaric acid than ethyl or methyl alcohol.

The isopropyl alcohol is evaporated under a vacuum to 50 ml volume, and the first crop of white crystals of tartaric acid collected. This amounts to about 4 grams after drying. Further evaporation yields additional crops of crystals. Vacuum evaporation is used so that

heating does not contribute to the formation of the ester isopropyl tartrate.

5
Lysergic Acid

All of the production methods from here on out use lysergic acid as the starting material. These methods may be preferable if the alkaloids have been extracted from seeds rather than ergot, because the crystallization of lysergic acid affords an excellent opportunity to remove the clavine alkaloids present in the seeds.

Two methods will be presented here. Method number one uses easily available KOH and methanol to cleave the amides to lysergic acid. Method number two uses hydrazine hydrate, which can be made from bleach and ammonia according to the directions in the previous chapter. The first method gives about 50% yield, while the yield in the second method is better. Both methods give a mixture of regular and iso lysergic acid, leading to mixtures of regular and iso-LSD. This makes the chromatographic separation procedure a must for all methods using the lysergic produced according to the directions given here.

Method One

Ten grams of lysergic amides extracted from the crops are dissolved in 200 ml of methanol containing 11 grams KOH. The methanol is

then removed at once by distillation under a vacuum. To the residue in the flask, then add 200 ml of an 8% solution of KOH in water. This mixture should then be heated on a steam bath for one hour.

Next, the reaction mixture should be cooled, and sulfuric acid added to it until it reaches pH 3. This results in the precipitation of crude lysergic acid having a dark color.

The acid solution should next be extracted several times with ether. These extractions remove a lot of the lopped off portions of the lysergic amides, and lighten up the color of the lysergic acid. The acid suspension should next be filtered to yield dark colored crude crystals of lysergic acid.

These crude crystals should be transferred to a beaker, and taken up in solution with two 200 ml portions of ethyl alcohol containing a few mis of strong ammonia. The residue which does not dissolve is inorganic, and can be discarded.

The alcohol solution of lysergic acid should be evaporated to dryness under a vacuum. The crystals should be ground quickly while soaking for a short period of time in 50 ml methanol to remove colored impurities, then filtered. This yields about *2Y2* grams lysergic acid. It should be dried in a vacuum dessicator, then stored in the freezer. The lysergic acid even after vacuum-drying holds one molecule of water as part of the crystal structure. This is not a problem if the method given in Chapter 6 is used. Other synthesis methods require the removal of this water of crystallization, and it is tough. A vacuum of 2 mm Hg and a temperature of 140° C is needed to remove it. Such methods are best avoided if possible. Reference: *Journal of Biological Chemistry*, Volume 104, page 547.

Method Two

As mentioned before, this method gives higher yields, and so it is highly recommended. An increase in yield from 50% to 75% translates into 50% more LSD produced from the crops. This is well-worth the hassle involved with scrounging up or making some hydrazine hydrate.

To do the hydrolysis, 15 grams of lysergic amides from the crops is put into a 500 ml flask along with a solution made up of 150 ml ethyl alcohol, 150 ml water, and 100 grams KOH. Next, 15 ml of hydrazine hydrate is added. This hydrazine should be the monohydrate, which is 64% hydrazine. If a weaker variety has been scrounged up, this can be made to work by adding more, and using less water.

Now the flask should be fitted with a condenser, and flushed with nitrogen. Then heat the flask in an oil bath to gentle boiling for 4 hours. A slow stream of nitrogen to the flask during the reflux averts the danger from hydrazine.

The flask is next cooled, and the contents poured into a sep funnel of at least 1000 ml capacity. The batch is then extracted with 600 ml ether, followed by 600 ml of an 85-15% mix of ether and alcohol. Finally, one more extraction with 600 ml of 85-15% ether-alcohol is done.

All of the desired product should now be extracted into the solvent, and out of the water. This fact should be checked using a black light to look for the characteristic blue fluorescence.

The combined solvent extracts should now be lowered to a pH of about 2 using HC1. At this point, a precipitate should form, and it should be filtered out. The precipitate should be washed free of entrained product with 4-1 ether-alcohol, and the washing added to the rest of the filtered solvent.

Now 2750 ml of water should be added to the solvent, and the mixture placed in a gallon and a half glass jug or 5000 ml beaker. To this should be added 3 portions of cation exchange resin in H* cycle. Cation exchange resin is a common item of commerce used in deionized water systems. Check the yellow pages under "water" and see which of the local Culligan men offer deionized water systems. The deionizers come in two-tank systems with one tank packed with cation exchange resin to remove calcium, magnesium and sodium from the water. The other tank has an anion exchange resin to remove chlorides, sulfates, and so on. It is no great task to buy cation exchange resin from these outlets. The resin consists of tiny plastic beads coated with the exchanger. In the case of the cation exchangers,

this is generally a sulfonate. "In H* cycle" means that the resin is charged up and ready to go. This is generally done by soaking the resin in 20% sulfuric acid in water for a while, then rinsing with distilled water. Check the directions on the container of resin. Steer clear of mixed resins that contain both anion and cation exchangers. If the Culligan man is too stupid to know the difference, or doesn't know what he has, keep looking until you find one who knows his business.

The treatment with three portions of cation exchange resin in H* cycle should be done as follows: Each portion of resin should weigh about 15 grams. The first portion is added, and then the mixture should be stirred strongly or shaken for about 10 minutes. The product will come out of the liquid, and stick to the resin. The resin should be filtered out, and kept in the fridge while similar treatment proceeds with the next two portions of cation exchange resin.

All of the product should now be out of the liquid and on the resin. This should again be checked using the blacklight.

The resin portions are now combined, and soaked in 300 ml of 10% NRjOH in water for 30 minutes with stirring. This brings the product off the resin, and into the ammonia solution. The slurry should now be filtered to give a brown liquid which is kept in the fridge. The resin should be treated again with 300 ml of 10% NHtOH, and filtered.

Now the 600 ml of ammonia solution containing lysergic acid should be evaporated down in a vacuum to a volume of 50 ml, and this remaining liquid kept in the fridge overnight at 4 C to yield a precipitate of about 5'/z grams of 96% pure lysergic acid. It consists of lysergic acid and iso-lysergic acid in about a two-to-one ratio.

The resin can be used over and over again by recharging in 20% sulfuric acid solution, and rinsing with distilled water.

Reference: *Chem Abstracts,* Volume 69, column 36323 (1968) Czech patent 123,689

Notes:

1. The blacklight is your friend, and is very useful in spotting the product, but don't overuse it as UV is quite harmful to the product. The blacklight should be a fluorescent tube, and not some black painted light bulb.
2. All work described in this chapter should be done under red or yellow darkroom lighting.

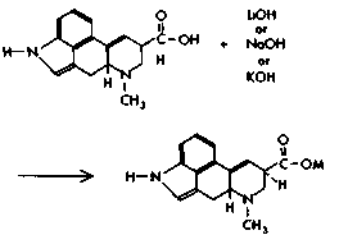
6
LSD from Lysergic Acid and SO_3

This is the second of the two excellent methods of LSD synthesis. It gives very good yields of high-quality product, if two precautions are followed. The first point on which success hinges is the requirement that a rather strict stoichiometry (stoichiometry concerns the proportions of different chemicals used in reactions) be followed in both the amount of alkali reacted with the lysergic acid to form the salt of lysergic acid, and the amount of SOs then added to form the mixed anhydride of lysergic acid.

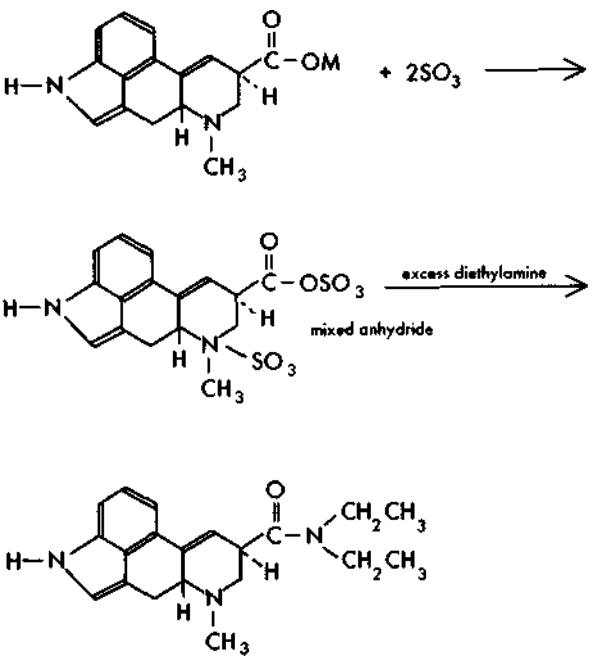
The other key precaution is the need to maintain strictly anhydrous conditions in both the production of the SO_3-solvent complex, and the reaction of that complex with the lysergic acid salt to produce the mixed anhydride. The reason for this is that SOs is the anhydride of sulfuric acid, and any traces of moisture will react with it to produce sulfuric acid. Sulfuric acid does not react with lysergic acid to form an anhydride. Instead, it just messes up the stoichiometry of the reaction, leading to greatly reduced yields.

To prevent moisture from interfering with the reaction, glassware should be baked in an electric oven for an hour or so, and then allowed to cool down in a dessicator. High humidity must be avoided, so this is not work suitable for a damp basement or even reasonably

humid days. Air conditioning, or winter's dry indoor heated air are best. Solvents and reagents must be free of water. The reaction works as follows:



Preparation of Sulfur Trioxide Complex



Work begins with the preparation and standardization of SOs-solvent complex. SOs is available from a couple of sources. There is a

form of pure stabilized SOs called Sulfan B. If this material can be had off of some unguarded shelf, it is superior to the other source of SOa, fuming sulfuric acid.

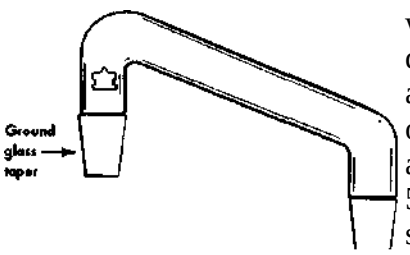
To make the SOs-solvent complex using Sulfan, a 2000 ml flask is charged with a magnetic stirring bar and 1000 ml acetonitrile. Dimethylformamide can also be used as the solvent, but the authors of the patent for this process evidently preferred acetonitrile for the production of LSD. The solvent should come from a freshly-opened bottle made by a reputable manufacturer. The bottle will list the water content, generally a few-hundredths percent. This amount of water will not pose a problem.

Next, the flask is fitted with a condenser and a dropping funnel, both being equipped with a drying tube to prevent the atmospheric moisture from infiltrating the reagents. The flask is nestled into a plastic or Styrofoam tub containing ice water, and the solvent allowed to cool down. When the temperature in the flask gets down to *5*- C, stirring is begun, and 40 grams of Sulfan should be put into the dropping funnel. The Sulfan should be dripped into the solvent slowly and cautiously over a period of an hour or two, while maintaining the temperature inside the flask in the 0-5° C range. A crystalline precipitate may form during the addition. If it does, continue stirring for another hour or so to bring it into solution. If it still fails to dissolve, add more solvent. Acetonitrile-SOs complex is generally used at a strength of .5 molar, while dimethylformamide-SOs complex is used at 1 molar strength. 80 grams per liter SOs is 1 molar. Using Sulfan fresh from the bottle, it is not necessary to analyze the strength of the resulting SOs-solvent complex so long as complete dissolution is achieved.

The procedure for making SO_3-solvent complex from fuming sulfuric acid is more complicated, but less likely to arouse suspicion since fuming sulfuric acid has a lot more uses than Sulfan. It is also far more likely to be available via the five-finger discount method.

Fuming sulfuric acid comes in a variety of strengths, but the ACS reagent contains 30% SO_3 or oleum. Pure SOs boils at 45° C, and at room temperature has a vapor pressure of over 400 mm Hg. That is

why the stuff fumes, and why the stuff can be removed from the sulfuric acid in which it is dissolved. A simple although time-consuming method for preparing SOa-solvent complex from fuming sulfuric acid is to use an adapter such as the one pictured in Figure 2.



Adapter used in preparing SOj-solvent complex from fuming sulfuric acid.

With all glass ware thoroughly dry, one can attach a 1000 ml flask on one side of this adapter and put 500 ml of fuming sulfuric acid in it. figure 2 '— On tne other side of the adapter, a 2000 ml flask can be attached containing 1000 ml of acetonitrile or dimethylformamide. The use of stopcock grease should be avoided, as SOs will attack it. Rather the joints should be sealed by wrapping parafilm around them.

There will be a tendency for the two solutions to come into a vapor equilibrium. 30% oleum contains about 580 grams per liter SOa. The vapors will over time work their way into the solvent and form complexes. It will take some time, depending upon the temperature, for enough fumes from the sulfuric acid to work their way out of the acid and into the solvent. Slow magnetic stirring in the solvent helps to maintain a homogenous mixture, and speeds absorption of SOa fumes. Cooling the solvent in ice can't hurt either.

Analysis of the solvent should be done after about 12 hours have passed. The need for stirring is especially crucial here so a representative sample is taken. To analyze, remove exactly 2 ml of solvent with a pipette and squirt it into 50 ml of distilled water. Add some phenolphthalein indicator, or monitor pH with a meter. Now titrate with .IN NaOH (prepared by dissolving exactly 4 grams of NaOH pellets in one liter of water) until the color of the solution turns

pink, or the pH meter shows pH 7. Record the amount of NaOH solution used.

Molarity SO, in solvent = mis NaOH used / 40

So a 1-molar SO_3 complex will require 40 ml of .IN NaOH to neutralize it. Two equivalents of NaOH react per sulfuric acid.

If after 12 hours, the solvent has still not absorbed enough *SO),* just let the process continue. The complex formed need not be exactly .5M in acetonitrile, or 1 M in dimethlyformamide, just close to those values. What is important is that the exact strength of complex formed be known, because that dictates just how much of SOa solution is used. That is crucially important to the success of the reaction.

When the SOa-solvent complex has reached the desired strength, the flask containing it should be stoppered with a glass or Teflon stopper, and kept in the fridge. It will gradually darken first to yellow and then orange, but it is good for at least 3 or 4 months.

The argument can be made that this procedure is wasteful of fuming sulfuric acid. After all, maybe only 2 liters of 1-molar SO_3 complex can be reasonably made from a pint of fuming sulfuric acid by this passive fume-absorption method. When one considers that this is enough SO_3 to make 3 million doses, however, such objections are silly.

Batch Production

With SO_3 complex in solvent prepared and carefully standardized to evaluate its exact strength, attention can be turned to LSD synthesis using lysergic acid and SO_3 complex. Exact weighing of ingredients, and assuring that they are free from water are the two main concerns in this synthesis. To that end, the lysergic acid crystals obtained by the methods given in Chapter 5 should be dried without heating under a vacuum for about an hour. This will remove all but the water of crystallization, which poses no problem. The scale used to portion out

the ingredients for this synthesis should at least be a very sensitive triple-beamer, and its accuracy should be checked using new corrosion-free brass weight standards. Atmospheric humidity is a very real threat. NaOH, KOH, and lysergic acid will all pull water from the air. This not only makes accurate weighing impossible, but it also introduces water to the batch. For this reason, air conditioning or the dry indoor heat of winter are best during the unavoidable handling and weighing of reagents.

Two methods will be presented here, the first being the specific synthetic method for LSD given in example ten of US Patent 2,774,763. The other is the general method given in *Journal of Organic Chemistry* Volume 24, pages 368 to 372. Both are authored by William Garbrecht, a true hero of LSD synthesis. The patent dates from 1955, while the *Journal* article dates from 1958.1 leave it to the serious experimenter to decide which is more advanced. No doubt, both are operable.

Patent Method

15 grams of lysergic acid is quickly weighed out, and placed in a dried 1000 ml flask equipped with a magnetic stirring bar. 200 ml of methanol is added to dissolve the acid, then the flask is stoppered while either 2.22 grams lithium hydroxide hydrate, or 2.09 grams sodium hydroxide pellets or 2.94 grams KOH pellets is weighed out and dissolved in 200 ml methanol. The use of lithium hydroxide is preferred because it doesn't absorb water from the air, thereby messing up the weighing. Lithium hydroxide, on the other hand, is not a very common item, and will raise red flags that attract unwelcome attention.

NaOH and KOH, however, are very mundane items. Further, a freshly opened bottle containing them can safely be assumed to be free of water. Quick weighing under low humidity will not add appreciable amounts of water to it. If the choice was mine to make, I would use NaOH or KOH.

The LiOH or NaOH or KOH solution is now added to the methanol solution containing lysergic acid. After a period of stirring to assure complete reaction to the metal salt of lysergic acid, the solvent is distilled off under a vacuum, leaving a bubbly residue clinging to the glass at the bottom of the flask. If the lysergic acid is pure, such as that made by method 2 in Chapter 5, this residue will have a glassy appearance. No heat stronger than steam or hot water should be used to drive the distillation.

The residue in the flask still contains traces of water and methanol. The water comes from the reaction of the hydroxide with the acid, and from the lithium hydroxide, if that was used. This is removed azeotropically. Add 500 ml of hexane to the flask, and distill off about half of it, using a fractionating column. Both water and methanol form azeotropes with hexane.

The approximately 250 mis of solution left in the flask is now cooled in an ice bath to about 5° C. When that temperature is reached, . 1 mole of SOa-acetonitrile complex is added. If the solution prepared is .5-molar strength, that requires the addition of 200 ml. This addition should be done with strong magnetic stirring, and slowly enough that the temperature does not climb too much. After the SOa has been added, allow the reaction to come to completion for about 5 minutes, then add 18 grams of diethylamine (26 ml) dissolved in 250 ml of anhydrous ether.

A further 5 minutes of reaction time is then allowed with stirring, before pouring the whole reaction mixture into a 2000 ml sep funnel. Now 1000 ml of water is slowly poured into the sep funnel with swirling. This addition of water generates a lot of heat as the SOs reacts to make sulfuric acid, and then gets diluted. Over a period of time work up to shaking the sep funnel. The LSD goes into the water layer. Separate it off, and extract four more times with 1000 ml portions of water.

The combined water extracts (5000 ml in all) are now saturated with salt, then extracted five times with 1000 ml portions of ethylene dichloride (1,2-dichloro-ethane). Ethylene dichloride is heavier than water, so it forms the lower layer in the sep funnel.

The ethylene dichloride now contains the LSD. Check the extracted solutions with a blacklight to make sure they have been completely extracted. This solvent is now removed under vacuum (a rotovap makes this much easier, but is not the sort of thing one gets at a garage sale). Warm water can be used to heat the flask during the vacuum evaporation.

The residue in the flask is a mixture of LSD and iso-LSD. The isomeric mixture comes from using isomerically-mixed lysergic acid. The iso-LSD is separated from the LSD using the chromatographic method given in Chapter 4, and the iso-LSD converted to LSD by the method also given in that chapter. Conversion to the tartarate salt is also done in the same way as described in Chapter 4.

Journal Method

In this method, the formation of the metal salt of lysergic acid is done exactly as given above. Now to the residue left in the flask after vacuum evaporation of the methanol, add 500 ml of dimethylformamide. Half of the dimethylformamide is now distilled off under a vacuum through a fractionating column to remove traces of water and methanol. Aspirator vacuum is strong enough for this distillation, but beware of the tendency for formamides to bump during vacuum distillations. The vacuum should be strong enough that the dimethylformamide distills at around 50° C.

Now cool the formamide solution, and when it has cooled to 5° C, add 100 ml of 1M SCvformamide complex. Allow 10 minutes of stirring in the cold before then adding 25 ml of diethylamine.

Stir for an additional 10 minutes, then pour the batch into a 2000 ml sep funnel. Now to the sep funnel add 800 ml of water. Mix this in thoroughly, then add 400 ml of saturated salt solution in water. Mix this in, then extract out the LSD by repeated extraction with 250 ml portions of ethylene dichloride. Check with a blacklight for complete extraction.

The combined ethylene dichloride extracts should be evaporated under a vacuum as above, and the residue of LSD and iso-LSD should be separated and treated as above.

1

LSD From Lysergic Acid And Trifluoroacetic Anhydride

This method is a little bit lame, but it may be the method of choice if trifluoroacetic anhydride or trifluoroacetic acid should happen to fall from the sky into one's hands. The reason why this method is a bit lame is threefold. Anhydrous lysergic acid is required for this reaction. To obtain anhydrous lysergic acid, the lysergic acid hydrate yielded by the methods in Chapter 5 must be baked under high vacuum for a couple hours. This is obviously not good for such a delicate molecule. The water molecule will be shed by a baking temperature of 120° C at a vacuum of 1 mm Hg, 140° C at 2 mm Hg, and still higher temperatures at less perfect vacuums. A MacLeod gauge is the only instrument that I know of which is capable of accurately measuring such high vacuums.

Another reason why this method is lacking is that the yields are not so good as those achieved by the other synthetic routes presented in this book. It is possible to recover the unreacted lysergic acid at the end of the process, but this does not make up for the initial lower yield, not to mention the added hassle of recovering and redrying the lysergic acid.

Strike number three for this route is its propensity to give by-products that are difficult to separate from the desired product. I am

not talking here about the large amount of iso-LSD that this method makes. That molecular jumbling is inconsequential, because the lysergic acid used is itself an isomeric mixture. Rather, what can occur here is the production of LSD and other by-products.

The mechanics of this reaction are similar to the reaction with SOs, in that two molecules of the anhydride react with the lysergic acid molecule to form the mixed anhydride. In this reaction, there is no need to first react the lysergic acid with hydroxide to form the metal salt. Also, the need to follow exact stoichiometric quantities of reactants is not as pressing as in the *SO$* method.

To do the reaction, into a 1000 ml flask (carefully dried and equipped with a magnetic stirring bar) place 16 grams of lysergic acid and 375 ml of acetonitrile. The lysergic acid will not dissolve. Stopper the flask and place it in the freezer to cool the contents to -20fi C.

Next, remove the flask from the freezer, and nestle it in an ice-salt bath. Now with stirring add a solution of 26'/i grams (17.8 ml) trifluoroacetic anhydride in 225 ml acetonitrile. The trifluoroacetic anhydride solution should have been previously cooled down to -20° C in the freezer before adding. The resulting solution is stirred in the cold and in the dark for a couple of hours, during which time the suspended lysergic acid dissolves and forms the mixed anhydride.

Now the mixed anhydride solution is poured into 450 ml of acetonitrile containing 23 grams diethylamine. This mixture is stirred in the dark at room temperature for a couple of hours.

To get the product, the acetonitrile is evaporated off under a vacuum. The residue is then dissolved in a mixture of 450 ml of chloroform and 60 ml ice water. The chloroform layer is then separated, and the water layer is then extracted four times with 150 ml portions of chloroform. The combined chloroform layers are then dried with a little sodium sulfate, and the chloroform evaporated away under a vacuum to give a solid residue weighing about 10 grams which is a mixture of LSD and iso-LSD. These are separated by chromatography as described in Chapter 4, and the iso-LSD converted to LSD as also described in that chapter.

The water layer from the extractions contains about 6 grams unreacted lysergic acid. It can be recovered by acidifying with sulfuric acid to pH 3, and filtering. This material should be purified by recrystallization from hot water, then dried again under high vacuum.

Preparation of Trifluoroacetic Anhydride

The simplest method for making trifluoroacetic anhydride is to dehydrate trifluoroacetic acid with phosphorus pentoxide. One is more likely to come across a bottle of trifluoroacetic acid than the anhydride, so knowledge of this method has a definite value.

To do this reaction, grind 25 grams phosphorous pentoxide with a mortar and pestle, and place it in a 500 ml flask. Next add a magnetic stirring bar, and 30 ml of trifluoroacetic acid. Rig the flask for simple distillation using glassware that has been baked to ensure freedom from traces of water. Flow ice water through the condenser, nestle the receiving flask in ice, and attach a drying tube to the vacuum adapter of the glassware.

Now with stirring, heat the flask with hot water — about 50-60° C. Trifluoroacetic acid has a boiling point of *12*- C, while the anhydride has a boiling point of 40° C. The anhydride as it is formed will boil out of the flask, to be collected in the receiving flask nestled in ice. When no more anhydride is produced, the crude product should be redistilled through a fractionating column. This product must then be immediately transferred to a dried container, or kept in its receiving flask tightly stoppered to protect from moisture. The yield is about 10 ml (15 grams).

8
LSD From Lysergic Acid And Phosgene

This method also appears to work via some kind of mixed anhydride. The authors of the US patent 3,141, 887 from which this is taken didn't investigate the nature of the intermediate formed between anhydrous lysergic acid and phosgene, but the similarities between this method and those using SOs or trifluoroacetic anhydride are obvious. As in those methods, lysergic acid reacts with about two molecules of phosgene to form an intermediate which is then reacted with diethylamine to yield LSD. According to the patent, it is not crucial for success to use the exact stoichiometric amount of phosgene in reaction with lysergic acid. A ratio of about 2-1 phosgene to lysergic acid gives best results, but anything fairly close to that works just fine too.

This is not a method to get excited about. Phosgene is a very sneaky poison which is best suited to assassination or wholesale chemical assault, not the home synthesis of drugs. Phosgene is not irritating when inhaled, and has delayed effects which easily lead to death. For a complete treatment of the poisonous properties of phosgene, read *Silent Death* by me. This substance should not be used without very effective ventilation. Smoking while in its presence serves as a warning device, as phosgene makes the smoke taste bad. One can also prepare a warning paper by soaking said paper in an

alcohol solution containing 10% of an equal mixture of p-dimethyl-aminobenzaldehyde and colorless diphenylamine. This paper is then dried. It will turn yellow to deep orange in the presence of the maximum-allowable concentration of phosgene. It is a good idea to wear this paper while working. The only justification of choosing this method is if a cylinder of phosgene gas is very easily available at work or school.

To do this reaction, a carefully dried 500 ml flask is charged with a magnetic stirring bar, 5 grams of anhydrous lysergic acid dried under heat and high vacuum as described in the previous chapter, and 100 ml dimethlyformamide. Stopper the flask, and cool it to -10° C in a salt-ice bath. The lysergic acid will not dissolve.

Next to this flask attach a dropping funnel, and drip in 20 ml of dimethylformamide containing 3.4 grams of phosgene. This solution is best prepared by taking 200 ml of dimethylformamide and slowly bubbling into it dimethylformamide phosgene from a cylinder until the solution gains 34 grams weight. Strong stirring during the bubbling helps to ensure that most of the phosgene goes into solution and not the surrounding air. The exact concentration of this phosgene-DMF complex is unimportant; what *is* important is that the weight gain be known, and the amount then portioned out into the batch contain 3.4 grams phosgene. The addition of the phosgene complex into the lysergic acid suspension should take at least 20 minutes.

The addition of phosgene should bring the lysergic acid suspension into solution. Continue the stirring in the cold and dark for half an hour, then add a previously-cooled solution of 21 grams diethylamine in 100 ml dimethlyformamide. Continue stirring in the cold for half an hour, then allow the flask to warm to room temperature while stirring for a couple of hours.

Next, the batch should be poured into a 1000 ml sep funnel, and diluted with 400 ml chloroform. When a thorough mixing is achieved, wash the chloroform with some 1-molar NaOH solution in water, and then some plain water. The chloroform contains the product. It is next evaporated off under a vacuum to yield an oily residue which is a mixture of LSD and iso-LSD. They are separated chromatographically

as in the other methods, the iso-LSD converted to LSD as in the other methods, then converted to tartrate salt as in the other methods.

9
Method X

About 1980, a major LSD-manufacturing operation was busted in England in a police action called Operation Julie. This name was derived from the undercover agent who infiltrated the manufacture group, and who spent a major part of her time milking the genitals of those involved. At the trial, it was revealed that the chief cook of the group had made a major advance in the field of LSD manufacture.

The nature of this innovation had remained a nagging mystery throughout the writing of this book. Searching the *Chem. Abstracts* for entries under LSD turned up nothing. After 1965, when acid became illegal, the entries under LSD no longer included improved cooking procedures. Rather, the section was filled with references to studies showing that massive doses of LSD are bad for mice, and forensic techniques for detecting LSD. This was clearly a waste of time.

A close reading of the listed chemicals in the "Love Letters From the Heat" section at the end of this book provided the clues I needed to solve the mystery. Note that propionic anhydride is a listed chemical under the Chemical Diversion Act, with a reporting threshold of 1 gram. There is only one substance in the field of

clandestine drug manufacture where 1 gram is a significant amount — LSD.

Could it be that propionic anhydride forms a mixed anhydride with lysergic acid? I returned to the *Chem. Abstracts* and searched under lysergic acid and closely related compounds for references to the formation of mixed anhydrides with propionic anhydride. I also looked for listings under substances related to LSD referring to the use of propionic anhydride in their manufacture. On this I hit paydirt!

Beginning in the late 70s and continuing through the 80s there were several references to the use of propionic anhydride to form mixed anhydrides with substances closely related to lysergic acid, mostly the 9,10-dihydro derivative of lysergic acid where the double bond two spaces upstream from the carboxyl group has been reduced.

The Operation Julie cooker had made the obvious analogy that if the procedure works for these substances closely related to LSD, it should also work for LSD. This type of underground research and discovery is not at all unusual. If you look through the *Chem. Abstracts* for references to the use of hydriodic acid and red phosphorus in the reduction of ephedrine to meth, you will find nothing. This procedure is a general method of reducing alcohols to alkanes, and was applied by clandestine chemists to ephedrine with excellent results. Ditto for the lithium-metal-in-liquid-ammonia reduction of ephedrine to meth.

To get the full details of the following procedure, your command of Russian or Hungarian had better be firmer than mine. All this research came out of Eastern Europe. For example, see *Chem. Abstracts,* Volume 93, column 186636. This will then direct you to: *Otkrytiya, hobret., Prom. Obraztsy, Tovarnye Znaki* 1980, (19), 303. Also Italian patent application 76/50,746 dating to Dec. 6, 1976.

For this method to be superior to the procedures given in the earlier chapters of this book, the need for a close stoichiometric quantity of anhydride added would have to be done away with. It must be possible to add a healthy excess of the propionic anhydride to get 100% conversion of the lysergic acid to the mixed anhydride. It would

further be nice if the procedure works with the hydrous form of lysergic acid, doing away with the need to bake it under high vacuum. Further advances in LSD manufacture taken from analogy to closely related compounds can also be found in the hydrazide "one-pot shot" route to LSD. It would appear that lysergic acid hydrazide can be reacted with the very common chemical sodium nitrite, and then diethylamine to give LSD. This eliminates the need to synthesize or otherwise obtain 2,4-pentanedione. (For synthesis of 2,4-pentanedione, see U.S. Patent 2,737,528 and 2,834,811.) See *Chem. Abstracts* Volume 94, column 209051 (1981) and German Patent 2,924,102. Another analogy can be found in *Chem. Abstracts* Volume 99, column 71069 which then refers you to German Patent DE 3,239,788. It would appear that phosgene, as used in Chapter 8, can be replaced with oxalyl chloride. This substance is much less dangerous than phosgene, and more easily measured out.

Preparation of Propionic Anhydride

Propionic anhydride is obviously going to be impossible to purchase without getting busted. It is, however, not too difficult to make in good yield and high purity. The simplest method of preparation is via the general method found on page 28 of *Organic Synthesis Collective* Volume 3. In this method propionic acid reacts with propionyl chloride in the presence of pyridine to yield propionic anhydride. Propionyl chloride is at present an easily obtained substance, but in the future, this may change. When that time comes, propionyl chloride can be easily made from propionic acid by the directions found in *The Journal of the American Chemical Society* Volume 60, page 1325 (1938). Propionic acid will never be a restricted chemical because it has such wide use as a means to kill fungus and mold growing on stored grain.

To do the reaction, a 250 ml flask and a dropping funnel are first thoroughly dried, then a magnetic stirring bar is placed in the flask, followed by 16 ml of pyridine and 25 ml of benzene. If there is a question as to whether the pyridine or benzene are completely free of

water, the pryridine should be dried by adding some KOH pellets to the jug of pyridine, and the benzene dried azeotropically by distilling off 10% of it, and using the residue.

Now to the stirred solution, rapidly add 9.25 grams (8.75 ml) of propionyl chloride. This causes a small rise in temperature, and pyridium complex conies out of solution. Then, with continued stirring, add 7.4 grams (7.4 ml) of propionic acid over a period of 5 minutes from a dropping funnel. This causes the solution to get hot, and pyridine hydrochloride comes out of solution.

The stirring is continued for an additional 10 minutes, then the pyridine hydrochloride is filtered out in a Buchner funnel. This should be done rapidly, and on a dry day, because the pyridine hydrochloride is very hygroscopic, and will melt. The filter cake of pyridine hydrochloride should then be quickly rinsed with dry benzene, and the combined filtrate should be concentrated under a vacuum, using steam or hot water to heat the flask. When the benzene and pyridine have distilled off, they will be followed by the product, propionic anhydride, boiling at about 70° C under a typical aspirator vacuum of 20 torr. This product may be contaminated with some propionic acid, and it can be removed by redistilling the product through a fractionating column, either at normal pressure or under a vacuum. Propionic acid boils at 141° C, while the anhydride boils at 168° C at normal pressure.

10 Solvent Management

A cursory reading of this text will make it plain to everyone that the production of LSD involves heavy usage of solvents. From the defatting and extraction of the crops to the crystallization of pure LSD, a variety of solvents must be used in large amounts relative to the product to get a fairly pure product.

"Fairly pure product"... how we starved masses long for such a thing. Back in the 70s when I dropped my first doses of acid, the stories were already impossibly ingrained in the consuming public's mind that the acid was cut with speed or strychnine. All of the stories are easily disproved, yet they persist to this day. If the entire weight of a blotter paper was made of pure meth or strychnine, its effect would be less than pronounced. The truth of the matter is that lysergic-similar compounds contaminating the LSD are responsible for these undesirable effects. From clavine alkaloids to unhydrolysed ergot alkaloids, to unreacted lysergic acid, or lysergic acid hydrazides to iso-LSD and God knows what substances created by the mishandling of the raw materials and product, a contaminated product is much easier to make than a pure one.

The use of large volumes of solvents poses twin problems: obtaining them and disposing of them. Both problems are made vastly

simpler by recycling the solvents. Just because a solvent has been used once in a given stage of the process does not mean its useful lifetime is over. For example, the solvent used for defatting the crop is easily made as good as new by distilling it to free it of its load of fat. Other solvents are not so easily recovered for re-use because the procedure calls for the given solvent to be removed from the product by vacuum evaporation. In this case, the solvent can be collected in a cold trap placed along the vacuum line on its way to the vacuum source. If a pump is used to create the vacuum, such a trap is vital to prevent solvent vapors from getting into the pump oil, thereby ruining the lubrication and the vacuum created.

A cold trap can be constructed of either glass or steel; it need only be large enough to hold the solvent collected, and airtight so as not to ruin the vacuum with leaks. This cold trap is then cooled down with dry ice during vacuum evaporations to condense the solvent vapors in the trap.

The solvent recovered in the trap can be re-used in the given stage of the process from whence it came. I would not co-mingle recovered solvents from different stages. For example, chloroform from the alkaloid extraction of the crops should be kept for that usage, and not be used for LSD crystallization, because it will also contain some ammonia and methanol.

The recovery of ether, for example, from method 2 of lysergic acid production, poses a special problem. This problem is the formation of explosive peroxides in ether during storage. Ether containing water and alcohol, as would be the case for this recovered solvent, does not form much peroxide. There is a possibility that dry ether can be made free of peroxides by shaking the ether with some 5% ferrous sulfate ($FeSO_4$) solution in water prior to distilling. Failure to do this may expose the operator to a fiery explosion during distillation. Ice water flowing through the condenser, and an ice-chilled receiving flask, are required to get an efficient condensation of the ether during distillation.

II
Keeping Out Of Trouble

The dangers of LSD manufacturing do not end with the possibility that the cooker may spill some of the stuff on himself and fry his brain. There is a much more malignant danger facing those who embark upon this course: Johnny Law.

The conduit through which those shit-eating dogs travel to get to you is your associates. If you are cooking alone with no partners in crime, your safety has been improved immeasurably. Partners in crime are too easily turned against you and transformed into star witnesses. Don't deceive yourself by thinking that your friends would never do such a thing. This country is populated with sheeple who lick the boots of their masters at the drop of a hat. The added incentive of avoiding jail time turns these bleating sheeple into singing stool pigeons nearly every time.

Along with partners in crime, one's customers for the product are a prime source of snitches. The first and foremost rule in contacts with one's customers is that they have no business knowing that you are cooking the product yourself. The reason for this, beyond their babbling their mouths to their friends, is that if they get themselves into trouble they then have a lot more leverage for cutting themselves a snitching bargain with the heat if they say that they can deliver up an LSD lab. More leverage for them turns into more time and freedom

for this turncoat to work at setting you up, because the heat sees a bigger pot of gold at the end of the rainbow. If all he has to offer to the heat is just another LSD connection, they will get frustrated with him if he does not immediately deliver on your demise, and will put his squealing butt in the slam where it belongs.

Several further tactics are called for to protect yourself from treachery emanating from your customers. If the heat succeeds in turning your customer against you, they will first try to get themselves in on a transaction, and failing this, try to make what is called a "controlled buy" whereby thek traitor buys while they watch and maybe record.

To foil such tactics, you must be in control of setting up transactions, not your customers. They do not call you to set up deals; in fact, it's best that they not even have your number, address or real name. Know well the schedules and habits of your customers, and simply call them with very short warning times of your arrival and readiness to do business. Third parties are not invited, wanted or allowed. If they don't have all the cash ready at hand, just front the remainder with an understanding of how long it will take to gather up the balance. Then return similarly unannounced to collect what is owed. By this I don't mean to come back in a couple hours to pick up the marked bills. Rather, the time frame must be sufficiently long so as to make a stake-out by the enemy a real pain and not worth their bother.

Explicit telephone conversations with one's customers are a definite no-no, and such an understanding must be reached with them from the outset. Rather, the conversations should be friendly, filled with small talk, and mostly held to make sure the guy is home. Use of codewords and other such nonsense is for idiots. If one's customer breaks these pre-agreed-upon rules, it is cause for suspicion.

The delivery machine of choice is a street-legal dirt bike. This vehicle is to be preferred because if the heat jumps you while on the way to a delivery, you can take off and travel routes they can't through backyards, ditches and cross-country, making a life-or-death drive for the nearest body of water. If you're in the desert you deserve what you

get for living where people aren't meant to be. Once a body of water is reached, the contraband can then be disposed of. A proper excuse for fleeing is that you thought they looked like a bunch of assassins. With all the black-hooded ninja-wanna-be police these days, this is a most believable excuse.

Setting up shop and getting chemicals is another source of exposure to the forces of our enemy, the state. See the "Love Letters from the Heat" section at the end of the book. Listed there are the required snitch-list chemicals. A series of tactics are used to circumvent the reporting requirements. Sensitive chemicals are home-synthesized according to the directions given in this book. The five-finger-discount method of acquisition is practiced to the fullest extent possible at work or school to obtain chemicals and equipment. Where an inside job will not yield the desired results, an actual heist at some plant may be called for. This is a reasonable course of action only if you know through a person inside the target about the availability of desired items, and the presence of security measures. Burglary is not the sort of thing to do hit-and-miss.

Other good sources of equipment and chemicals are the surplus market and waste exchanges. Dealers in the surplus market can be found in trade publications for the chemical industry and those industries which use a lot of chemicals. The surplus people buy the chemical stock of defunct businesses, or chemicals no longer wanted by other businesses, and re-sell them. The typical surplus dealer is more concerned with moving his stock than with brown-nosing the feds. A company letterhead and a phone will open the door to most of these people.

The waste exchanges came about as a result of hazardous-waste laws which prevent the dumping of chemical waste and unused chemicals. The waste exchanges act as matchmakers to bring together those with unwanted chemicals and those who want them. A list of waste exchanges is included at the back of this book. A company letterhead gets you into the waste exchange network, a world filled with eager chemical-holders who will generally send you their chemicals if you pay shipping.

When these measures fail, setting up a front operation using chemicals opens the legitimate pipeline to your door. One such business which can be founded and then subverted to the needs of LSD synthesis is metal plating. From the stocking of plating baths, to analytical chemicals to monitor the composition of these baths, to waste water treatment chemicals, the electroplating field is awash with chemicals useful for making LSD. The plating field is also underserved because so many shops have been put out of business due to tough environmental regulations. There are many people looking for somebody to plate their old car or bike parts, and the one-man plating shop is an old and respected tradition in the industry.

Metal plating uses all sorts of solvents, including all the ones mentioned in this book, to clean and degrease the metal parts prior to plating. Hydrazine is used to reduce hexavalent chrome in wastewater to the trivalent state so that it may then be removed from the wastewater by precipitation as the hydroxide. Hydrazine is also used in electroless nickel baths which plate pure nickel, not the nickel phosphorus alloy obtained from those baths which use hypophosphite as the reducer. Hydrazine is also used in boilers to prevent oxygen pitting. Chlorine and 12V4% bleach are used to destroy cyanide in the wastewater. The lab of a plating shop can be stocked with items such as 2,4-pentanedione which is a transition metal chelator, and many other items. I wouldn't try for diethylamine though.

The use of a false identity when founding a front operation adds a layer of security for the operator. Loompanics has the most complete selection of books covering this topic.

During the actual cooking process, I have emphasized the need to keep making progress and not fiddle around. One must present as small a target as possible by getting the stuff made, moved, and operations shut down as rapidly as is compatible with the production of quality acid. When you have made your million-dose score, don't go back to the well for another try the next year. Take a vacation.

Due to the very small dosage size of acid, any reasonable lab-scale production will produce at least tens of thousands of doses. Be prepared to be able to move that much without having to meet

"friends of friends." If all you want is some high-quality trips for yourself and a close circle of friends, you are much better served with TMA-2 made from calamus oil, or MDA made from sassafras oil.

I have long been an outspoken advocate of the need for a self-destruct device in a lab. One serves a great deal less time for acts of mayhem than for drugs. An ideal self-destruct device is a stick of dynamite already armed with fuse and cap, stored inside a metal can. The can protects against small accidental fires leading to the big one.

If a squad of goons starts pounding down the doors, the self-destruct sequence is initiated by lighting the fuse, and then diving out the window. The ensuing blast and solvent fire will erase all evidence of drugs. Explaining why the blast coincided with the arrival of the enemy is best left to your lying lawyer, but if you can't wreck your own place, what has this country come to?

A bit of perimeter security is called for to slow up the aforementioned goon squad, and allow sufficient time and warning so that the self-destruct sequence can be initiated. A dog with a bad disposition posted outside will warn of the approach of strangers, and some "anti-burglar" strengthening of the doors will further slow up the forces of evil.

At the time of this writing (fall '94), federal and most state courts that I know of have mandatory minimum sentences for LSD that count the weight of the carrier in the total weight of the drugs seized. Only politicians could be so stupid and still keep their jobs. This screwed-up state of affairs has a strong bearing on the best way to move the acid. It means that large blocks of acid are best sold as grams of the crystal sealed in glass to someone who will then make blotter out of them. The time-exposure is thereby greatly cut down, even if a lower price is obtained.

Smaller operators looking to turn on a few thousand of their closest friends would do best to drip the product onto sugar cubes, freeze them during storage and move the product as a high priced gourmet treat. Dilution with alcohol and moving the stuff as liquid is not good, as even at freezer temperatures acid does not keep well in solution. Once locked up in a sugar cube, the tender molecule is

protected. Producing thousands of sugar cube doses in one day is an easy, though tedious, operation. One starts with a burette and lots of sugar cubes (not purchased at the same place, for God's sake!).

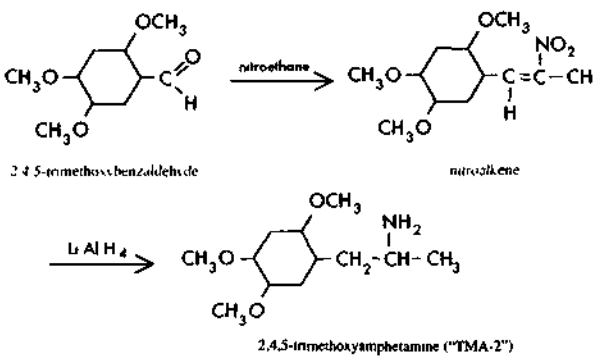
Next, the average size of droplet delivered from this burette must be measured, and the concentration of LSD tartrate in water solution calculated so that one drop contains 100 micrograms of acid. The burette in my lab delivers 188 drops per 10 ml, so each drop is .0532 ml. The size of the drops delivered from a burette depends upon the size of the drip-tip on the burette, the viscosity of the liquid, its surface tension and the molecular attraction of the fluid to the drip-tip. The addition of a little acid to the water solution may change these factors, so the preliminary results obtained from pure water should be checked against the size of droplet one gets with LSD solution. In any case, the calculation goes like this:

$$\frac{100 \text{ mikes}}{.0532 \text{ ml}} = \frac{.0001 \text{ gr}}{.0532 \text{ ml}} = \frac{? \text{ gr LSD}}{1 \text{ ml solution}} = \frac{.00188 \text{ gr LSD}}{1 \text{ ml solution}}$$

$$= \frac{1.88 \text{ gr LSD}}{\text{liter of solution}}$$

The weight measurement assumes LSD of high purity. Proper dose size should be checked by dropping a test sugar cube. This bio-assay should be done by someone other than the cooker, as he may have been chronically exposed to LSD during manufacture, and immune to its effect.

12
Studies On The Production Of TMA-2



2,4,5-trimethoxybenzaldehyde

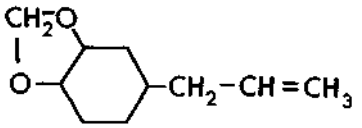
nitroalkene

2,4,5-trimethoxyamphetamine ("TMA-2")

That route has several drawbacks which make it impractical for clandestine synthesis. The first and most important problem is the availability of 2,4,5-trimethoxybenzaldehyde. This substance is not exactly a linchpin of chemical commerce. So far as I know, it has one use: making TMA-2. Those same folks who gave me the hassle over the purchase of Rochelle salts will certainly report a shipment of 2,4,5-trimethoxybenzaldehyde, and the heat will not be far behind. Further chemical supply problems arise from this method's use of large amounts of anhydrous ether or THF in the LiAlHj reduction. This too will be duly noted by the heat, especially in combination with buying LiAlHt.

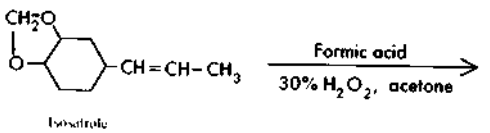
A much more low-profile synthetic route is possible using calamus oil as the raw material. A couple of patents granted in the late 80s have completely changed the field of psychedelic amphetamine manufacture from the way Dr. Shulgin knew it during his days of cooking in the 60s. Previous to the publication of these patents, the Knoevenagel condensation of benzaldehydes to yield the nitroalkene, followed by the reduction of the nitroalkene to the amphetamine, was far superior to an alternative route making use of the common essential oils.

Many essential oils have as major components substituted allylbenzenes. For example, sassafras oil is 80-90% safrole:

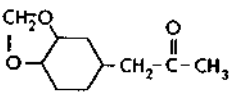


The alternative route was to take this substituted allylbenzene, move the double bond to the propenyl position by heating with anhydrous alcoholic KOH, yielding in the case of safrole, isosafrole. Then a messy, tedious and low-yield reaction was used to convert this propenylbenzene to the corresponding phenylacetone. All we veteran speed cooks love phenylacetones, because they offer the cleanest and best route to the amphetamines, but the old-fashioned method of

converting propenylbenzenes to phenylacetones made this route impractical:



Isosafrole



Methyl dioxyphenylacetone

My own experience with this reaction dates to the early 80s, when I decided to torment myself by trying it. Detailed cooking procedures using it can be found in *Pikhal* under MDMA. My experience with the KOH isomerization was that the conversion of safrole to isosafrole went cleanly at about 100% yield, as long as traces of moisture were excluded from the reaction. The conversion of isosafrole to methylenedioxy-phenylacetone is another matter. The yields are low, a lot of work is required because the formic acid and hydrogen peroxide must be removed from the reaction mixture under a vacuum before final treatment with sulfuric acid solution to yield the phenylacetone, and these vapors corrode the aspirator supplying the vacuum. This method stinks!

Two patents dating to the late 80s, and to a lesser extent a journal article dating back to 1970, have turned the situation around. The first patent I will cite is US patent 4,638,094, titled "A Process for Producing Phenylacetones." This patent reveals, using many different examples over the course of 36 pages, the best general method for converting allylbenzenes to the corresponding phenylacetone in very high yields.

This procedure reacts the allylbenzene (for example safrole, as obtained in pure form by vacuum distilling sassafras oil) with methylnitrite in methanol solution containing water and a palladium catalyst to yield the phenylacetone. The palladium catalyst can be used in a variety of forms, as detailed in the patent. The best choices

for use with safrole are palladium bromide, chloride, or a mixture of palladium chloride and copper chloride. Of the three, the mixture catalyst is better for reasons which will be explained in the following cooking example:

In a 4000 ml beaker, or one-gallon glass jug, is placed 3000 ml methyl alcohol, 150 ml safrole, 300 ml distilled water, and the chemist's choice of either 20 grams palladium bromide or ten grams of palladium chloride or a mixture of one gram palladium chloride and 4.25 grams copper chloride (CuCk). The catalyst choices have been given here in order of good to best. The reason why the last choice is best is because of the very high cost of palladium salts. Palladium chloride is preferred over the bromide because palladium chloride finds use in the electroplating field. It is used there in baths to plate palladium, and as part of the activation process to prepare plastics to be plated. The bromide is not as commonly used.

Next, a methyl nitrite generator is rigged up as shown in Figure 3:

Into the 2000 ml flask is placed one pound of sodium nitrite, 225 ml of methyl alcohol, and 260 ml of water. They should be swirled around for a while to mix. Then 680 ml of cold dilute sulfuric acid (made by adding 225 ml of sulfuric acid to 455 ml of distilled water, mixing and chill-ng) is put into the dropping funnel.

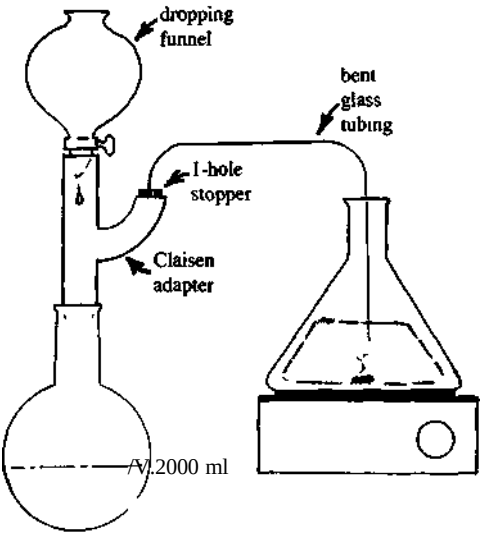
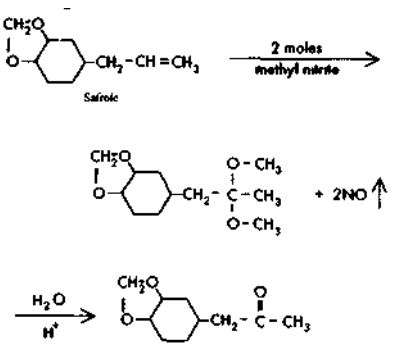


Figure3
Methyl nitrite generator

Now vigorous magnetic stirring is begun in the beaker or glass jug containing the allylbenzene-alcohol-pal-



In the 1-mole batch given in this example, about 6 moles of methyl nitrite are bubbled into the reaction mixture, while only 2 are required for the reaction. The reason for the excess is because methyl nitrite is not held in solution very well on account of its very low boiling point. If ethyl nitrite was used instead, then only three or four moles would be needed.

While the reaction is being done, the mixture takes on the appearance of mud if palladium bromide is being used. A fizzing also

occurs, which gives the reaction mixture the appearance of freshly poured Coke. Note above that a bit of acid is required to get hydrolysis of the intermediate dialkoxyphenylpropane to the phenyl-acetone. The best pH for this reaction is between 4-7. If palladium chloride or the mixed catalyst PdCh-CuCla is being used, the pH of the reaction mixture can be adjusted to this range by adding a small amount of HC1. If $PdBr_2$, is used, it is best to wait until the catalyst is filtered out before adding HC1, as the HC1 could form PdCh and complicate catalyst recovery. The pH of the reaction mixture is best measured by first dampening some indicating pH paper with distilled water, then putting a drop of reaction mixture on the paper. The preferred temperature for this reaction is about 25° C throughout.

When all the methyl nitrite has been bubbled into the reaction mixture, stirring should be continued for another hour. Then, if palladium bromide was used, it should be filtered out. Repeated filtrations will be needed to remove all of the catalyst, because it gets quite finely divided during the course of the reaction. This leaves a clear light-reddish solution. If palladium bromide was used, now adjust pH to 4-7, and allow another hour to complete the hydrolysis.

If palladium chloride or the mixed catalyst was used, these substances are soluble in alcohol. In this case, the catalyst will be recovered later. Here, check the pH of the solution again to be sure it is in the proper range before proceeding.

Now the alcohol solvent must be removed. This is best done by pouring the reaction mixture into a large filtering flask, stoppering the top of the flask, and removing the solvent under a vacuum. Use of a hot-water bath to speed evaporation is highly recommended for this process. It is not OK to distill off the alcohol at normal pressure, as the heat will cause the nitrite and NO in solution to do bad things to the product.

To the residue left in the flask after removal of the alcohol, add some toluene to rinse the product out of the flask into a sep funnel. Next, put 300 ml of water into the flask to dissolve the catalyst if PdCla or the mixed catalyst was used. Add the water solution to the sep funnel to dissolve carried-over catalyst there, then drain this water

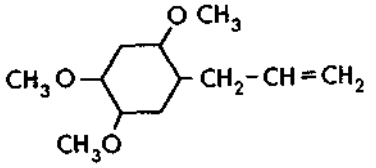
solution of catalyst into a dark bottle and store in the dark until the next batch. If PdBr2 was used, this step can be skipped. Just store the filtered-out PdBra under water in the dark.

Now the toluene-phenylacetone solution should be distilled through a Claisen adapter packed with some pieces of broken glass to effect fractionation. The first of the toluene should be distilled at normal pressure to remove water from solution azeotropically. The b.p. of the azeotrope is 85° C, while water-free toluene boils at 110° C. When the water is removed from solution, turn off the heat on the distillation, and carefully apply a vacuum to remove the remainder of the toluene. Then with the vacuum still on, resume heating the flask, and collect the substituted phenylacetone. Methylenedioxyphenyl-acetone distills at about 140° C and 160° C using a good aspirator with cold water. A poor vacuum source leads to much higher distillation temps and tar formation in the distilling flask. The yield from the reaction is close to 150 ml of phenylacetone. Its color should be clear to a light yellow. The odor of methylenedioxyphenylacetone is much like regular phenylacetone, with a trace of the candy shop odor of the safrole from which it was made.

A higher-boiling phenylacetone like 2,4,5-trimethyloxyphenyl-acetone is better purified as the bisulfite addition product, unless a vacuum pump giving high vacuum is available. To make the bisulfite addition product, take the residue from the filtering flask, dissolved in some toluene and freed from catalyst as described above, and pour it in a beaker. Next, add 3 volumes of sodium bisulfite solution prepared by adding sodium bisulfite or metabisulfite to water until no more dissolves. Shake or vigorously stir for a couple of hours to convert the phenylacetone to the solid bisulfite addition product. Filter out the solid, then regenerate pure phenylacetone by putting the solid into a round-bottom flask, adding an excess of saturated solution of sodium bicarbonate in water, and refluxing for a couple hours. After cooling, the phenylacetone should be extracted out with some toluene. The toluene should then be removed under a vacuum, and the residue stored in a freezer until conversion to the amphetamine. All

phenylacetones are sensitive to light, and should be stored in the freezer.

The above cooking procedure is the best way to process allylbenzenes to the corresponding phenylacetones. Sassafras oil, as previously mentioned, is 80-90% safrole. Calumus oil, if its country of origin is India, consists of about 80% of the allyl isomer of asarone:



It too can be purified by distillation under a vacuum to yield fairly pure allyl-asarone. Its boiling point is 296° C at normal pressure and about 170° C with aspirator vacuum. More details on this Indian calamus oil can be found in *Chetn. Abstracts* column 6585 (1935), also *Current Science*, Volume 3, page 552 (1935).

My search for calamus oil of Indian origin came up empty. In fact, the health-food store in my town, which is well-stocked with various oils for use in aromatherapy, had never heard of the stuff, nor was it listed for sale in their catalogs. This left one alternative: dig up the roots of North American calamus, and steam-distill the oil out of them.

While searching for calamus in my area's swamps, bogs and ponds, the damaging effects of the spread of purple loosestrife was obvious. This imported plant from Europe has taken over much of the former habitat of the calamus plant. Here in America, the loosestrife is free from the insect that keeps it under control in Europe by feeding on its seeds. The state paper-pushers have been thinking for years about importing the bug, without ever getting off their butts and doing it. I suggest this project to somebody out there in the reading public so that it can finally get done while there is still some native flora left.

After a lot of searching, I finally found a large patch of the American calamus. (See Figure 4.)

The time for harvesting the roots of the calamus is in the fall after the killing frost. The frost brings the oil down out of the leaves and

into the root for winter storage. The roots are about a foot long, an inch or so in diameter, and run horizontally in the soil at a depth of a few inches. They are best dug out using a fork, taking care not to pierce the root, as this will cause loss of oil during drying. The dug-up roots should be rinsed free of dirt, and the tops cut off there in the field. (See Figure 5.) The roots should then be taken home and allowed to dry at room temperature for a week or two. Take care that they do not get moldy!

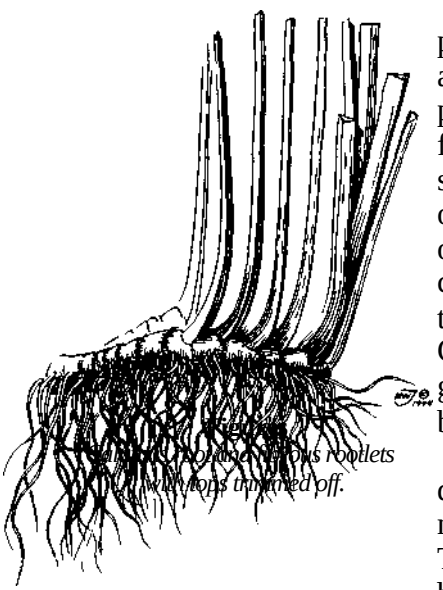
Figure 4

Calamus plant root and fibrous rootlets.

Once dried, oil can be distilled from them. This is done by first grinding up the roots in a blender or with a Salad Shooter, and piling the ground-up roots into a large pressure cooker. A good-sized pressure cooker will take a load Of 10-15 pounds Of root. Next, add a few gallons of water, a couple handfuls of salt, and mix.

The oil can now be distilled. Attach a five-foot length of copper tubing to the steam exit on the lid of the pressure cooker. Its diameter should match that of the steam exit so that steam is not lost here, and should be tightened into place with a pipe clamp. The tubing should then be led downward into a pail of ice water, and back up into a

dark-glass 40 or 64 ounce beer bottle. The ice water cools the steam, turning it into water which collects in the bottles.



Calamus rhizomes with rootlets and tops trimmed off.

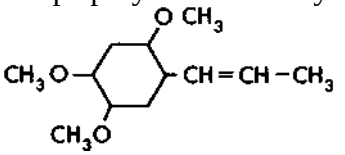
Heat is applied to the pressure cooker, bringing it to a boil. Heat as fast as is possible without bringing over foam or having uncondensed steam escape. When a couple of gallons have been distilled out, stop the heating and add a couple more gallons of water to the pressure cooker. Continue this process until 4-5 gallons of water have been collected.

This process is a steam distillation, and is the way most plant oils are obtained. The steam distillate in the beer bottles contains calamus oil floating on top of the water and clinging to the glass. Calamus oil produced from American plants is reddish brown, and has a strange, pleasant and sweet odor. For more detailed information on calamus oil see *The Chemergic Digest* August 30, 1943, pages 138-40, and *Soap, Perfumery and Cosmetics* August 1939, pages 685-88.

The oil is obtained by first saturating the steam distillate with salt, then extracting the oil with toluene (obtained off the shelf in the hardware store's paint section). About a gallon of toluene is plenty to effect the extraction. Then the toluene is removed by vacuum evaporation in a large filtering flask to yield the calamus oil as a

residue in the filtering flask after the toluene has been evaporated. The yield is about 200 ml from 15 pounds of roots.

Calamus oil obtained from sources other than India differs from the Indian oil in two important respects. The amount of asarone in the oil is much lower than the 80% found in the Indian oil, and the position of the double bond is propenyl rather than allyl:



The asarone is obtained in pure form from the oil by fractional distillation under a vacuum. Asarone boils at about 170° C under good aspirator vacuum of 15-20 torr. The asarone fraction should be collected over a 20-degree range centered on 170° C. I found the yield of asarone from American plants to be about 15% of the oil, giving 30 ml from 15 pounds of root.

Asarone is a light-sensitive material, and as such, should be stored in the fridge or freezer. Upon standing in the fridge, it will crystallize, allowing further purification by filtering. The m.p. of the pure substance is 67° C. Asarone is listed as a cancer-suspect chemical, along with half the other substances in the world. In reality it is not particularly harmful. See *Chem. Abstracts* 1931, page 169. It also doesn't have any pronounced drug effect at reasonable oral dosage. See Dr. Shulgin's comments on the substance in *Pikhal.*

With the double bond in the propenyl position, we come to the next major advance over the disappointing procedure cited in the beginning of this chapter. See European Patent 0,247,526 titled "A Process for 3,4-dimethoxyphenylacetone Preparation." This process uses a simple electrochemical cell to convert the propenyl-benzene to the corresponding phenylacetone in very high yield. The procedure given also works with 2,4,5-trimethoxypropenylbenzene (asarone), and probably also with iso-safrole. It is my opinion that it will work with all propenylbenzenes.

There are great advantages to the use of an electrochemical cell in clandestine synthesis. The solvents and the salts can be reused over

and over again, making for a very low profile. The reagent doing the transformation is electricity, available at the nearest wall socket. The transformer, multimeter and alligator-clip wiring can all be obtained at Radio Shack with zero suspicion attached. This method comes with my highest recommendation.

To do the reaction, a 1000 ml beaker must be rigged up as shown in Figures 6 and 7.

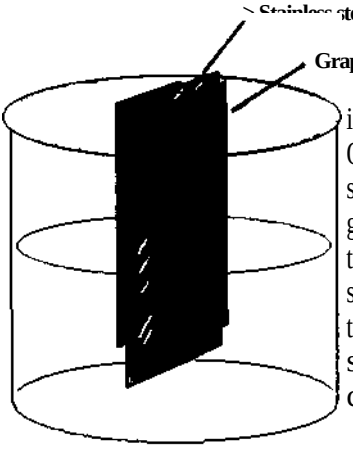


Figure 6

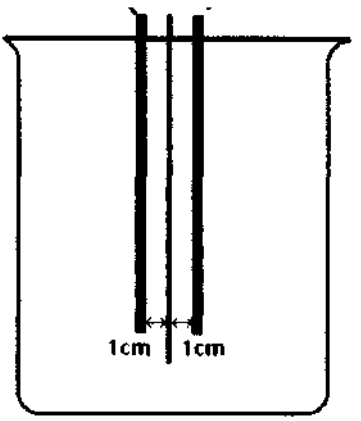
Electrochemical cell used to convert a propenylbenzene to the corresponding phenylacetone.

A central piece of stainless steel having a surface area of about 100 cm^2 actually in contact with the solution is securely clamped into place down the center of the beaker. On each side of this stainless steel piece, securely clamp into place two pieces of graphite, roughly equal in size, having a total surface area in contact with the solution of about 70 cm^2. All three of these electrodes should run straight down into the flask, and a constant distance of 1 cm should separate the surface of the anodes from the cathode. This is very important, as the anode-to-cathode distance determines the voltage at which this cell runs. It is also very important that shorts between the anode and cathode be prevented. The current must flow anode-to-cathode through the solution, not through a short!

Then into the beaker place a magnetic stirring bar, 25 grams of NaBr dissolved in 100 ml of water, 500 ml of acetonitrile, and 20 grams of asarone. Note now the depth of the solution in the flask, and

be sure that the required amount of electrode surfaces are in the solution. I depicted graphite sheet anodes, in Figures 6 and 7, but the more commonly available graphite rods will work as well.

Now, using alligator-clip wiring, attach one clip to the central stainless steel cathode, and run it to your DC transformer where it is connected to the black or negative pole. Another approximately one-foot long section of alligator-clip wiring is attached to each of the graphite anodes; *i.e.* the alligator-clip on one end gets attached to graphite anode A, while the alligator-clip on the opposite end of the wire gets attached to graphite anode B. Then remove some insulation in the center of the wire, and make an electric connection to the positive and red pole on the DC transformer.



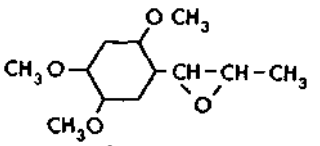
Figure?

Side view of electrochemical cell.

Next, begin vigorous magnetic stirring of the solution, turn on the transformer, and adjust the output of the transformer so that it is pushing a constant current of about 3.4 amps. All three of the electrodes should be fizzing away at this point. If one appears dead, dig the alligator-clip into it to make better contact. Continue passing electricity until 24,000 coulombs have been passed through the solution. A coulomb is defined as 1 amp-second, so this takes about 2 hours at 3.4 amps. The patent states that the temperature must be kept in the range of 10-30^{6} C, so watch to make sure that the current

doesn't heat up the solution too much. Surround the beaker with ice if this occurs.

The electrochemical cell makes the following compound, an epoxide:



When the required amount of current has been passed, turn off the juice and the stirring, and pour the contents of the beaker into a sep funnel. Allow it to stand for about *Vi* hour for the phases to fully separate. An aqueous phase settles out at the bottom of the sep funnel, in spite of the fact that water and acetronitrile are miscible. This water phase contains the NaBr. It should be separated off and saved for reuse.

The acetonitrile phase contains the product. It should be poured into a distilling flask, and the solvent removed under a vacuum. By packing the receiving flask in dry ice during this process, the acetonitrile can be recovered for reuse.

The residue of epoxide product left in the flask should be diluted with 150 ml of ethyl acetate, and poured into a 500 ml flask. Flush the flask with nitrogen, then add 1.5 grams lithium iodide, and reflux for 5 hours. The lithium iodide catalytically transforms the epoxide to the phenylacetone.

After the 5 hours of reflux are over, allow the mixture to cool, then pour it into a sep funnel. Wash the ethyl acetate solution with 50 ml of water to recover the lithium iodide into the water solution. Separate off the water layer, and evaporate the water to recover the lithium iodide for reuse. The ethyl acetate solution should be dried over some anhydrous sodium sulfate, then the ethyl acetate evaporated off to give about 20 grams of 2,4,5-trimethoxyphenlyacetone. This light-sensitive substance should be stored in the freezer.

Method Two

Acetonitrile is a quite poisonous solvent, dangerous both in inhalation from the fizzing electrochemical cell and by absorption through the skin. It has been my experience that just spilling a little bit of it on your skin is enough to give you head rushes and make you feel uncomfortable. The use of acetonitrile can be avoided without loss of yield by using the alternative procedure in Example 6 in the patent.

The electrochemical cell is constructed in exactly the same way as in the first method. Then into the electrochemical cell put 400 ml of dimethylformamide, 200 ml of water containing 27 grams NaBr, and 20 grams asarone. Check the level of the solution, and make sure that the amount of electrode surfaces are the same as in the first method. Then begin stirring, and pass the current through the solution exactly as in the first method.

When the 24,000 coulombs have been passed, pour the contents of the beaker into a sep funnel, dilute with 1000 ml of a 20% solution of salt in water, and extract four times with 200 ml portions of ethyl acetate. The combined extracts, amounting to 800 ml, should be washed twice with 200 ml portions of a 20% solution of salt in water. The ethyl acetate solution contains the product epoxide. It should be evaporated under a vacuum to a volume of about 200 ml, then reacted with lithium iodide just as in the first method to yield about 20 grams of 2,4,5-trimethoxyphenylacetone.

Recycling of solvents is possible with this method too. Ethyl acetate can be recovered during the vacuum evaporation by use of a dry-ice trap. The dimethyl-formamide can be recovered by vacuum distillation.

The Journal Method

A very effective alternative method exists for converting propenyl benzenes to phenylacetones. I know through mail received from the reading public that this method gives a yield of about 80% when used

with isosafrole. Similar results can be expected when used with asarone.

In spite of the high yields and simplicity of this reaction, I can't recommend its use. That's because this procedure uses thallium(III) nitrate to oxidize the propenylbenzene to the corresponding phenylacetone. The thallium(III) nitrate gets reduced to thallium(I) nitrate. Both of these heavy-metal compounds are very poisonous and, unlike organic chemicals, the heavy metals persist forever in the environment, and accumulate in the body. You want a bunch of thallium around the house about like you want to be kicked in the teeth with a heavy pair of boots.

A further bad aspect of this method is its high cost. 100 grams sell for $150, and the high molecular weight of the compound means that a lot of it has to be used to get a moderate amount of product. One pound of thallium(ni) nitrate is required for a 1-molar batch.

This method can be found in *Tetrahedron Letters* No. 60, pages 5275-80 (1970). To produce a one mole batch, dissolve one mole of propenylbenzene in some methanol, and put it into a one-gallon glass jug. In a beaker, dissolve one mole (448 grams) of thallium(HI) nitrate trihydrate in methanol. Then pour the thallium solution into the jug with the propenylbenzene, and stir at room temperature for 5 minutes. The thallium(I) nitrate formed by the reaction comes out of solution. It is removed by filtration.

The propenylbenzene has at this point been converted to a ketal. This is hydrolyzed to the phenylacetone by shaking the filtrate with about 2000 ml of 1 molar sulfuric acid solution in water for about 5 minutes. The phenylacetone is then extracted out with a couple of portions of tolulene. This extract is then washed with 5% NaOH solution, then distilled or purified by conversion to the bisulfite addition product.

Production of TMA-2, MDA, etc. from the Corresponding Phenylacetone

There are three good methods for converting the phenylacetone to the psychedelic amphetamine. Choice number one is to use reductive amination with a hydrogenation bomb with Raney nickel, ammonia and alcohol solvent. See *Journal of the American Chemical Society,* Volume 70, pages 12811-12 (1948). Also see *Chem. Abstracts* from 1954, column 2097. This gives a yield of about 80% if plenty of Raney nickel is used. The preferred conditions for use with MDA is a temperature of 80 C, and a hydrogen pressure of 50 atmospheres.

The drawback to this method is the need for a shaker device for the bomb, and also a heater. The use of platinum as the catalyst in the bomb works great when making MDMA, but gives lousy results when making MDA. There may be a way around this, however, for serious experimenters. It has been found in experiments with phenylacetone that a mixture of ammonia and ammonium chloride produces good yields of amphetamine (50%) when used in a bomb with platinum catalyst. Methylenedioxyphenylacetone is quite likely to behave similarly, along with other phenylacetones.

To use this variation, the following materials are placed in the 1.5 liter champagne bottle hydrogenation device described in Chapter 11 of *Secrets of Methamphetamine Manufacture,* Third Edition: .5 gram platinum in 20 ml distilled water. If this platinum is in the form of PtO_2 instead of reduced platinum metal catalyst obtained with borohydride, the experimenter must now reduce the platinum by pressurizing the bottle with hydrogen and stirring for about an hour. Next 100 ml of methylenedioxyphenylacetone is added along with 40 grams NHUCl, 500 ml methyl alcohol saturated with ammonia gas, and 50 ml NHjOH. The bottle is then set up as seen in Figure 17 in *Secrets of Methamphetamine Manufacture,* Third Edition. The hydrogenation is done as described in that section.

When the reduction is over, the contents of the flask are filtered to remove the platinum metal for reuse. Some crystals of NH4C1 are also filtered out; they are rinsed down with some water to remove them.

Next the filtered batch is poured into a 1000 ml round-bottom flask, a few boiling chips are added, and the glassware is set up for refluxing. Plastic tubing is attached to the top of the condenser and led outside. The mixture is boiled under reflux for one hour to force out the excess ammonia.

Next, the solution is allowed to cool, and made acid to congo red (about pH 3) with hydrochloric acid. Now the glassware is set up as shown in Figure 3 of *Secrets of Methamphetamine Manufacture,* Third Edition, and the solution is evaporated to about one-half its original volume under vacuum. A fair amount of crystalline material forms during the acidification and vacuum evaporation.

Next, 400 ml of water is added to the solution, and then it is extracted with about 100 ml of toluene. The toluene layer is thrown away because it contains garbage. The batch is now made strongly basic by adding lye water to it. It should be remembered here that it is very important to shake the batch well once it has been basified, to make sure that the MDA hydrochloride gets neutralized. Finally, the MDA is extracted out with a few hundred ml of toluene, and distilled under vacuum. The boiling point is about 160fi C under aspirator vacuum. The yield is about 50 ml.

Another very good choice of a method for converting methylenedioxyphenylacetone to MDA is the Leuckardt reaction. In this case formamide is used instead of N-methyl formamide. When used with phenylacetone to make amphetamine, only the very high-grade 99% material will work. In the case of methylenedioxy-phenylacetone, however, the much more commonly available 98% formamide works just fine. See *Chem. Abstracts* from 1952, column 11246, and Austrian patent 174,057. In this variation, 40 ml of methylenedioxyphenylacetone is mixed with 100 ml of freshly vacuum-distilled formamide, 2 ml glacial acetic acid, and 20 ml water. This mixture is heated up to about 130° C, at which point bubbling should begin, then the temperature is slowly raised to keep

the bubbling going, as described in Chapter 5 of *Secrets of Methamphetamine Manufacture,* Third Edition, until a temperature of ISO° C is reached. This should take at least 5 hours. The yield is 70%.

Processing is then done just as in the case of meth. The formamide is destroyed by boiling with lye solution. In this case, the ammonia gas produced is led away in plastic tubing. The formyl amide is then separated, and hydrolyzed by refluxing in a mixture of 60 grams KOH, 200 ml alcohol, and 50 ml water for an hour. After the reflux, the mixture is made acid with HC1, and the alcohol evaporated away under a vacuum. The residue is then diluted with water, and the freebase obtained by making the solution strongly alkaline to litmus by adding lye solution. The freebase is then extracted out with some toluene, and distilled.

This procedure is no doubt applicable to all phenylacetones. In the case of 2,4,5-trimethylphenylacetone, I would first try this with only half as much added water. Those phenylacetones containing the methylenedioxy grouping, I would use just as stated.

The last choice is a very simple, but also very time-consuming (several days!) reaction. Sodium cyanoborohydride in methanol with ammonium acetate and methylenedioxyphenylacetone at pH 6 react to give disappointing yields of MDA. See *Pikhal* by Dr. Shulgin in the section under MDA for full cooking instructions.

This method is general for all phenylacetones, as Dr. Shulgin used it on quite a variety of them, all with similar low yields.

In all of these methods, once the freebase is obtained in pure form by distillation (the boiling point of the amphetamine is similar to the phenylacetone), the freebase should be converted to the crystalline hydrochloride derivative. This is done by dissolving about 50 ml of freebase in about 400 ml ether or toluene, then bubbling dry HC1 gas through the solution, and filtering out the crystals to dry. See Chapter 5 of *Secrets of Methamphetamine Manufacture,* Third Edition for a full description.

Appendix

Know Your Essential Oils

Sassafras Oil — contains about 80-90% safrole. This is purified by fractional vacuum distillation. Boiling point of safrole is 234° C at normal pressure, about 120° C with an aspirator, and 105° at 6 torr. Yields MDA with ammonia, or MDMA (XTC) with methylamine. Dosage 1/10 gram.

Calamus Oil — that of Indian origin contains 80% ally! asarone. Oil from other areas contains much less asarone. Boiling point is 296° C at normal pressure, and 167° C at 12 torr. Yields TMA-2. Dosage is 40 rag.

Indian Dill Seed Oil — contains up to 53% dill apiol (3,4-methylene-dioxy-5,6-dimethoxy-alIylbenzene). Boiling point is 296° C with decomposition at normal pressure. Aspirator vacuum will distil! it at about 170° C. Yields DMMDA-2, dosage about 50 mg.

Nutmeg OH — contains 0-3% safrole, and 0-13% myristicin (3,4-methylene-dioxy-5-methoxy allylbenzene. The boiling point at 15 ton is ISO° C. Yield MMDA, dosage 80 mg.

Mace Oil — contains 10% myristicin.

Parsley Seed Oil — contains 0-80% parsley apiol (2-methoxy-3,4-methylene-dioxy-5-methoxy-allylbenzene). Its boiling point is 292° C at normal pressure, and 179^{2} C at 34 torr. It yields DMMDA, dosage about 75 mg. This oil may also contain 10-77% myristicin.

References: *Pikhal* by Dr. Shulgin, and *The Essential Oils* by Ernest Guenther.

Precursor and Essential Chemicals

Listed Precursor Chemicals

Domestic, Import and Export Distribution

Chemical	Thresholds by Base Weight
Anthranilic acid and its salts	30 kilograms
Benzyl cyanide	1 kilogram
Ephedrine, its salts, optical isomers, and salts of optical isomers	1 kilogram
Ergonovine and its salts	10 grams
Ergotamine and its salts	20 grams
N-Acetylanthranilic acid and its salts	40 kilograms
Norpseudoephedrine, its salts, optical isomers, and salts of optical isomers	2.5 kilograms
Phenylacetic acid and its salts	1 kilogram
Phenylpropanolamine, its salts, optical isomers, and salts of optical isomers	2.5 kilograms
Piperidine and its salts	500 grams
Pseudoephedrine, its salts, optical isomers, and salts of optical isomers	1 kilogram
3,4-Methylenedioxyphenyl-2-propanone	20 kilograms

Listed Essential Chemicals

Import and Export Distribution

Chemical	Thresholds By Volume	Thresholds By Weight
Acetic anhydride	250 gallons	1,023 kilograms
Acetone	500 gallons	1,500 kilograms
Benzyl chloride	N/A	4 kilograms

Chemical	Thresholds By Volume	Thresholds By Weight
Ethyl ether	500 gallons	1,364 kilograms
Hydriodic acid	40 liters (57%)	22.8 kilograms
Potassium permanganate	N/A	500 kilograms
2-Butanone (MEK)	500 gallons	1,455 kilograms
Toluene	500 gallons	1,591 kilograms

Domestic Distribution

Chemical	Thresholds By Volume	Thresholds By Weight
Acetic anhydride	250 gallons	1,023 kilograms
Acetone	50 gallons	150 kilograms
Benzyl chloride	N/A	1 kilogram
Ethyl ether	50 gallons	135.8 kilograms
Hydriodic acid	10 liters (57%)	5.7 kilograms
Potassium permanganate	N/A	55 kilograms
2-Butanone (MEK)	50 gallons	145 kilograms
Toluene	50 gallons	159 kilograms

The cumulative threshold is not applicable to domestic sales of Acetone, 2-Butanone (MEK), and Toluene.

A total of 20 precursor and essential chemicals have been listed. The Administration may add or delete a listed chemical by publishing the proposed change in the Federal Register with at least a 30-day comment period prior to the publication of the final rule. A chemical handler may petition to have a chemical added or deleted from the list by following the procedures in 21 CFR 1310.02.

Waste Exchanges

Alberta Waste Materials Exchange
Jim Renick Red Deer ARC
Provincial Building, #303A
Edmonton, Alberta Canada
T6H 5X2 (403) 450-5461

Arizona Waste Exchange
Barrie Herr
4725 East Sunrise Drive, Suite 215
Tucson,AZ85718
(602) 299-7716

B.A.R.T.E.R. Waste Exchange
Jamie Anderson
MPIRG
2512 Delaware Street South East
Minneapolis, MN 55414
(612)627-6811

By-Products & Waste Search Service
Susan Salterberg Iowa Waste
Reduction Center University of
Northern Iowa Cedar Falls, IA
50614-0185 (319) 273-2079

California Materials Exchange (CALMAX)
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Interstate Waste Management Board
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Sacramento, CA 95826
(916) 255-2369

Canadian Waste Materials Exchange
Dr. Robert Laughlin
Ortech International 2395
Speakman Drive
Mississauga, Ontario
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(416)823-4111

Hawaii Materials Exchange
Jeff Stark P.O. Box 1048 Paia,
HI 96779 (808) 579-9109

Indiana Waste Exchange
James Britt
Recycler's Trade Network, Inc.
P.O. Box 454
Carmel, IN 46232
(317)574-6505

Industrial Material Exchange Service
Diane Shockey
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2200 Churchill Road #34
Springfield, IL 62794-9276
(217) 782-0450

Montana Industrial Waste Exchange
Montana Chamber of Commerce
Don Ingles P.O. Box 1730 Helena,
MT 59624 (406) 442-2405

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Dwight Long 10
Four Comers Recycling 3
P.O. Box 904
Farmington, NM 87499
(505) 325-2157

Northeast Industrial Waste Exchange
Carrie Pugh
620 Erie Boulevard West, Suite 211
Syracuse, NY 13204-2442
(315)422-6572

Pacific Material Exchange
Bob Smee
E4708 Jaremko Drive
Mead, WA 99021 (509)
466-1019

RENEW
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Texas Water Commission
P.O. Box 13087
Austin, TX 78711
(512)463-7773

Southeast Waste Exchange
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Urban Institute, UNCC Station
Charlotte, NC 28223 (704) 547-
2307

Southern Waste Info Exchange
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960 Tallahassee, FL
32302 (904)644-5516

Distributors

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AR Industrial Development Commission
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Little Rock, AR 72201
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John Konefes
IA Waste Reduction Center
University of Northern Iowa
75 Biology Research Complex
Cedar Falls, IA 50614-0185
(319)273-2079

Kentucky
Charles Peters
Division of Waste Management
Department of Environmental Protection
18 Riley Road
Frankfort, KY 40601
(502) 564-6761

Missouri
Tom Welch
Missouri Environmental
Improvement Authority
325 Jefferson Street
Jefferson City, MO 65101
(314)751-4919

North Dakota
Robert Tubbs-Avalon Division of
Waste Management 1200 Missouri
Avenue Bismarck, ND 58202-5520
(701) 221-5166

Oklahoma
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OK Waste Exchange Program P.O.
Box 53551 Oklahoma City, OK
73152 (409) 271-5338

Wisconsin
Sam Essak
Bureau of Solid Waste Management
P.O. Box 7921
Madison, WI53707
(608) 267-9523

All Other Locations
Diane Shockey
IMES
2200 Churchill Road, #34
P.O. Box 19276
Springfield, IL 62794-9276
(217) 782-0450
Fax (217) 524-4193

Love Letters From The Heat

UNITED STAnS DEPARTMENT OF JUSTICE
MUG INfORCIMINI ADMINISTRATION !2«A
FEDERAL ILDG AND U S COURTHOUSE 517 EAST
WISCONSIN AVINUt MILWAUKEE. WISCONSIN
53202

Dear Sir:

The United States Congress recently passed the Chemical Diversion Trafficking Act of 1988 (Public Law 100-690). This Act requires in part, that any person who manufactures, distributes, imports or exports certain precursor or essential chemicals identify their customers, maintain retrievable records, report suspicious or unusual orders, and provide advance notification of imports and exports. The requirements for maintaining records and reporting suspicious or unusual orders also apply to tableting and encapsulating machines.

In order to determine if you will be subject to the provisions of the law, we ask that you complete the attached questionnaire and return it to as in the enclosed envelope within two weeks. If it appears that you will be subject to this Act, you will be contacted and provided with further information. If you have any questions, please contact Investigator Marilyn J. Sumner or Investigator Kathy L. Edwards-Federico at our office (414) 297-3395.

Thank you for your cooperation in this matter.

J. E. Snyder
Resident Ag*nt in Charge

***415.1**

QUESTIONNAIRE

NAME:_____ ADDRESS:

According to information that DEA has obtained, you purchased one or more of the following precursor and essential chemicals. Please indicate which chemicals have been purchased in threshold or larger quantities.

PRECURSOR CHEMICALS		ESSENTIAL OEHmu s	
Anthranilic Acid and its salts.	___	Acetic Anhydride	__
Benzyl Cyanide	___	Acetone	__
Ephedrine, its salt, ootical isomers, and their salts	———	Benzyl Chloride --nionoe	———
Ergonovine and its salts	___	Et$_y$i Ether	
Ergotamine and its salts	__	Hydriodic Acid	___
N-Acetylanthranilic Acid 3nd U° 3alt°	———	Potassium Permanganate	———
Norpseudoephedrine, its salts, optical isomers and their salts	——	2-8utanone fnr Kut-h i rn, ———	
		or	
Phenylacetic Acid and its salts optical iaomers, and their salts	————	Toluene	——
Phenylpropanolanine, its salts, optical isomers, and their salts	———		
Plperidine and its salts	___		
Pauedoephedrine, its salts, optical ' isomers and their salts	———		
5,*-Methylenedioxyphenyl-Z propanone (Piperonyl methylketone)	———		

DO V_{OU} MANUFACTURE OR DISTRIBUTE TABLET1|!G_ OR EIICAPSULATIHG '^

Briefly describe your uaes of these chemicals:

If you use these chemicals in a manufacturing process do you salvage any of the chemical for future sale or redistribution? Yes No --—'

Do you redistribute any of these chemicals in any manner? (Not including as a component of an end product mixture) Yes ___ No —-

If yes, please explain:

Please provide the name, title and telephone number of a contact person:

NAME AND TITLE: _ _ _ ________ ___ ________

TELEPHONE NUMBER: _ __ _ _______

Thank you for your cooperation in this matter.

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SUPPLEMENTAL LISTED
CHEMICAL QUESTIONNAIRE

BUSINESS NAME: _ __ Address:

1. Do you currently 'rr in the past **two years) handle any of the following chemicals** in threshold **quantities or above?**

CHEMICAL	THRESHOLD (BY WEIGHT)	YES/NO	BUSINESS ACTIVATION CODES
METHYLAMINE AND ITS SALTS	1 KG.	NO	
ETHYLAMINE AND ITS SALTS	1 KG.	NO	
D-LYSERGIC ACID, ITS SALTS, OPTICAL ISOMERS, AND **SALTS OF OPTICAL ISOMERS**	**10 GRAMS**	NO	
PROPIONIC ANHYDRIDE		NO	
ISOSAFROLE		NO	
SAFROLE		NO	
PIPERONAL		NO	
N-METHYLEPHEDRINE, ITS SALTS, OPTICAL ISOMERS, **AND SALTS OF OPTICAL** ISOMERS	1 KG	NO	
N-ETHYLEPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS	1 KG	NO	
N-METHYLPSEUDOEPHEDRIHE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS	1 KG.	NO	
__*£ N-ETHYLPSEUDOEPHEDRIHE ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS	1 KG.		
HYDRIOTIC ACID (HYDRIODIC ACID) (previously listed as an essential chemical with a threshold of 22.8 KGS.)	1.7 KG. (1 Liter)	NO	
3,4-METHYLENEDIOXPHENYL-2-PROPANONE	4 KGS.	l(Ji>	__^»^__

(previously listed as a threshold of 20 KGS.)

2. Handling status of previously controlled precursor and essential chemicals:

LISTED PRECURSOR CHEMICALS

Domestic. Import and Export Distribution

YES/HO BUSINESS ACTIVITY CODE!51			
ITS SALTS	30 KGS.	*A/0*	
BENZYL CYANIDE	1 KGS.	</j	
EPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS	1 KG.		
ERGONOVINE AND ITS SALTS	10 CMS.		
ERGOTAMINE AND ITS SALTS	20 CMS.		
N-ACETYLANTHRANILIC ACID AND ITS SALTS	40 KGS.		
NORPSEUDOEPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS	2.5 KGS-		
A"> PHENYLACETIC ACID AND ITS SALTS	1 KG.	*M*	
PHENYtPROPANOLAMINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS	2.5 KGS.		
a>0 PIPERIDINE AND ITS SALTS	500 CMS.	*nil*	
PSEUDOEPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS	1 KG.		
Ak> LISTED ESSENTIAL CHEMICALS			

ACETIC ANHYDRIDE	1,023 KGS.		
ACETONE	1,500		
BENZYL'CHLORIDE	4 KG		
ETHYL ETHER	1,500		
POTASSIUM PERMANGANATE	500 KG		
2-BUT.ANONE (HER)	1,500		
TOLUENE	1,500		
DOMESTIC DISTRIBUTION			
ACETIC ANHYDRIDE	1,023 KGS		
ACETONE	150 KGS.		
BENZYL CHLORIDE	1 KGS.		
ETHYL ETHER	135.8 KGS		
POTASSIUM PERMANGANATE	55 KGS.		

»nd **Export** Distribution

2-BUTANONE (MEK) 145 KGS.
TOLUENE 159 KGS.

(The threshold is cumulative by calendar month except for domestic sales of Acetone, 2-Butanone (MEK), and Toluene for which sales of 50 gallons or more are regulated.)

3. Please provide the name, title, and telephone number of a contact person.

NAME AND TITLE:

TELEPHONE:

A Few Words Concerning Calamus by Cousin Lester

Acorus calamus L (also known as Sweet Rag, Sweet Sedge and Rat Root); *Araceae;* Arum Family. Calamus is a native perennial grasslike plant with sword-shaped leaves and thick, cylindrical spikes of tiny, brown flowers. It possesses a horizontal jointed rhizome of spongy texture, from one-half inch to an inch in thickness that sometimes attains a length of several feet Calamus grows in marshy or wet habitats, primarily in the Prairie Bioregion. The dried root (rhizome or rootstock) has long been used in medicine and as an ingredient of certain flavors, liqueurs and perfumes. The rhizome contains a volatile oil, which can be obtained by steam distillation, and that has a peculiar, but pleasant, rather sweet odor and flavor. The rhizomes are collected in the spring or late fall, and are washed, dried artificially at moderate heat and freed of fibrous rootlets. The fiber-like rootlets can be removed before drying, but are usually removed after drying because they become brittle and are more easily dislodged. The "stripped" roots are more aromatic than those which have been peeled.

The dry, unpeeled footstocks are known to have both carminative (prevents the formation or causes the expulsion of gas or air in the intestinal tract) and anthelmintic (destroys or expels intestinal worms) properties.

Calamus was prized by the Native Americans of the prairies for its medicinal, ritualistic and dietary uses. The Pawnee name for the plant is "kahtsha itu," which means "medicine lying in the water." The Osage know calamus as "peze boao'ka," or "flat herb." To the Lakota Sioux, the plant is "sinkpe tawote," which translates as "muskrat food." They also refer to the root as "sunkace," or "dog penis," probably because of the shape of the flower stalk.

The Osage chew the root for its distinctive flavor, while the Lakota Sioux eat the leaves, stalks and roots (the plant's young, tender leaves

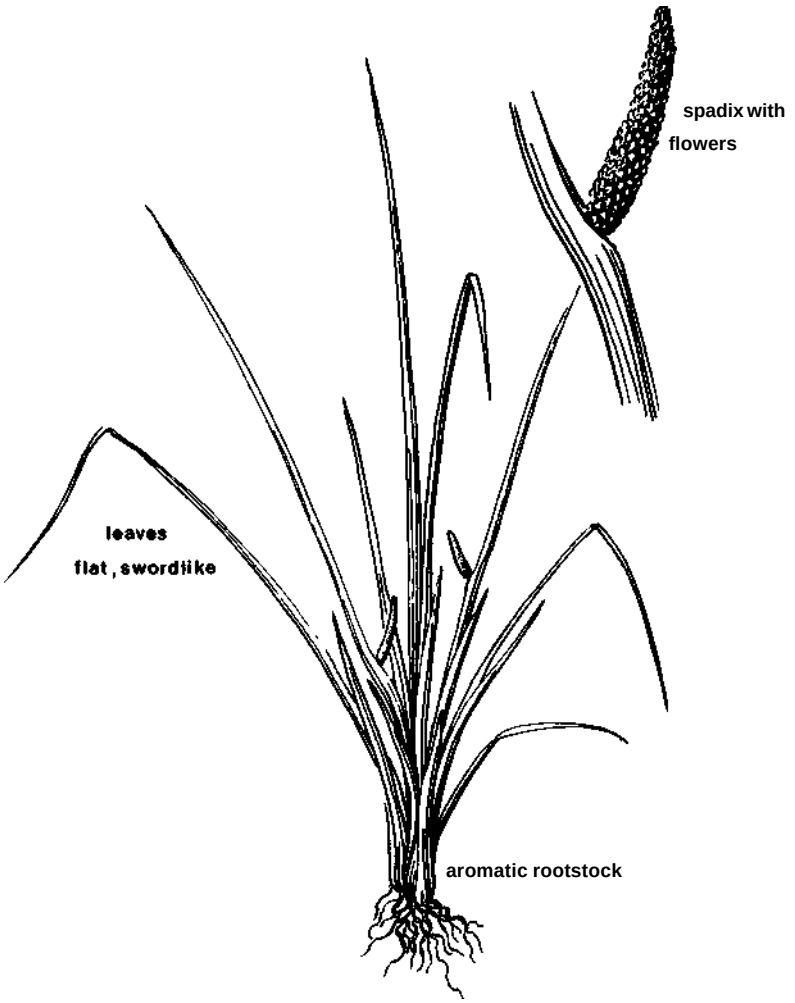
are a welcome addition to tossed green salads). The Omaha ingest boiled roots, often for medicinal reasons.

Calamus grows in the wild in water, but can be cultivated in practically any good, fairly moist soil. It usually fares well in moderately dry soils which would sustain crops of com or potatoes. The plants can be readily propagated from divisions of old roots. They should be set out early in the fall, planted one foot apart in rows and adequately covered. During the growing season, the plants require frequent and thorough cultivation.

In the fall, the roots are harvested. A spade or plow may be used. The tops, along with about an inch of the rootstock, are cut off and used for new plantings.

Calamus can be grown from seeds, which are commercially available in many parts of the world. Burma and Sri Lanka are two countries where the plant is widely cultivated. Seeds are available from a number of sources in North America, including: Prairie Moon Nursery
Route 3, Box 163 Winona, MN 55987 (507) 452-1362

L.E.R. (Legendary Ethnobotanical Resources)
PO Box 1676
Coconut Grove, FL 33233
(305) 649-9997, is a source for calamus roots.


spadix with
flowers
leaves
flat, swordlike
aromatic rootstock

Uncle Fester has done it again! The underground mastermind of psychedelic cookery has provided up-and-coming Owsley-wannabes with *Practical LSD Manufacture,* the most detailed, comprehensive and concise description *ever* of several of the methods employed in the preparation *of lysergic acid diethylamide,* or *LSD,* from natural sources. Uncle Fester also offers a breakthrough in psychedelic literature: a simple process for extracting the hallucinogenic drug *TMA-2* from the commonly-grown calamus plant. *Practical LSD Manufacture* contains:

- An overview of LSD production
- Natural sources of the lysergic amides, including harvesting procedures for ergot-infested rye and Spartina marsh-grass
- Methods of extraction and isolation of the lysergic acid amides
- An interpretation of LSD-progenitor Albert Hofmann's patented "one-pot shot" method of LSD synthesis, plus purification and storage techniques
- A never-before-published presentation of "Method X," wherein a propionic anhydride mixes with lysergic acid, allowing for a much-improved synthesis
- A section on solvent management, a crucial but often-overlooked detail all chemists should be aware of
- How to manufacture the hallucinogen 2,4,5-trimethoxy-amphetamine (TMA-2) from the calamus root
- Detailed growing, harvesting and availability information on the calamus plant
- Cautionary notes on keeping out of trouble
- And much, much more!

Loompanics Unlimited is proud to offer Uncle Fester's complete, illustrated guide for anyone who is interested in *Practical LSD Manufacture! Sold for information purposes only!*

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