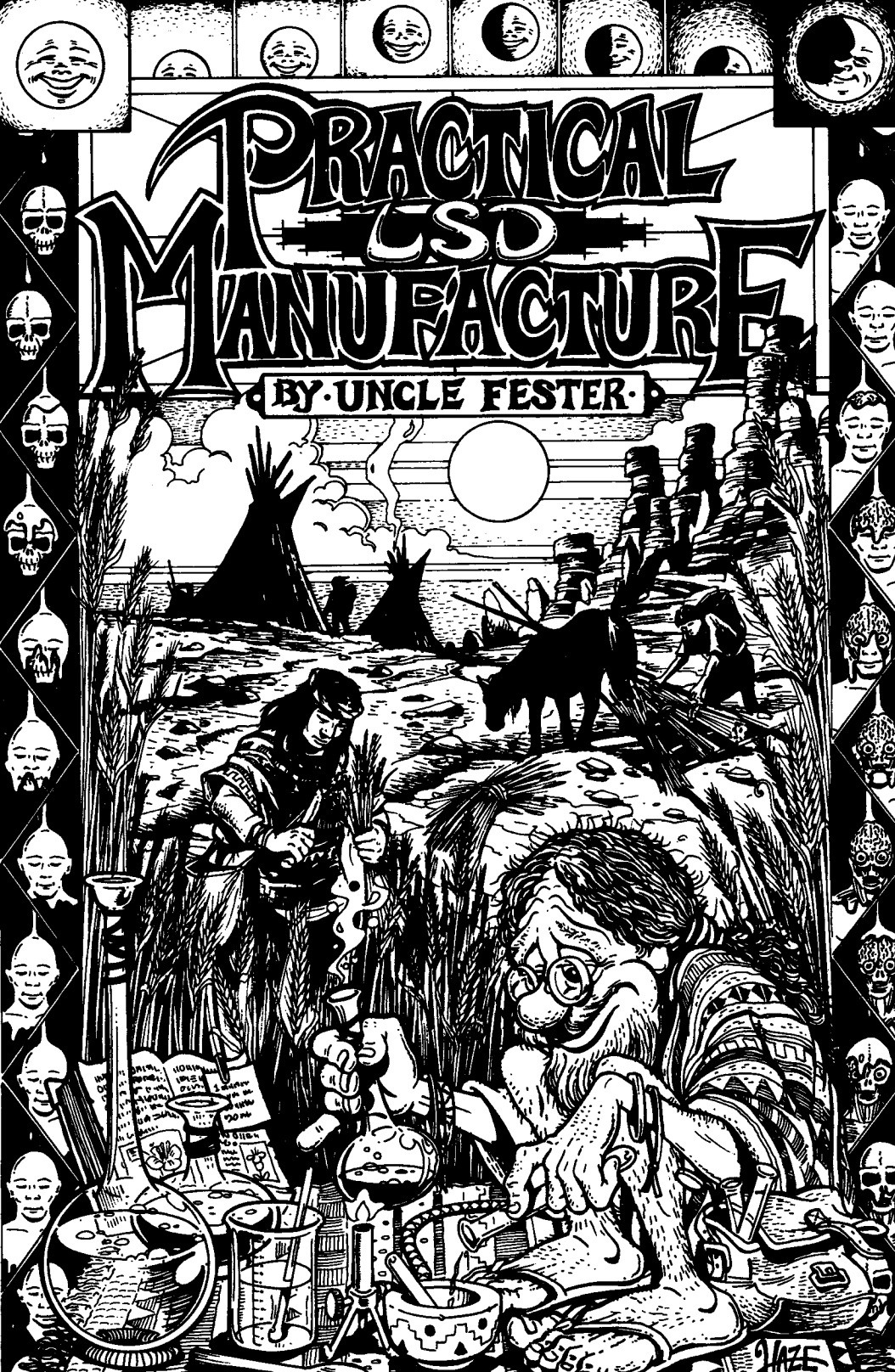
PRACTICAL
LSD
MANUFACTURE
BY·UNCLE FESTER·

# Practical LSD Manufacture

## 3rd Edition

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**by Uncle Fester**

Festering Publications
Green Bay, WI

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# Contents

# Preface

The DEA 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. The vast majority of acid is uniform, bland, low powered and uninspiring.

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. The most convenient and direct starting materials for acid production such as lysergic acid or lysergamide are controlled substances and unavailable directly to the public in any quantity. These roadblocks,however, pale in comparison to the most important factor - the inaccessibility of good information to those motivated to put it into action.

**Practical LSD Manufacture**

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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

# One: 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 Seventh 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.

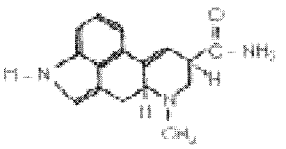
A small scale experimenter can turn his or her prescription for the migraine medicine ergotamine into a few thousand hits of acid by extracting the ergotamine from the pills and converting it to LSD by the methods which will be detailed in this third edition. A few like minded migraine sufferers could pool their prescriptions and greatly multiply the yield of product. Boutique acid brews may be ready for a comeback!

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.

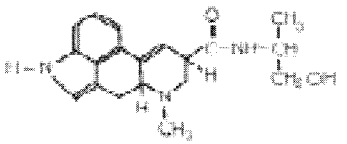
Even a seemingly very simple reaction cannot escape this law. Case in point is the hydriodic acid and red phosphorus reduction of ephedrine to methamphetamine. If in this reaction the ephedrine is not fairly free of the fillers and binders found in the stimulant pills from which it is extracted, the 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 some 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.

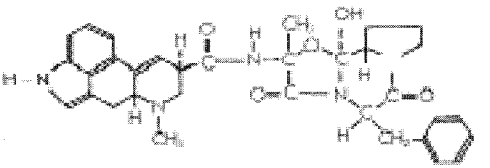
Mother Nature's bounty is then squirreled off to the lab site for the biochemical phase of the process - the isolation of the lysergic alkaloids. Here one or more of a series of alkaloids are freed from the very complex plant matrix and hopefully isolated in a pure form. These alkaloids all have one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



ergine

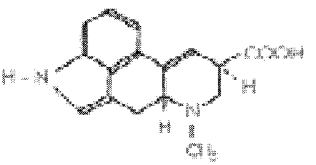


ergonovine

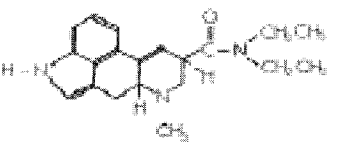


ergotamine

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 clavinet 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 hornshaped 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 modern 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.

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.

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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. Animals or children living in the house greatly exacerbate contamination problems. 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 ½ 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 seventh 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."

# Three: 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 1/4 to 1/2% alkaloids by weight. A 200 pound harvest of ergot will, after extraction, yield 1/2 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.

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 naturally occurring ergot alkaloids come in isomeric pairs. The carboxyl grouping of the amides can be in the desired and very physiologically active configuration. Or they can be in the inactive "iso" configuration. The amides in the "iso" configuration will not form crystalline precipitates with tartaric or malic acid.More on this later. 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:

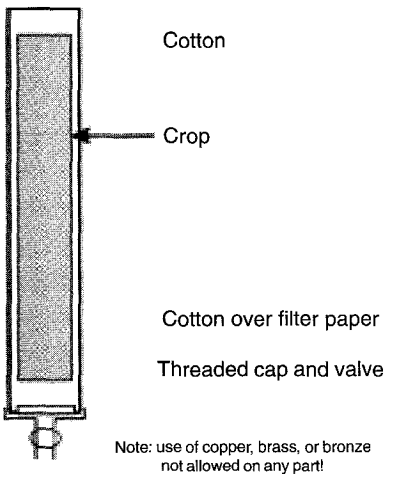



**Figure 1**
***Apparatus for large-scale defatting***

The extraction solvent is made up by adding one-tenth gallon strong ammonia (28% $NH_3OH$; 56% $NH_4OH$) 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. Wrapping the flask in foil will also protect the flask contents from light damage. 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 DEA. The serious experimenter may wish to try substituting toluene for ether, since it is not now on the mandatory snitch list. Ether starting fluid would work fine for smaller batches.

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 can be a hassle to buy tartaric acid sometimes.

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. This conversion is an equilibrium reaction in which only partial reversal to the iso form will occur. Heating is also needed to get the reaction moving, but it should be avoided.

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 1/4 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.

Now as was mentioned previously, the lysergic amides occur in pairs in nature. This extraction procedure was designed to isolate the "active" members of the pairs and leave behind the inactive "iso" alkaloid. Hunting for this "iso" material should double one's yield of product whether one is extracting ergot or seeds.

Where would the iso alkaloid be? Since the iso member of the alkaloid pair doesn't form salts easily, it should still be in the ether and chloroform solution that is left from the extraction of the ergot or

seeds. The active member of the alkaloid pair was extracted into the decimolar acid solution earlier in this procedure, but the inactive iso alkaloids should still be in the solvent.

If one would evaporate away most of this solvent, isomerization can be done very easily by the action of KOH in alcohol. The procedure can be found in the Journal of the *Chemistry Society*, Volume 139, page 1168 and 1440 (1936).

One molar KOH solution in methanol is made by adding 5.6 gr KOH to 100 ml of methanol or 56 gr KOH to 1000 ml methanol, depending upon batch size. The residue left after most of the chloroform and ether is evaporated away from the original extraction solvent can then be dissolved in this alcoholic KOH solution. It should next be filtered and put into a boiling flask. A stream of nitrogen is passed through the boiling flask, and the mixture is brought to reflux for on half hour. The alcohol is then removed under a vacuum at room temperature, and the residue extracted into acid solution as described earlier in this section. One must use extra acid to compensate for the KOH in the residue. Then filter the solution and extract out garbage with ether. The acidic water can then be based with ammonia as before, and the fresh portion of product extracted with ether as described earlier in this section.

## Ergotamine Pill Extraction

Ergotamine tartrate is often prescribed to people who suffer from migraines. If a few like minded migraine victims get together and pool their prescriptions, they could very easily produce thousands of doses of LSD.

Ergotamine tartrate pills come in a variety of forms. The pill generally contains one or two mg of the active ingredient. Obviously, the latter are preferable for extraction. Pills which also contain caffeine should be avoided because they would make the extraction more complicated. Pills which contain the hydrogenated form of ergotamine, the dihydroergot pills, should never be extracted because they are valueless for producing LSD.

To extract the pills, grind them up in a blender. Then extract out the ergotamine tartrate using rubbing alcohol or whatever other source of alcohol is convenient. One gram of ergotamine tartrate will dissolve in 500 ml of alcohol, but it will take more alcohol than that to completely extract the pills. The general procedure for pill extraction is to use several portions of alcohol, and to filter the extract off the settled pill mass, then extract the pill mass again with a fresh portion of alcohol. Protect the extracts from contact with light.

When several extractions of the pills have been done, the pooled alcohol extracts should be placed into a vacuum flask. Then the alcohol should be boiled off under a vacuum. As the alcohol evaporates away, ergotamine tartrate will start to come out of solution because it will have exceeded its one gram per 500 ml limit of solubility. It should be filtered out and rinsed with ether. This fairly pure ergotamine tartrate should then be stored in a glass container in a cool, dark place.

# Four: 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.

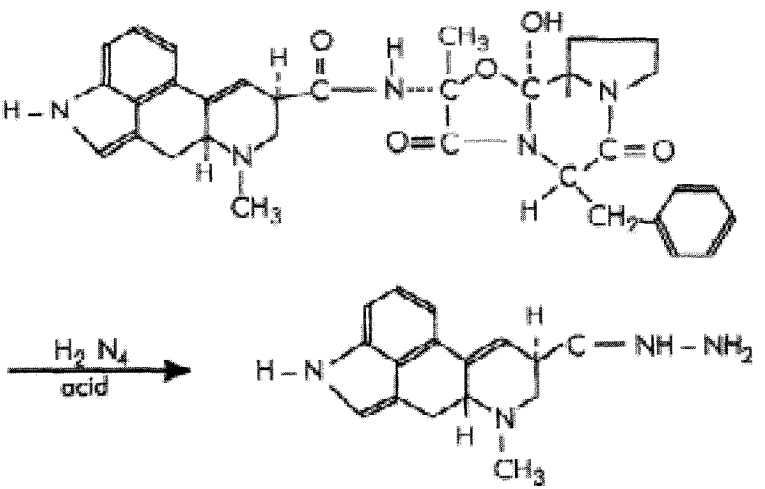
There are several possible methods to follow in the conversion of the lysergic amides to LSD. The first two presented in this book are pretty good, and highly recommended. The third one is OK. The fourth one may kill you with phosgene gas, but seems to work well. The Method X procedures don't have direct citations to their use in making LSD, but should be the best of all methods. 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 isolysergic 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 iso- LSD. A bit of warm KOH in methanol then gives conversion of the iso-LSD to the active form of LSD.

This method at first glance seems complicated, but the actual chemical chemical manipulations involved are easier than those found in Chapter 6. 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 may catch the attention of those shit-eating dogs at the DEA. It is fortunate that only small quantities are required to do the reaction. 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 iso-Lysergic Acid Hydrazide



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 200 ml of decimolar tartaric-acid solution (3 grams tartaric acid in 200 ml water) to the flask, and 200 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 500 ml sep funnel, and drain the lower layer (water solution of iso-lysergic acid hydrazide tartarate) into an 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 iso-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 (42 grams per liter) sodium bicarbonate 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 iso-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.

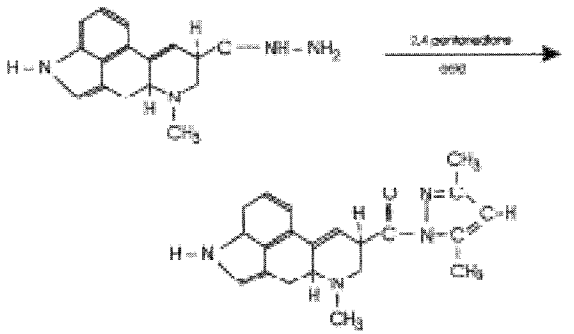
Both of these choices are really very poor, because lysergic acid hydrazide, unlike most other lysergic compounds, crystallizes very well with negligible loss of product. At the hydrazide stage of LSD manufacture, one has a perfect opportunity to get an exceedingly pure product, freed from clavine alkaloids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



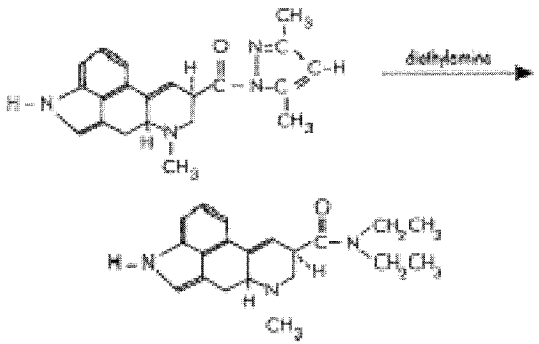
In this reaction, one mole of iso-lysergic acid hydrazide is dissolved in an inert, water-miscible solvent like ethanol. Then an excess of 1-molar hydrochloric acid is added to form a salt with the iso- lysergic acid hydrazide. To this mixture is then added two moles of acetylacetone (2,4-pentanedione), 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:
## Iso-LSD CH,




This simple and easy reaction is done as follows: In a flask wrapped in a single layer of foil are placed 1 gram iso- lysergic acid pyrazole, and 20 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 iso- LSD.

## Purification and Storage

At this point, the process has yielded iso- LSD freebase. In this state, the substance is quite unstable and not suitable for storage, and it is in the inactive iso form. Conversion of all lysergic compounds from the iso form to the active form is easy. The procedure first mentioned in the last chapter is used, whereby heating the iso form product in some alcoholic KOH solution results in conversion of the iso LSD to active form LSD.

One molar KOH solution is prepared by dissolving 5.6 gr KOH in 100 ml of methanol. One gram of the iso LSD is then dissolved into the KOH solution, and the resulting mixture is poured into a boiling flask. A condenser is attached to the flask and a stream of nitrogen is begun through the apparatus. Wrap the flask in foil, then heat the mixture to 50 C for half an hour using a boiling water or steam bath. This results in a mixture which is roughly two thirds active form LSD and one third iso LSD.

After the half hour reaction time has passed, the methanol should be boiled or evaporated away under a vacuum. In a well equipped lab, a rotovap would be used to do this task and a good aspirator would be used as the source of vacuum. One can use warm water as a heat source on the flask to speed evaporation of the methanol.

Once all the methanol has evaporated away, it is time to separate the active form LSD from the iso LSD. There are a few ways to do this task, and here the procedure using chromatography will be illustrated.

The following procedure is taken from US patent 2,774,763. 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 thesolvent 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. This is done simply by heating it with more one molar KOH solution. In this way, virtually all the iso LSD will be converted to the active form LSD.

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 first removing the solvent from the benzene-chloroform elute from the chromatography column. Evaporation of the solvent to a low volume under a vacuum gives a residue which is LSD free base. Dissolve this free base in about 4 ml of warm methanol for each gram of LSD free base. Then slowly add the d-tartaric acid and allow it to react in the dark for half an hour. Then dropwise with stirring add ether to the mixture. The originally clear mixture will become milky as the ether content of the solution

rises. This is crystalline LSD tartrate coming out of solution. Continue adding ether until the milkiness of the solution will not clear on continued stirring. This will take around 10 volumes of ether for each volume of methanol. Let the crystals fully form by putting the mixture in the freezer overnight, then filter them out and dry them under a vacuum. This should result in a mass of crystals at the bottom of the flask, and a clear solution. 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. It will take 4 or 5 ml of warm methanol to dissolve the LSD. 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 (iso-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, pages 1168-1169 and 1440-1444(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.

This general procedure will be referred to over and over again in this book as the way to convert iso product into active product. The conversion of iso Product to active form is often called epimerization. It is just switching around the orientation of the carboxyl group by means of KOH and some heating in methanol solvent to yield the active isomer.

There is a competing side reaction, and that reaction is hydrolysis of the amide LSD to lysergic acid. In the Smith and Timmis works cited, they found that very little hydrolysis was done to complex lysergic amides like ergotamine or ergometrine within one hour of boiling with one molar KOH, but they got the steric inversion they wanted.

A simple amide like LSD would be more vulnerable to KOH hydrolysis than the bulky and sterically hindered ergotamine. They found for example that ergine was almost half hydrolysed to lysergic with an hour. LSD would give results intermediate between the two. One would be well advised to be gentle with the heating during this isomerization of LSD. How gentle? Well your Uncle has spent the past twenty years fighting in the trenches of the meth wars, and so can't give you an exact answer?but heating below the boiling point of the methanol solvent will cause very little hydrolysis of any amide, including LSD.

## 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 warmed to about 40 or 50 C under 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

Into a 3-quart-capacity glass baking dish (Pyrex) put 750 ml strong ammonia (28% NH3), 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


The reaction which produces diethylamine also yields as byproducts 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 2 or 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 ½ 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 °C below 0 °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 0 °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, especially if you are Fester! For most of you?getting tartaric acid will be easy. Enjoy the synth?

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% labgrade 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 CaCl2- 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 CaCl2.

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.

# Five: 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. The regular or D-lysergic acid is the only isomer which will form salts and be precipitated from solution with acid at the end of the hydrolysis, and it will be formed in the ratio of roughly three to one over the isolysergic acid.

## 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 a few hours under a nitrogen atmosphere.

Next, the reaction mixture should be cooled, and sulfuric acid added to it slowly until it reaches pH 3. This results in the precipitation of crude lysergic acid having a dark color. The beaker should be allowed to sit over night in the fridge to let the crystals fully form. Then the crystals of crude D-lysergic acid should be filtered out and rinsed with a little ether.

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 mls of strong ammonia. The residue which does not dissolve within an hour of stirring is inorganic, and can be discarded. Filtration or letting the beaker sit to settle the sludge followed by decantation is the way to remove this insoluble material.

The alcohol solution of lysergic acid should next be acidified to roughly pH 3 using diluted sulfuric acid once again. 2 or 3 N sulfuric acid is roughly the proper range of dilution of the sulfuric acid. The pH of the solution is best tracked using pH papers which have been moistened with some water. Then a glass rod is dipped into the lysergic acid solution, and a bit put onto the indicator strip of the pH paper. Meters don't work well in alcohol solution. The crystals of lysergic acid will form while the beaker is sitting in the fridge overnight. Then filter or centrifuge, and rinse with some ether. This yields about 3 or 3.5 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.

To increase the yield, it would be worthwhile to pull out the isolysergic acid and convert it to lysergic acid. The isolysergic acid should still be in the filtered mother liquor from paragraph two of this

section. If this pH 3 solution is based with bicarb and then extracted with ether or chloroform, the isolysergic acid should extract out. Removal of this solvent under a vacuum followed by another heating in alcohol solution containing KOH should then give a fresh portion of D-lysergic acid. Then add water and cool the solution and slowly add sulfuric acid solution to pH 3. Remove the alcohol under a vacuum, but leave a good portion of the water remaining. Then chill overnight in the fridge and collect the crude crystals.

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 entrain ed 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% by weight 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% NH4OH 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% NH4OH, 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 1/2 grams of 96% pure lysergic acid. The resin can be used over and over again by recharging in 20% sulfuric acid solution, and rinsing with distilled water.

The mother liquor of this process also contains isolysergic acid which should be recovered. This mother liquor would be the water left from the cation exchange extraction in paragraph 6. Basing this water with bicarb and solvent extraction and a KOH isomerization as in the first method should yield another measure of D-lysergic acid.

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

## Racimization free hydrolysis

This variation comes in very handy when one is starting with pharmaceutical ergotamine tartrate as no isolysergic acid is formed. All the product from the hydrolysis is D-lysergic acid. See Chem Abstracts Vol. 95 entry 133217 (1981) and Hungarian Patent 19,274. These inventors found that the addition of some methanesulfonic acid to a hydrolysis solution made up of a 50-50 alcohol water mixture with the usual amount of KOH gave none of the isolysergic by-product. Unfortunately, the Chem Abstracts article doesn't state just how much methanesulfonic acid they added to the aqueous alcoholic KOH solution, but it couldn't have been nearly enough to neutralize the KOH. They state an 81% yield with a workup procedure very similar to that in Method One. It's worth a bit of experimentation if one should have a bunch of those ergotamine tartrate pills to work with.

## 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, or wrap the flask in foil.

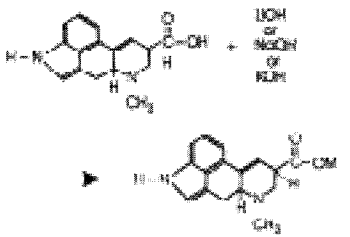
# Six: LSD from Lysergic Acid and $SO_3$

This is the second of 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 $SO_3$ then added to form the mixed anhydride of lysergic acid.

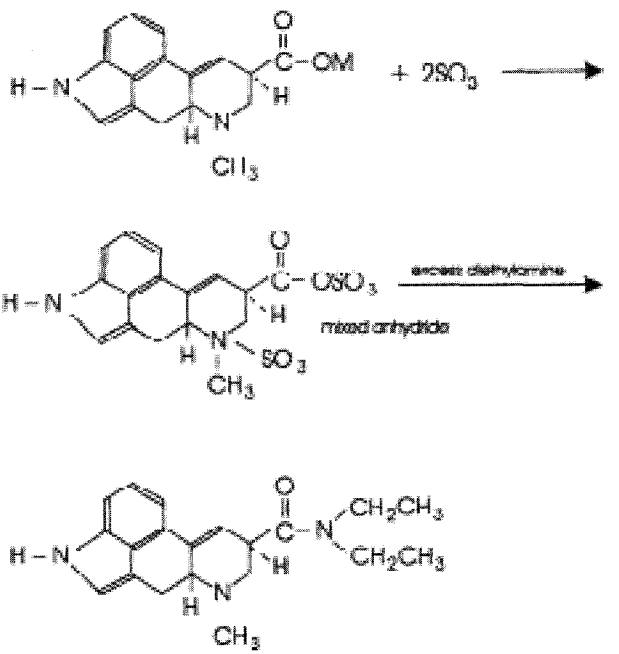
The other key precaution is the need to maintain strictly anhydrous conditions in both the production of the $SO_3$-solvent complex, and the reaction of that complex with the lysergic acid salt to produce the mixed anhydride. The reason for this is that $SO_3$ is the anhydride of sulfuric acid, and any 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 $SO^3$ solvent complex. $SO^3$ is available from a couple of sources. There is a

form of pure stabilized $SO_3$ called Sulfan B. If this material can be had off of some unguarded shelf, it is superior to the other source of $SO^3$, fuming sulfuric acid.

To make the $SO^3$-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-$SO_3$ complex is generally used at a strength of .5 molar, while dimethylformamide-$SO_3$ complex is used at 1 molar strength. 80 grams per liter $SO_3$ is 1 molar. Using Sulfan fresh from the bottle, it is not necessary to analyze the strength of the resulting $SO_3$-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 $SO_3$ 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 $SO_3$-solvent complex from fuming sulfuric acid is to use an adapter such as the one pictured in Figure 2.

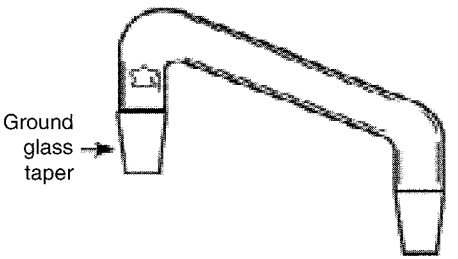



**Figure 2:**
***Adapter used in preparing $SO_3$-solvent complex from fuming sulfuric acid.***

With all glassware 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.

On the 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 $SO^3$ 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 $SO_3$. 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 $SO_3$ 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 .1N NaOH (prepared by dissolving exactly 4 grams of NaOH pellets in one liter of water) until the color of the solution turns 50 Adapter used in preparing SO3-solvent complex from fuming sulfuric acid.

## Molarity SO3 in solvent = mls 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 $SO_3$, 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 $SO_3$ solution is used. That is crucially important to the success of the reaction.

When the $SO_3$-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 SO3 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 SO3 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.I 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 may become a source of suspicion for the clandestine cook.

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 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 mls 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 SO3-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 $SO^3$ 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 $SO_3$ 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 is an unavoidable complication encountered in

this and most other methods of making LSD. Fortunately, the active LSD isomer is formed predominately over the inactive iso isomer.

The iso-LSD can 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.

There is a simpler alternative method which doesn't make use of chromatography. It takes advantage of the fact that only the active form of LSD will form salts with tartaric acid or malic acid and be precipitated from solution as crystals.

Dissolve each gram of LSD and iso-LSD mixture into roughly 4 ml of warm methanol. A yield of roughly 15 grams of crude product could be expected from this example batch, so 60 ml of warm methanol would be sufficient. Then a little less than 4 grams of d-tartaric acid should be added and stirring continued until a clear solution is obtained. Next ether is slowly added with strong stirring just as in Chapter 4. Addition is continued until the milky appearance caused by the addition of ether doesn't dispel with continued stirring. It may take roughly 10 volumes of ether for each volume of solution if the similar recrystallization of meth from such solutions can be taken as a guide.

The solution is allowed to sit overnight in the fridge as in the earlier example, and the crystals filtered out and rinsed and dried as before. The iso-LSD is still in the mother liquor as it refuses to form crystals and come out of solution. If one would evaporate off the solvent from the mother liquor to leave a residue of iso-LSD, one could then use either method One or Two as given in Chapter 4 to convert this iso-LSD to the active form. There is excess tartaric acid in the mother liquor, so the first portions of KOH added to the mother liquor will be consumed neutralizing the tartaric acid. This is no problem, just use a bit more KOH to compensate. The total yield from this example batch should be close to 15 grams of LSD. This example batch can be scaled up or down depending upon how much lysergic acid one has.

## 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. A pump will be required if an aspirator of this high quality is unavailable. Ditto for the cold running water needed to make it work.

Now cool the formamide solution, and when it has cooled to 5 °C, add 100 ml of 1M $SO_3$-formamide 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.

# Seven: 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 byproducts 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 iso-LSD can be pretty easily converted to the active form LSD. 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 $SO^3$, 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 $SO_3$ 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 -20 °C.

Next, remove the flask from the freezer, and nestle it in an ice-salt bath. Now with stirring add a solution of 26.5 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 chloroform extracts are next combined with the original chloroform layer. This combined chloroform solution is next washed a couple of times with 100 ml portions of cold water to remove amine salts. 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 and other by products. The "other by-products" are the result of non-specific acylation as reported by Garbrecht in the Journal of Organic Chemistry Volume 24, pages 368-

372 (1959). These are separated by chromatography as described in Chapter 4, and the iso-LSD converted to LSD as also described in that chapter. The tartrate salt of LSD is then formed as described earlier. Read US Patent 2,736,728 for more information on this reaction route.

The water layer from the extractions contains about 5 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 72 °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).

# Eight: 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-

dimethylaminobenzaldehyde 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 for choosing this method is if a cylinder of phosgene gas is very easily available at work or school. A well equipped lab with an experienced operator can handle the smaller amounts of phosgene used to make batches of LSD.

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 prepared by taking 200 ml of dimethylformamide and slowly bubbling into the dimethylformamide some phosgene from a cylinder until the solution gains 34 grams weight. Strong stirring and cooling during the bubbling helps to ensure that most of the phosgene goes into solution and not the surrounding air. Another method is to cool said cylinder of phosgene down in a freezer until the phosgene liquifies (b.p.8 °C) and then invert the cylinder and crack open the valve to drip some liquid phosgene into the dimethylformamide. Both options are pretty dangerous without strong ventilation and protective gear such as gas masks. 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 7 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 or by precipitation of the LSD tartrate salt 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. The water and one molar NaOH solution washes should contain unreacted lysergic acid which can be recovered by the methods in the earlier chapters ,i.e. acidify to pH 3 and recover.

# Nine: Method X

In the late 1970's, 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.

The evidence introduced at trial that I have been able to uncover is similarly unhelpful. The English police stated that the chef was supplied with 7.5 kilos of ergotamine tartrate, and from that he produced 1.7 kilos of acid. This yield is unremarkable. Let's say for example that some route going through lysergic acid was used by this master acid chef. Ergotamine tartrate has a molecular weight roughly twice that of lysergic acid, so if the racemization free hydrolysis method cited earlier in this book had been used, the amount of lysergic

acid obtained would be roughly 3 kilos. That could easily convert to about 2.5 kilos of LSD tartrate.

Similarly, if the Hoffman hydrazide One Pot Shot method was used, one would expect yields higher than stated at trial. There are a couple of explainations for this somewhat disappointing yield. Point number one is that not very batch turns out as one would hope. This is especially true when one is just beginning doing a certain reaction and getting to know it's quirks. The next point is that this cook produced the most pure LSD in the world. He may have trashed some material that didn't meet his standards.

All of this still leaves us wondering what new procedure the Operation Julie acid chemist came up with that so wowed the world. Once he was eliminated from production, the worldwide price of acid roughly doubled, quality took a tumble, and those very generous doses he provided on a blotter were a thing of the past.

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.

So what new method did the victim of the Operation Julie bust apply to his acid production operation? One possible explaination can be found in US Patent 3,084,164 issued in 1963. It never mentions LSD, so a cursory lit search for acid recipes would never turn it up. This Patent details how to produce lysergic acid chloride. The acid chloride is an easy and high yielding general route to produce amides just by reacting this acid chloride with diethylamine, but it had previously been thought to be impossible to produce the acid chloride of lysergic acid. For example, see the *Journal of Organic Chemistry* article by Garbrecht which has been cited several times in this book. He states in his article that attempts to produce the acid halide of lysergic acid by standard procedures just makes the lysergic acid molecule fall apart.

The Patent states that by using both phosphorous oxychloride (POCl3) in combination with phosphorous pentoxide (PCl5), he was able to produce the acid chloride of lysergic acid. He took one half gram of lysergic acid and ground it finely. He doesn't specify in his Patent that this lysergic acid should be the anhydrous and baked under a vacuum form of lysergic acid rather than the dihydrate form, but I'll bet it is. Then to this lysergic acid in a round bottom flask with stirring he added 10 ml of phosphorous oxychloride and .4 grams of phosphorous pentachloride. Then he shook the reaction mixture for a couple of minutes, and then heated it to 90 C for two minutes, presumably while still shaking or stirring...then let it cool..

He evaporated off the phosphorous oxychloride using vacuum from an aspirator to flush the fumes down the drain. A good aspitirator will pull a vacuum under 50 torr if it has cold water flowing through it, so he hardly had to heat the mixture at all to rid himself of that reactant. That left him with the unreacted phosphorous pentachloride and the product which is the lysergic acid halide. To recover that he dissolved the residue from the vacuum evaporation in hot hexane (think naptha!) as the acid chloride dissolved in that and left the phosphorous pentoxide behind. After filtering to rid himself of the PCl5, he could then evaporate down the the hexane and cool it to yield a grey colored powder of lysergic acid chloride which he filtered out.

## Lysergic acid chloride

The reaction to produce LSD from the acid chloride is then a very simple one. A very standard and time tested procedure would be to mix about 1.5 ml of diethylamine into 50 ml of chloroform for this one half gram example recipe. Then slowly and with stirring the approximately one half gram of lysergic acid halide obtained from the preceeding recipe should be added in small portions to the diethylamine in chloroform. It would be best if the Lysergic acid halide was in solution when it is added to the reaction mixture. We know it dissolves into naptha or hexane, so that is one solvent choice. It probably dissolves into chloroform as well. Once all the lysergic acid

halide has been added, warm the mixture to boiling under reflux and in the dark for half an hour...or at least wrap the flask in foil during the whole procedure. Then cool and pour the batch into 50 ml of one molar ammonia solution. This solution is made by adding one part of strong ammonia (30% $NH_3$ , 56% $NH_4OH$) to ten parts water, but the amounts aren't critical, just be close so the HCl produced from the reaction gets neutralized and one still has an alkaline solution.

Shake the mixture in a sep funnel, and drain off the chloroform layer. It will be on the bottom and will also be the layer which contains the LSD. Then extract the water layer once with a bit of chloroform to get any LSD which may have escaped the first extraction and add this extract to the main charge of chloroform-LSD solution.

To then isolate and recover the LSD product, the chloroform solution is evaporated away under a vacuum. The residue left is LSD, and the Patent holder claims that very little iso-LSD is made during the process. This residue can then be converted to the crystalline tartrate salt by the methods covered earlier in this book. They are either directly dissolving the residue in some warm methanol and adding tartaric acid, or first chromatographing the residue, and then going on to the tartaric acid crystallization.

This method is a very strong contender for being the mystery process used by the Operation Julie acid chemist. It would have been considered "outside the box" by almost everyone at the time, yet it was published well before he set up his operation so he could well have known about it.

Another possible new route used by this acid chef can be found in Chem Abstracts Volume 69, entry 106934 (1968) and Czech Patent 125,498. The Czechs have done a lot of work in the field of lysergic chemistry, and it is quite understandable because at one time they produced a lot of ergot.

This method uses an entirely novel route to LSD. It is mentioned in this reference and never again. One general method of producing amides, such as LSD, from organic acids such as lysergic acid is to simply heat the organic acid and an amine inside a sealed tube. In this case the reaction would be between lysergic acid and diethylamine to give LSD, and they claim yields of 90 to 100%! There is one caveat

on this procedure. They used the hydrogenated form of lysergic acid in which the double bond at the 9-10 position has been reduced. Medicines made with this bond hydrogenated are less likely to cause a puking stomach upset in the person taking them, and a lot of lysergic acid chem is to be found under this "9-10 dihydro" subset. It may be that this hydrogenated form of lysergic acid tolerates the very high temperatures used in the reaction better than natural lysergic acid. There are some very high temps used in this process, but they are below the melting point of lysergic acid. The natural acid decomposes at its melting point of roughly 240 °C, and this process gets close to that temperature.

In their Patent, the inventors took some threaded stainless steel pipe and securely attached a stainless steel cap onto the bottom of the pipe. They put an excess of amine into the pipe along with a couple volumes of acetone, then added the lysergic acid. They bubbled nitrogen down into the solution in the pipe so that all oxygen would be flushed out, then they screwed on the top cap of the pipe.

They next proceeded to heat this mixture at 195 to 200 °C for a prolonged period of time. For benzyl amine they heated for 8 hours, and for cyclohexylamine they heated for 60 hours. They never tried diethylamine in the experimental results published in Chem Abstracts, but I would think that 24 to 60 hours should be enough for diethylamine. It's not as reactive as benzylamine, but it should be pretty similar to cyclohexylamine. The temperatures used are below the critical temperature of diethylamine, so if the natural lysergic acid holds up to the heat, this method would be the winner of the simplest, best, and highest yielding recipe contest. There is no mention in the Patent as to whether the lysergic acid should be the baked dry form. There is water formed as the result of the reaction anyway, so it seems that small amounts of water carried in from the lysergic acid would be OK.

To isolate the product, simply cool the tube and evporate off the excess diethylamine under a vacuum. Take up the residue in chloroform, and wash it with dilute ammonia or bicarb water. Then the chloroform extract can be evaporated down and either chromatographed or directly crystallized as the tartrate.

If this was the method used in that famous acid lab, he certainly was a pioneer. A more likely choice for the mystery method used can be found in the Journal of Medicinal Chemistry, Volume 16, pages 532-537 (1973). This published paper fits easily into the timeframe in question, and it also devulges what is generally considered to be the best acid recipe of them all. This recipe easily gives 70% yield from lysergic acid on a consistent basis, and produces very little iso-LSD by-product. It has the further advantage of using the hydrate form of lysergic acid, so there is no need to go about attempting to remove the water of crystallization from the lysergic acid by means of heat and high vacuum.

A stirred slurry of three grams of lysergic acid (as the hydrate) and 7 grams of diethylamine in 150 ml of chloroform was brought to reflux inside a 500 ml round bottom flask equipped with a condenser. Once the mixture has been brought to a boil, the external heating is removed, and 3.3 gr of POCl3 (phosphorous oxychloride) was added over the course of two minutes at such a rate to keep the mixture boiling. The heating is then recommenced to keep the mixture boiling for an additional five minutes. Everything should now be in solution rather than being a slurry, and the batch should have an amber color.

The batch is allowed now to cool to room temperature. As with all LSD recipes, the reaction should be protected from exposure to light. Once the batch has cooled down, it should be poured into 200 ml of an ammonia solution made by diluting one part of strong ammonia with 11 parts of water. This is 1 N ammonia solution. Shake the mixture in a sep funnel, then let the layers separate. The chloroform layer will be on the bottom of the sep funnel, and it has the product. Drain it off and dry the solution with a little bit of anhydrous magnesium sulfate. Filter to remove the magnesium sulfate.

Now the chloroform is removed under a vacuum. The paper specifies that he heated the mixture no hotter than 40 C to help with the vacuum evaporation, and that he used a strong vacuum of two to five torr to remove the last bits of solvent.

The residue left in the flask after evporating away the solvent should be a very thick liquid. It is fairly pure LSD free base. To get pure LSD from it, one could dissolve it in solvent and chromatograph it, then

evaporate away the solvent and proceed with crystallization as the tartrate or maleate salt as usual. The authors of the paper simply proceeded directly to the crystallization. They dissolved the residue in the minimum amount of warm methanol, and then acidified the solution by adding a freshly prepared 20% solution of malic acid in methanol to the LSD free base with stirring. They state that they got instant crystallization of the product which they filtered out. They then rinsed with a bit of cold methanol and air dried the product.

Note that this procedure for crystallization is a bit different from the ones given earlier in this book using tartaric acid. The authors claim instant crystallization without the need for dilution with ether or for letting the crystals grow overnight in a freezer. I have no idea which procedure is better, or which one gives the more pure product in highest yields. It could well be that they both work just fine and it is more a matter of taste than anything else.

That recipe is considered to be the pinnacle of acid synthesis. It is beloved for its simplicity, its quickness and its high yields. There have been some other ones developed since the mid 70's, but none of them are reasonable candidates to be the method used by the Operation Julie chef since they fall outside the timeline in which he was brewing acid.

For example, one might want to check out US Patent 4,035,501. It has the disadvantage of requiring the anhydrous form of lysergic acid and it is also very sensitive to other sources of moisture entering the reaction system. From what I can gather reading the Patent, this method is very similar to the phosgene procedure given in Chapter Eight. The big improvement is that the phosgene has been replaced with oxalyl chloride. This substance is far less likely to kill you through accidental inhalation than phosgene.

In a three necked flask under a nitrogen atmosphere cool a mixture of 75 ml of absolute (water free) dimethyl formamide and 150 ml of absolute acetonitrile to minus 30 C. Then with stirring slowly add a solution of 4.28 ml of oxalyl in 30 ml of absolute acetonitrile dropwise to this solution. The oxalyl chloride solution should similarly be chilled to around minus 30 C before it is added to the reaction mixture. Allow this mixture to react for five minutes.

Now the lysergic acid is added by slowly sprinkling 14.1 grams of anhydrous lysergic acid over the surface of the solution with stirring and while maintaining the cold temperature. This grey green suspension is then stirred for half an hour while keeping the temperature below minus 10 °C.

The reaction mixture is subsequently cooled back down to minus 30 °C, and 25 ml of cold absolute pyridine is added, and then immediately a cold mixture of 10 ml of diethylamine in 100 ml of dimethylformamide is added. It is crucial that all moisture be kept out of the reaction system. Stir at zero degrees for two hours.

Now the reaction mixture can be worked up and the product recovered. Pour the reaction mixture into 500 ml of 10% sodium hydroxide solution. Shake it to get all the acid neutralized, then extract with methylene chloride. This solvent will contain the product. Wash it with water, then dry the methylene chloride solution with some sodium sulfate. After filtering that off, the methylene chloride is evaporated away under a vacuum using no source of heat hotter than 50 °C. The residue of oily LSD free base and other materials can be purified as usual by chromatography and crystallization as the tartrate or maleate salt.

There have been other methods developed over the years which offer new ways to make LSD, but none of them are improvements over the methods already presented in this book. For example, if one would check out Chem Abstracts Volume 90, entry 168,959 there can be found detailed references to the carbodiimide route to making acid. Just because it is new and different doesn't make it better. Similarly, there have been new refinements in the old azide route to the lysergic acid amides. Even with these improvements, I consider these methods less useful than the ones presented in detail thus far in this book.

# Ten:
# 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 lysergicsimilar 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 (FeSO4) 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.

# Eleven: 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 fivefinger-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 oneman 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 12% 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. Take heart in the fact that just a small amount of chemical feed goes a long way in the LSD field. One gram of acid produces thousands of trips.

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:

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

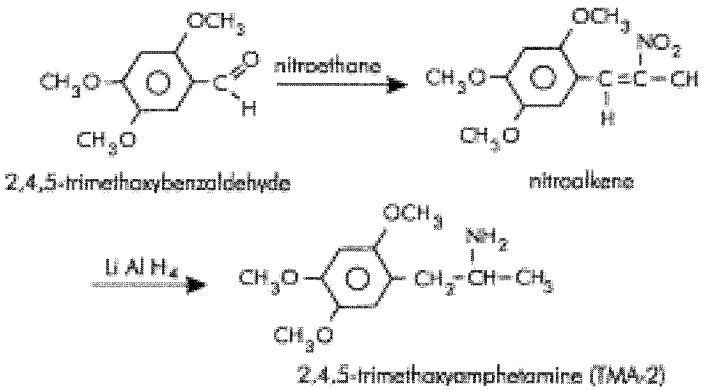
$$= \frac{1.88 \text{ gr LSD per liter}}{\text{liter of solution}}$$

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.

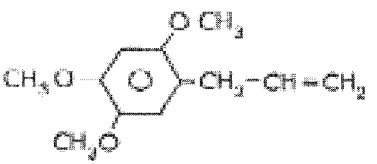
# Twelve: Studies On The Production Of TMA-2

My interst in this subject was stimulated after reading Dr. Shulgin's description of the effects of this wonderful substance in his book PIKAL. The good doctor describes it as being just like mescaline, without mescaline's color intensification. Oh well, you can't have everything!

The route Dr. Sgulgin took for the production of TMA-2 was to condense 2,4,5-trimethoxy-Benzaldehyde with nitroethane in a Knoevenagel reaction to yield the nitroalkene, which he then reduced with lithium aluminum hydride to yield 2,4,5-trimethoxyamphetamine.



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 LiAlH4 reduction. This too will be duly noted by the heat, especially in combination with buying LiAlH4.



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 less than satisfying.

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 article I will cite is Organic Syntheses , Volume 62 and a recipe titled "A General Method for the Preparation of Methyl Ketones from Terminal Olefins : 2-decanone." This article reveals, 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 oxygen or hydrogen peroxide in dimethylformamide or alcohol 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 chloride, or a mixture of palladium chloride and copper chloride, or palladium acetate. Of the three, the palladium acetate catalyst is best because it uses so little of that expensive metal. It does however require the use of t-butyl alcohol as solvent. In Secrets of Methamphetamine Manufacture 7th ed I give several procedures for using this Wacker oxidation, and I also detail how to make palladium acetate catalyst from a commonly available ingot of palladium metal. Here is a recipe using palladium acetate:

In a 2000 ml beaker place one liter of t-butyl alcohol and about one mole of an allyl benzene such as safrole. That would be roughly 160 ml of safrole obtained from sassafras oil in this instance. The

mixture is heated to 80 °C with stirring, and then a gram and a half of palladium acetate catalyst is added to the beaker. Once the catalyst is in the beaker, about 500 ml of 30 to 35% strength hydrogen peroxide is dripped in with stirring over the course of half an hour. The temperature of the reaction is maintained at 80 °C for about 6 hours and an orange colored solution should result.

After the six hour reaction period, allow the mixture to cool. Then pour it into 5000 ml of water. Stir it around, then let it sit for a while. The product..in this case methylenedioxyphenylacetone...will float to the top. Separate it off, and extract the t-butyl alcohol and water solution left with a couple portions of toluene to get the remaining product out. Add these extracts to the methylenedioxyphenylacetone originally separated off. Now wash this toluene solution of methylenedioxyphenylacetone a couple of times with 5% NaOH solution to remove t-butyl alcohol. Finally, distill off the toluene at normal pressure. Then vacuum distill the md-phenylacetone to get 80 to 90% yield of product.

There are other example procedures given in Secrets of Methamphetamine Manufacture for this same Wacker oxidation using dimethylformamide or methyl or ethyl alcohol. They use much more of the palladium catalyst to get the same or lesser results. The listed yield using DMF is more like 50%, so pick and choose one's recipes.

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. Methylenedioxyphenylacetone 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-trimethyloxyphenylacetone 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 carbonate 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 80-90% asarone. This is a propenyl benzene like isosafrole, and is often called beta asarone. Indian calamus oil also contains a few percent of allyl asarone. This substance is often referred to as gamma asarone. It too can be purified by distillation under a vacuum to yield fairly pure 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 *Chem. Abstracts* column 6585 (1935), also *Current Science*, Volume 3, page 552 (1935). Note that in the original articles the researchers thought that the Indian oil was almost pure allyl asarone. A few years later, however, they decided that what they had originally identified as allyl asarone was really the cis-trans isomer of ordinary asarone. The double bond was in the propenyl position. See *J. Chem. Soc.* 1937 page 1338 to 1340.

The general rule is that calamus oil coming from tropical regions will be almost pure asarone. In temperate regions the total asarone content will fall to about 20%, with allyl asarone making up a fair portion of the total asarone. Both asarones have similar boiling points, so no separation by distillation is possible nor desirable. Rather, these temperate oils from regions like Europe or Japan should be isomerized to all beta asarone (propenyl position) by heating with KOH. This is a simple procedure, and I have experience with it converting safrole to isosafrole. Traces of water prevent the conversion. The procedure I used with safrole involved adding some 10% KOH in methanol to safrole, and heating the mixture above the boiling point of safrole but below the boiling point of isosafrole. That way it was easy to tell if you were getting conversion because if you can't reach the higher temperature above the boiling point of safrole, you aren't getting conversion to isosafrole. In the case of calamus, the more general procedure given by Dr. Shulgin in PIKAL would be more appropriate. See the recipe for DMMDA for the general method to convert a mixture of allyl and propenyl asarones into all propenyl position beta asarone. Keep water out of the mix!

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 was in the days before search engines made on line shopping so easy. Now in 2006, the listed price of a liter of Indian calamus oil is $110. What a bargain! This left one alternative: dig up the roots of North American calamus, and steam-distill the oil out of them. It was a great plan, but it had one drawback. There are three varieties of calamus plant. The diploid american type, the triploid European type, and the tetraploid Asian type. The diploid American race of calamus produces next to no asarone of any type. One could however, plant the seeds of the Asian variety in a warm weather area and harvest great quantities of high grade oil if the supply of calamus oil ever gets shut off like has happened to sassafras oil.

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.)

**Figure 4**
***Calamus plant root and fibrous rootlets.***

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 dugup roots should be rinsed free of dirt, and the tops cut off there in the field. (See Figure 5)

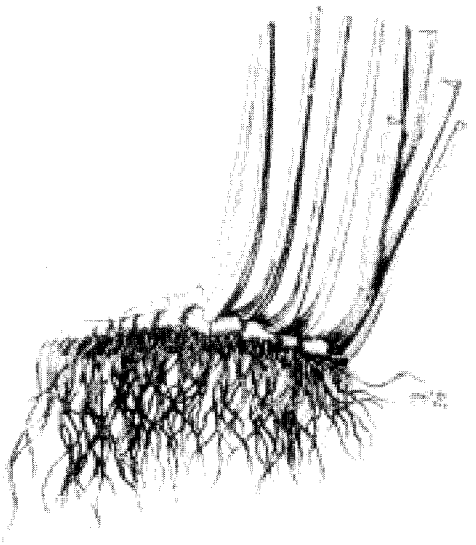


**Figure 5**
***Calamus root and fibrous rootlets with tops trimmed off.***

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! 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 Calamus. 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.

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

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.

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, so long as one isn't comsuming it regularly. 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. 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.

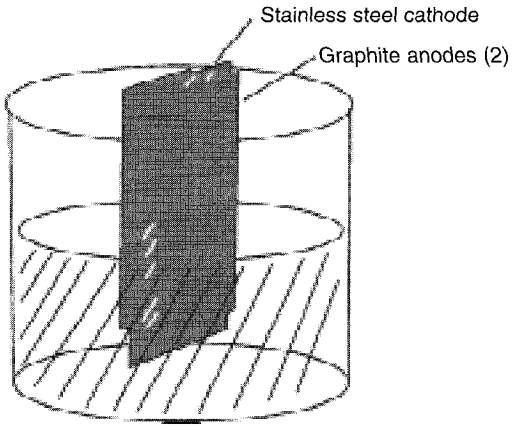



**Figure 6**
***Electrochemical cell used to convert a propenylbenzene to thecorresponding phenylacetone.***

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 onefoot 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.

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 °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:

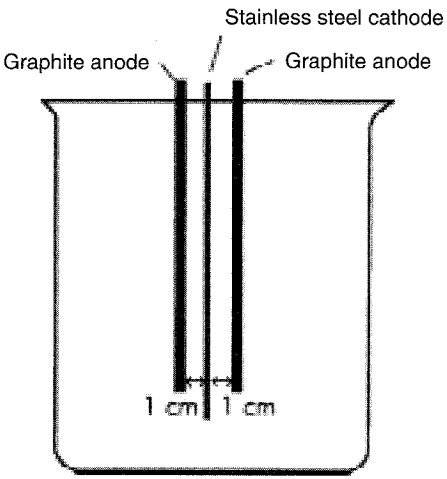



**Figure 7**
***Side view of electrochemical cell.***

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 one half 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(III) 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.

A much better hydrogenation mixture is revealed in US Patent 3,187,047. In this Patent, the inventor substitutes ammonium acetate for ammonia and gets really impressive results in the reductive amination of substituted phenylacetones. He used the following mixture: 3 kg of dimethoxyphenylacetone, 1.2 kg of ammonium acetate, 180 ml of acetic acid, 9.5 liters of methanol, 300 ml of water and 500 gr of Raney nickel. He got a 95% yield of dimethoxyamphetamine!

Raney nickel isn't convenient or even practical for most clandestine chemists to use. The shaking and heated hydrogenation device is a big home construction project and Raney nickel isn't easily made or purchased.

These problems with the usage of Raney nickel can be avoided if one substitutes a type of catalyst designated P-1. It's non-magnetic, and so magnetic stirring can be used rather than shaking. This allows one to put to use a suggestion I got from a reader who calls himself "The Iceworm" for what he calls his "poor man's hydrogenation device". The reader uses an old fire extinguisher as his hydrogenation bottle. He makes sure the bottle is made of aluminum or stainless steel by checking it with a magnet. If the

magnet doesn't stick to it, it is stainless or aluminum. Then he empties it out, screws off the cap, cleans it out and tosses in a magnetic stir bar to check to see if it will spin properly when placed on his magnetic stirrer, then he is ready to go. The ingredients are loaded into the extinguisher, the cap is screwed back on, and the air is sucked out of the extingisher and replaced with hydrogen under a pressure of at least 100 pounds using the spray nozzle as hydrogen inlet. The pressure guage is standard equipment with the fire extinguisher, allowing hydrogen uptake to be watched and more hydrogen introduced as needed. Simultaneous heating and stirring is no problem with this set-up.

For production of P-1 nickel catalyst see *Journal of Organic Chem* Vol. 35, page 1900 (1970). This recipe can be scaled up or down as needed. Dissolve 5 milimole of nickel acetate, chloride or sulfate in in 50 ml of water and pour it into a reaction vessel flushed with hydrogen gas. Then with high speed stirring add 10 ml of one molar sodium borohydride in one tenth molar NaOH solution over 30 to 45 seconds. When the fizzing stops, another 5 ml of this solution is added. Then when the fizzing stops again, the stirring is halted and the precipitated nickel catalyst is allowed to settle. The liquid is then decanted off, and the precipitate washed twice with alcohol. Then the catalyst is ready to use.

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, Seventh Edition: .5 gram platinum in 20 ml distilled water. If this platinum is in the form of PtO2 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 $NH_4Cl$, 500 ml methyl alcohol saturated with ammonia gas, and 50 ml $NH_4OH$. The bottle is then set up as seen in Figure 32 in Secrets of Methamphetamine Manufacture, Seventh 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 $NH_4Cl$ 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 160 °C under aspirator vacuum. The yield is about 50 ml.

Another 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 highgrade 99% material will work. In the case of methylenedioxyphenylacetone, 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, Seventh Edition, until a temperature of 150 °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 HCl, 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.

## *Dear Uncle Fester:*

Thanks for the information. The magic in the reaction is to put the phenylacetone and the formamide together a couple days before doing the reaction. One part phenylacetone and two parts formamide must stand at room temperature at least 24 hours or more. I think one week is best. It should be shaken twice a day or more. I believe the formamide is slowly reacting with the phenylacetone, so the longer it stands, the better!

Maybe if the temperature is higher, it reacts faster. Some people leave it for only one day, but if you have time, why not let it stand for one or two weeks?

Just before I got arrested, I was measuring the pH of the mixture and I believe it changes every day. With a little patience, you should be able to figure out the best length of time to let it stand.

WARNING: NEVER put the formic acid with the mixture. It will ruin everything and you will have to start all over.

So now you have waited a week, and you're ready to go for it. Put your mixture into a glass flask along with boiling chips. You should use as many as possible, because you need small bubbles. This is very important!! With a condenser on top for refluxing, the water must not be very cold because the ammonia will block the hole in the condenser, so 30 °C is a good temperature for it. You can even let all the water out if you don't mind the smell of ammonia.

Just before you start, you take a little bit of formic acid and add it to the mixture. You only need a little bit, for instance with a 3 liter mixture +5 cc. More won't do any harm. If you put too much, just heat the flask without the condenser until the temperature has gone up to 160-165 °C, then put the condenser back on.

Now you raise the temperature to 180 °C, and sometimes a bit higher, depending upon the quality of your formamide. Here they use industrial grade. I've never had any problems, as long as it is clean.

Let the mixture reflux for one hour after it has reached the desired temperature. The mixture will change from light yellow to dark yellow. If it starts to darken, your temperature is too high.

After one hour, let it cool down, or when it is a small batch, take a sep funnel filled with water and mix in your batch. Don't use any lye at this stage.

Now take out the oil and mix it with twice the amount of hydrochloric acid. Let it reflux for one hour.

Now you have to separate this with lye. The best thing is to let it cool down first, but if you have a small batch in a big sep funnel, you can take the risk. Also, very good shaking is needed here.

Take the oil layer and start to distill it under maximum vacuum.

Take the distilled product and mix it with some toluene and bubble dry HCl gas through it and filter.

Big batches take a long time to dry, so people here put it in a centrifuge. Take a bedsheet, but you batch in there and let it spin. Be carful with sparks and such!

Instead of formamide, you can also use ammonium formate. You don't need formic acid here.

If you have any questions, just write me and ask. Keep up the good work and have a good time.

*Geert Hendricks, Eindhoven, Holland*

Ps...this reaction also works with methylenedioxyphenylacetone

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, Seventh Edition for a full description.

## Alternative Route #I

Applicable to all allylbenzenes and illustrated for MDA

A synthetic route suitable to the clandestine manufacture of MDA or MDMA (Ecstasy) is the reaction of sassafras oil (which contains 80-90% safrole) with hydrobromic acid to yield bromosafrole, and then the consequent reaction of this bromosafrole with either ammonia or methylamine to yield MDA or MDMA respectively.

A Serious problem with the use of this route is the very poor yields one obtains when using 48% HBr. This is the standard ACS reagent, and is most easily obtained without arousing suspicion, yet it is too dilute and hydrated to add well to the double bond of the safrole. Standard methods to dehydrate 48% hydrobromic acid fail. Adding sulfuric acid to 48% HBr breaks it down to bromine. Distillation also fails to dehydrate it, because the 48% acid is a constant boiling mixture. I have solved this problem by using dry HC1 gas to dehydrate 48% HBr, and it is the purpose of this paper to demonstrate the proper use of this procedure.

The reader should refer to Ch*em Abstracts* from 1961, column 14350, and *Journal of the American Chemical Society,* Volume 68, pages 1009 to 1011 (1946) for background on this synthetic route. Into a suitable size Erlenmeyer flask or volumetric flask place a magnetic stirring bar and one volume of sassafras oil with one volume of glacial acetic acid and two volumes of 48% HBr. Chill this mixture down in an ice bath, then, with strong stirring, pass dry HC1 gas into the reaction mixture slowly over a period of an hour or two. For a batch using 50ml sassafras oil, the amount of dry HC1 gas produced by dripping 75-100 ml of sulfuric acid onto a 500 ml flask half full of salt dampened with concentrated hydrochloric acid is about right. The reaction mixture first turns green then blue, then purple, and finally burgundy. The temperature shouldn't be allowed to go above 10-15° C, and periodic fresh ice will be required, as the dehydration of the acid by HC1 generates a fair amount of heat. After the bubbling of the HC1 is over, stopper the reaction flask and continue to stir in the cold for about a day. About half a day into the stir, it should be notice that when the stirring is stopped, a homogenous mixture is maintained, i.e. an organic layer doesn't float on top of the acid.

The amount of dry HC1 produced by dripping sulfuric acid onto salt will vary with the exact conditions, so the batch should be checked for reaction before quenching it on ice. It doesn't hurt to add too much dry HC1, within limits, but too little won't dehydrate the acid sufficiently. To check this, after the day of stirring is done, pour some of the reaction mixture into a beaker. Then, from the beaker, return it to the reaction vessel. This leaves a coating of the reaction mixture on the glass in the beaker. Fill the beaker with

water to rinse away the fuming acids, empty it, and sniff inside the beaker for the aroma of organics clinging to the glass. If it still smells like the candy ship fragrance of sassafras oil, an additional bubbling with dry HC1 is going to be required, followed by another day of stirring in the cold. After the first batch or two, it's easy to gauge how much dry HC1 one is getting. If the aroma has changed to something more chemical and fruity, (yes, just like phenylacetone), sufficient HC1 has been added.

Now pour the reaction mixture onto crushed ice. For the batch mentioned above using 50 ml of sassafras oil, about 300 grams of crushed ice is enough. Wait for the ice to melt, and then transfer to a sep funnel. Add a portion of toluene about equal to the amount of sassafras oil used, and shake the mixture. The product organic layer should be floating on top of the aqueous layer, and it should still be colored burgundy. Drain off the aqueous layer then wash the organic with a few hundred ml of water for the batch size mentioned above. Follow this with the addition of small portions of bicarb, and swirl around the mixture until the addition of more bicarb doesn't produce fizzing. Then all the cared-over acid will be neutralized. Drain off the water layer and keep the toluene and bromosafrole mixture.

Now the mixture should be distilled. The toluene should be mostly distilled at atmospheric pressure to dry the solution azeotropically. When most of the toluene has distilled off, vacuum distillation may be commenced. An oil bath is preferred to heat the flask, as direct heat generally causes bumping with this substance. A vacuum, which distills safrole at about 110°C, will distill bromosafrole at about 140-145°C. Some chlorosafrole will come over at about 125°C. The Yield is about 66-75% conversion to bromosafrole, with the remainder being unconverted safrole, and some chlorosafrole. It smells like phenylacetone, and may turn pink on standing. It's recommended to store it in the freezer.

There are other ways to dehydrate the 48% hydrobromic acid. For example, slow addition of phosphorus pentoxide to stirred and chilled HBr will dehydrate it. This method would create far less fumes, but phosphorus pentoxide is more difficult to obtain than sulfuric acid and salt.

With bromosafrole obtained, one can convert it to MDA or MDMA. The first thing one needs is a 20% solution of either ammonia or methylamine in alcohol. The best way to get this is to purchase 91% isopropyl alcohol at the drug store. I prefer this because isopropyl alcohol is less likely to make the ether byproduct mentioned in the *JACS* article. 91% strength is best, because the presence of water in the reaction mixture is harmful, although some can be tolerated. Now cool this alcohol down in the freezer. A cylinder of anhydrous ammonia should next be obtained at the welding-supply shop. Then pour the cold alcohol into a beaker or other container. Next, tip the cylinder upside down and crack open the valve a bit to dribble out ammonia into the alcohol until its volume has increased 20%. Good ventilation is needed for this operation, and one should stay upwind. This solution should be immediately transferred to a champagne bottle with plastic stopper for storage.

40% methylamine and 56% ammonium hydroxide can also be used. In these cases, one puts the fuming liquid into a round bottom flask, attaches a condenser at least 1.5 feet long, and while running ice-cold water through the condenser, heats the methylamine or ammonium hydroxide. The dried and cooled vapors which come out the top of the condenser should then be piped into the ice-cold isopropyl alcohol, with strong stirring to catch the vapors. Continue bubbling until the volume increases 20% and store in the champagne bottle. Be aware that methylamine is on the reporting list and should never be purchased. For synthesis, see *Organic Syntheses*. You'll find methylamine hydrochloride in the table of contents.

The next thing one needs is a section of steel pipe, threaded at both ends, with a screw-on cap for each end. This is the pressure reaction vessel. Steel pipes are generally heavily galvanized with zinc. Zinc reacts with ammonia to form a crystalline mass inside the pipe, and generally messes up the reaction. The zinc on both the pipe and end caps must be stripped off by immersion in a 5% hydrochloric acid solution until the fizzing slows to a crawl.

Once the zinc is off the steel, rinse the parts thoroughly in clean water. Then screw the cap onto the bottom of the section of pipe. A Pipe wrench will be needed to get the cap on tight enough to prevent leaking when it's heated.

Now, almost fill up the pipe with a mixture of one part bromosafrole with eight to nine parts of the 20% alcoholic ammonia or methylamine solution. Then screw the top cap onto the pipe, and tighten it down with a pipe wrench, too.

The pipe reaction vessel must now be heated to about 130 C for about 3 hours. The best and safest way to do this is to put the pipe inside a pressure cooker, and enough water for a three-or four-hour boil, and boil the pressure cooker at 15 pounds pressure for three to four hours. At 15 pounds pressure, the temperature inside the cooker will be 120 C to 125 C. This is close enough to the prescribed temperature, with somewhat longer heating making up for the few degrees it's short of 130 C.

Once the pipe has cooled off after the cooking, it should be put in a vise, and the top screwed off with a pipe wrench. The contents should then be poured into a beaker. The color of the reaction mixture should be a semi-transparent brownish yellow. It contains MDA or MDMA in alcohol, along with unreacted ammonia or methylamine and the gunky by-products of the reaction.

Heat the beaker to just about boiling. The ammonia or methylamine will quickly evaporate away, along with some alcohol. When the volume of the liquid in the beaker has been reduced by one third to one half, all of the ammonia or methylamine will be evaporated away. This can be confirmed by sniffing the vapors. At this point, add an amount of concentrated hydrochloric acid roughly equal to the amount of bromosafrole used in the batch. Mix it in, and continue the gentle heating of the contents of the beaker to evaporate the alcohol. One wants to reduce the volume of the solution to at least one-fourth its original amount. One must also be careful to avoid burning the batch. This isn't a concern when using a vacuum.

When the alcohol has been mostly evaporated away, one should pour the batch into a sep funnel, and rinse the beaker with 5% HC1 solution, pouring the rinse also into the sep funnel. The total amount of the rinse with 5% HC1 solution should be about double the volume of the batch that went into the pipe. For example if the bromosafrole-alcoholic ammonia mixture in the pipe occupied a volume of 200m; then about 400 ml of 5% HC1 should be used.

Now shake the contents of the sep funnel strongly for a few minutes. This puts the MDA or MDMA into water solution in the sep funnel as the hydrochloride salt. After the shaking is done, extract the acid solution with three portions of toluene. This removes most of the by-products. The amount of toluene used isn't critical, but for 400 ml of acid, extraction with 100-ml portions of toluene is more than sufficient. Throw away the toluene extracts, keep the acid solution.

Next the acid solution must be free based. To do this, add lye or NaOH pellets, slowly and with stirring. This makes a lot of heat, so take your time. Continue adding lye or NaOH pellets until pH paper says that the solution is strongly basic. Then shake the solution in the sep funnel strongly for a couple of minutes to ensure complete reaction of all the hydrochloride to free base.

The free bases of MDA or MDMA should now be extracted from the water with a couple of portions of toluene. For the abovecited-size batch, two 75-ml portions of toluene are plenty. Pour the toluene extracts into a beaker, and be sure they are free of entrained water. The color of the extracts should be pale yellow. It is best to distill this toluene extract to get pure MDA or MDMA free base. A vacuum that distills safrole at 110 C will distill about 10 volumes of toluene. Bubbling dry HCI through this solution yields nice white crystals of MDA or MDMA. Filter them out, and spread them out to dry.

With smaller batches, distillation may not be practical. In that case, the toluene extract may be bubbled with dry HC1. This yields off white- tending to tan-colored crystals of MDA or MDMA.

They can be cleaned up after filtration and drying by adding a little chloroform to the crystals to make a slush, then filtering. This leaves nice white crystals. The yield is about 3 grams of MDA or MDMA for each 7-ml of bromosafrole used.

## Alternative Route #2

Applicable to all propenylbenzenes except isosafrole

Note: the formation of the pseudonitrosite derivative is a good way to judge the amount of a- and B-asarone in the product fraction of distilled calamus oil.

My many thanks to a correspondent for making me aware of this alternative procedure for making TMA-2, and my further thanks to Wolf Eichberger for his translation from the German. This is an old recipe, directly written towards to object of making TMA-2 with simple chemicals and equipment. It also involves the use of an electric cell, but I don't find that at all inhibiting. Perhaps my employment in the electroplating field skews my perspective, but passing current through a solution is a simple act.

I must further comment on the old German scientific literature. They always add pertinent cooking detail often lacking in other countries' journals. On the other hand, their patents are vague at best. Enjoy the details translated to the English correctly for the first time, to the best of my knowledge. *Psychedelic Chemistry* makes a stab at this procedure, but leaves out half of it. See page 81 of that book for comparison. This procedure is taken from *J. Pract. Chem,* Vol. 138, Page 271 (1933). See also *J. Chem. Soc.* (1937) p. 1338. Relevant to other propenylbenzenes as well is *Gazz. Chim. Ital.* (1932) Vol. 62 p. 370, and *Liebig's Ann.* Vol. 332, p. 331 (1904). Note that a correspondent informs me that the second stage of the reaction, that with KOH, doesn't produce product with isosafrole. Perhaps it is selectively general. In any case, this production scheme, which is quite well-suited for clandestine manufacture of TMA-2, involves the reaction of the propenylbenzene, asarone, with nitrous acid to make the pseudonitrosite, in high yields.

The formation of the pseudonitrosite seems to be general to all propenylbenzenes. It's the next stage of the reaction, turning the pseudonitrosite to the nitroalkene, where isosafrole bows out. I judge my correspondent to be reliable on this point. This then leaves us with the nitroalkene, which can be reduced by a variety of methods. This seemingly complicated but simple-to-do procedure is illustrated below.

The first step is the reaction of asarone with nitrous acid. Bruckner, in the *J. Pract. Chern* article does it as follows: In a 500-ml flask, place a saturated water solution of sodium nitrite containing 40 grams of sodium nitrite. This means mix 50 ml of water with 40 grams of NaN02. Then pour in 10% ether solution of asarone. This solution is made up by dissolving ten grams of asarone in about 140 ml of ether. It floats as a layer above the nitrite solution. If ether isn't easily available to you, ether starting fluid can be used. First spray some out and watch it evaporate to make sure the brand doesn't contain oil or grease. If it evaporates cleanly, it can be captured by spraying it down and ice-chilled copper tube into an ice-chilled beaker. This isn't a good project for indoors or in densely populated areas. The smell of ether carries, and has gotten more than a few people unwelcome attention. A Grolsh beer bottle with a resealable cap is a good container for ether, as is a champagne bottle with a plastic stopper.

Now with the asarone floating on top of the nitrite, 75-ml of a 20%-by- weight solution of sulfuric acid in water is added slowly from a dropping funnel over the period of 4-5 hours. The article

doesn't mention stirring during this add. Provision also has to be made to keep the ether from evaporating away. This can be done by cooling the mixture in an ice-salt bath. The product precipitates out of solution as crystals, and this process is complete in about 12 hours. Filter the mixture to recover these crystals, and wash them with some water, then alcohol, and finally ether. Drying in a vacuum dissicator yields 80% (in theory) of a yellow powdery product. It decays slowly on exposure to air, so the next step in the reaction should be done as quickly as possible, or the product should be stored in a freezer. Asarone pseudonitrisite doesn't dissolve in the usual organic solvents, with the exception of chloroform. Be aware that nitric oxide fumes are likely to be given off in this reaction. Make provision to pipe them away through plastic tubing, as they are quite poisonous.

The *J. Chern. Soc.* Article uses a somewhat different procedure to make the asarone pseudonitrisite. They use an ice-salt bath to cool sodium nitrite in suspension in ether (5gr. Asarone in 50 ml ether with 20 gr. Sodium Nitrite). They too don't mention stirring, but to keep the solid NaN02 in suspension, some stirring must be involved. Then 30 ml of 20%-by-weight sulfuric acid in water is added dropwise over 5 hours. After standing overnight, the crystals of product are filtered out and washed and dried as in the above example. The claimed yield here is 3 grams from 5 grams of asarone, or a yield of a little over 50%.

The *Essential Oils* reports that the best general method for making pseudonitrosites is to first dissolve the asarone in petroleum ether, hexane, or even mineral spirits, and then add a concentrated aqueous solution of sodium nitrite. Then after cooling the mixture, acid is slowly introduced, and the whole mixture shaken. The batch is then allowed to stand in the cold, and the crystals filtered out later. With the pseudonitrosite thusly obtained, it's time to move on to the next stage of the reaction scheme, that of producing the nitroalkene B-nitro asarone. Put 10 grams of the pseudonitrosite into any suitable flask which can be

stoppered, then add 60 ml of an 8% solution of KOH in ethanol. The most convenient source of 95% ethanol is 190- proof vodka. Check at your liquor store. A stock solution of 8% KOH in ethanol would be make by taking 8 grams of KOH, and adding 95% ethanol until a volume of 115 ml is reached, then shake to dissolve.

The mixture of the pseudonitrosite and the 8% KOH in ethanol is then shaken to dissolve the pseudonitrosite. A little bit of warming may be required to get the dissolution to occur, but never let the mixture go above 30 C. If you live in one of nature's steam baths, the reactants must be cooled. Just shake long enough to get it dissolved. There is a by-product from this reaction, asarone aldehyde, and the warmer the solution gets, the more of it is made. Also, prolonged shaking beyond that needed to dissolve the pseudonitrosite makes more of the substance.

The benzaldehyde by-product is the starting material that Dr. Shulgin uses in his synthesis. If you really want this material, the better way to make it directly from asarone is with chromic acid. See *Die Atherischen Ole* 3rd Edition, Vol. I, page 618. Then nitroethane is condensed with the asarone aldehyde to five the nitroalkene produced in this step. Nitroethane is now on the Chemical Diversion Reporting list, so I would skip that procedure unless the nitroethane is pretty handy.

Once the pseudonitrosite has dissolved in the 8% KOH solution, about 150 grams of crushed ice should be added. Then make the solution acidic by adding 100 ml of dilute hydrochloric acid. This acid is made by cutting the concentrated lab HC I in half by mixing it with water. It's a little over 15% HCI. If you have hardware- store HC1, dilute accordingly to get the proper strength. After the HCI add, mix it around, and nestle the reaction flask in ice, then filter it to recover the nitroalkene product. Wash this product with some fresh water. Any unmelted ice should be allowed to melt as additional wash. Finally, vacuum-dry the product in a dessicator to give a little over 7 grams of crude product. This crude material should be recrystalized from ethanol or methanol to give either a
canary- yellow product or an orange-red product. The color will vary depending upon the concentration of alcohol solution from

which it is precipitated, and the speed of precipitation. Often one gets both color varieties. Both melt at 101 C.

The production of the nitro alkene at this point offers the chance for the clandestine chemist to produce the phenylacetone. The recrystallization could be skipped, and instead the crude nitro alkene could be placed into a flask along with some hydrochloric acid and iron filings and boiled to give the phenylacetone in high yield. Complete directions can be found in chapter nine of my book Secrets of Methamphetamine manufacture.

Now with the nitroalkene obtained, one has choices as to the means of reduction for turning it into TMA-2. Dr. Shulgin uses lithium aluminum hydride in anhydrous THF and ether. If you have these materials in reach, and have done a few reductions with LAH, the directions in *Pihkal* under TMA-2 are recommended. Another possibility is to be found in *Synthetic Communications,* Volume 14 (12), page 1099-1104 (1984), using borane THF with a little sodium borohydride. This isn't practical for most of us either, although one would think that the procedure could be tweaked to use sodium borohydride in alcohol solvent. If anybody makes this work, let me know. Finally, we come to the procedure used in the *J. Pract. Chem.* article cited. It uses an electric cell to reduce the nitroalkene to TMA-2, and claims a good yield. The most important feature which separates it from the electric cell used by Gordon Alles to reduce the nitroalkene to amphetamine given in US Patent 1,879,003 and *JACS* Vol. 54 page 271-4 (1932) is the divided electric cell. The solution containing the nitroalkene doesn't come in contact with the anode. At the anode, oxidation would occur, and make by-products. Commercially produced electric cells with dividers made of porous porcelain are available. See *Reduction in Organic Chemistry* by Hudlicky, page 25, for a general diagram of one. Let your Uncle be your guide here. A small and thin walled earthenware pot for plants with the surface glaze removed is an effective cell divider. Make sure the drain holes in the bottom of the pot are well plugged. The yield in electric reductions is often dependent upon the purity of the starting material, so the recrystallization step shouldn't be skipped.

Now to do the electric reduction, 30 grams of the B-nitro asarone are dissolved in 100-ml ethanol and 100 ml of acetic acid. Then add 50 ml concentrated hydrochloric acid. This is the catholyte. Both cathode and anode are made of lead. If you don't happen to have this metal on hand, a lead sinker can be hammered flat, or it can be obtained from, for example, Kocour Co. (Telephone [312] 847-1111) or Larry King Corp. (Telephone 718-481-8741). Ask for the "lead Hill cell anodes" price- about $9 each. You like to play around with plating chrome, don't ya know? These chunks of pure lead, about 2 inches square and about .25 inch thick, can be beaten easily into thinner material. The actual size of the anode isn't specified, but it should have a surface area almost as big as the cathode, and be as large as will fit into the beaker used as the reaction container. For a batch using 30 grams of nitro alkene, a cathode of 200 square centimeters total surface area is about right. See below. Make sure they can't contact each other to make a short, as the current must flow through the solution. The cathode is cylinder shaped, the metal about 1mm thick, with holes drilled in it to allow passage of current from the solution to the inner surface, and the flow of catholyte. Alternatively, one could just line the inner surface of the beaker with a sheet of lead and use that as the cathode. The anode is a sheet of lead. The anolyte inside the earthen ware pot in contact with the anode is 20% by weight sulfuric acid diluted with water.

Just prior to doing the reduction, the surface of the lead cathode should be coated with a layer of lead oxide. This is done in a separate beaker filled with 5% by weight sulfuric acid solution diluted with water. The cathode for the reaction is immersed in the dilute sulfuric acid and attached to the positive pole of a CD power source such as a car battery. Now into the solution put a negative counter pole, such as for example the metal end of a jumper cable attached to the negative counter pole, such as for example the metal end of a jumper cable attached to the negative pole of the previously mentioned car battery. The solution will fizz as the current flows through it, and the cathode lead sheet will become coated with oxide within a minute or two. Pull it out and rinse with water. This oxide coating must be done before each run of this electric cell. Then it can be placed into the electric reduction vessel and the catholyte and the anolyte poured into their respective places.

The anode is connected to the DC+ pole of a transformer, and the cathode is connected to the DC-pole of the same transformer. A good amp meter should be put in line to measure current, as this is crucial to control. All this electric equipment can be had at Radio Shack.

Chill the reaction mixture in ice, then with vigorous magnetic stirring, apply .03 amps per square centimeter of the cathode immersed in the solution. When measuring this area, measure cylinder surface on the outside and inside of the hollow cylinder cathode, only count the side facing inwards. When making contact with alligator clips to the anode and cathode, be sure that both present a clean metal surface, and dig them in well. Lack of current passage will result from not making a good contact on both poles. Turn up the applied voltage until the desired amount of current is flowing. The meter reading will make this obvious.

Pass current until the initially yellow- or orange- colored solution clears. At a current of 5 or 6 amps, this will take about 12 hours. Keep the solution cooled to 20 C for the first 6 hours, then let it rise towards 40 C during the last 6 hours or so. Maintain magnetic stirring of the catholyte during the reduction. Then take the catholyte, and reduce its volume by about 150 ml under a vacuum. Add roughly 300 ml of water to residue after evaporating off the alcohol, and mix it in well. Then extract out the unreacted nitroalkene with 50 ml of ether. Save these extracts for the next run on the electric cell after the solvent has evaporated away. Make the residue alkaline to + 12 to litmus with NaOH solution, with good shaking to ensure conversion of the salt to the free base, then extract out the TMA-2 free base with ether or toluene. The ether or toluene solution should then be bubbled with dry HCI to yield crystals of TMA-2 hydrochloride. Dry under a vacuum, and store in a freezer. If they are colored or sort of gooey and sloppy, recrystallize by dissolving the HCI salt in the minimum amount of alcohol then add 20+ volumes of ether with shaking. Then filter and dry under a vacuum.

The total yield will be about 80% and the materials are more commonly available to we ordinary folks. One's situation should decide the course of action.

# Appendix

## Know Your Essential Oils

**Sassafras Oil** - contains about 80-90% safrole, and it is now regulated as a List One chemical just like 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%-90% asarone. Oil from other areas contains much less asarone. Boiling point is 296° Cat normal pressure, and 167° C at 12 torr. Yields TMA-2. Dosage is 40 mg.

**Indian Dill Seed Oil** - contains up to 53% dill apiol (3,4-methylenedioxy-5,6-dimethoxy-allylbenzene). 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 Oil** - contains 0-3% safrole, and 0-13% myristicin (3,4-methylene-dioxy-5-methoxy allylbenzene. The boiling point at 15 torr is 150 °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 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.

## Listed Precursor Chemicals (List One)

*Domestic, Import and Export Distribution*

*Thresholds by Chemical Base Weight*

1. Anthranilic acid and its salts.....30 kilograms
2. Benzyl cyanide.....1 kilogram
3. Ephedrine, its salts, optical isomers, and salts of optical isomers all sales reported if ephedrine is the only pill ingredient. When mixed with guiafenesin or other active ingredients the reporting threshold is.....24 grams
4. Ergonovine and its salts.....10 grams
5. Ergotamine and its salts.....20 grams
6. N-Acetylanthranilic acid and its salts.....40 kilograms
7. Norpseudoephedrine, its salts, optical isomers, and salts of optical isomers.....2.5 kilograms
8. Phenylacetic acid and its salts.....1 kilogram
9. Phenylpropanolamine, its salts, optical isomers, and salts of optical isomers.....2.5 kilograms
10. Piperidine and its salts.....500 grams
11. Pseudoephedrine, its salts, optical isomers, and salts of optical isomers.....all sales recorded
12. 3,4-Methylenedioxyphenyl-2-propanone.....4 kilograms
13. Methylamine and its salts.....1 kilogram
14. Ethylamine and its salts.....1 kilogram
15. Propionic anhydride.....1 gram
16. Isosafrole.....4 kilograms
17. Safrole.....4 kilograms
18. Piperonal.....4 kilograms
19. N-methylephedrine and its salts.....1 kilogram
20. N-methylpseudoephedrine and its salts.....1 kilogram
21. Hydriodic acid.....1 liter
22. Benzaldehyde.....4 kilograms
23. Nitroethane.....2.5 kilograms
24. Gamma butyrolactone.....all sales reported

25. Red Phosphorus.....all sales reported
26. White or yellow phosphorus.....all sales reported
27. Hypophosphorous acid and its salts.....all sales reported

## Listed Essential Chemicals

Import and Export Distribution Thresholds ...
Chemical By Volume By Weight (List Two)

Acetic anhydride 250 gallons.....1,023 kilograms
Acetone 500 gallons.....1,500 kilograms
Benzyl chloride N/A.....4 kilograms
Ethyl ether 500 gallons.....1,364 kilograms
Potassium permanganate N/A.....500 kilograms
2-Butanone (MEK) 500 gallons.....1,455 kilograms
Toluene 500 gallons.....1,591 kilograms

Domestic Distribution Thresholds ....
Chemical By Volume 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
Potassium permanganate N/A.....55 kilograms
2-Butanone (MEK) 50 gallons.....145 kilograms
Toluene 50 gallons.....159 kilograms
Iodine.....400 grams
Anhydrous HCl.....no threshold

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)
Joyce Mason
Interstate Waste Management Board
8800 Cal Center Drive
Sacramento, CA 95826
(916) 255-2369

Canadian Waste Materials Exchange
Dr. Robert Laughlin
Ortech International 2395
Speakman Drive
Mississauga, Ontario
Canada L5K1B3
(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
P.O. Box 19276
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

New Mexico Material Exchange
Dwight Long
Four Comers Recycling
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
Hope Castillo
Texas Water Commission
P.O. Box 13087
Austin, TX 78711
(512)463-7773

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

Southern Waste Info Exchange
Eugene Jones P.O. Box
960 Tallahassee, FL
32302 (904)644-5516

## Distributors

Arkansas
EdDavis
AR Industrial Development Commission
#1 Capitol Mall
Little Rock, AR 72201
(501) 682-7322

Iowa
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
Fenton Rude
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 STATES DEPARTMENT OF JUSTICE
DRUG ENFORCEMENT ADMINISTRATION
FEDERAL BLDG. AND U U.S. COURTHOUSE
517 EAST WISCONSIN
AVENUE 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 Agent 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 CHEMICALS** | |
|---|---|---|---|
| Anthranilic Acid and its salts | _____ | Acetic Anhydride | _____ |
| Benzyl Cyanide | _____ | Acetone | _____ |
| Ephedrine, its salt, optical isomers, and their salts | _____ | Benzyl Chloride | _____ |
| Ergonovine and its salts | _____ | Etyl Ether | _____ |
| Ergotamine and its salts | _____ | Hydriodic Acid | _____ |
| N-Acetylanthranilic Acid and its salts | _____ | Potassium Permanganate | _____ |
| Norpseudoephedrine, its salts, optical optical isomers and their salts | _____ | 2-butanone (or Methyl Ethyl Ketone or MEK) | _____ |
| Phenylacetic Acid and its salts optical isomers, and their salts | _____ | Toluene | _____ |
| Phenylpropanolanine, its salts, optical isomers, and their salts | _____ | | |
| Piperidine and its salts | _____ | | |
| Psuedoephedrine, its salts, optical isomers and their salts | _____ | | |
| 3,4-Methylenedioxyphenyl-2 propanone (piperonyl methylketone) | _____ | | |

**DO YOU MANUFACTURE OR DISTRIBUTE TABLETING OR ENCAPSULATING MACHINE**

YES_____ NO_____

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.

**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. | | |
| ETHYLAMINE AND ITS SALTS | 1 KG. | | |
| D-LYSERGIC ACID, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 10 GRAMS | | |
| PROPIONIC ANHYDRIDE | 1 GRAM | | |
| ISOSAFROLE | | | |
| SAFROLE PIPERONAL | | | |
| N-METHYLEPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 1 KG. | | |
| N-ETHYLEPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 1 KG. | | |
| N-METHYLEFEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 1 KG. | | |
| N-METHYLEPSEUDOEPHEDRIHE ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 1 KG. | | |
| N-ETHYLPSEUDOEPHEDRINE ITS SALT, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 1 KG. | | |
| HYDRIOTIC ACID (HYDRIODIC ACID) | 1.7 KG. (1 Liter) | | |
| (previously listed as an essential chemical with a threshold of 22.8 KGS.) | | | |
| 3,4-METHYLENEDIOXPHENYL-2-PROPANONE | 4 KGS. | | |

(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/NO | BUSINESS ACTIVATION CODE(S) |
|---|---|---|---|
| ANTHRANILIC ACID AND ITS SALTS | 30 KGS. | _____ | _____ |
| BENZYL CYANIDE | 1 KGS. | _____ | _____ |
| EPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 1 KG. | _____ | _____ |
| ERGONOVINE AND ITS SALTS | 10 CMS. | _____ | _____ |
| ERGONAVINE 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 KG. | _____ | _____ |
| PHENYLACETIC ACID AND ITS SALTS | 1 KG. | _____ | _____ |
| PHENYLPROPANOLAMINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 2.5 KGS. | _____ | _____ |
| PIPERIDINE AND ITS SALTS | 500 CMS. | _____ | _____ |
| PSEUDOEPHEDRINE, ITS SALTS, OPTICAL ISOMERS, AND SALTS OF OPTICAL ISOMERS | 1 KG. | _____ | _____ |

**LISTED ESSENTIAL CHEMICALS**
**Import and Export Distribution**

| | | | |
|---|---|---|---|
| ACETIC ANHYDRIDE | 1,023 KGS. | _____ | _____ |
| ACETONE | 1,500 KGS. | _____ | _____ |
| BENZYL CHLORIDE | 4 KGS. | _____ | _____ |
| ETHYL ETHER | 1,364 | _____ | _____ |
| POTASSIUM PERMANGANATE | 500 KGS. | _____ | _____ |
| 2-BUTANONE (MEK) | 1,455 KGS. | _____ | _____ |
| TOLUENE | 1,591 KGS. | _____ | _____ |

**DOMESTIC DISTRIBUTION**

| | | | |
|---|---|---|---|
| ACETIC ANHYDRIDE | 1,023 KGS. | _____ | _____ |
| ACETONE | 150 KGS. | _____ | _____ |
| BENZYL CHLORIDE | 1 KGS. | _____ | _____ |
| ETHYL ETHER | 135.8 KGS. | _____ | _____ |
| POTASSIUM PERMANGANATE | 55 KGS. | _____ | _____ |
| 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 fiberlike 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.

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

**Practical LSD Manufacture**

134

- 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 oftenoverlooked detail all chemists should be aware of
- How to manufacture the hallucinogen 2,4,5-trimethoxyamphetamine (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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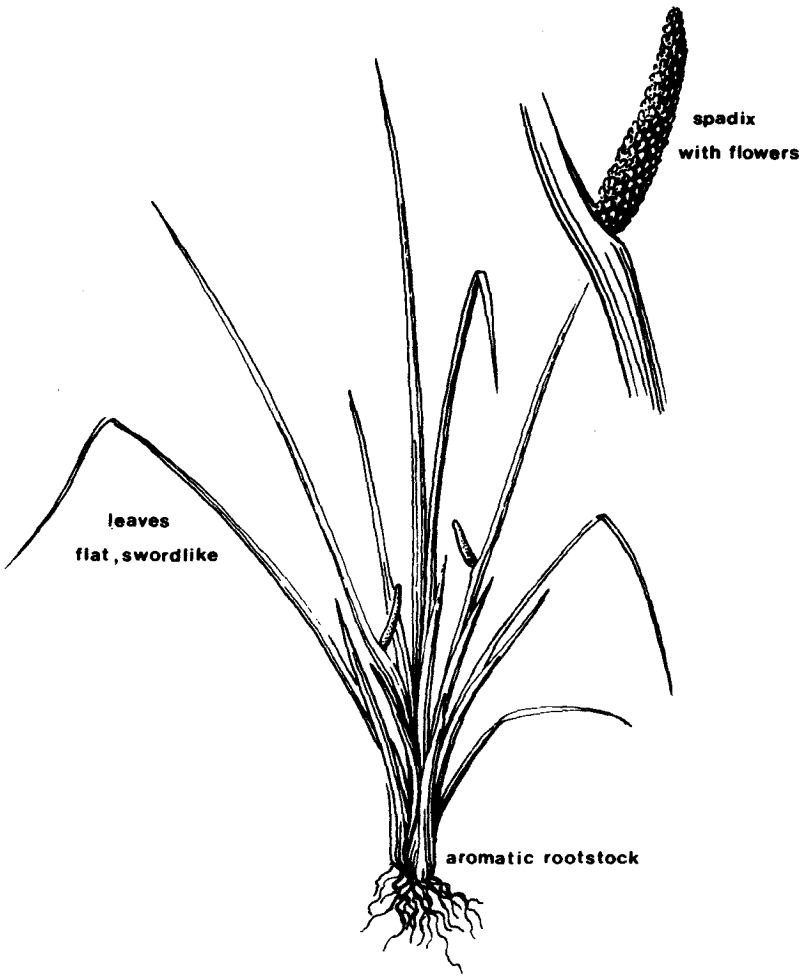


*Acorus calamus Lt*