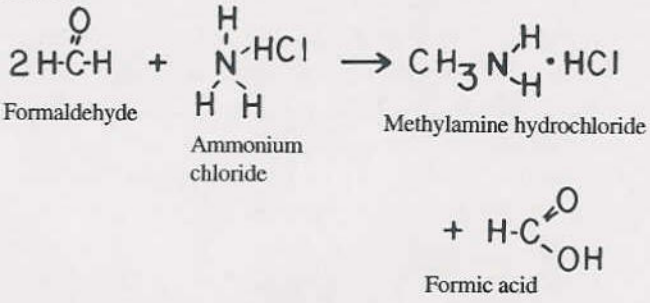
Chapter Thirteen
Methylamine

Methylamine is very high on the do-not-ever-purchase-through-regular-commercial-channels list. As such, any meth production scheme that uses the phenylacetone route will also have to produce its own methylamine. This is no great challenge. In the days before methylamine became commercially available, researchers and practical cookers in industry always had to make their own. To our benefit, they left good directions for us to follow. See *Organic Syntheses*, Collective Volume 1, pages 347-9.



Formaldehyde

Ammonium chloride

Methylamine hydrochloride

Formic acid

The reaction to produce methylamine is cheap, but requires a lot of labor. Two molecules of formaldehyde react with ammonium chloride to produce a molecule of methylamine hydrochloride and formic acid. Both starting materials are easily obtained in 5-gallon-pail or 50#-bag sizes from commercial chemical outlets serving industry.

The glassware is set up as shown in Figure 11 in Chapter Three. The chemist places 1000 grams of ammonium chloride and 2000 ml of 35-40% formaldehyde in the 5000 ml flask sitting in the pan of oil. (These chemicals need not be a very high grade; technical grade is good enough.) He puts a thermometer in the oil next to the flask and heats the oil to 105° C or so, with the aim of heating the contents of the flask to about 100° C or so. A thermometer inserted into the flask is used to monitor its temperature. A bubbling reaction kicks in, and a condensate made up of formic acid and methylal collects in the receiving flask. When this distillation slows in a couple of hours, raise the temperature inside the flask to 104° C, but no higher. Continue heating at this temp until no more distillate comes over (4 to 6 hours). Periodic applications of aspirator vacuum to the batch will increase yield of methylamine because it pulls the CO_2 out of the reaction mixture.

Then he turns off the heat and removes the flask from the pan of oil. Some liquid will have collected in the 2000 ml flask; he throws it out and rinses the flask with water. The 5000 ml flask is set in a pan of room temperature water to cool it off. A good amount of ammonium chloride crystals precipitate from the solution. He does not want these chemicals, so he filters them out. He returns the filtered reaction mixture to the 5000 ml flask and again sets up the glassware as shown in Figure 11. A 250 ml flask is used as the collecting flask. The reaction mixture should be clear to pale yellow.

He turns on the vacuum source and attaches it to the vacuum nipple of the vacuum adapter. He boils off the water and formic acid in the reaction mixture under a vacuum. Heating the flask in the oil pan speeds up the process, but the oil is not heated

above 100° C. When the volume of the contents of the flask is reduced to about 1200-1300 ml, he turns off the vacuum and removes the flask from the oil pan. The flask is put in a pan of room temperature water to cool it off. Some more crystals of ammonium chloride come out of solution. He filters out these crystals and pours the filtered reaction mixture into a 2000 ml flask. He sets up the glassware as before, and again boils off the water and formic acid under a vacuum. He does not heat the oil above 100° C.

When the volume of the reaction mixture has been reduced to about 700 ml, crystals of methylamine hydrochloride begin to form on the surface of the liquid. It looks a lot like a scummy film. When this happens, the vacuum is disconnected and the flask is removed from the oil bath. The flask is placed in a pan of room temperature water to cool it off. As the flask cools down, a lot of methylamine hydrochloride crystals come out of the solution. When the flask nears room temperature, it is cooled off some more with some cold water. This will cause even more methylamine hydrochloride to come out of the solution.

The chemist filters out the crystals and puts them in a mason jar. The crystals look different from the crystals of ammonium chloride, so he should have no trouble telling the two apart. These crystals soak up water from the air and melt, so he does not waste time getting them in the mason jar after they are filtered.

He pours the filtered reaction mixture into a 1000 ml round bottom flask and again sets up the glassware as shown in Figure 11. He reattaches the vacuum and continues boiling off the water and formic acid under a vacuum. When the volume of the mixture reaches 500 ml, he removes the flask from the hot oil and places it in cool water. As it cools off, more crystals of methylamine hydrochloride appear. He filters the cold reaction mixture to obtain these crystals. He transfers them to a beaker and adds 200 ml of cold chloroform to the beaker. He stirs the crystals around in the chloroform for a few minutes, breaking up any chunks. This dissolves any dimethylamine hydrochloride in the product. He filters the crystals in the beaker, then puts them in the mason jar along with his first crop of methylamine hydrochloride crystals. He throws away the chloroform and returns the reaction mixture to the 1000 ml flask.

He boils the reaction mixture under a vacuum again. When its volume reaches about 150-170 ml, he turns off the vacuum and removes the flask from the hot oil. He pours the reaction into a beaker and stirs it as it cools down, to prevent it from turning into a solid block. Once it has cooled down, he adds 200 ml of cold chloroform to the slush. He stirs it around with a glass rod for a couple of minutes, being sure to break up any chunks. The mixture is then filtered. The crystals of crude methylamine hydrochloride are kind of gooey, so it may not be possible to filter out all the chloroform.

This batch of crystals is added to the mason jar along with the rest of the crude product. The yield of crude product is around 425 grams. It absorbs water easily from the air, and melts. Its smell has been described as "like old woman's pussy." The main contaminant of the crude product is ammonium chloride, along with some dimethylamine hydrochloride, and some of the reaction mixture. The 425 gram yield of crude product is therefore deceivingly high.

Purification would best start with drying under a vacuum. This could be conveniently done by placing the crude crystals into a large vacuum flask, stoppering the top of the flask, and applying aspirator vacuum for about half an hour. Gentle heating of the flask with warm water during the vacuum drying helps speed along the process, as does some shaking around of the contents of the vacuum flask. If one has an aspirator that likes to spit back water into flasks under vacuum, then one should use a vacuum pump.

Now to get nice and pure crystals of methylamine hydrochloride, we leave those crude crystals in the filtering flask, and add around ¾ of a quart of 190 proof vodka to the crystals. 190 proof vodka won't dissolve ammonium chloride, but it will dissolve methylamine hydrochloride when it is hot. Leave the top of the filtering flask stoppered to prevent steam from getting into the flask, then warm up the flask using hot water. Water fresh off

the stove, almost boiling hot, would be best. Swirl around the flask as it warms to get the methylamine hydrochloride dissolved.

Once the alcohol solution gets hot, stop swirling to let suspended crystals settle out. Then decant off the alcohol solution, taking care to keep the crystals inside the flask. Filtering is necessary. Then put the alcohol which has been decanted from the flask in the freezer. As it gets cold, methylamine hydrochloride crystals will come out of solution. When the alcohol is good and cold, filter to collect these pure crystals of methylamine hydrochloride. Store them in a mason jar with lid.

Return the filtered cold alcohol to the filtering flask containing the crude product. Once again heat the alcohol with swirling to dissolve some more methylamine hydrochloride. Then let the suspended crystals settle once again, and decant the alcohol as before, and cool that down in the freezer to get another crop of pure methylamine hydrochloride. A few cycles through this process will get all the methylamine hydrochloride soaked out of the crude product and recovered as pure recrystallized methylamine hydrochloride. The yield of pure methylamine hydrochloride will be around 350 grams or so.

Sometimes, the methylamine hydrochloride is used directly as such in the reaction, such as, for example, in reductive alkylation using aluminum foil as the reducer. More generally, the free base is used. To obtain a strong solution of methylamine in water, 100 grams of methylamine hydrochloride is placed in a flask with 50 ml water. This is chilled in an ice-salt bath to a temperature nearing 0° F. Then a cold solution of 60 grams of NaOH in 100 ml water is slowly added with stirring. The addition must be slow enough, and the cooling strong enough, to avoid losing the free base as a gas. Methylamine solution produced in this way is roughly comparable to the commercial 40% methylamine, except that it also contains salt, and maybe a little NaOH if too much was added.

This solution should either be used immediately, or stored in a tightly stoppered bottle. Refrigeration of the solution is optional, but desirable.

The reader should be aware that chloroform is a poison for Raney nickel catalyst, so if that particular method is going to be used in meth production, the crystals must be vacuum-dried. Also, it is possible that the excess NaOH may interfere with methods using catalytic hydrogenation. I can't say. If it does, an apparatus like that in Figure 17 can be used to boil out the methylamine free base into a stirred chilled solution of alcohol.

Other methods of making methylamine exist, but they are not well-liked by the pioneers mentioned at the beginning of the chapter. Presented here is their preferred method. For example, it can be made in 71% yield by reacting methyl iodide with hexamine, also known as hexamethylene tetramine. Good directions for making this substance from ammonia and formaldehyde can be found in *Home Workshop Explosives* by yours truly. The production details for methylamine are found in the *Journal of the American Chemical Society,* Volume 61, page 3585 (1939). The authors are Galat and Elion.

It can also be made by degrading acetamide with Clorox. See *Journal of the American Chemical Society*, Volume 63, page 1118 (1939). The authors are Whitmore and Thorpe, and the yield is 78%.

It can also be made via the Curtius reaction in a yield of 60%. See *Helvetica Chimica Acta*, Volume 12, page 227 (1929). The authors are Naegeli, Gruntuch and Lendorff.

References

Journal of the American Chemical Society, Volume 40, page 1411 (1918).

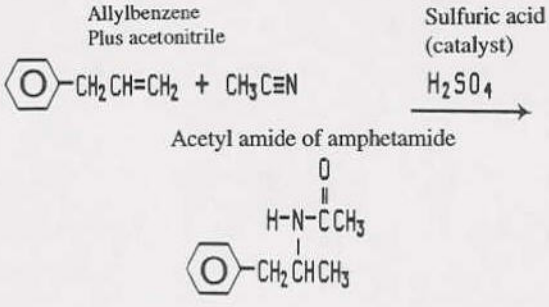
Chapter Fourteen
The Ritter Reaction: Amphetamines Directly From Allylbenzene

A most interesting sidelight appears in an article by Ritter and Kalish found in the *Journal of the American Chemical Society*, Volume 70, pages 4045-50 (1948). This sidelight was a bit of research done by a grad student as part of his master's thesis. The grad student just happened to work out the experimental details for converting allylbenzene directly into amphetamine.

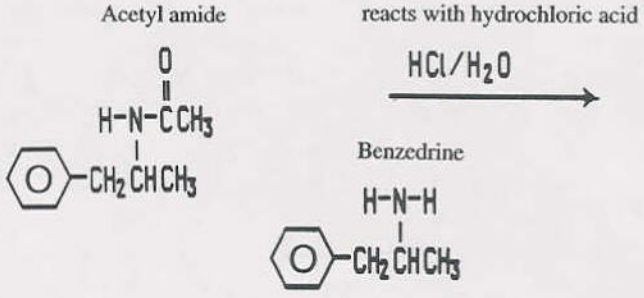
The main thrust of the article was the good Dr. Ritter telling of his new method for converting double bonds into amines. The method which he pioneered has since come to be known as the Ritter reaction. This versatile reaction can well serve the underground operator as an alternative pathway to the amphetamines.

The Ritter reaction in general is a reaction whereby amides are made by adding an alkene to a mixture of a nitrile in sulfuric acid. After the amide is made, it is then boiled in hydrochloric acid solution to give the corresponding amine.

The particular variation on this theme in which we are interested deals with the case in which the alkene is the now familiar and highly useful allylbenzene. When it is added to a solution of acetonitrile in sulfuric acid, the following reaction takes place:



The acetyl amide thusly produced is not isolated and purified. Rather, it is added in the crude state to hydrochloric acid, and boiled for several hours. A hydrolysis reaction almost identical to the one seen in Chapter Five takes place producing the prototype amphetamine, benzedrine.



The acetyl amide of amphetamine is very similar to the formyl amide of methamphetamine produced by the Leuckardt-Wallach reaction. Its main difference is that it is more difficult to hydrolyze to amphetamine by the action of boiling hydrochloric acid. It must therefore be boiled with the acid for a longer period of time than the formyl amide. The manufacturer may well find it to his advantage to boil the tar left over at the end of the process once more with fresh hydrochloric acid. This will likely yield an additional measure of amphetamine from the stubbornly unreactive amide.

This small hassle with the hydrolysis process could be avoided if HCN were used as the nitrile in sulfuric acid solution. However, the extreme danger of dealing with hydrogen cyanide more than out-

weighs the additional work needed when using acetonitrile.

To do the reaction, a solution of 450 grams (243 ml) of concentrated sulfuric acid in 400 grams (530 ml) acetonitrile is made by slowly adding the acid to the acetonitrile. Both ingredients are cold when they are mixed together, and the temperature of the mixture is kept in the 5-10° C range during the mixing by setting the reaction container in ice. An admirable reaction vessel is a glass beer pitcher.

When the addition of the acid to the nitrile is complete, the pitcher is taken out of the ice, and 236 grams (262 ml) of allylbenzene is slowly added to it with stirring. The mixture quickly turns an orange color, and begins to warm up.

Stirring is continued and the temperature of the mixture followed. It slowly climbs to 50° C, and then more rapidly to 80° C, as the color of the mixture darkens.

This is a tenfold scale up of the original recipe, so be watchful and protected in case the reaction gets out of control. One wouldn't want this mixture to go postal on you. Once the 80° C temperature is reached, pour the mixture out of the pitcher, and onto a few pounds of ice cubes. Smaller batches can be cooled just by immersing the reaction vessel in ice, but on this scale, go right onto ice.

Once the reaction mixture has cooled down, the acid should be neutralized by slowly pouring it into a 15% solution of lye dissolved in water. About a pound of lye will be required to neutralize all the sulfuric acid and produce an alkaline solution. Most of the unreacted acetonitrile will end up in the water layer, but some will evaporate during the neutralization. Stay upwind!

The neutralization of the acid by the lye solution produces a great deal of heat. The lye solution is gently stirred during the addition, and then stirred more vigorously during the following minutes. After a few minutes of stirring, the mixture is allowed to sit for a few minutes. A yellow oily layer floats on the top of the solution. This yellow oil is the crude amide. If the oil were to be allowed to sit for a while longer, it would begin to form crystals of crude amide. There is no need for this, however, so the processing continues immediately.

The top yellow layer is poured off into a sep funnel, and any water carried along is drained off. Then the yellow oil is poured into a 2000 ml round bottom flask. It is now ready for hydrolysis with hydrochloric acid solution to make amphetamine. The approximate volume of the crude amide is determined, and five times that volume of 15% hydrochloric acid solution is added to it. Fifteen percent hydrochloric acid solution is easily made by starting with the 28% hardware store hydrochloric acid, and adding just about an equal volume of water to it. A wise move here is to rinse the inside of the sep funnel with acid. This rinses off the amide clinging to the glass insides of the sep funnel.

When the acid has been added to the amide, the mixture is swirled. They usually mix together well. If they don't, stronger acid is used. Adding some full strength acid to the mix should do the job. Then a few boiling chips are added to the flask, a condenser attached to the flask, and heat applied to boil the mixture at reflux.

The reflux boiling is continued for 10 hours. During this time the mixture will turn black. At the end of the boiling period, the mixture is allowed to cool down. When it is cool, 200 ml of toluene is added to the flask. The mixture is shaken well for a couple of minutes, then allowed to sit. The toluene floats up to the top, and has dissolved in it most of the unreacted amide, and other unwanted garbage.

The toluene layer is then poured off into a sep funnel, and any water layer carried along drained back into the flask. The toluene layer is poured off into another container for future processing. It may be difficult to tell exactly where the toluene layer ends and the water starts because of their similar color. A sharp eye and good lighting help to spot the interface of the two fluids.

The acid solution of the amphetamine is now made alkaline to liberate the free base for distilling. To do this, lye is added to the acid solution in the 2000 ml flask. Assuming the use of about 1200 ml of 15% hydrochloric acid solution, one 12 oz. can of lye does the job. The mixture is first swirled to release heat, then shaken vigorously for five minutes. I cannot emphasize enough the importance of vigorous and prolonged shaking here because the

amphetamine base initially formed tends to dissolve unneutralized amphetamine hydrochloride. The oily droplets protect the hydrochloride from contact with the lye solution unless the shaking is strong and prolonged.

When the shaking is completed, the mixture is allowed to cool down. Then 300 ml of toluene is added to the flask, and shaking continued for a minute or two. After sitting for a couple of minutes, a toluene-amphetamine layer floats above the water layer. This is poured off into a sep funnel, and the toluene-amphetamine layer poured into a 1000 ml round bottom flask.

The amphetamine-toluene mixture is distilled in exactly the same manner as described in Chapter Five. The boiling point of benzedrine is 10° to 20° C lower than meth. The yield of benzedrine is in the range of 100 to 150 ml.

The benzedrine produced by this reaction is either used and removed as is, or it is converted to methamphetamine. A very good and simple process for doing this can be found in the *Journal of the American Chemical Society*, Volume 62, pages 922-4. The author is Woodruff. The yield for this process is over 90%, so a greater volume of methamphetamine comes out of the reaction than the benzedrine input. This is because the gain in molecular weight achieved by adding the methyl group outweighs the small shortfall from 100% yield.

For those who have difficulty reading the Woodruff article, meth is described as B-phenylisopropylmethylamine. The amine is benzedrine.

If the benzedrine product is used as is, the producer makes it as the hydrochloride salt. This is made the same way as methamphetamine hydrochloride. An alternative to the hydrochloride salt is the sulfate salt. This more hasslesome procedure calls for the use of cooled solutions of amphetamine base in alcohol and cooled solutions of sulfuric acid in alcohol. Furthermore, a recrystallization from alcohol-ether is required because trapped excess sulfuric acid in the crystals causes them to turn to mush or worse. By using HCl gas, the excess acid floats off as gas.

An excellent review of this reaction can be found in *Organic Reactions*, Volume 17. Nearly double these yields should be obtained if the underground chemist is willing to risk using hydrogen cyanide instead of acetonitrile. The hydrogen cyanide is made inside the reaction flask from sodium cyanide and sulfuric acid. For complete directions, see *Organic Syntheses*, Collective Volume 5, pages 471 to 473. The name of the compound is alpha, alpha, Dimethyl beta phenethylamine.

My opinion is that anyone attempting this variation with hydrogen cyanide in any place other than a well ventilated shed, well upwind from the batch, is just nuts. This variation isn't recommended, nor do I know if it has been specifically tested for efficacy with allylbenzene. It sure as hell is worth checking out, if the required precautions are taken for dealing with hydrogen cyanide solution. This is not for beginners!

Chapter Fifteen
Methamphetamine From Ephedrine or Pseudoephedrine; Amphetamine From PPA

Ephedrine and Pseudoephedrine

Ephedrine and pseudoephedrine are structurally mirror images of each other. This is possible because they have a chiral center, the carbon atom attached to the alcohol group of these two substances. Theoretically, reduction of these two materials should both give the same product, the "d" isomer of meth because when the alcohol group is reduced, the chiral center disappears.

Theory and reality aren't quite in agreement. Pseudoephedrine is less willing to undergo reduction in several of the methods given in this chapter than is ephedrine. This gives a lower octane product which had myself and several of my correspondents from the pen convinced that pseudoephedrine was giving the low octane "l" isomer of meth. The reality was that this low octane product was a mixture of meth and unreduced pseudoephedrine.

Several of the methods given in this chapter are equally effective for both ephedrine and pseudoephedrine. Foremost among these is the lithium metal in liquid ammonia reduction to meth. Some methods that work just fine for ephedrine fail miserably when used on pseudoephedrine. The prime example of this situation is the "Cat" recipe given in the next chapter.

When one can only get pseudoephedrine, and only has access to chemicals best used to reduce ephedrine, an alternative is to racemize the pseudoephedrine to racephedrine. Reduction of racephedrine to meth will give the "d,l" isomer mix which produces a buzz superior to the pure "d" isomer. The same "d,l" isomer mix is produced when phenylacetone is used to make meth. A racemization procedure is given in this chapter.

Procedure for Obtaining Pure Ephedrine, Pseudoephedrine or PPA from Stimulant Pills

No aspect of methamphetamine manufacture has changed so radically in the past few years as the composition and availability of the OTC "stimulant" pills which are useful as raw-material feedstock for methamphetamine production. Back in early '92, when I penned the third edition to this book, ephedrine pills were available by mail order. These pills were quite well-suited for a very simple water extraction to get out the active ingredients, because they were about 30-40% active ingredient, and the fillers were mostly non-water soluble.

Since that time, the mail-order outfits have been heavily leaned upon. Several of their heads are now in the slammer. Other such companies have been taken over and are now agents of the enemy. I wouldn't trust most of them as far as I could spit. All ephedrine orders now must be accompanied with photocopies of driver's licenses, etc. Ephed-

rine is now on the chemical diversion list with no minimum threshold quantity. To quote one of the DEA's top dogs on this matter: "We're keeping track of where they are going."

Pseudoephedrine, aka Sudafed, and PPA, aka phenylpropanolamine or Dexatrim, are now subject to close sales scrutiny as well due to the Meth Act of 1996, passed in response to the 4th edition of this book. A single sale limit of 24 grams of base, which corresponds to a little under 500 of the 60 mg pseudoephedrine pills or a little over 300 of the 75 mg phenylpropanolamine pills, has been established. Mail-order pill companies are now required to turn over, on a regular basis a complete customer list with names and addresses and amounts purchased.

As a result, it must be emphasized that the procedures given in this chapter are most suitable for making "stash" amounts of meth or dexedrine. Large pill purchases will attract the heat like blood in the water does sharks. The recipes given in this chapter can easily be scaled down to whatever amount of pill feedstock one is able to obtain. Retail store sales aren't federally regulated, but the prices there are very high compared to the mail-order prices.

Starting in 1998, Wal-Mart began limiting sales of all cold medicines (read: ephedrine, pseudoephedrine and phenylpropanolamine) to three packages per customer. Try to buy more and the cash register throws a fit. Some localities in areas such as California have passed ordinances decreeing similar retail sale limits. State laws may be coming imposing similar limits. Unless one is willing to spend all day shopping for pills, it may be difficult to accumulate thousands of them.

An alternative to the use of ephedrine pills is what are called "herbal extract" pills. These don't at present come under reporting requirements because they wear that "herbal" label. These pills are loaded with ephedrine, so expect laws to be passed shortly bringing them under reporting requirements. They are at present available by mail order at a reasonable price. Ads can be found in our favorite sleazy magazines. I would suggest using a fake name and a rented mailbox when ordering them.

Another source of ephedrine is the herb Ma Huang, which is the source of those herbal extract pills, and the natural source of ephedrine. It contains around 8% ephedrine, along with some pseudoephedrine. This material too isn't under reporting requirements as I write this, but stay tuned. That could change at any moment. I've looked for this herb at my local health food stores with no success, but your area may be better stocked.

The "doctoring" of the pills over the past several years has been similarly dramatic. In the case of ephedrine pills, the first thing which was done was to add more filler to make them less suitable to a simple water extraction. The more filler, the more water required to extract the active ingredient, and the more inert pill-gunk co-extracted. This gunk had a bad effect on the meth-production reactions, which follow the GIGO principle.

Then the pill doctoring became more scientific. The insoluble filler was replaced by some type of water-soluble fiber which played much greater havoc on the ensuing reaction if no purification beyond just water extraction was done. Even in the case of making cat, it would screw up the reaction by causing the entire reaction mixture to take on a milkshake consistency upon neutralization with NaOH. The gunk equally filled the water and toluene layer (which, by the way, were pretty hard to spot because of the floating gunk). The floating gunk could be filtered out of the toluene layer, and the hydrochloride then precipitated, but the octane numbers were greatly reduced below normal.

The next version of ephedrine pills contained 25 milligrams of ephedrine along with 100 milligrams of guaifenesin. They were available at gas stations and other stores because they came under the definition of "mixture" in the Chemical Diversion Act, and so were not regulated.

These 100 milligram guaifenesin pills were followed by 200 milligram guaifenesin pills, which is what are on the market now. The definition of "mixture" has been changed so that ephedrine pills containing guaifenesin are just as reportable as the others.

The best extraction procedure to use will vary with the pill type. Let's start with the basic pill extraction procedure. It's an extension of the old standard water extraction procedure that was so successful with the old mini-thin ephedrine pills. It will work fine with the typical pseudoephedrine pills now on the market along with ephedrine pills containing less than 200 milligrams of guaifenesin, and phenylpropanolamine pills. By substituting 1-2% HCl solution for the water in this extraction, it can also be used to extract Ma Huang, and the "herbal extract" pills.

The first step is to grind the pills. A mortar and pestle gives the best grind size, as overly fine grinding makes the subsequent filtering steps more difficult. With herbal extract pills and Ma Huang, the initial grind should be done in a blender, mixing the substance to be ground with its initial grind charge of 1-2% hydrochloric acid (hardware store muriatic acid diluted 15-30 fold) and blenderizing at medium high speed until small particles are obtained.

The next step after the grind is to determine whether these pills need to be degummed and desplooged.

The ephedrine-guaifenesin pills I've come across are really loaded with gum. They must be now degummed and desplooged by soaking the ground up pills in toluene, then filtering. Other types of pills I've come across aren't so loaded with this ingredient, although it may be more prevalent in the future. Failure to degum and desplooge these pills results in a milkshake later when the water extract is made basic and extracted with solvent. It also really slows up the filtering of the water extract.

I think this emulsion-forming ingredient is some type of fatty acid which forms a soap when sodium hydroxide is added later on to free base the ephedrine, or whatever. Toluene is also quite good at removing guaifenesin from pills. Colored pills should be tested with solvent. If toluene is going to be used as the extractant at the end of this procedure, check to see if toluene dissolves the coloring matter. If it does, then soak the ground up pills in toluene and filter to remove the color. Ditto if Coleman camper fuel is going to be used as the final extractant. Allow the ground up pills to dry after desplooging so that the solvent is removed from them.

Then water extraction is done. Mix 1,000 ground up pills with 350 ml of water, and stir for about an hour. Another variation is to just mix 1,000 pills with 350 ml water, and after the pills have softened, mush them up and stir for an additional hour.

Now the pill mush should be filtered. Vacuum filtration through a Buchner funnel is greatly preferred, because it will suck the filter cake dry, giving better extraction with less use of water. The need to keep the amount of water used to a minimum arises from the fact that the "pill extraction deterrents" are less soluble in water than the desired ingredient, so the more water used, the more effective they are. It may be difficult to get the mush to filter easily through filter paper, so a preliminary filtering through clean white cotton cloth cut like a filter paper will be helpful in these cases.

The filtrate should be clear, and very bitter tasting, as it contains the active ingredient. Hopefully, most of the pill fillers didn't dissolve, and they are sitting in a filter cake in the Buchner funnel.

Now take this filter cake of pill sludge, remove it from the filter, and mix it with an additional 300 ml of water. Stir this around for an hour, then filter this. If a Buchner funnel was used, this is enough water to extract the pills. If only gravity was used to aid filtration, then the pill sludge should be soaked with a final 100 ml portion of water, and filtered.

To the combined filtrates, add a dash of hydrochloric acid to suppress steam distillation, and boil its volume down to about 200 ml. With pseudoephedrine, this isn't so important because it isn't as water-soluble as ephedrine or PPA free bases, but the volume should be reduced some for it, too.

Now let the solution cool, and then add 20% NaOH or lye solution with stirring or shaking until the solution is strongly alkaline to litmus paper. Indicating pH paper should say 12+. A pH meter may not be as useful as paper for this reading. The

solution at this point should smell strongly of the kind of fishy free bases.

Extract the water solution with about 100 ml of toluene. This solvent can be found in the paint-thinner section of the hardware store or paint-supply outlet. If you can't find this solvent, Coleman camper fuel will work almost as well. The water layer should remain a liquid, and the toluene layer should be clear and transparent. If the particular "deterrent" formulation results in a milkshake consistency, just estimate how much is that top 100 ml of solvent, separate it off, and filter it. Rinse the filtered out gunk with solvent. Repeat this extraction with two additional portions of toluene. With the ephedrine-guaifenesin pills, extract with petroleum ether, hexane or Coleman camper fuel.

The combined toluene extracts should be placed in a 400 ml beaker and allowed to sit for a few hours. This serves two purposes: first, entrained water will settle to the bottom of the beaker and stick to the glass. When it is poured into a fresh beaker, the water will be removed. The second reason involves an observation I made some time ago with one particular "deterrent" formulation. In that case the water layer became almost solid after the second toluene extraction, because the solvating action of ephedrine free base was lost for these fillers. The toluene extract in this case, upon standing, grew a mat of white solid about ¼- to ½-inch thick on the bottom of the beaker. After letting this mat grow, and pouring the solution off of it, all proceeded well from that point.

Once the toluene has been poured into a fresh beaker, dry HCl gas should be bubbled through it to precipitate pure ephedrine, pseudoephedrine or PPA hydrochloride. This is done just like the bubbling to get meth hydrochloride in Chapter Five. The yield from 1000 25 mg ephedrine pills is about 20 grams, from 1,000 60 mg pseudoephedrine pills about 50 grams, and from 1,000 75 mg PPA pills is about 65 grams.

With Ma Huang and herbal extract pills, the dilute acid solution used as extractant should be boiled down to concentrate it, just as with pills. Then take some toluene and extract the acid solution concentrate. This will remove coloring and other plant material, but not the desired ephedrine. One, of course, has to wait for the solution to cool before doing a solvent extraction or the solvent will boil and fume and make a mess on you.

Then after extracting the acid concentrate, this concentrate should be made strongly alkaline by adding lye solution and shaking. We now have free base as in the pill example. It can be extracted out with toluene, just as with pills, and the hydrochloride collected by bubbling with HCl, just as with pills.

Another pill extraction procedure which was briefly touched upon in the fourth edition of this book has proven quite useful when extracting those 200 milligram guaifenesin pills. It can also be used on other pills, but it is with those bulky 200 mg guaifenesin — 25 mg ephedrine pills that its advantages really shine through.

First the pills are finely ground in a blender. Shaking the blender some while it is running will help to get large pill chunks off the bottom of the blender and into the blades. Next one can pour the powdered pills into a beaker, and desplooge with roughly one ml of toluene for each pill used. Stir it around for about half an hour, then filter. Spread the pill mass out to air dry. I'm not really certain if this step is absolutely necessary. Feel free to skip the toluene desplooge step, and see if it makes any difference.

Return the dried pill mass to the beaker, and add about 4 grams of lye for each 100 of the 25 mg ephedrine pills used. Stir this in. Then slowly add 91% isopropyl rubbing alcohol, or hardware store denatured alcohol or 190 proof vodka, with stirring, until a moderately runny paste is achieved. Too much alcohol could make it difficult to precipitate the hydrochloride crystals at the end of this process. Using water instead of alcohol can result in a regrettable mess, especially if too much water is used. That extraction deterrent formulation really kicks in with water, and a horrendous milkshake emulsion easily forms. Stick to alcohol.

Stir this fairly light paste for about half an hour. The lye dissolves, and produces the free base of the

ephedrine. We now extract out the ephedrine free base.

Add 50-75 ml of Coleman camper fuel for each 100 pills used, and stir this mixture for about half an hour. Then filter the mixture. Doubled up coffee filters or lab filter paper will be fine enough to catch the pill particles. A clear blue filtrate should result. The blue color is from the camper fuel; it causes no problems.

Return the pill mass to the beaker, and add another 50-75 ml of Coleman camper fuel for each 100 pills used. Stir this for about half an hour, then filter.

The combined clear blue filtrate is now ready for bubbling with dry HCl. This is done just like in all the other examples where we bubble dry HCl to get the crystalline hydrochloride product. The blue color of the camper fuel doesn't color the crystals at all, so long as it is sucked away using a Buchner funnel and vacuum flask. If you don't have such equipment, a final rinse of the crystals with toluene will wash off the camper fuel.

Camper fuel evaporates quickly, and doesn't leave a lingering smell on the crystals of ephedrine hydrochloride. One can expect to get close to 100% extraction of the pills by this method, so long as the pills were finely ground in the first place. Your Uncle has tried and likes this method!

Racemization of Pseudoephedrine

This procedure is taken from *Chemical Abstracts*, Volume 23, pages 3452-4 (1929). It yields racemic ephedrine or racephedrine from pseudoephedrine, thereby allowing the use of pseudoephedrine to get d,l-meth.

Pseudoephedrine hydrochloride prepared as described above is dissolved in 25% hydrochloric acid solution. Stronger acid must be avoided, as the use of this stronger acid would produce a significant amount of chloroephedrine. 100 ml of 35% lab-grade HCl can be diluted to 25% by adding 40 ml of water. In the example given in the *Chemical Abstracts*, a fairly dilute solution of pseudoephedrine was used, but I can't think of any reason why one can't mix this solution much stronger. Adding more pseudoephedrine to a given volume of HCl solution allows much more material to be processed at once, and also makes recovery by extraction at the end of the process much easier.

This solution of pseudoephedrine in HCl is then heated at 100° C for at least one day, preferably two. Heating the solution to reflux is to be avoided, as correspondents have informed me that reflux temperatures lead to the burning of the product. Simply heat the flask in an oil bath whose temperature is kept at about 100° C. A reflux condenser must be attached to the flask to keep the acid from evaporating away. In the example from *Chemical Abstracts*, the acid was heated inside a sealed tube, but I can't see why this simpler procedure won't work just as well.

At the end of the heating period the solution is cooled, and then sodium carbonate or bicarbonate is added to the acid solution a bit at a time until all of the acid is neutralized. This point can be spotted because the carbonate will stop fizzing once all the acid is gone. Now shake the solution strongly for a few minutes to ensure that all of the racephedrine hydrochloride has been converted to the free base. Then extract a couple of times with toluene. The pooled toluene extracts can then be bubbled with HCl gas to precipitate the product as the hydrochloride.

The best use for this procedure is probably to isomerize the "l" meth extracted from Vick's inhalers to "d,l"-meth.

Indirect Reduction

A popular alternative method for making methamphetamine uses ephedrine as the starting material. This method was not covered in the original edition of this book. It is now presented in all its glory for the education of the reader.

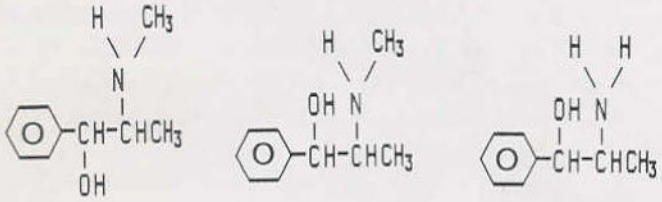
The reasons for the popularity of this method are twofold. Firstly, this method does not require the use of methylamine because the methylamino group is already incorporated in the ephedrine molecule.

The utility of this method is not limited solely to ephedrine. Pseudoephedrine and phenylpropanolamine can also be used as starting materials. This means that Sudafed and Dexatrim, and their ge-

neric equivalents, can be used as raw materials for clandestine amphetamine manufacture. The active ingredient is easily separated from the diluents in the pills by the method given in this book.

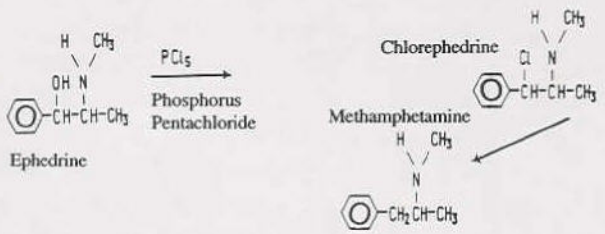
The bad thing about this method is that foul impurities generated during the manufacturing process are easily carried into the final product. Due care must be practiced by the chemist during the purification to exclude this filth. Unscrupulous and/or unskilled manufacturers turn out large volumes of crank containing this abomination. The impurities not only ruin the finer aspects of the meth high, but they also have a pronounced deleterious effect on male sexual function.

Study the compounds pictured below, and compare them to the meth molecule:



Pseudoephedrine Ephedrine Phenylpropanolamine

One can quickly see that all a chemist needs to do to turn ephedrine into meth is to replace the alcohol OH grouping with a hydrogen atom. This is not done directly. Instead, a two-step process is used whereby the OH is first replaced by a chlorine atom, and then this chlorine is removed by one of several reductive processes, to be replaced with a hydrogen atom. To illustrate:



There are several general methods for converting an alcohol group into a chlorine atom. Substances such as thionyl chloride ($SOCl_2$), phosphorus pentachloride (PCl_5), phosphorus trichloride (PCl_3), phosphorus pentabromide (PBr_5) and phosphorus tribromide (PBr_3) can all be used to convert the alcohol group to either a chloride or bromide. Essentially the same reaction conditions are followed when using any of the above listed substances. The only difference is how much ephedrine or PPA (phenylpropanolamine) the substance can chlorinate or brominate. See the table below:

Substance	Molecular Weight	reacts with this many moles of ephedrine
$SOCl_2$	119	1
PCl_3	137	2
PBr_3	271	2
PCl_5	208	3
PBr_5	430	3

molecular weight of ephedrine HCl=202,
PPA-HCl = 188

Using the above table, a person can quickly calculate how much ephedrine or PPA will react with a given amount of chlorinating agent. Use of excess chlorinating agent will result in a higher percentage yield based on the ephedrine used, but after a point, this is wasteful. The following example takes this largess to an extreme, but achieves 100% conversion of ephedrine to chlorephedrine. This procedure can be followed with all the chlorinating agents. The reaction is fairly easy to do. The main precautions are to make sure that the glassware is free of water, and taking one's time to be sure the mixture stays sufficiently cold. It is also wise to avoid doing this reaction in very humid conditions.

The following procedure for the conversion of ephedrine, racephedrine or PPA to the chloro compound and then its reduction can be found in *Chemical Abstracts*, Volume 23, page 3453. It results in a little racemization, but mostly keeps the structure of the starting material.

To do this reaction, a 2000 ml Erlenmeyer flask is filled with 360 ml of chloroform and 360 grams of PCl_5. This mixture is then cooled down in ice water, and once it has cooled down, 240 grams of ephedrine HCl is added in little portions, with shaking of the slushy reaction mixture after each

add of ephedrine, racephedrine or PPA. The addition should be completed in about ½ hour. Then, for an additional two hours, the reaction mixture should be shaken to mix around the contents. Cooling in ice must be continued throughout the reaction time to keep the contents from overheating.

When two hours of reaction time has passed, let the contents settle in the flask. After about 45 minutes, when all has settled inside the flask, the mixture is carefully decanted off into a one-gallon glass jug. Great care is taken during this decanting to make sure that all of the settled PCl_5 remains behind. If any of it were mixed in with the product chlorephedrine it would be reduced in the succeeding hydrogenation to phosphine, PH_3, an exceedingly deadly gas. If it appears any is being carried along, the mixture is filtered.

The PCl_5 left in the flask should be rinsed with 150 ml of chloroform to get the trapped product out of it. This is done by adding the chloroform, shaking the sludge to mix, allowing it to settle, then decanting off the chloroform as before into the glass jug.

There will be a lot of unused PCl_5 in the flask, and it would be a shame to just trash it. The obvious thing to do is to save it by stoppering the flask, and try using this material to run another batch.

Next, the product is precipitated from the chloroform solution in the gallon jug. This is done by slowly adding ether or, better still, mineral spirits (cheap and easily available in large amounts) to the gallon jug until it is nearly full. For best results, the mixture in the gallon jug is continuously stirred during the addition of the ether or mineral spirits. Chlorephedrine does not dissolve in ether or mineral spirits, so as the solution changes from chloroform to predominantly ether, the product is thrown out of solution in the form of crystals. If an oily layer forms at the bottom of the jug, this means a dirty batch. The oil may eventually crystallize, but more likely it must be separated, dissolved in an equal volume of chloroform, and precipitated once again by adding ether or mineral spirits.

After the addition of the ether or mineral spirits, a large mass of crystals fills the jug. This is the product. The jug is stoppered, and put into the freezer overnight to let the crystals fully grow. The crystals are then filtered out and rinsed down with a little bit of cold acetone. Then the crystals are spread out to dry on china plates or glass baking dishes. The yield of chlorephedrine hydrochloride is in the neighborhood of 250 grams.

Production of Meth

To make meth from chlorephedrine, the chlorine atom is replaced with a hydrogen. This reduction is accomplished by any of several methods. Lithium aluminum hydride does the best job of completely converting the chlorephedrine into meth, but it is very expensive, and a watched chemical. Zinc dust, on the other hand, is cheap and easily available, but it leaves a large proportion of the chlorephedrine trashed. The most practical and effective way to turn out large volumes of meth is by catalytic hydrogenation. It is possible to use Raney nickel as the catalyst for this hydrogenation, but it has to be used in quite large amounts to do a good job. Ammonia, amine or some other base also has to be added to the bomb in an amount equal to the chlorine given off by the chlorephedrine, i.e., one mole of chlorephedrine would require one mole of ammonia, amine or other base added. Platinum can also be used to reduce the chlorephedrine, but it too has to be used in large amounts to get good results. Furthermore, it is rapidly poisoned by the hydrochloric acid generated by the removal of the chlorine atom from chloroephedrine unless one mole of base per mole of chloroephedrine is included in the hydrogenation mixture. But I have seen one example in which good yields were obtained with Pt and chloroephedrine without addition of any base.

The best catalyst to use for this reduction is palladium, in the form of palladium black on charcoal, or palladium on barium sulfate. The palladium stands up well to the chlorine, and can be used to run many batches before it needs to be recycled. Palladium works fine at low pressures of

hydrogen, and can be used with the champagne bottle hydrogenation system pictured in Chapter Eleven.

Important: *The valve on the hydrogen tank is only opened when adding more hydrogen to the bomb. Otherwise, it's kept closed. Failure to do this may result in explosive accidents!*

"The Poor Man's Hydrogenation Device" is a good deal more resilient than a champagne bottle, and it will only accept a feed of hydrogen when its valve is opened. It also is much easier to seal against leaks. So long as the inside surface is coated either with Teflon or high phosphorous electroless nickel, this container for hydrogenation is superior to the champagne bottle used in the following example.

To do the reaction, a champagne bottle of at least 1.5 liters volume is filled with 50 grams sodium acetate (anhydrous) and 700 ml of distilled water. The pH of this solution is then made neutral (pH 7) by dripping in diluted acetic acid. This forms an acetic buffer which prevents the solution from becoming acidic when chlorephedrine hydrochloride is added to it. It also neutralizes the hydrochloric acid formed when the chlorine atom is removed from the chlorephedrine molecule. Then 40 grams of 5% palladium black on charcoal (palladium content 2 grams) is added, and finally 125 grams of chlorephedrine hydrochloride is added.

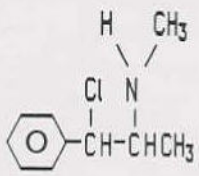
Palladium on $BaSO_4$ catalyst gives a faster reduction, but barium compounds aren't as easily available as activated C. The choice is up to the reader.

Sodium acetate is now on California's list of less restricted chemicals, so it is wise to avoid using sodium acetate as such. This is not the least bit troublesome, and shows just how stupid the people are who put it on the restricted list. To avoid the need for sodium acetate purchases, acetic buffer is made from vinegar and sodium hydroxide. To do this, 700 ml of vinegar is used instead of distilled water. It should be the cheapest grade of white distilled vinegar, because this is likely to be made just by diluting glacial acetic acid with water down to a 5% strength. Then to this 700 ml of vinegar, sodium hydroxide pellets are slowly added until the pH of the solution is around 7. This takes about 22-23 grams of NaOH.

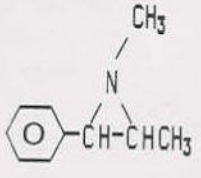
The champagne bottle is then attached to the hydrogen line pictured in Figure 30 in Chapter Eleven, and the air is sucked out and replaced with hydrogen as described in that chapter. Then the pressure of hydrogen is increased to 30 pounds, and magnetic stirring is begun. The solution soaks up hydrogen for several hours, during which time the pressure is maintained around 30 pounds by letting more hydrogen into the bottle.

When absorption of hydrogen ceases after several hours (up to one day for Pd/C), the reaction is complete. The hydrogen valve is turned off at the cylinder, and hydrogen inside the bottle released outside through a line of tubing as described in Chapter Eleven. Stirring is stopped, and the palladium on charcoal catalyst is allowed to settle in the bottle. When it has settled, the solution is carefully poured out of the bottle into a beaker, taking care to try to leave all the catalyst behind in the bottle. The solution is then filtered to remove suspended Pd on charcoal catalyst. The catalyst is returned to the bottle, which is then refilled with a fresh batch, or filled with hydrogen to protect the catalyst. If another batch isn't going to be done soon, rinse the catalyst with some water.

Before proceeding further with the processing of the filtered batch, it is wise to look more closely at the nature of the byproducts produced by this method of making meth. There are twin villains to be dealt with here:



Chlorephedrine



1,2-dimethyl-3-phenyl Aziridine

These substances, or closely related ones, will always be formed when making meth by this method. The chlorephedrine is the result of incomplete reduction to meth, and the aziridine the result of an intermolecular reaction between the chlorine

atom and the nitrogen atom of the chlorephedrine. It is likely that the aziridine byproduct is more easily formed when the bromoephedrine variation of this synthetic route is chosen. There are two things which aid in the formation of the aziridine. They are exposure to strong bases such as lye, and heat. To minimize formation of the aziridine, one first of all aims for as complete a reduction as possible of the chlorephedrine to meth. Next, during processing, one backs off on the heavy duty use of lye, using bicarb instead to neutralize the last of the acid. Finally, the distillation is done as quickly as feasible under vacuum to get the least heat exposure to the unreduced chlorephedrine. Obviously, the first point is the most important.

To proceed, the filtered batch is reacted with lye with strong shaking until litmus paper says that the pH is around 7. Then bicarb is added to finally make the solution basic. One needs only go over pH7 here. Neutralization is complete when fizzing stops when adding bicarb. The fizzing and venting of CO_2 gas is a hassle at this point, but it is worth it to avoid the formation of the aziridine. A 2000 ml flask is a good vessel in which to do the neutralization procedure. One must periodically vent off the built up CO_2 gas after bicarb has been added.

Upon standing after the shaking, a layer of meth floats on top of the water layer. Then 200 ml of benzene or toluene is added, and the jug is shaken again. After standing for a couple of minutes, the benzene-meth layer floats nicely upon the water. This is carefully poured off into a sep funnel, and the benzene-meth layer is poured into a 500 ml round bottom flask. The water layer is discarded.

Next, the product is distilled as described in Chapter Five. Here also is a point at which lazy or under-equipped operators err and thereby leave their product polluted with chlorephedrine. You see, it is next to impossible to completely convert the chlorephedrine into meth. The conversion can be encouraged by using plenty of catalyst, sufficient pressure, and ample reaction time in the bomb, but there will still be some left unreacted. As the catalyst wears out from doing repeated batches, the proportion of chlorephedrine in the product will increase. Only by doing careful fractional distillation, can the chlorephedrine be removed. Chlorephedrine's solubility characteristics are so similar to meth's that it can't be removed by crystallization or rinsing the crystals. When doing the distillation, the meth distills at the usual temperature range. The next fraction which distills is chlorephedrine. Since this chlorephedrine can then be cycled back into the hydrogenation step, it makes both economic and ethical sense to remove it from the product. By skipping the fractional distillation, lazy operators cost themselves an added measure of meth yield from their raw material inputs.

The chlorephedrine free base thusly obtained is too unstable to keep as such. It must immediately be reacted with HCl to form the hydrochloride.

It has become kind of obvious that you wonderful readers out there have been having trouble using the table presented earlier in this chapter, so some examples of the use of other chlorinating agents other than PCl_5 are called for. See *Chemical Abstracts*, Volume 23, page 3453. For example, with thionyl chloride ($SOCl_2$), one puts into a flask 100 ml of chloroform, 100 ml of thionyl chloride and a magnetic stirring bar. The contents are chilled in an ice bath, then 50 grams of ephedrine or racephedrine is slowly added. Stirring in the ice bath is then continued for a few hours, as the reaction of $SOCl_2$ is slower than that of PCl_5. After the reaction time is up, about 500 ml of ether or mineral spirits is slowly added with stirring to precipitate the chloroephedrine hydrochloride. This is filtered out, rinsed with a little cold ether, and spread out to dry as in the previous example.

In the above example, the 100 ml of $SOCl_2$ could have been replaced with 60 ml of PCl_3, or 65 ml of PBr_3.

Along a similar line, a correspondent named Yehuda has written to tell of his experience with the use of trichlorethane as a chloroform substitute. He was not pleased with the results, and wrote with his homebrew method for making your own chloroform. It's interesting and I'll pass it along. In a sep funnel, he puts 35 ml of acetone (hardware store) and either 500 ml of Clorox bleach or 170 ml of

15% sodium hypochlorite solution. This 15%-strength bleach is easily available from swimming-pool suppliers. The sep funnel is shaken vigorously with frequent breaks to vent the gas from the sep funnel. The solution gets pretty warm. The shaking is continued until it stops producing gas. Then let the solution sit for a few minutes for the chloroform produced to settle to the bottom. Drain it off. Then shake the sep funnel again to get a little more chloroform. Total yield: about 15 ml of chloroform. The crude product should be distilled. Then preserve the distilled chloroform by adding .75 ml of ethyl alcohol to each 100 ml of chloroform. The boiling point of chloroform is 61° C. Yehuda also writes to remind the readers that all of the chlorinating agents in this section produce noxious fumes, and should be handled with extreme care. Good ventilation, gloves, protective clothing and eye protection are highly recommended.

Palladium Black on Carbon Catalyst

Since palladium black on carbon catalyst is on the narcoswine's watch list of chemicals, it is wise for the operator to make his own supply. Luckily, this is not too difficult, and gives a catalyst that is fresher and more active than off the shelf catalysts.

To make the catalyst, the chemist first obtains Norit or Darco brand activated charcoal, and washes it with nitric acid. This is done by measuring out about 100 grams of the charcoal, and then putting it into a beaker along with 10% nitric acid. They are mixed together into a watery slurry, and heated on a steam bath or in a boiling water bath for 2 or 3 hours. After the heating, the carbon is filtered and rinsed liberally with distilled water until the last traces of acid are rinsed from it. This requires about a gallon of water.

The acid washed carbon is then transferred to a 4000 ml beaker. A few grams of the carbon sticks to the filter paper and is otherwise lost, but this is OK since the idea is to get about 93-95 grams of carbon into the beaker. 1200 ml of distilled water is added to the beaker, and it is heated with stirring to 80° C. When this temperature is reached, a solution of 8.2 grams of palladium chloride in 20 ml of concentrated hydrochloric acid and 50 ml of water is added. This acid solution of palladium chloride is heated for a couple of hours before it is added, because $PdCl_2$ dissolves slowly in the acid solution. It is not added until all the $PdCl_2$ is dissolved. If $PdCl_2$ dihydrate is used, the amount used is increased to 10 grams.

When the $PdCl_2$ solution has been added and stirred in, 8 ml of 37% formaldehyde solution is added and mixed in. Next, the solution is made slightly alkaline to litmus by adding 30% sodium hydroxide solution to the beaker dropwise with constant stirring. Once the solution has become slightly alkaline to litmus paper, the stirring is continued for another five minutes.

Next, the solution is filtered to collect the palladium black on charcoal catalyst. It is rinsed ten times with 250 ml portions of distilled water. Then after removing as much water as possible by filtration, the catalyst is spread out to dry in a glass baking dish. It is not heated during the drying process since it could burst into flames. When it has dried it is stored in a tightly stoppered bottle and used as soon as possible. This process gives about 95 grams of 5% palladium black on charcoal catalyst.

An alternative to the filtration and rinse is the settle, rinse, and decant procedure used in the $Pd/BaSO_4$ procedure which follows.

Palladium on Barium Sulfate Catalyst

As mentioned earlier, $Pd/BaSO_4$ catalyst will reduce the chloroephedrine to meth a good deal faster than Pd/C. It is useful in other reduction methods in this chapter, so its preparation will be covered here.

$PdCl_2$ is used to make this catalyst, just like the Pd/C catalyst, so some more mention should be given to sources of supply for this very useful material. As I mentioned before, $PdCl_2$ and H_2PtCl_6 are both used in the plating industry. $PdCl_2$ is used to activate plastics so that they can be electrolessly plated. It is also used to electroplate palladium.

The typical bath formulation is 50 gr/1 $PdCl_2$, 30 gr/1 NH_4Cl and HCl to adjust the pH to 0.1 to 0.5. Similarly, a platinum plating bath is mixed up with 10 gr/1 chloroplatinic acid and 300 ml/1 HCl. Companies which supply platers carry these materials. See the *Metal Finishing Guidebook and Directory*. Your library can get it by interlibrary loan if they don't carry it. Turn to the back of the book to the "product, process, and service directory" and look under palladium and platinum to get a list of suppliers. This is far better than dealing with a scientific supply house loaded with snitches, and their prices are much better. For example, 10 grams of $PdCl_2$ was going for just under $60 in 1995. By naming yourself XYZ Plating instead of Joe Blow, easy access to these materials is assured.

To make about 45 grams of 5% $Pd/BaSO_4$, a solution of about 5 grams of $PdCl_2$ dihydrate (the usual form) in 10 ml concentrated hydrochloric acid and 25 ml water is made. The $PdCl_2$ will take a while to dissolve, and heating the solution to about 80° C speeds the process. It can take about 2 hours. Once it is dissolved, set this solution aside.

Then in a 2000 ml beaker, a solution of 600 ml distilled water and 63 grams barium hydroxide octahydrate is made and then heated with stirring to 80° C. When this temperature is reached, 60 ml of 6N sulfuric acid (3M; 160 ml concentrated H_2SO_4 diluted to one liter with distilled water is 3M or 6N) is added all at once to the barium hydroxide solution with rapid stirring. Then some more 6N sulfuric acid solution is slowly added to the barium suspension until it is just acid to litmus.

Now add to this suspension of barium sulfate the $PdCl_2$ solution prepared earlier. Stir it in, then follow it with 4 ml of 37% formaldehyde.

After the formaldehyde has been stirred into solution, make the barium sulfate suspension slightly alkaline to litmus by cautiously adding 30% NaOH solution with constant stirring. Stir for an additional 5 minutes, then let the $Pd/BaSO_4$ catalyst settle to the bottom of the beaker. Decant off the water (it should be clear) and pour fresh distilled water into the beaker. Stir up the settled catalyst to rinse it off. Then let the catalyst settle again, and decant off the clear rinse water. This rinsing procedure is repeated about ten times to get clean catalyst. It can then be poured into the champagne bottle hydrogenation bomb.

Reference

See *Organic Syntheses,* Collective Volume 3.

Direct Reductions

This section deals with the direct conversion of ephedrine, pseudoephedrine, or phenylpropanolamine to meth or dexedrine respectively. This conversion can be accomplished by one of five methods. These conversions are all possible because ephedrine, pseudoephedrine, and phenylpropanolamine are all benzyl alcohols, and benzyl alcohols are the easiest of all alcohols to reduce to the corresponding hydrocarbon.

These methods all have the advantage of being quick and simple, but they also have their unique disadvantages, along with the general shared disadvantage that the starting material must be gathered bits at a time from bottles of pills.

Method 1:
Lithium Metal in Liquid Ammonia Reduction

This is a new method, and is the best one I've seen come down the pike in ages. This procedure was pioneered by a clandestine operator in California. Unfortunately, he was busted because he bought a jug of ephedrine to use as his starting material. Had he been more cautious, and isolated the ephedrine from legal pills, he may well have gone undetected. This method is ideally suited for the rapid production of truly massive amounts of crank. It suffers from the need to use liquid anhydrous ammonia. This is very smelly stuff, especially in the quantities needed to make large amounts of meth. The smell problem means that this method can only be used in countryside locations, preferably in a large shed with a strong breeze passing through it. In this way, the production masters can position the reaction so that they are upwind from the fumes.

The countryside location has the further advantage that tanks of anhydrous ammonia are not at

all out of place in such a location. In every agricultural area, tanks of anhydrous ammonia ply the roads all through the growing season. Farmers use it for nitrogen fertilizer on their crops, especially corn. The local co-op hauls out the tank to the farmer, who then applies it to his crops at his leisure. The implication of this is obvious. A well thought out large scale meth production scheme would center upon renting some nondescript piece of land, planting some corn on it, and then getting a tank of "anhydrous" to fertilize the crop. The resulting product will pay much better than corn. A less well thought out plan would involve getting a tank of anhydrous ammonia from a chemical or welding supplier and taking it to a countryside location for further use. In either case, the ammonia is of the same grade.

This method of making crank is based on the research of Gary Small and Arlene Minnella as published in the *Journal of Organic Chemistry*, Volume 40, pages 3151 to 3152 (1975). The article is titled "Lithium-Ammonia Reduction of Benzyl Alcohols to Aromatic Hydrocarbons. An Improved Procedure." It results in the 100% conversion of ephedrine, pseudoephedrine or PPA in a reaction time of 10 minutes or so.

This method requires the use of the free base rather than the hydrochloride salt. Both the hydrochloride salt and water act as quenchers to the so-called dissolved electrons formed by either lithium or sodium metal in ammonia. This quenching activity could probably be overcome by using more lithium or sodium metal in the reaction, but this is wasteful of a very valuable commodity.

Lithium ribbon currently sells for about $33 per 25 grams, and sodium sticks sell for about $70 per pound. These prices won't break a cooker, but purchasing large amounts of them may result in unwanted attention. Sodium metal can be easily made at home from lye, using DC current in a version of the Down's cell. How to do this will be covered at the end of this section.

A detailed procedure for disassembling lithium batteries to get the lithium metal contained within them is given in *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture.*

This method works equally well with either ephedrine, pseudoephedrine, or phenylpropanolamine as the raw material. A high quality product is obtained that doesn't cause the dreadful hangovers that one will experience from crude undistilled meth made from the HI/red phosphorous method.

The need for free base in this reaction is no real problem. The toluene or mineral-spirits extract of the pills contains the free base in solution. This can just be added directly to the reaction mixture. This in fact saves the added work of bubbling HCl through the toluene or mineral spirits solution to precipitate the hydrochloride. Correspondents also indicate that the ether or THF used as co-solvent in the reaction mixture with ammonia can be entirely replaced with toluene or mineral spirits. Of these two, mineral spirits is preferred, such as, for instance, Coleman camper fuel.

With a supply of free base in hand, it is now time to consider the lithium metal in ammonia reduction method. A very good review of this procedure can be found in the book *Reduction: Techniques and Applications in Organic Syntheses,* by Augustine, pages 98 to 105. At the heart of this method is the fact that lithium metal, or sodium metal, or even potassium metal can dissolve in liquid ammonia to form blue colored solutions that have powerful reducing properties. Such solutions are often referred to as "dissolved electrons." These solutions are stable unless water gets in them, or unless they are contaminated with iron from the ammonia tank. When the free bases of ephedrine or PPA are added to these "dissolved electrons," they are quickly and easily reduced to meth or dexedrine respectively. To do the reaction, a 3000 ml round bottom 3 necked flask is set inside a Styrofoam tub. The purpose of the tub is to provide insulation, because once liquid ammonia gets out of the cylinder it starts to rapidly boil away until the liquid is lowered to its boiling point of -33° C. This boiling can be kept under control by adding dry ice to the tub. If a cylinder of ammonia is being used, it is a good idea to cool it down before use by putting it in a freezer. With a tank from the co-op, this is not practical. To get the liquid ammonia out of the tank or cylinder, ei-

ther clear plastic tubing or rubber tubing is placed over the exit valve of the tank or cylinder, and run into the 3 necked flask. Use of metal, and especially copper, is to be avoided. Then the cylinder is tipped upside down, so that the valve is at the bottom of the cylinder. This assures that liquid comes out rather than gas. Next the valve is cautiously cracked open, and liquid ammonia is run into the flask until it is about ½ full. It will quickly boil away until the volume of the ammonia is down to about 1000 ml, and then more slowly because the ammonia has cooled to its boiling point. Then, wearing rubber gloves and eye protection to keep the fumes out of the eyes, a magnetic stirring bar is placed in the flask, and the tub is put on a magnetic stirrer, and stirring is begun. Now 7 grams of lithium metal is put into the flask. Lithium usually comes in the form of turnings inside a sealed glass ampule under inert atmosphere. It can be used directly as such. If lithium wire is being used, it should be cut into short lengths, and rinsed off with petroleum ether prior to use. The lithium metal quickly dissolves, forming a blue solution. Next, 500 ml of tetrahydrofuran is added to this solution. The purpose of the THF is to aid in the dissolution of the ephedrine or PPA which is to be added next. I can see no reason why anhydrous ether can't be used instead of THF, if it is easier to obtain. Next 55 grams of ephedrine (or 50 grams of PPA) is dissolved in 500 ml of THF or ether, and this solution is added to the lithium in ammonia solution over a period of 10 minutes.

Some care should be taken to make sure that this solution of the free base of ephedrine in solvent is pretty much free of water, which would quench the dissolved electrons. If one has, for example, pill extract dissolved in camper fuel, the simplest way to assure dryness of this solution would be to add a couple of grams of drying agent such as magnesium sulfate to the extracted solution, stirring it around for a few minutes, then decanting the extract solution off the settled drying agent. Magnesium sulfate drying agent can be made by pouring a layer about ¼ inch thick of Epsom salts into the bottom of a glass baking dish, and baking at 450° F in an electric oven for about an hour. Allow the dish to cool to the point where you can handle it with oven mitts, then pour the baked Epsom salts into a glass jar and seal the top.

After allowing the reaction to proceed for an additional 10 minutes, the reaction is quenched by slowly adding water to the ammonia. This is done dropwise at first, and then more rapidly until the blue color disappears from the ammonia solution. The flask is then taken out of the Styrofoam tub, and the ammonia is allowed to evaporate overnight. When the ammonia is gone, some more water is added to the remaining ether (or THF) solution to dissolve the salts of lithium in the bottom of the flask. After separating the water layer, the ether layer is dried using anhydrous sodium sulfate, and the meth or benzedrine is obtained as the hydrochloride salt by bubbling HCl gas through the ether solution as described back in Chapter Five. Distillation is unnecessary because of the lack of formation of byproducts in this reduction. It would just be a colossal waste of ether.

An alternative procedure has become popular among clandestine cookers. In this alternative procedure, liquid ammonia is first added to the reaction vessel, followed by free base dissolved in solvent. Some go so far as to add solvent and then ground up pseudoephedrine or phenylpropanolamine pills. That this works is a testimony to the power of this alternative procedure.

Then to the liquid ammonia containing solvent and ephedrine, pseudoephedrine or phenylpropanolamine, they add lithium metal obtained by taking apart lithium batteries. It takes roughly 5 minutes to dissect a lithium battery and pull out the lithium metal. See *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture* for the details. The extra long life lithium battery contains a bit under .4 grams of lithium metal. They just take apart the batteries one at a time, and add the lithium metal to the pot. They continue adding the lithium until the solution takes on a blue color, rather than the blue color from the dissolving lithium being rapidly sucked up by the solution. Once the blue color persists for a bit, the addition is complete. Then after a few minutes fur-

ther reaction time, the water can be added as in the first example, and processing done as usual.

This variation probably works better because one isn't relying upon the stability of a pot full of dissolved electrons. As they form, they can just go on to react with ephedrine. In this way, the reaction becomes more tolerant to the presence of potential quenching agents. People like this variation.

One may justifiably ask now, "How is this such a great mass production method, when one is only getting 50 grams of product out of each batch?" The answer is that the work can easily be organized so that one batch after another is quickly turned out by this method. Each individual batch only requires a few minutes of attention. After one flask is filled with ammonia, another may be set up and filled, resulting in a virtual assembly line procedure.

Before moving on, there is a possible complication which must be addressed. This is the possibility that a tank of ammonia may only be putting out ammonia gas, rather than spewing liquid. This is no great hassle. In that case, the 3000 ml 3-necked flask is well packed in dry ice, and rubbing alcohol poured on the dry ice to create a very cold bath. When the ammonia gas hits the very cold flask, it will be condensed to a liquid. This may actually be a better procedure because it will assure that the ammonia does not have dissolved iron in it from the tank. Iron interferes with some lithium in ammonia reductions. I am not sure whether that is the case with this particular reaction. Input from serious experimenters is welcome.

Sodium metal can be used with just as good of results as lithium. The higher atomic weight of sodium requires that 23 grams of sodium metal be used instead of the 7 grams of lithium used in the preceding example. Potassium metal probably works too, but is not so common as sodium metal.

Before leaving this topic, a couple of issues should be addressed. Issue number one is the very bad effect that the presence of water in the reaction mixture has upon the success of the reaction. Lower levels of water will quench the dissolved electrons, preventing that beautiful blue color from forming in the reaction mixture. This low level of water contamination can probably be overcome by using more lithium metal.

At higher levels of water contamination, the lithium metal just seems to fizz away in the liquid ammonia, forming hydrogen gas and lithium hydroxide from the water contaminant. At this level of water contamination the batch is guaranteed to be a failure.

Where is this water coming from? Assuming that the pill extract has been dried of water, either by use of a drying agent or distilling the water off the extract as the water-solvent azeotrope, then the water can only be coming from the "anhydrous ammonia" used. It must not be so anhydrous, so get another source of anhydrous. Low levels of water contamination can also come from absorption of water from the air. Liquid ammonia will pull water out of the air. This method shouldn't be done on very humid days.

Issue number two is the smell produced by this reaction. If this is going to be tried in any place other than a remote country location, the ammonia fumes will alert anyone nearby to the goings on. They'll also knock you over without really good ventilation. The fumes can be held down during the course of the reaction by cooling in a dry ice bath, but after the reaction, the liquid ammonia must be allowed to evaporate away. There comes a cloud of ammonia fumes! On a small scale, these can be sucked up with an aspirator, and flushed down the drain. Simply run some clear plastic tubing from the aspirator to the top of the reaction vessel, and turn on water flow to the aspirator. This will control the smell from small scale batches.

Another way to address the strong smell and actual physical assaultiveness of liquid ammonia is to replace it as the material in which the dissolved electrons are generated. The most clandestine suitable substitute for liquid ammonia is ethylenediamine. This substance was mentioned earlier in the Knoevenagel reaction section. It can be obtained from laboratory chemical suppliers for around $20 per liter. It is also used in the electroplating industry as a complexor in nickel stripping solutions. Component A of these strips is m-nitrobenzenesulfonic acid, component B is ethylenediamine.

This industrial grade of ethylenediamine will cost about $20 per gallon. See the *Metal Finishing Guidebook and Directory* under "stripping solutions," for suppliers.

The industrial grade of ethylenediamine should first be fractionally distilled (boiling point 116° C) to see if there is a significant amount of water in the material. We do this by looking for the toluene-water azeotrope, which boils at 85° C. Mix two parts ethylenediamine with one part toluene, and fractionally distill at atmospheric pressure through a good fractionating column, such as a claisen adapter packed with broken glass. If an insignificant amount distills at the azeotrope temperature, there isn't much water. If a fair amount does distill, the solution can be dried by distilling off the azeotrope. The toluene left in the mixture can just be left there as co-solvent for the reduction.

If only a small amount of water was found in the industrial grade ethylenediamine, then in the next runs drying can be done by letting the ethylenediamine sit in contact with KOH pellets for about half a day, then distilling the dried ethylenediamine. The amine has to be dry, because water really fucks with this reaction! Once distilled, the ethylenediamine should be stored in a tightly stoppered bottle to prevent absorption of water from the air.

Then to use ethylenediamine in this reduction, I would put ethylenediamine into the reduction flask in place of liquid ammonia, along with a stir bar. Then I would add the co-solvent, followed by a dried solution of the free base of ephedrine, or whatever. The free base solution should be dried by distilling off the water-solvent azeotrope, or by use of magnesium sulfate drying agent.

With the materials in the flask, I would next flush the reduction flask with nitrogen. This is because oxygen is a quench to the dissolved electrons, and we don't have the boiling liquid ammonia here to keep oxygen away from the reaction. One can get cylinders of nitrogen, or argon, at the welding supply shop. Once the flask has been flushed, I would begin taking apart lithium batteries, and tossing the lithium metal into the reaction mixture with magnetic stirring. The blue color from the dissolving lithium will quickly be sucked up at first, then more slowly. Once the royal blue color persists in the solution, then the end point will have been reached, and no more lithium metal needs to be added to the reduction mixture.

Once the end point is reached, stop the slow flow of nitrogen into the flask and allow atmospheric oxygen to quench the dissolved electrons. When the blue color disappears, water can cautiously be added to the reduction mixture. Stir it in!

Then pour the reduction mixture into 10 volumes of water, and shake. After shaking, the co-solvent-amphetamine layer will float on top of the water. Separate it off. Then extract the water with about 200 ml more toluene. Separate it off too, and add this extract to the first extract.

Now the combined extracts should be washed a couple of times with water. This will remove most of the ethylenediamine. One can next either distill the extracts to get pure amphetamine free base, which is then bubbled with dry HCl, or the extracts can be bubbled directly with dry HCl after they have set for a while to allow entrained water to settle out of solution. This method should work quite well, and not stink to high heaven.

If the clandestine cooker wants to substitute sodium metal for lithium, he may find it difficult or risky to buy sodium metal through standard channels, so a procedure to manufacture sodium metal from household items is of definite value. A process called the Downs cell has been in use since the early days of this century to make sodium on an industrial scale. In its early versions, sodium hydroxide (lye) was the raw material used, but in later versions this was replaced with salt. By using salt instead of lye, another valuable material — chlorine gas — was also obtained. In this procedure the earlier version will be used because we don't want the poisonous chlorine, and because lye has a much lower melting point.

Some basic precautions must be followed with this cell. The last thing someone wants on their skin or in their eyes is molten lye or sodium metal, so gloves, protective clothing, and a face shield must be worn. Also, molten lye gives off fumes,

especially when a current is being passed through it, so good ventilation is mandatory.

To make sodium metal, a steel or copper pan about 1 pint in capacity is placed on a stove burner. A gas stove shouldn't be used here because of its open flame. Also, we want to take advantage of the insulation on the electric heating element. An aluminum pan can't be used, nor is Teflon coating acceptable.

Now, into this pan put lye until it's ¾ full. Begin heating this lye at medium to medium-high heat, and melt it. More lye can then be added to get the volume of lye back to about ¾ full.

When the lye has melted, the standard DC electrochemical cell wiring is done. A graphite anode is inserted into the molten lye. It should be about 1 inch away from the side of the pan, and it must not touch bottom, or a short will occur. Clamp it in place with a ringstand and insulated clamp. Make an electric contact with the graphite anode by wrapping some bare copper wire around the top of the graphite anode, and twisting it down tight. Run a wire from the positive pole of the DC source, through an amp meter, and to the graphite anode. Then run wiring from the negative pole of the DC source to the pan near where the graphite anode is located.

Now turn on the DC power source. The best source would be a 0-110 volt transformer. Also workable would be a DC arc welder with variable voltage control. One could also hook a couple of car batteries in series to raise the applied voltage to 24 V. I have heard from reliable sources that 12 volts isn't enough to push current through the molten lye.

Now turn up the applied voltage from the DC source until a current of one or two amps flows. This is enough current to give a good rate of sodium production. How much current can be passed is largely limited by how big the graphite anode is. As in so many other things, bigger is preferable to smaller. The molten lye obeys Ohm's law, so increasing voltage gives higher current flow.

As the current flows, little globules of shiny metallic sodium should start floating up to the top of the molten lye. They will mostly show up along the edge of the pan near the handle. Sodium is very light, so it floats. As they appear, scoop them up with a stainless steel spoon. Separate them from the molten lye also scooped up by rolling them around in the spoon, and once separated, pour them into a jar of mineral spirits. Sodium metal reacts rapidly with air to make sodium peroxide, so this should be done as fast as they appear. The mineral spirits will protect the product. It's important to keep the lye out of the product, as this would mess up its intended use in the reaction. It's probably quite possible to separate the two later by melting the sodium (it melts below the boiling point of water) and pouring it off the entrained lye, but this hazardous procedure is best avoided in the first place.

Current flow is continued until one has as much sodium metal as needed. The serious experimenter will want to try using iron or steel as the anode if graphite can't easily be found. The serious experimenter will also note that the amount of current that he can pass through the molten lye is related to the size of the anode used. After a certain current density, the electricity gets wasted by fizzing at the anode, which produces water and hydrogen.

Before leaving this topic, another warning should be included. When scooping up the sodium metal with the spoon, don't make contact with both the anode and the pan at the same time! You will short the cell, and make sparks that will startle you to no end! This could lead to spills and messes and burns! Be careful!

Method 2:
Wolff-Kishner Reduction

This method of directly reducing ephedrine, pseudoephedrine, or phenylpropanolamine to meth or dexedrine uses hydrazine hydrate as the reducing agent. The Wolff-Kishner reduction is generally used to deoxygenate ketones to the corresponding hydrocarbon, but in this case, it can be used on these particular substances to reduce them. No doubt this is because the benzyl alcohol grouping has a ketone nature due to tautomerism.

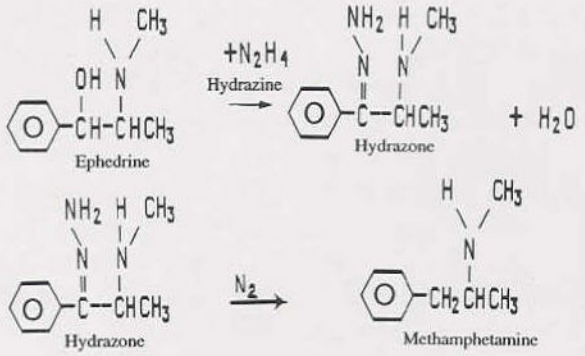
I came across the reference for this reduction several years ago, but have been unable to find it

since. I've also received mail from the pen from cookers telling me that they have had some success with this reduction. This method should be considered a minor pathway to meth, as there are better and more convenient routes.

The Wolff-Kishner reduction has the advantage of not producing great plumes of stink. It could likely be done in an urban setting without arousing the suspicions of nosey neighbors. Further, the reactants are only moderately expensive, and not tightly controlled at present. Fair amounts of product can be turned out at a rate of one batch per day.

The disadvantages of this method are twofold. First, hydrazine is a carcinogen. The chemist must wear gloves while doing the reaction, and do a careful clean-up when finished. If any should be spilled on the skin, a serious, prolonged, and immediate shower is called for. Care must further be taken that the fumes of hydrazine are not breathed in, as this could cause the same problem. Ever try giving your lungs a shower? The other disadvantage to using this method is that the free bases must be used. This necessitates the free basing and distillation procedure described in Method 1.

The mechanism by which this procedure works involves first the formation of a hydrazone by reaction between the ephedrine and hydrazine. Then at the high temperatures at which this reaction is done, the hydrazone loses nitrogen (N_2) to form meth. This is illustrated below.



To do the reaction, a 3000 ml round bottom flask is placed on a buffet range, and then 1500 ml of diethylene glycol and 336 grams of KOH (potassium hydroxide) pellets are put in the flask. Next a condenser is attached to the flask, and water flow is begun through it. Gentle heating of the flask is now begun, with occasional swirling of the flask to try to dissolve the KOH pellets. The operator must be ready here to quickly remove the buffet range, because once the solution warms up, and the KOH pellets start to dissolve, a great amount of heat is released which could cause the solution to boil wildly and squirt out the top of the condenser. Since diethylene glycol has a boiling point of 245° C, this would definitely not be good stuff to be splashed with. Eye protection is, of course, necessary. The heat source is periodically removed, and then reapplied until the dissolution of the KOH pellets is complete.

Once the KOH pellets have dissolved, the heat is removed, and the temperature of the solution is allowed to fall to about 80° C. Then 300 ml of hydrazine hydrate (85% to 100% pure material is OK) and either 303 grams of PPA free base or 332 grams of ephedrine free base is added to the flask. The condenser is then immediately replaced, and the mixture is heated with great caution until any exothermic (i.e., heat generating) reaction has passed. Then stronger heat is applied to maintain gentle boiling for one hour.

Now heating is stopped, and as soon as boiling ceases, the condenser is removed, and the flask is rigged for simple distillation as shown in Figure 11 in Chapter Three. The stillhead should have a thermometer in it reaching down into the middle of the liquid mass in the flask. A cork or rubber holder for this thermometer is unacceptable because hydrazine attacks these materials. The holder must be made of all glass only.

Now the heat is reapplied, and distillation is commenced sufficiently slowly that the froth does not rise out of the flask. Froth can be broken up by occasional applications of weak vacuum, as mentioned back in Chapter Five. When the temperature of the liquid has reached 200° C or so (around 200 ml of distillate will have been collected by that point), the heating is stopped. Once boiling ceases, the stillhead is removed, and the condenser is rein-

serted into the flask. Now heat is reapplied, and the mixture is boiled gently for 3 additional hours.

The reaction is now complete, and it is time to get the product. The heating is stopped on the flask, and once it has cooled down, the contents of the flask are poured into 2000 ml of water. The 200 ml of distillate obtained earlier is also poured into the water. This mixture is stirred to get the hydrazine out of the meth layer which floats on the top, and into the water. The solution of KOH in water makes the water fairly hot. Once it has cooled down, 500 ml of toluene is added, and the mixture is shaken. A one gallon glass jug is a good vessel to do this in. The top layer of meth dissolved in toluene is then separated and distilled as described earlier. The yield is 250 to 275 ml of meth. If a careful fractional distillation is not done, the product may be contaminated with a small amount of hydrazine. This is definitely not good, and may be avoided by shaking the separated meth dissolved in toluene layer with a fresh portion of water.

Method 3:
Direct Reduction of Ephedrine With Palladium

These methods are pretty similar to the indirect reduction of ephedrine, racephedrine, pseudoephedrine and PPA presented earlier. The difference here is that these variations on that theme are one-pot methods. For example, chlorination and reduction can be done simultaneously in a solution containing dry HCl gas and palladium in a hydrogenation bomb. Other variants use sulfuric, perchloric or phosphoric acid to either first form an ester with the ephedrine, or whatever, which is then reduced to meth in the hydrogenation bomb, or these same substances act as promoters to cause the direct reduction of the benzyl alcohol (ephedrine, or whatever) to the meth or benzedrine. The exact mechanism of how this actually works is a matter for debate in the patent literature.

These direct routes have the advantage of using very common materials as feed stocks. The various chlorinating agents given in the indirect-reduction section aren't particularly common lab chemicals. Also, chloroform is becoming less commonly used in industry with the new ozone-depletion rules.

There are a few drawbacks to this method. First and foremost, in one of these procedures the contents of the bomb must be heated to about 80-90° C during the reaction. This leads to the danger that the champagne bottle hydrogenation bomb may crack and burst due to heat stress. This is a possibility even if the outside of the bottle is coated with fiberglass resin. Another problem is the high cost and suspiciousness of purchases of the various palladium catalysts used in these methods. This can be avoided by getting one's palladium chloride from the plater's supply outfits mentioned earlier in the palladium-catalyst section. One note on this source of palladium. Platers tend to use archaic or weird technical language. For example, a company may offer what he calls 60% $PdCl_2$. This refers to the Pd content of the $PdCl_2$, and indicates that it is actually quite pure.

A big improvement to this procedure is to use "The Poor Man's Hydrogenation Device" detailed in the second edition of *Practical LSD Manufacture* and also in *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture*. With this improvement, the danger of breaking champagne bottles under heat is removed.

For this particular application, I would use a fire extinguisher having a bottle made out of a non-magnetic stainless-steel. The technical sheet that comes with the extinguisher will state what the bottle is made out of, and a magnet will tell you if it is a non-magnetic alloy. A non-magnetic stainless-steel will pass a magnetic field, so magnetic stirring will be possible.

The reason for choosing stainless steel over aluminum in this application is the corrosive nature of the hydrogenation mixtures used in these direct reductions. Acetic acid is the solvent used in these procedures, and it will dissolve aluminum. The promoters used in these procedures, either HCl, sulfuric or perchloric acid, all eat away at aluminum as well. If the coating should fail inside the bottle, then a hole could be rapidly eaten through the pressure bottle if it were made of aluminum.

To coat the inside of the stainless-steel pressure bottle, one has the choice of Teflon and Teflon-based paint, or high phosphorous electroless nickel. To pursue this latter alternative, one goes to the Yellow Pages and looks under electroplaters. Ask them if they do electroless nickel, if they have a high phosphorous (phosphorus content in alloy of about 12%) plating bath, and if they are set up to plate over stainless-steel. To plate stainless, a Wood's nickel strike must first be applied to the part.

If they answer yes to the above questions, you are set to go. Say you want the plating to make the extinguisher match your techno décor or some such cock and bull story. I work in this business and I've heard it all by now. This request will raise no eyebrows. It's important that the inside of the extinguisher be plated well, so an auxiliary electrode placed inside the bottle will have to be used during the Wood's nickel strike to get that surface activated. Make sure you specify that. Also ask that threads be masked off so that you can screw the top back on the extinguisher after plating. Also ask for a plating thickness of about one thousandth of an inch for good protection.

Let's look at variation number one of this procedure.

To do this reaction, the chemist first prepares palladium black catalyst. This is done as follows: In a 2000 ml beaker, 50 grams of palladium chloride is dissolved in 300 ml of concentrated hydrochloric acid (laboratory grade, 35-37%). Once it has all dissolved, it is diluted with 800 ml of distilled water. Next, the beaker is nestled in a bed of ice that has been salted down. This is an ice-salt bath. The contents of the beaker are stirred occasionally, and once it is cold, 300 ml of 40% formaldehyde solution is added with stirring. After a few minutes, a cold solution of 350 grams KOH in 350 ml distilled water is added slowly over a period of 30 minutes. The palladium solution must be vigorously stirred during the addition. Now the beaker is removed from the ice and warmed up to 60° C for 30 minutes with occasional stirring during the heating.

When the heating is complete the beaker is set aside to cool and to let the catalyst settle. Once the catalyst has settled, the chemist pours off as much of the water solution as possible, without losing any catalyst. Then fresh distilled water is added to the beaker, the catalyst is stirred up to wash it off, then the chemist lets it settle again and pours off the water. This washing is repeated a total of six times. Finally, the catalyst is suspended in a bit of fresh distilled water, and filtered, preferably through sintered glass to be sure of catching all the catalyst. Any catalyst still clinging to the sides of the beaker are rinsed down with water and poured in with the main body of catalyst. It is wise to rinse off the catalyst again with still another large portion of water while it is in the filtering funnel. This process yields 31 grams of palladium black catalyst, once it has dried. It is important that the catalyst be allowed to dry completely, because the presence of water in the reaction mixture is to be avoided.

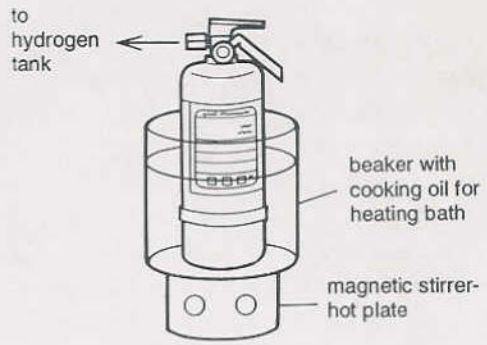


Figure 34

With a supply of catalyst on hand, the chemist can move on to production. To begin, 600 ml of glacial acetic acid is poured into a 1000 ml beaker. Now the glassware is set up as shown in Figure 19 back in Chapter Five. The glass tubing is led into the acetic acid, and bubbling of dry HCl gas into the acetic acid is begun as described in that chapter. It is a good idea here to magnetically stir the acetic acid solution during the bubbling. The whirlpool formed will help the bubbles of HCl gas to dissolve in the acetic acid, rather than escape

and waft away on the breezes. This bubbling is continued until the acetic acid solution has gained 30 grams in weight.

Next, this acetic acid-HCl mix is poured into the 1.5 liter hydrogenation device along with 60 grams of either ephedrine, pseudoephedrine or PPA (sulfate or HCl salt OK for any of these), and 50 grams of palladium catalyst. Since the mixture is going to be magnetically stirred, a magnetic stirring bar, of course, is put in the bottle. Now the apparatus is set up as shown in Figure 30 in Chapter Eleven. The air is sucked out of the bottle as described in that chapter, and then hydrogen is put into the bottle at a pressure of 30-40 pounds per square inch. Stirring is then begun, and the oil heating bath warmed to the 80-90° C range. Hydrogen absorption begins, and fresh hydrogen is put into the bottle to keep the pressure in the prescribed range. In an hour or two, hydrogen absorption stops, and the reduction is complete.

The heating is then stopped, and the stirring is halted. The hydrogen is vented outside as described back in Chapter Eleven, and the product solution is carefully poured out of the bottle, taking care not to pour out the palladium catalyst. If any comes out, it is filtered, and the palladium returned to the bottle for the next run.

The product mixture is poured into a 1000 ml round bottom flask along with a few pumice chips, and the glassware is set up as shown in Figure 11. The chemist distills off 500 ml of acetic acid (b.p. 118° C). This acetic acid can be used over and over again just by bubbling some more dry HCl through it to dry the solution and to recharge its HCl content.

The residue left in the distilling flask has the product. Once it has cooled down, lye water is added to it, and it is shaken vigorously. The solution should be strongly basic. Now toluene is added, the top layer separated off, and this top layer is distilled as described so often in this book to yield a little over 50 grams of meth (or dexedrine if PPA was used). This is about 95% yield.

If there is a high boiling residue in the flask, it's likely to be the acetyl amide of meth or dexedrine formed by heating with the acetic acid. This can be hydrolyzed by boiling with concentrated hydrochloric acid, then making the solution basic with lye, and extracting out the product as described in Chapter Five.

You thought that method was a hassle? You ain't seen nothing yet. Check out the method given in *Journal of Medicinal Chemistry,* Volume 9, pages 966-67. In this variation, the phosphoric acid ester of ephedrine, pseudoephedrine is reduced to meth or phenylpropanolamine is reduced to dexedrine by hydrogenation with Pd on carbon catalyst. The phosphoric acid ester isn't made just by reacting the ephedrine with phosphoric acid. No, that would be too easy. Instead, phosphorus oxychloride($POCl_3$) must be used. The free base of the ephedrine, or whatever, must be used to make the ester. Here's how they did it:

165 ml of freshly distilled ephedrine free base was put in a flask fitted with a drying tube. Then with stirring, 300 ml of triethylamine was added along with 5000 ml of benzene. The triethylamine absorbs HCl given off in the reaction. The benzene could be replaced with toluene. Water in the reaction mixture must be avoided. Then with vigorous stirring, 100 ml of $POCl_3$ dissolved in 500 ml of anhydrous benzene is added dropwise at such a rate that the temperature stays below 50° C. Stirring was then continued for 4-5 hours.

Next, the reaction mixture was filtered and the solvent was removed under a vacuum. The residue left in the flask was next extracted with boiling petroleum ether. Several portions of petroleum ether were used to do the extraction, a total of about 6000 ml.

The petroleum ether extracts were next combined and put in a freezer for 24 hours. The product, 2-chloro-3,4-dimethyl-1,3,2-oxazaphospholine 2-oxide precipitates out in the cold, and it is filtered out. It is stable only in a dry inert atmosphere. The yield is about 165 grams.

This product is then put in a flask, along with 5 ml of 1N hydrochloric acid for each gram of the 2-oxide. 1N hydrochloric acid is made by adding 11 volumes of water to one volume of concentrated hydrochloric acid. This reaction mixture is heated on a hot water bath for one hour. The reaction

mixture becomes clear. It should be decolorized with some activated charcoal, filtered, and then the solution is evaporated away under a vacuum, with a warm water bath no hotter than 40-45° C to speed along the evaporation. The residue is the phosphate ester of ephedrine. This material will hydrogenate to meth.

The residue can be dissolved in 15 ml of ethanol for each gram of residue, and then add one tenth gram of 5% Pd on C for each gram of residue. It is hydrogenated at room temperature, and they claim no more than atmospheric pressure of hydrogen was required to get 85% yield of meth in about 2 hours.

The hydrogenation mixture was filtered to remove catalyst, and the filtrate was evaporated under a vacuum. The residue was dissolved in water, then the water solution was made strongly alkaline by adding sodium hydroxide solution. The free base was then extracted out using toluene, and this solution can either be distilled to yield the pure free base, or the toluene extract could be directly bubbled with dry HCl to get crystals of meth hydrochloride. That was some pain in the ass, huh?

It's time to move on to some good and convenient hydrogenation methods, isn't it?

See *Chemical Abstracts* from 1949, column 1025 to 1026. Here meth was made in 95% yield by placing 40 grams of ephedrine HCl in 900 ml of glacial acetic acid with 47 grams of 84% sulfuric acid and 10 grams of palladium. Concentrated sulfuric acid can't be used because it breaks down under hydrogenation to H_2S, which poisons the catalyst. 84% sulfuric acid is made by taking 84 grams of concentrated sulfuric acid (46 ml) and adding 16 ml of water. The palladium catalyst specified is Pd wool, but Pd black will work as well. This solution was hydrogenated at about 30 pounds per square inch at room temperature for a few hours until hydrogen absorption ceased to give meth with no change in its stereochemistry.

If the hydrogenation is done at room temperature, a champagne bottle would be good enough to do the reaction in. One has the option of heating the hydrogenation mixture to get a much faster reduction time. See *Chemical Abstracts,* Volume 38, column 1219 for this example. In this case, the heated hydrogenation should be done in "The Poor Man's Hydrogenation Device," preferably one with a non-magnetic stainless steel bottle coated on the inside with Teflon or high phosphorus electroless nickel.

I'm pretty sure that the mechanism whereby this hydrogenation works is that the sulfuric acid acts as a catalyst for the formation of an ester between the acetic acid solvent and the alcohol group in the ephedrine, pseudoephedrine or PPA. This benzyl ester then undergoes what is termed hydrogenolysis to yield meth or dexedrine. See *Organic Reactions*, Volume 7, for an article on hydrogenolysis of benzyl esters. This reaction mechanism is the basis for the Fester Formula, revealed in *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture*, and also the Advanced Fester Formula, given later in this chapter.

Workup of the reaction mixture to isolate the meth product consists of first either letting the catalyst settle to the bottom, or filtering it out. It can be reused and when it wears out it can be reworked like platinum catalyst.

Now the acetic acid solution containing the meth is poured into a distilling flask, and with the aid of vacuum, the acetic acid is distilled off. If the vacuum isn't really high, and if the condenser and receiving flask are pretty cold, then most of the acetic acid should be recovered for reuse.

Then to the residue left in the flask after most of the acetic acid has distilled away, add a 20% solution of lye in water until the mixture is strongly alkaline. This would be best done in a sep funnel, with shaking between adds of lye solution. You now have meth free base. When the solution has cooled down a bit, add 200-300 ml of toluene and shake to extract the meth into the toluene, which will float on top of the water after the shaking stops. Separate this top layer of meth in toluene, and after any entrained water has settled out, bubble HCl gas through it to get meth hydrochloride crystals. Filter them out, and air dry as usual to get the product.

My commentary on this procedure? That sure is a lot of acetic acid used, a lot of expensive catalyst

used, and heating the hydrogenation bomb is a hassle that prevents the use of glass hydrogenation vessels. Also that need for distilling off all that excess acetic acid at the end of the hydrogenation is a lot of work, and it requires that one have a set of distillation glassware to do the hydrogenation. Let's get around all that.

This hydrogenation works by way of the hydrogenolysis of the acetic acid ester of the benzyl alcohol of the ephedrine, or whatever. This hydrogenolysis is very easy, even easier than reducing a double bond by hydrogenation. It's the formation of the acetic acid ester that requires the heating of the hydrogenation mixture. Also, ephedrine hydrochloride isn't very soluble in cold acetic acid. That's why such a large amount of glacial acetic acid is used in the old standard recipes found in the patent literature. Let's get around all the practical cooking problems in one broad stroke just by preforming the acetic acid ester prior to hydrogenating the solution.

Into an appropriately sized Pyrex glass vessel put however much ephedrine hydrochloride you are using in the batch. Then add 5 or 6 ml of glacial acetic acid for each gram of ephedrine hydrochloride to the glass vessel. This is one quarter of the amount of glacial acetic acid needed for the standard procedure. Begin heating this mixture with stirring. An oil bath is best as a heating medium. Warm the solution to around 90° C. As the mixture warms up, the ephedrine hydrochloride dissolves in the acetic acid. When it has mostly all dissolved, drip in with stirring two or three drops of concentrated sulfuric acid for each gram of ephedrine hydrochloride used. Heat to around 90° C for a period of two or three hours. The acetic acid ester of ephedrine will be made. It will stay in solution when the mixture cools down.

When the heating period is over, allow the solution to cool down to room temperature. Then with stirring, one drop of water for each three drops of concentrated sulfuric acid is added to the mixture. This will prevent the breakdown of the sulfuric acid to hydrogen sulfide during the hydrogenation. The sulfide would poison the catalyst.

Now one is ready to hydrogenate the mixture. A champagne bottle, "The Poor Man's Hydrogenation Device," or even a filtering flask with a rubber stopper wired into the top can be used as hydrogenation vessel. Pour the ester reaction mixture into the hydrogenation vessel, add a stir bar, and one gram of palladium catalyst for each 8 to 10 grams of ephedrine used. This is a reduction of half or so of the amount used in the more dilute solution in the standard procedure. One could probably get away with using even less catalyst.

Suck the air out of the hydrogenation vessel, and replace it with hydrogen. With a champagne bottle or "The Poor Man's Hydrogenation Device," around 30 pounds of hydrogen pressure can be applied. With a filtering flask, a few pounds of pressure in excess of atmospheric pressure can be used. Hydrogenate until the uptake of hydrogen by the catalyst stops. This shouldn't take too long, even at low pressure. That hydrogenolysis goes very easily. Stirring the mixture by use of magnetic stirrer is required to bring the catalyst into contact with hydrogen.

When the hydrogenation is complete, allow the catalyst to settle out, and decant the solution off the catalyst. It can be reused in the next batch! Filter the decanted solution to get back all the catalyst, as palladium is getting expensive these days.

Now the hydrogenation mixture can be put into a large sep funnel, and 10-20% lye solution can be added slowly with shaking to make the mixture strongly alkaline. This will make a good amount of heat, so take a break if need be.

When enough lye solution has been added to make the mixture strongly alkaline, the meth free base will float on top of the water solution. When it is cool, extract out the meth with 5 to 10 ml of toluene for each gram of ephedrine used. Separate off the toluene layer, wash it with a little water, then let the separated toluene layer set in a beaker for a few hours to settle out any entrained water. Pour the meth in toluene solution into a fresh beaker, and bubble dry HCl through the solution as usual to get crystals of meth hydrochloride which are filtered out, rinsed with clean toluene, and

spread out to dry on a plate. The yield should be about the same as with the standard procedure — around 90%.

Since the fourth edition of this book came out in 1996, I've been hearing the narcoswine rant about some Nazi Meth recipe, and this Nazi Meth recipe has been a topic of speculation on the Internet for some time. These narcoswine aren't exactly clear as to what they mean when talking about this recipe. Could they be talking about the lithium metal reduction in liquid ammonia? That wouldn't fit because that reduction technique was invented long after the Nazis went defunct. The following recipe would fit, as it dates to the correct time period, and was invented by guys named Karl Kindler, Erwin Karg and Ernst Scharfe, and published in *Berichte der Deutschen Chemischen Geshellschaft*. That fits, but since when do narcoswine and reality have anything in common?

See *Chemical Abstracts*, Volume 38, column 1219. In this variation, the 84% sulfuric acid is replaced with 70% perchloric acid. The specified catalyst is palladium on barium sulfate, but I would think any palladium catalyst would work. The reaction mechanism would be the same as in the earlier example, with the perchloric acid acting as catalyst to form the ester between the acetic acid solvent and the ephedrine, and this ester then gets reduced to meth. Care must be taken not to use too much perchloric acid, as this over-hyped-up reaction mixture would also reduce the benzene ring to cyclohexane.

The hydrogenation using perchloric acid as the activator is complete in about ¾ of the time required when using sulfuric acid, but other than that, the reaction is pretty much identical to the earlier example. It can be done at room temperature, or heated to around 80° C to speed up the hydrogenation. Workup of the hydrogenation mixture to get the meth product is the same as in the earlier example. My opinion is that since sulfuric acid works just as well, but a little bit slower, one is better off using the more common acid : sulfuric. The reference has been cited if you want to read the details.

In my book, *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture,* I revealed the Fester Formula. This is an electrocatalytic hydrogenation where one takes an ingot of palladium metal, anodizes the surface of the metal in dilute sulfuric acid to coat the surface of the metal with black palladium, and then in an electric cell this ingot of palladium hydrogenates the acetic acid ester of ephedrine, pseudoephedrine or PPA by means of electrically generated hydrogen at the surface of the palladium ingot. This is a good and very low-profile method for making stash quantities (a gram or two at a time) of meth. It does, however, have a few drawbacks.

The primary drawback to this method is that the small size of the ingot (submerged face surface area about 6 square cm) limits the batch size to a gram or two at a time. That is fine for stash, but not production of sizable amounts. Drawback number two is the kind of low specific catalytic activity of the anodized palladium metal ingot. These two roadblocks to production using this method are now addressed here with the Advanced Fester Formula.

Starting back in the middle '70s, it was discovered that a black palladium electroplated in a very thin layer upon graphite produced an electrode for electrocatalytic hydrogenations that was much more active than a simple piece of palladium metal. In the scientific literature, the solid piece of metal is termed a mass electrode. Handling and using the mass electrode (an ingot of Pd) is simpler than Pd electroplated onto graphite, but the higher activity and unlimited size of the plated graphite has almost completely displaced the mass electrode in truly modern methods. One has, in effect, Pd on carbon with the plated graphite electrode.

Electroplating graphite with a thin layer of black palladium is no great challenge. Your Uncle's day job is as a chemist in the electroplating industry, so count on me to walk you through the procedure with no hitches. Very small amounts of palladium are consumed in the plating of this electrode, so this is a very economical method as well as being stealthy and clean in both product and waste generated. The very small amount of Pd catalyst used

requires that the ephedrine feed for the reduction be very pure to avoid fouling out the catalytic surface.

For some examples to read using this production method, see *Journal of Applied Electrochemistry*, Volume 5, pages 125-28 (1975) and *Electrochimica Acta*, Volume 21, pages 449-59 (1976), and for a 50 amp scale up, see *Transactions of the SAEST*, Volume 13, pages 161-67 (1979). The author of all these articles is Krishnan, a pioneer in the use of palladium plated graphite electrodes.

Let's take the case of a piece of graphite with a surface area of about 100 square cm on a side. We only consider the surface area of the graphite which will face the anode in the electric cell, because the current can't reach the back side. See the discussion earlier in Chapter Ten for sources of graphite bars, which can be cut as desired. Graphite rods could also be used; just hook a bunch of them together using copper wire to make electrical contact with the rods. The graphite must be clean to plate properly. Any grease on them should be removed by scrubbing with hot dish soap water, then after rinsing and drying, a solvent rinse such as acetone would ensure grease and dirt removal. Have clean hands or wear rubber gloves. Foul and polluted graphite can be cleaned by soaking in hydrochloric acid (10%), rinsing and letting dry. This would remove metal contaminants, but good unused graphite will be pretty much free of these materials.

We're now ready to electroplate the graphite with that thin layer of black palladium. The plating solution is made up by starting with 350 ml of 3N hydrochloric acid. This is roughly 90 ml of concentrated (36%) HCl diluted to 350 ml with water. To this acid we add .35 grams of palladium chloride and 1.75 grams of ammonium chloride, and stir until the $PdCl_2$ has all dissolved to give a reddish brown colored solution. The palladium chloride can be purchased as such from those suppliers of precious metal salts to electroplaters mentioned earlier, or from photography shops or their suppliers. It can easily be made by the electrical method given in *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture* from an ingot of palladium. See the discussion there for calculating how much $PdCl_2$ is dissolved in the hydrochloric acid solution. One could also calculate how much $PdCl_2$ is in solution by means of current consumed. .35 gr $PdCl_2$ is .002 mole. It is a 2 electron oxidation to $PdCl_2$ from Pd ingot metal. This would require 386 seconds at a current of one amp, assuming 100% efficiency. Let's figure on getting 75% efficiency. Then at a current of one amp on the ingot, that would require 515 seconds (roughly 8½ minutes) to dissolve .35 grams of $PdCl_2$ into solution.

The ammonium chloride is very important to the plating solution, and shouldn't be left out! It acts as a complexor, preventing the hydrogen generated at the graphite during plating from sludging out the palladium solution. You want electroplate on the graphite, not sludge on the bottom of the beaker! If you don't have ammonium chloride right at hand, the same effect could be obtained by adding a couple ml of strong (28% NH_3) ammonia to the plating bath.

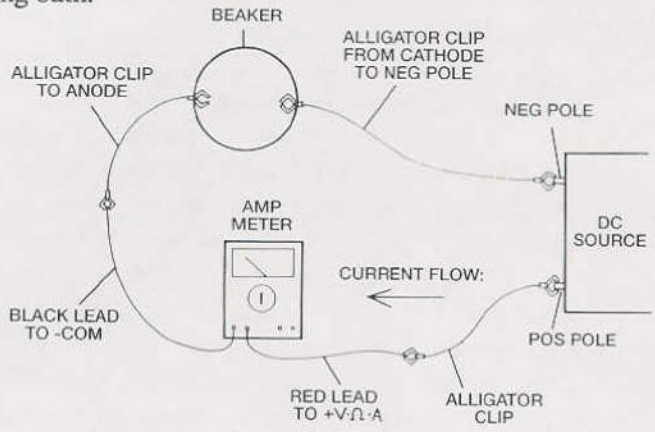


Figure 35

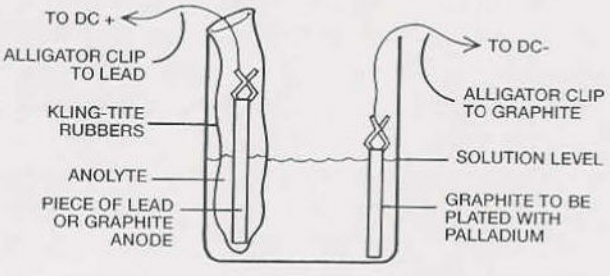


Figure 36

We're now ready to plate the graphite with palladium. Wire up your plating cell as shown in Figures 35 and 36.

I'll make the assumption that the reader is already familiar with the basic Fester Formula given in *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture*, so I won't go through the complete explanation of all the terms and other details given there. A DC power source is needed to do the electroplating. A DC rectifier, such as a Hull Cell rectifier sold by suppliers to electroplaters for around $500, is perfect. See the *Metal Finishing Guidebook and Directory* for suppliers. A battery can also be used, such as a 1½ volt "D" cell battery. This will deliver about the right amount of voltage and current. This is very important! The Hull Cell rectifier has controls that easily adjust the applied current to the desired level, and meters which display both voltage and current.

Wiring is run from the positive pole of the DC source, to the amp meter. In Figure 35 I show the $50 Radio Shack multi-tester, which works fine for the purpose. Then from the amp meter, wiring is run to the anode. Lead, or preferably graphite, is acceptable to use as the anode. It functions solely as a current pump into the solution, and at the low currents used for plating, its size relative to the graphite to be plated isn't very important. The anode is placed inside two scrubbed Kling-Tite Naturalamb rubbers. One rubber is placed inside the other to give a double wall of separation of the anode and its associated anolyte from the plating solution (catholyte).

The anolyte is a 3-5% by volume solution of sulfuric acid in water. Pour it inside the Kling-Tite rubber, and put some of it in the space between the two layers of rubbers to carry current. The anolyte should fill the rubbers to about the same depth as the plating solution fills the beaker. A clothes pin can be used to hold things in place.

The piece of graphite to be plated should be almost completely immersed in the plating solution, with just enough sticking out of the solution to keep the alligator clip which makes electrical contact with the graphite out of the plating solution. The plating solution will dissolve the clip, and result in an alloy being plated rather than pure palladium, so this is important! The alligator clip from the graphite being plated then goes to DC negative to complete the circuit.

A 10 cm by 10 cm piece of graphite has the face area of 100 square cm mentioned in this example. A 1000 ml to 2000 ml beaker or similar size plastic measuring cup or pitcher are suitable for doing the plating in. If the 350 ml of plating solution doesn't fill up the container enough to completely submerge the graphite to the extent shown in the drawing, switch to a different container, or dilute the plating solution with water or dilute HCl.

A moderate rate of stirring is begun on the plating solution, preferably with a magnetic stirrer. Then the DC power is turned on. With a rectifier, you just turn on the power switch. With a battery, the circuit is completed by making contact with the negative pole of the battery. The current output is adjusted to 2 or 3 milliamps per square centimeter of graphite actually facing the anode. With the 100 square cm graphite example, the output is 200-300 milliamps (.2 to .3 amp).

Output on a DC rectifier is adjusted just by turning up or down the voltage output. E=IR. Output from a battery is adjusted by choosing a battery with the right voltage output.

After a few minutes of plating at this rate, enough palladium has deposited on the graphite surface to begin the formation of hydrogen gas on the surface. It bubbles off the surface at a moderate rate. As the palladium plates out of solution, the initially reddish brown colored plating solution begins to lighten in color. Continue plating at the prescribed current density until the plating solution becomes clear, indicating that all the palladium has plated out of solution onto the graphite.

How long does this take? In my experiments at the prescribed current density, plating was complete within three hours. I started plating at the beginning of a Packers game, went to my favorite pub to guzzle beers and watch the game, and when I got back home after three hours, plating was complete.

The palladium plates from this bath at the prescribed current density of 2-3 milliamps per square cm, is a black form of palladium. It's next to impossible to distinguish it from the graphite itself. Mark the side of the graphite facing the anode, as this is the side you will want facing the anode when doing the electrocatalytic hydrogenation.

When plating is complete, remove the plated graphite piece from the plating solution, rinse it off with water, and stand it up to dry on a piece of wax paper. Don't put it in contact with paper, as it will discolor the paper.

In this plating cell, I used dilute sulfuric acid as the anolyte, and a double layer of Kling-Tite rubber as the cell divider. The reason is that one doesn't want to generate chlorine gas at the anode. It would work its way through a single layer of rubber into the plating solution and make an unplatable mess out of it. HCl working its way into the anolyte would also start to dissolve the lead anode, which would be bad. It might get into the plating solution, and do bad things to the catalytic activity of the plated graphite. It would also contribute to pollution in a minor way.

Now that the graphite has been plated with palladium, it can be used as a catalytic cathode in exactly the same manner as in the basic Fester Formula. Once again, I refer you to *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture*. The sole difference is that the palladium plated graphite electrode doesn't need to be anodized in dilute sulfuric acid, because the palladium has been electroplated in the form of palladium black.

Let's do a 10 gram batch example. First the acetic acid ester of ephedrine, pseudoephedrine or phenylpropanolamine must be made. We do this by taking 10 grams of ephedrine hydrochloride, or whatever, isolated from pills by the extraction procedures given earlier in this chapter, and we place it in a glass reaction vessel such as an Erlenmeyer flask or a volumetric flask. Then for each gram of ephedrine hydrochloride, we add 5-7 ml of glacial acetic acid. Swirl to mix, then stopper the opening of the flask with a cork. Heat the mixture with water heated to just about boiling, and swirl the contents of the flask to dissolve the ephedrine hydrochloride. When it has just about all dissolved, add two or three drops of concentrated sulfuric acid for each gram of ephedrine hydrochloride. Swirl this in to mix, then continue the heating in the nearly boiling hot water for about 4 hours. Keep the opening of the flask stoppered with the cork during the heating to keep steam from getting into the ester reaction mixture. This would lower the yield of product. After the heating period, remove the flask from the hot water, and allow it to cool. The mixture should be perfectly clear.

The electrocatalytic hydrogenation cell is then wired up. It is identical to the cell used in the basic Fester Formula, and also, by the way, identical to the plating cell given earlier in this section. The anolyte inside the Kling-Tite rubber(s) is 3-5% by volume sulfuric acid diluted with water. The catholyte, which is the solution in contact with the catalytic cathode, is either 3-5% sulfuric acid solution, or probably more preferably 2N to 3N hydrochloric acid. This is concentrated hydrochloric acid diluted to four to six times its original volume with water. If sulfuric acid is used as catholyte, then a single layer of Kling-Tite rubber is OK as a cell divider, and either lead or graphite or platinum can be used as anode. If hydrochloric acid is used as catholyte, then a double layer of Kling-Tite rubber must be used as a cell divider, and only graphite or platinum can be used as anode. The reasons for this have been mentioned earlier — if hydrochloric acid infiltrates into the anolyte then chlorine gas will be made, and we want to keep that out of the catholyte. HCl would also etch the lead anode. We don't want that to happen either.

The most convenient container to do this reduction in is a rectangular shaped plastic measuring cup. Stand your catalytic cathode up on one side of the measuring cup, hold it in place with a clothes pin, and make contact with it using an alligator clip. The anode inside the rubber(s) goes on the other side of the measuring cup, and one can hold it in place as well with a clothes pin, and make electrical contact with it. Put your stir bar in the cup to stir the catholyte, and turn on the DC power source. Apply 50 to 100 milliamps for each square

centimeter of the cathode surface facing the anode which is actually immersed in the catholyte. If you have about 75 square cm immersed, then flow 3.75 amps through the cell. The cathode will fizz off hydrogen, and you will know that you are making good contact with anode and cathode. If you don't see fizzing, or read current flow on the amp meter, check the surfaces on which electrical contact has been made for dirt.

Now that you have current flow, pour the cooled ester reaction mixture into the catholyte, and continue moderate stirring. Adjust current flow to 30-50 milliamps per square cm of cathode immersed by the catholyte. Only count the area on the side facing the anode! For instance, if 90 cm^2 of cathode is now immersed, flow 2.7 to 4.5 amps through the cell. When doing this reduction, one should choose the catholyte volume so that the ester reaction mixture is diluted at least five fold when it is poured into the cell, but no more than 10 fold. For this example, with an ester reaction mixture having a volume of about 80 ml, one would be using 400 to 800 ml of dilute sulfuric or hydrochloric acid as catholyte. Obviously, one wants as much of the catalytic cathode immersed into the catholyte as possible, without getting the alligator clip contact down into the solution. Choosing the proper size and shape of plastic cup makes this possible.

Continue current flow through the cell at this rate until at least 3000 coulombs per gram of ephedrine has passed. This will take roughly 2½ to 3 hours. Only a DC rectifier or car battery will be able to supply this much power for the required time period. The reduction mixture shouldn't be allowed to heat up, as that would encourage the hydrolysis of the ester of ephedrine back to ephedrine. If necessary, cool the reaction in an ice bath.

After the reduction is complete, the catholyte, which now contains the product, should be poured into a large sep funnel. The solution is made strongly alkaline by adding a 20% solution of lye in water. The lye solution should be added slowly, with shaking between adds of lye. When the solution is strongly alkaline (pH 13+ to pH papers) the meth will come out of solution as free base, which floats on top of the water. When the solution has cooled down, extract out the meth with 200-300 ml of toluene. Separate the toluene-meth layer from the water, and then bubble dry HCl through the toluene to get crystals of meth as usual.

The catalytic cathode can be reused at least a few times before it loses its catalytic activity. When this happens, the palladium black can be sanded off, and the graphite replated with new palladium black. Alternatively, one could just toss away the whole electrode, and start over by plating a new one.

This procedure can be scaled up as much as desired. One could conceivably line the walls of a fish tank with palladium plated graphite cathodes, and just cook away! One would need to use considerably heavier wiring than alligator clips to carry that much current. The 3 amps used in this example is near the upper limit for those flimsy little things. One might be advised to double up the alligator clips so that each clip is only carrying 1½ amps in this example.

The cooker will note that the product produced by the Advanced Fester Formula has higher octane numbers than the product made by the basic Fester Formula. That is because of the higher catalytic activity of the Pd on C used here versus blackened palladium metal. Passing more current through the basic Fester Formula should raise the octane rating to a similar level, but there is that competing reaction mentioned before. That reaction is the hydrolysis of the acetic acid ester of ephedrine back to ephedrine. In that form, it's not going to be easily reducible, and will act as a filler in the product.

As mentioned before, eventually the catalytic cathode will lose activity. In the early phases of this process, one can just use more current to get the same results. Eventually, one will just want to recover the product mixture in the usual way, and react it with fresh acetic acid and sulfuric to reform the ester, and run it through the electrocatalytic hydrogenation using newly plated Pd on graphite. Quality control is one of the joys of cooking. There's just nothing like the thrill of sampling batches, is there? Dirty ephedrine feed will quickly kill the catalytic activity of the cathode.

Someone who calls himself WizardX has suggested an improvement to this procedure. His suggestion is to add a little bit of palladium on carbon catalyst to the electrocatalytic hydrogenation mixture. About ½ gram of at least 10% Pd on C catalyst added to the reduction mixture would be plenty to increase the efficiency of the process. The directions for making this catalyst are found in this chapter. One could also vary the standard directions to make 20 or even 30% Pd on C for added power.

The addition of the catalyst to the reduction mixture results in much quicker and more efficient hydrogenation of the acetic acid ester of the ephedrine. The hydrogen which would be fizzed off by the palladium cathode can be caught by the catalyst and put to use doing the reduction rather than being wasted. This improvement would have the most striking beneficial effect on the basic Fester Formula procedure, as this basic procedure is more wasteful in its use of hydrogen than the Advanced Fester Formula given here.

One would simply add the catalyst to the reduction mixture during the charging of the cathode with hydrogen. This will give time for the Pd on C catalyst to also absorb hydrogen. At the end of the electrocatalytic hydrogenation, one would then just filter out the Pd on C catalyst for reuse in the next batch. Work-up of the reaction mixture then proceeds as usual. Your Uncle thinks this is a really good idea, and it comes with my highest recommendation.

Before leaving this topic, I have to report that some guys on the Internet have found that the two makeshift alternative power sources for doing electrocatalytic hydrogenation are completely unsuitable for use. A toy train transformer usually puts out a set voltage, around 10 or 12, and turning the knob on the transformer just causes the current output to be raised or lowered. This is way too much voltage. As a result, the reaction media gets fried, the sulfuric acid in solution gets reduced to hydrogen sulfide, and the catalyst surface gets poisoned.

Using a 12 volt battery is just as bad, as the dashboard dimmer knob similarly just turns the current up and down, with voltage staying at 12 volts.

The correct way is to start at zero applied volts, and then crank up the voltage a notch at a time until the desired current flow is achieved. In this way the resistance of the cell and the electrolyte determines the voltage of the reaction. In this Fester Formula, applying around 3 volts to the cell will give the desired current flow. The cathode will then measure around -.6 to -.8 volts versus standard calomel electrode. This is the right range. One only wants generation of hydrogen at the cathode, not a bunch of other electric reactions caused by excessive voltage.

Batteries can be used as power sources if the voltage they produce is in the right range. For example, a six volt lantern battery can be used to anodize the ingot surface, and two "D" cell batteries hooked in series to give three volts can be used to generate hydrogen at the ingot surface.

I always use an electroplating rectifier. Just by turning the knob on the front of the rectifier, the voltage output is raised or lowered, and the amperage which flows through the solution is correspondingly increased or decreased. Used "hull cell rectifiers" or their generic equivalents are available cheap. See the *Metal Finishing Guidebook and Directory*. Look under Testing Equipment — plating cells, and also under Rectifiers. If your library can't get you a copy of the directory, call the publisher at 914-333-2500.

Method 4:
Reduction With Hydroiodic Acid and Red Phosphorus

In this procedure, the alcohol grouping of ephedrine, pseudoephedrine, or PPA is reduced by boiling one of these compounds in a mixture of hydroiodic acid and red phosphorus. Hydroiodic acid works as a reducing agent because it dissociates at higher temperatures to iodine and hydrogen, which does the reducing. This dissociation is reversible. The equilibrium is shifted in favor of dissociation by adding red phosphorus to the mixture. The red phosphorus reacts with the iodine to produce PI_3, which then further reacts with water to form phos-

phorus acid and more hydroiodic acid. Since the hydrogen atom of the HI is being absorbed by the ephedrine, the red phosphorus acts as a recycler.

In some reductions, the need for HI is dispensed with just by mixing red phosphorus and iodine crystals in a water solution. The red phosphorus then goes on to make HI by the above-mentioned process. With a small amount of due care, this is an excellent alternative to either purchasing, stealing, or making your own pure hydroiodic acid.

This method has the advantage of being simple to do. It was formerly the most popular method of making meth from ephedrine. Now red phosphorus is on the California list of less restricted chemicals, so an increased level of subterfuge is called for to obtain significant amounts. One might think that this is easily gotten around by making your own red phosphorus, but this is a process I would not want to undertake. Ever hear of phosphorus shells? I would much rather face the danger of exploding champagne bottles. Those who insist upon finding out for themselves, will see *Journal of the American Chemical Society,* Volume 68, page 2305. As I recall, *The Poor Man's James Bond* also has a formula for making red phosphorus. Those with a knack for scrounging from industrial sources will profit from knowing that red phosphorus is used in large quantities in the fireworks and matchmaking industries. The striking pad on books of matches is about 50% red phosphorus.

The determined experimenter could obtain a pile of red phosphorus by scraping off the striking pad with a sharp knife. A typical composition of the striking pad is about 40% red phosphorus, along with about 30% antimony sulfide, and lesser amounts of glue, iron oxide, MnO_2, and glass powder. These contaminants can be removed from the red phosphorus by soaking the mixture in a 10% solution of hydrochloric acid for about an hour with stirring, then filtering out the red phosphorus and rinsing it with some water. This procedure is very stinky as the antimony sulfide gets converted to hydrogen sulfide and antimony chloride. Ventilation is mandatory. Various correspondents have written to tell that the glue holding the red phosphorus striking pads to the paper is softened by soaking in rubbing alcohol, or acetone, or even hot water, and can then be easily scraped off. On a related note, iodine crystals can often be found for sale at aquarium shops.

Another problem with this method is that it can produce a pretty crude product if some simple precautions are not followed. From checking out typical samples of street meth, it seems basic precautions are routinely ignored. I believe that the byproducts in the garbage meth are iodoephedrine, and the previously mentioned aziridine. (See the earlier section concerning chloroephedrine.)

I don't think that even a good fractional distillation will remove these byproducts from the meth. The aziridine has to have a boiling point very near that of meth, and so be unremovable by that method. I also think that the heat of distillation would cause the iodoephedrine byproduct to form more aziridine, which would then distill with the meth.

To some extent, these byproducts can be avoided in the first place, if when making hydroiodic acid from iodine and red phosphorus, the acid is prepared first, and allowed to come to complete reaction for 20 minutes before adding the ephedrine to it. This will be a hassle for some, because the obvious procedure to follow is to use the water extract of the ephedrine pills to make HI in. This should never be done, especially with the doctored pills now on the market. Pure ephedrine, racephedrine or PPA hydrochloride made according to the directions at the beginning of this chapter must be used. Impure raw material leads to big reductions in yield, and isolation problems at the end of the reaction. Since the production of HI from iodine and red phosphorus gives off a good deal of heat, it is wise to chill the mixture in ice, and slowly add the iodine crystals to the red phosphorus-water mixture.

To do the reaction, a 1000 ml round bottom flask is filled with 150 grams of ephedrine hydrochloride (or PPA-HCl). The use of the sulfate salt is unacceptable because HI reduces the sulfate ion, so this interferes with the reaction. Also added to the flask are 40 grams of red phosphorus, and 340 ml of 50%+ hydroiodic acid. This same acid,

which by the way, is on the chemical diversion list and should never be purchased, can be made by adding 300 grams of iodine crystals to 50 grams of red phosphorus suspended in 300 ml water. A more refined procedure is to react 300 grams of iodine with 30 grams of red phosphorus in 300 ml water, and then distill this mixture, collecting the first $^2/_3$ of it, and leaving the phosphorus-acid by-product behind in the distilling flask. This home brew acid smells bad, but works really well. It loses its bad smell shortly after the beginning of the reflux in the reaction with ephedrine. When using this more refined procedure, remember that 20-30 grams of red phosphorus must be added to the reaction mixture. The 40 grams cited above is overkill, but the unused portion can be reused by filtering it out at the end of the reaction.

With the ingredients mixed together in the flask, a condenser is attached to the flask, and the mixture is boiled for one day. This length of time is needed for best yields and highest octane numbers on the product. While it is cooking, the mixture is quite red and messy looking from the red phosphorus floating around in it.

The progress of the reaction can be followed by watching the consumption of the red phosphorus. The majority of product is obtained in about 10 hours, after 16 hours over ¾ is obtained, and after 24 hours, the reaction is done.

When one day of boiling under reflux is up, the flask is allowed to cool, then it is diluted with an equal volume of water. Next, the red phosphorus is filtered out. A series of doubled-up coffee filters will work to get out all the red phosphorus, but real filter paper is better. The filtered solution should look a golden color. A red color may indicate that all the phosphorus is not out. If so, it is filtered again. The filtered-out phosphorus can be saved for use in the next batch. If filtering does not remove the red color, there may be iodine floating around the solution. It can be removed by adding a few dashes of sodium bisulfite or sodium thiosulfate.

Of these two, the thiosulfate is much preferred because it has the ability to destroy aziridine along with the iodine in the solution. You see, iodoephedrine makes aziridine (dimethylphenyl aziridine) by reaction between the iodine atom and the amino group. See earlier in this chapter for the drawing of the aziridine in question. The high temperature at which this reaction works encourages its formation.

The best way to add the thiosulfate or bisulfite is along with the sodium hydroxide or lye solution used to neutralize the reaction mixture. That is our next step. For the batch size given here, using 300 grams of iodine in 300 ml of water and about 150 grams of ephedrine hydrochloride, over 150 grams of lye or sodium hydroxide will be required to make the solution strongly alkaline (ph 13+). This amount of lye should be dissolved in about 600 ml of water and allowed to cool down.

Now the few grams of thiosulfate or bisulfite is put into a beaker, and about 50 ml of the sodium hydroxide solution is added to the beaker, and the two are mixed to make a solution. This is then added to the filtered reaction mixture, and swirled around or shaken. Some fizzing may be noted here as the reaction takes place. Then as the great heat produced by the neutralization reaction allows, the rest of the sodium hydroxide solution should be added with stirring or shaking to the reaction mixture. The meth free base which forms will float to the top of the water solution. Strong and prolonged shaking of the mixture is necessary to ensure that all the meth has been converted to free base.

Then check the pH of the water layer using pH papers. It should read strongly alkaline. If not, add more lye, and continue shaking.

With free base meth now obtained, the next step, as usual, is to form the crystalline hydrochloride salt of meth. To do this, a few hundred mls of toluene is added to the batch, and the meth free base extracted out as usual. If the chemist's cooking has been careful, the color of the toluene extract will be clear to pale yellow. If this is the case, the product is sufficiently pure to make nice white crystals just by bubbling dry HCl gas through the toluene extract as described in Chapter Five. If the toluene extract is darker colored, a distillation is called for to get pure meth free base. The procedure for that is also described in Chapter Five. The

yield of pure meth hydrochloride should be from 100 grams to 110 grams.

If gummy binders from the stimulant pills are carried over into the reaction mixture, they produce a next-to-impossible-to-break emulsion of meth, gum, toluene, and water when the reaction is done and it is time to extract out the meth. It is absolutely necessary that the pill-extraction procedure given in this book be followed. The pills must be extracted with water, the extract free based and extracted with toluene, and the hydrochloride then precipitated from the toluene extract. The crystals should be long, white, and needle-like. If this emulsion is encountered, the only way to break it is to first let the emulsion sit in a sep funnel for a few hours. Water will slowly work its way out and settle to the bottom where it can be drained away. The stubborn residual emulsion should be transferred to a distilling flask, and the toluene slowly distilled off through a fractionating column. This removes water from the emulsion as the toluene-water azeotrope. It may be necessary to add additional toluene to the distilling flask to get all the water removed. The gum sticks to the glass flask, and causes no further problem. Once the emulsion is broken, distilling should be stopped. The toluene-meth solution should be poured from the distilling flask, and the meth precipitated as hydrochloride as per the usual dry HCl bubbling method.

Two additional methods exist for making hydroiodic acid, and while not as convenient as just adding iodine to red phosphorus, they produce a more pure acid. Further, they don't require red phosphorus in the making of the acid, so the amount of that material needed for production is greatly reduced.

See *Inorganic Syntheses,* Volume 1. The first of these methods involves bubbling H_2S gas into a suspension of iodine in water. The H_2S gas is made by dripping HCl onto iron sulfide and piping the gas into solution just like the HCl gas made in Chapter Five. The excess H_2S gas dissolved in the product acid is then boiled out of solution under a reflux. This method is quite good.

The alternate method directly produces a real pure acid by direct union of H_2 gas with I_2 vapor in the Pt catalyst bed. This is really good if you aren't afraid of putting together some glass tubing.

WARNING! *Iodine and red P are now very "hot" items. Ordering them is likely to cause the cops to follow right behind!*

Method Five:
Zinc Reductions

Check this one out! In *Journal of Organic Chemistry*, Volume 26, page 99, the ester of a benzyl alcohol was reduced to the alkane by using zinc metal and formic acid. Not a bad idea, huh? Simple materials, non-toxic reaction materials and waste, and maybe good yields of meth.

There is a more general reduction method for benzyl alcohols which uses trifluoroacetic acid and sodium borohydride, but trifluoroacetic acid is a potent rat poison and pretty effective people poison as well. I'll be damned if I'm the one who gets blamed for introducing "the rat poison meth recipe." Try this method. If it works, fine. If not, you know how I like those hydrogenation methods. Stick with them.

In their procedure, put 20 grams of an ester of the benzyl alcohol in a flask along with 40 grams of zinc metal, 160 grams (about 130 ml) of 95% formic acid, and 40 ml of water. They refluxed the mixture for 5 hours, then after cooling, the zinc metal left over was filtered out. The filtered reaction mixture was then made strongly alkaline with sodium hydroxide solution, and the product extracted out with toluene.

How would one tinker with this recipe to use ephedrine? First, I would put the 20 grams of ephedrine hydrochloride in the flask. Then I would add the 130 ml of 95% formic acid. I would heat this mixture on a hot water bath for a few hours to make the formic acid ester of the ephedrine. Then I would add the zinc metal and the 40 ml of water, and heat the mixture to reflux for 5 hours.

After cooling, filtering and making strongly alkaline with sodium hydroxide solution, the toluene extract likely contains the formic acid amide of meth. This would be hydrolyzed by boiling with hydrochloric acid, just like in Chapter Five. The product is then recovered from the hydrochloric

acid just as in that chapter, distilled, and bubbled with HCl gas to get crystals of meth hydrochloride.

Would you like another recipe? I like to use this one for about 10 gram batches. It's fast and really good.

First, make about ½ gram of palladium black by adding sodium borohydride to a well stirred solution containing one gram of $PdCl_2$ in water. See *Journal of the American Chemical Society*, Volume 84 pages 1493-95. I suck up the settled black catalyst with a pipette and squirt it into a filtering flask. Then take a balloon and put the end of it over the vacuum nipple of the filtering flask. Tie it in place with some string so that it doesn't leak air when one blows air into the filtering flask.

Now take the ester reaction mixture made by heating 10 grams of ephedrine hydrochloride with about 60 ml of glacial acetic acid and a little sulfuric acid and pour this mixture into the filtering flask. Put a magnetic stir bar into the filtering flask, too.

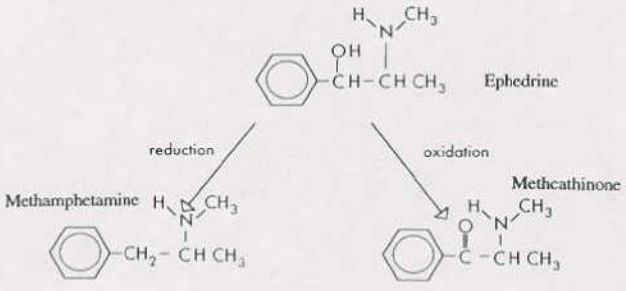
Begin fast magnetic stirring, then toss a piece of sodium borohydride about the size of a split pea into the flask, and quickly stopper the flask to hold in the hydrogen generated. When the fizzing stops, add another piece. Continue this until the balloon stays inflated with hydrogen.

Then heat the filtering flask in a pan of boiling water to make more ester. Then add more borohydride until once again the balloon stays inflated. Repeat the heating. Then add borohydride again.

Now filter out the palladium black for reuse. Put the filtered batch in a sep funnel, and add lye solution with shaking until the mixture is strongly alkaline. Extract out the meth with toluene, separate off the toluene layer, and bubble it with dry HCl gas to get about 10 grams of a very nice meth. You'll like this recipe! If you should have trouble getting the $PdCl_2$ to dissolve in the water, add a few drops of HCl. That will make it dissolve.

Chapter Sixteen
Methcathinone:
Kitchen Improvised Crank

One designer variant upon the amphetamine molecule which gained popularity and publicity a few years ago is methcathinone, commonly called "cat." This substance is remarkably similar to the active ingredient found in the leaves of the khat tree which the loyal drug warriors on the network news blame for turning peace loving Somalis into murderous psychopaths. The active ingredient in the khat leaves is cathinone, which has the same structural relationship to methcathinone that amphetamine has to methamphetamine. It is made by oxidizing ephedrine, while meth can be made by reducing ephedrine.



The high produced by methcathinone is in many ways similar to the one produced by methamphetamine. For something so easily made and purified, it is actually quite enjoyable. The main differences between the meth high and the methcathinone high are length of action and body feel. With methcathinone, one can expect to still get to sleep about 8 hours after a large dose. On the down side, it definitely gives me the impression that the substance raises the blood pressure quite markedly. This drug may not be safe for people with weak hearts or blood vessels. Be warned!

Chronic use of methcathinone causes a person to become very stinky, as the foul metabolic breakdown products of cat come out of one's pores. Be double-warned!

Cat is best made using chrome in the +6 oxidation state as the oxidizer. I recall seeing an article in the narcoswine's *Journal of Forensic Science* bragging about how they worked out a method for making it using permanganate, but that method gives an impure product in low yields. Any of the common hexavalent chrome salts can be used as the oxidizer in this reaction. This list includes chrome trioxide (CrO_3), sodium or potassium chromate (Na_2CrO_4), and sodium or potassium dichromate ($Na_2Cr_2O_7$). All of these chemicals are very common. Chrome trioxide is used in great quantities in chrome plating. The chromates are used in tanning and leather making.

For preparation of this substance, see US Patent 2,802,865. Formerly, back in the days when ephedrine pills were 30-40% active ingredients by weight, and the fillers were mostly water-insoluble, one could just extract these pills with water and directly perform the oxidation in the water extract. Now, with the garbage pills containing guaifenesin

and a water-soluble fiber filler, the extraction-freebasing-extraction-crystallization-isolation procedure given in this book absolutely has to be used. Using the old procedure, one gets a mess that look likes a milkshake at the end of the reaction.

Pseudoephedrine can also be used to make cat. The pills containing this starting material must be extracted according to the directions given in Chapter Fifteen, and then converted to racemic ephedrine (called racephedrine) by heating with HCl solution as also described in that chapter. This will yield dl or racemic cat, which is almost as potent as cat made from ephedrine.

Note in the patent that for each molecule of ephedrine or racephedrine in the reaction mixture, there are .66 atoms of Cr^{+6} in the solution. As a result, the amount of Cr^{+6} substance used in this reaction will vary with the compound used. For example, in the one-tenth mole-size batch given here, 20 grams of ephedrine or racephedrine hydrochloride will react with:

10 grams of $Na_2Cr_2O_7 \cdot 2H_2O$
10 grams of $NaCrO_4$
22.8 grams of $NaCrO_4 \cdot 10H_2O$
12.9 grams of $KCrO_4$
10 grams of $K_2Cr_2O_7$
6.6 grams of CrO_3

There are two main precautions to be adhered to in doing this reaction. The first one is the need to keep the temperature of the reaction mixture below 100° F. It is better to keep it well below that. To keep the reaction temperature down, the glass container in which the reaction is done should be packed in ice. I have also heard that very fast stirring will so speed up the reaction that the ice bath fails to keep the temperature down. This is only a problem with large-size batches about one mole in size or larger. In these big batches, a favorite agitation technique was to put the reaction mixture contained in a glass jug surrounded by ice in a cooler into the trunk of a car and spend a few hours driving on rough back roads to stir the mix.

The other main precaution is to add the Cr^{+6} solution slowly to the ephedrine or racephedrine with stirring. It is best to do the addition dropwise, but with larger batches, this is just not practical. In any case, use some common sense as to the rate of add for the chrome.

To do the reaction, 20 grams of ephedrine or racephedrine hydrochloride is dissolved in 50 ml of water, then 5 ml of concentrated sulfuric acid is slowly added to it with stirring. The beaker containing this mixture should then be nestled in ice to cool down. Then, in another beaker, mix 45 ml of water with 7.5 ml of sulfuric acid and the amount of Cr^{+6} compound listed above.

Begin stirring the ephedrine solution, and then dropwise add the Cr^{+6} solution to it. The addition should take about ½ hour. The chrome solution will be clear orange-red going into the mix, but it soon darkens to a blackish red. The stirring should be continued for a reaction time of four hours. Shortening this reaction time gives poor yields and incomplete oxidation. Exceeding this reaction time causes destruction of the product, and again poor yields. The preferred solution temperature during the 4 hour reaction time is 80-90° F. The amount of cooling required will depend on the batch size.

When the reaction time is over, a 20% solution of lye in water should be added to the reaction mixture with stirring until it is strongly alkaline to litmus. Now pour the mixture into a sep funnel, and shake vigorously for a couple of minutes to ensure complete conversion of the cat to free base. The chrome will come out of solution as a greenish sludge. Extract this mixture with two 50 ml portions of toluene. The extracts should have a mild yellowish tint. The pooled toluene should then be washed once with 100 ml of water, then the toluene layer should be poured into a beaker to sit for about an hour. This will allow entrained water to settle and stick to the glass. Now pour the toluene solution into a fresh, clean beaker and bubble dry HCl gas through it, as described in the Chapter Five, to get crystalline cat hydrochloride to precipitate out of solution. Filter this out as also described there, and spread the crystals out on a plate to dry. The yield of white to maybe slightly yellow-tinted crystals is a little over 10 grams.

The yield from this reaction is quite variable, ranging from 10% up to 80%. One often gets unreacted ephedrine instead of cat. Also, at times, it's

been reported to me, a very dangerous byproduct can be formed. This byproduct causes one's blood to gel if it is injected. The nature of this byproduct is unknown to me. If there's a possibility that the cat is going to be injected, then one should recrystallize the crude yellow-tinted cat from acetone.

So that is the standard cat recipe. How can it be improved? Let's consider the time at which this patent was filed, the mid-50s. Just after this time, it was discovered that using acetone as the solvent for Cr^{+6} oxidations gave much better results. It prevents over-oxidation of the product, which is the real problem with the original recipe. Pseudoephedrine is particularly vulnerable to the over-oxidation, so by using acetone solvent, pseudoephedrine could probably be used in the reaction.

In this acetone variation, I would put the 20 grams of ephedrine or pseudoephedrine in a beaker with 50-100 ml of hardware store acetone. Then I would mix 25 ml of water with 10 ml of sulfuric acid, and then mix the prescribed amount of Cr^{+6} compound into this acid water-solution. After cooling, the Cr^{+6} solution should be dropwise added to the acetone-ephedrine hydrochloride mixture with stirring.

After the reaction is complete in a few hours, I would pour the reaction mixture into about ten volumes of water. Then I would make the water solution strongly alkaline by adding lye water solution with shaking. Then I would extract out the cat with toluene, and then bubble this extract with dry HCl gas to get crystals of cat hydrochloride. The yield should be greatly improved, and the reaction should be more reliable.

If one really wants to get elegant, we could use the preferred oxidizer for benzyl alcohols, activated manganese dioxide. See *Journal of Organic Chemistry*, Volume 26, pages 2973-75 (1961).

To make activated MnO_2, one dissolves 1100 grams of manganese sulfate tetrahydrate or 833 grams of manganese sulfate monohydrate in 1500 ml of water. 1170 ml of a 40% solution of sodium hydroxide in water is also prepared. Finally, one makes a solution of 960 grams of potassium permanganate in 6 liters of water. The permanganate solution is heated to 80-90° C, and then the manganese sulfate solution and the sodium hydroxide solution are simultaneously added to the permanganate solution over a period of about one hour, while maintaining the temperature of the permanganate solution at 80-90° C. Strong stirring of the permanganate solution is done during the addition. Manganese dioxide precipitates out of solution as a fine brown solid. Continue stirring for an additional hour.

The hot solution is then filtered to collect the manganese dioxide, and the filter cake in the Buchner funnel is washed with water until the purple color of permanganate no longer comes out with the water rinse. The solid (manganese dioxide) is then baked in an oven at 110-120° C for about 4 hours to dry it. It is then ground finely, and returned to the oven for another bake at the same temperature. This is activated manganese dioxide.

Then a toluene or Coleman camper fuel extract containing about 20 grams of ephedrine or pseudoephedrine free base is diluted to a volume of about 500 ml with more of the same solvent. Then .4 mole of activated manganese oxide (35 grams) is added. The mixture is stirred, and preferably heated to reflux for 5 hours. A Dean-Stark trap to remove water will increase yields.

Then the cooled reaction mixture is filtered to remove the activated MnO_2 and the MnO formed by the oxidation. Then dry HCl is passed through the solution to collect the product, cat hydrochloride in about 80% yield. Your Uncle thinks this variation is more bother than it is worth, especially considering that permanganate is a List II chemical, and not easily available at the hardware store.

Chapter Seventeen
Brewing Your Own Ephedrine

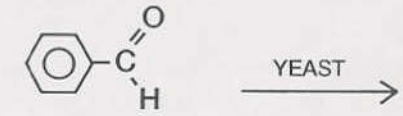
I love to guzzle beer. Not that mass produced swill, but real beer turned out in small batches by microbrewers and homebrewers. Beer that has some body, flavor, and a real kick! Homebrewing is just a joy, as lots of people have found out. Stores selling supplies to the homebrewer have sprung up in every backwater town. They are just all over the place, and newspapers catering to the homebrewer or fans of microbrews can be picked up for free at the local liquor store. The ads in these newspapers are predominately for mail-order brewing supplies at discount prices.

What a fortunate coincidence that the industrial process for making ephedrine is just a fermentation process using brewer's yeast. This process is much cheaper than extracting ephedrine from Ma Huang, and yields l-ephedrine as the product. Other chemical processes give product mixtures that consist of d and l ephedrine and pseudoephedrine. If one wishes to scale up production beyond that which can be sustained by scrounging pills and extracting them, this fermentation is a very viable alternative.

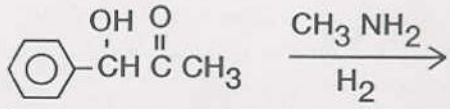
This process uses benzaldehyde as the starting material, so essentially one could consider this method as an alternative to the Knoevenagel reaction back in Chapter Nine. The fermentation action of the brewer's yeast takes the place of that List I chemical nitroethane.

Benzaldehyde is easily available, in spite of the fact that it too is a List I chemical. Oil of bitter almonds can be used as is, once the HCN it contains is removed by applying a vacuum to the oil. On a larger scale, the electric oxidation of toluene procedure given in Chapter 9 would give all the benzaldehyde that could ever be desired.

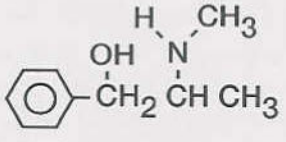
The fermentation action of brewer's yeast converts benzaldehyde to l-1-phenylpropanol-1-one-2 in a yield corresponding to about 80% by weight of the benzaldehyde added to the fermentation mixture. This phenylacetone derivative is then reductively alkylated with methylamine by any one of several procedures to give l-ephedrine.



BENZALDEHYDE



ℓ-I-PHENYL PROPANOL-I-ONE-2



ℓ-EPHEDRINE

One would think that the reductive alkylation of that phenylacetone derivative would yield d,l-ephedrine, and then that reduction of that d,l-

ephedrine would then give d,l-meth. That same racemic meth that results from reductive alkylation of phenylacetone. (Your Uncle prefers the buzz produced by the racemate over the harsher, more nerve jangling buzz produced by d-meth.) Apparently, this isn't the case. The references for this process claim that solely l-ephedrine is produced, and then reduction of this l-ephedrine, which is identical to natural ephedrine, yields that potent but harsh d-meth.

To start with this project, one would first want to read a home beer brewing book, such as (Storey Communications publishes one such book) *Better Beer and How to Brew It,* since the processes are so similar and much of the same equipment and materials will be used. I have this book, and it is good. This is all you need to sound like a real brewer when you head down to the Brew Shop in your town to pick up supplies.

As with regular beer brewing, one starts with a brew vat, five gallon plastic pails work just fine for this purpose. They should be cleaned, then rinsed with bleach diluted with several volumes of water to disinfect the surfaces, then rinsed some more with clean water to remove the bleach residue.

Next fill the pails with tap water until they are half to $^2/_3$ full. We are now ready to brew. See *Drug Trade News*, Volume 16, Number 16, page 27 (1941) (I love that reference) and *Wallerstein Labs. Commun.*, Volume 4, Number 13, page 213 (1941). Also see *Chemical Abstracts,* Volume 17, page 1484, and *Biochemische Zeitschrift*, Volume 115, page 282 (1921) and Volume 128, page 610 (1922). My German stinks, so I'm winging this a bit. If you can read those references, go for it.

Into the pail the brew mixture is made up by adding glucose, also called dextrose or corn sugar, available at the brew supply shop, until there is one part by weight of sugar for each 20 parts by weight of water. Then live brewer's yeast is added. The recipe states that an amount of live brewer's yeast should be added equal to the weight of sugar added. This stuff comes in small packages at the brewing supply shop, so it would no doubt be best to use what brewers call a starter bottle, which is a clean bottle containing some diluted malt extract (unhopped!). Pour the freeze dried yeast in there, and within a day at room temperature it will have grown many fold. Pour it into the brew pail while the yeast is actively growing, as shown by the bubbling and frothing of the starter bottle. I think that the brew mixture in the pail is very nutrient-poor. No nitrogen source for the yeast is in the solution. The brewing supply shop sells yeast nutrient, diammonium phosphate, and I think it would be advisable to add a teaspoon or so of it to roughly 3 gallons of water.

When the brew mixture in the pail has been fermenting for about 8 hours at room temperature, about one-twelfth as much benzaldehyde should be added as compared to the sugar. For example, let's just say you have 20 pounds of water in the brew vat, and that you added one pound of sugar to the water. Then you would add one-twelfth pound (about 40 ml) of benzaldehyde to the brew pail. Cover the pail to help prevent contamination, and allow the mixture to ferment for three days.

During the course of the fermentation, an enzyme called carboligase produced by the yeast converts the benzaldehyde to phenylpropanol-1-one-2. It is believed that the enzyme links acetaldehyde or acetic acid made by the fermenting yeast with the benzaldehyde to give the product. In any case, in a few days, one gets a yield of product amounting to 85% of the amount of benzaldehyde used.

When the fermentation is completed after about 3 days, it's time to recover the phenylpropanol-1-one-2 from the brew mixture. The yeast in the mixture is a problem. With regular beer brewing, the yeast just settles to the bottom of the fermenter when the fermentation is complete. Siphoning is then done to remove the clear beer from the settled yeast. Apparently, in this process, the yeast doesn't settle so well. The industrial process uses centrifugation of the fermentation mixture to force the yeast to the bottom. I'm sure that works well for them, because once the centrifuge is installed, no materials need be purchased from then on to settle the yeast. The centrifuge pays for itself.

Brewers approach unsettled yeast in two ways. On a small scale, they will add a material called

'finings' to the brew mixture which settles the yeast. On a larger scale, they will filter the brew. Yeast is some gummy stuff. It will plug a filter paper in no time flat, so in addition to the filter paper, they use filter aid.

Filter aid is stuff like Celite (diatoms), powdered cellulose, or even a bed of sand to catch those gummy yeast particles before they get to the filter paper and plug it up.

Once the yeast has been removed from the brew mixture, the phenylpropanol-1-one-2 can be extracted out of the solution. The original references used ether to do the extraction. I would suggest substituting hardware store toluene. Several extractions with a few hundred ml portions of toluene should be enough to completely remove the product from a 5 gallon pail fermenter.

Next the combined extracts should be distilled to remove the toluene. Once the toluene is mostly all gone, the residue should be fractionally distilled under a vacuum. The product, also called phenylacetylcarbinol, distills over the range of 100° C to 150° C under a vacuum of 14 torr. A really good aspirator using nice cold water will pull a vacuum this strong. Weaker vacuums will result in higher boiling ranges. The yield of distilled product amounts to around 85% of the amount of benzaldehyde added to the fermentation mixture.

Now the phenylpropanol-1-one-2 can be reductively alkylated to give l-ephedrine. Any one of several methods can be used, just as in the case of reductively alkylating phenylacetone to meth. Method number one has to be catalytic hydrogenation using platinum catalyst.

In the example taken from US Patent 1,956,950, the chemists place 300 ml of the distilled phenylpropanol-1-one-2 in the hydrogenation bomb along with one gram of platinum catalyst, and 85 grams of 33% methylamine solution. They state that it's advantageous to add some ether to the hydrogenation solution. How much is some, they don't say. They then hydrogenate the solution in the usual manner, with up to 3 atmospheres of hydrogen pressure, and magnetic stirring of the contents of the hydrogenation bomb.

When absorption of hydrogen stops in two or three hours, the platinum catalyst is filtered out. Then the ethery hydrogenation mixture is shaken with a volume or two of 10% HCl solution to pull the ephedrine out of the ether and into the acid water, forming the HCl salt of ephedrine. The ether layer is separated off with a sep funnel, then the dilute acid is boiled away. The residue is diluted with a little alcohol, and then a lot more ether. Passing dry HCl through this mixture then gives crystals of pure ephedrine hydrochloride. Their yield was around 110 grams.

My commentary on this hydrogenation? That yield is awfully low. Using phenylacetone as a guide, one should be expecting a yield around 300 grams of ephedrine. What's up? Check out the amount of methylamine used. There are about two moles of the phenylacetone derivative, but they don't even use one mole of methylamine. It should be the other way around, an excess of methylamine. Perhaps this is how they only get l-ephedrine from the phenylacetone derivative. In any case, I'd much rather have 300 grams of racephedrine than 110 grams of l-ephedrine. My thoughts are that one would be better served just going to Chapter Eleven, and just plug in this phenylacetone derivative for the regular phenylacetone. That means two or three moles of methylamine for each mole of phenylacetone, alcohol as solvent, and a bit more platinum catalyst in the mixture.

In the patent, they give another reductive alkylation example. They use amalgamated aluminum as the reducer, just like in Method Three in Chapter Twelve. They take 120 grams of the undistilled fermentation product containing the 1-phenylpropanol-1-one-2, and drip it over the course of two hours into a solution of 10 grams of methylamine in 500 ml of ether in the presence of 20 grams of activated aluminum amalgam. Simultaneously, they drip into the mixture 20 to 30 ml of water. Stirring of the mixture is required.

The vigorous reaction that sets in is moderated by periodic cooling. When the reaction is complete after a few hours, they filter the mixture to remove the aluminum. Then they shake the ether solution with 10% HCl solution to draw the ephedrine into

the water. The ether layer is separated, then the dilute acid boiled off. The residue is thinned with a little alcohol, then dissolved in a lot more ether. Bubbling with dry HCl gives 25 to 45 grams of l-ephedrine hydrochloride crystals.

My commentary on this procedure is identical to the last one. So little methylamine used! I haven't tried this, but I would be surprised to say the least if more methylamine didn't greatly increase the yield of product. I would also think that any one of the activated aluminum procedures given in Chapter Twelve could be used, just by plugging in this phenylacetone derivative for the regular phenylacetone. Also the use of ether is to be avoided when possible.

You don't like that recipe? Check out this one taken from *Chemical Abstracts,* Volume 47, column 3347. 20 grams of N-methyl-d,l-alanine and 50 grams of benzaldehyde are placed in a flask and heated on an oil bath at 150-160° C until the mixture stops fizzing off carbon dioxide.

The mixture is then cooled and mixed with a few hundred ml of toluene. Whatever doesn't dissolve in the toluene is thrown away. The product, which is a mixture of ephedrine and pseudoephedrine, is then extracted out of the toluene by shaking the toluene with about an equal volume of 10% HCl. The toluene can be distilled to recover unused benzaldehyde, if there is any in it.

The dilute hydrochloric acid solution which contains the products should be boiled down to concentrate it. The steam will also carry off some byproducts, so vent this steam outside.

Once the dilute acid has boiled down to a volume of 50-100 ml, allow it to cool. Then add a little activated carbon, and stir it around for a while. Then filter it out. This will decolorize the solution.

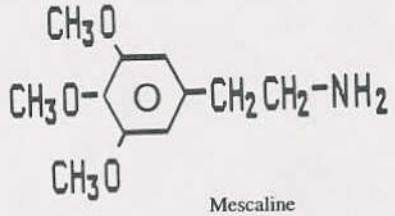
Add lye pellets a little bit at a time with stirring until the water solution is strongly alkaline. Extract the alkaline water a few times with toluene. The combined toluene extracts should next be bubbled with dry HCl gas to give a crystalline product amounting to about 12 grams. The product will be about 8 grams of d,l-pseudoephedrine, and 4 grams of d,l-ephedrine. It will yield racemic meth upon reduction.

This recipe should be easy to scale up. Replacing the N-methylalanine with the commonly available amino acid alanine should give one phenylpropanolamine instead. Reduction of that would give benzedrine.

Take note that recovering ephedrine from water solutions is a bit different than recovering meth. That's because ephedrine free base dissolves well in water, while meth doesn't. So for recovery of the ephedrine we take the dilute acid solution of the ephedrine and boil it down, just like in the pill extraction procedure using water. Once it is concentrated, then it is made alkaline with lye, and the ephedrine extracted out. In this way you get good recovery of the ephedrine. Use too much water, and it's difficult to extract it all out.

Chapter Eighteen
MDA, Ecstasy (XTC), and Other Psychedelic Amphetamines

The psychedelic amphetamines are a fascinating and largely ignored group of drugs. They all have the basic amphetamine carbon skeleton structure, but show effects that are more akin to LSD than to the amphetamines. The LSD-like effect is due to the presence of a variety of "add ons" to the benzene ring of the basic amphetamine structure. Generally, these "add ons" are ether groupings on the 3, 4, or 5 positions on the benzene ring. Because of these "add ons" one can consider these compounds more closely related to mescaline than to amphetamine. Consider the mescaline molecule pictured below.



Mescaline

Mescaline should by all rights be considered an amphetamine derivative. It has the basic phenethylamine structure of the amphetamines with methyl ether groupings on the benzene ring at the 3, 4, 5 positions. To be a true amphetamine, it would only need its side chain extended by one carbon, putting the nitrogen atom in the central, isopropyl position. Such a compound does in fact exist. It is called trimethoxyamphetamine, or TMA for short. Its effects are very similar to mescaline in much lower dosage levels than the ½ gram required for pure mescaline. Its chemical cousin, TMA-2 (2,4,5 trimethoxyamphetamine) has similar awe inspiring characteristics. More on this later.

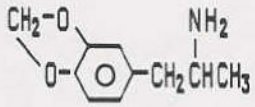
The most popular and, in my opinion, the best of the psychedelic amphetamines are the members of the MDA family. This family consists of MDA, and its methamphetamine analog, XTC, or Ecstasy, or MDMA. MDA (3,4 methylenedioxyamphetamine) gives by far the best high of this group. Its effects can best be described as being sort of like LSD without the extreme excited state caused by that substance. It was popularly known as "the love drug" because of the calm state of empathy so characteristic of its effect. It could also be a powerful aphrodisiac under the right circumstances.

This substance gradually disappeared during the early 80s due to an effective crimping upon the chemicals needed for its easy manufacture.

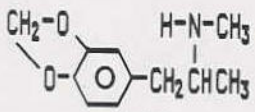
This crimping, and the drug laws in effect at the time, gave rise to a bastard offspring of MDA. This substance was XTC, or MDMA, the so-called Ecstasy of the drug trade. This material was a designer variant of MDA, and so was legal. The chemicals needed to make it could be obtained without fear of a bust. It also lacked the best qualities of its parent. While the addition of a methyl group of the nitrogen of the amphetamine molecule accentuates its power and fine effect, the addition of a methyl group to the MDA molecule merely served to make it legal. As fate would have it, the hoopla surrounding the subsequent outlawing of this bastard child served to make it a more

desired substance than MDA. This is typical of black-market, prohibition-driven demand.

To understand the various routes which can be followed to make these substances, note the structures of MDA and MDMA shown below:

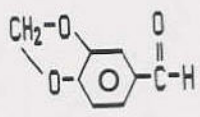


mda

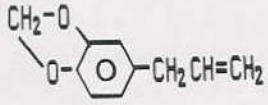


mdma

To make these substances, and the rest of the psychedelic amphetamines for that matter, the manufacturer has a choice of two starting materials. He can use the appropriately substituted benzaldehyde, which in the case of MDA or MDMA is piperonal (heliotropin), or he can use the correspondingly substituted allylbenzene, which in this case is safrole. These substances are pictured below:



Piperonal

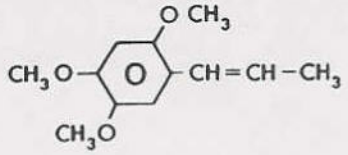


Safrole

Piperonal was the favored starting material for making MDA, as were the other substituted benzaldehydes for making other psychedelic amphetamines. The supply of these raw materials was effectively shut off. Piperonal does find legitimate use in making perfumes, but considerable determination is needed to divert significant amounts of the stuff into clandestine operations.

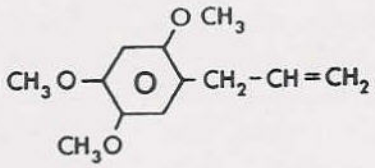
Once obtained, these substituted benzaldehydes could be converted into amphetamines by an interesting variant of the Knoevenagel reaction as described in Chapter Nine. They could be reacted in a mixture of nitroethane and ammonium acetate to form the appropriately substituted 1-phenyl-2-nitropropene. This nitropropene could then be reduced to the amphetamine by using lithium aluminum hydride. For this recipe, see *PIHKAL* under MDA. The nitroalkene obtained by the reaction of piperonal and nitroethane can also be reduced by the hydrogenation methods given in the Knoevenagal reaction section of this book in a yield of around 50%. Similarly, the electric reduction method given in that section can also be used. Now that both piperonal and nitroethane are List I chemicals, we would have to concede that the narcoswine have won this round, and that this pathway can be considered for all practical purposes to be dead.

This left safrole, and the other substituted allylbenzenes, as starting materials for psychedelic amphetamine manufacture. This route had the advantage of having a raw material source that was nearly impossible to shut down. For instance, sassafras oil consists of 80-90% safrole. One merely has to distill the oil under a vacuum to get very pure safrole. Similarly, other psychedelic amphetamines can be made using essential oils that contain the appropriately substituted allylbenzene or propenylbenzene as a major substituent. For instance, calamus oil contains a large proportion of B-asarone, the starting material for TMA-2. Nutmeg contains a mixture of myristicin (potential MMDA) and elemicin (potential TMA). These oils are all available from herbal supply shops and dealers in the occult. Even without this source, the oils can be easily obtained from the plants.



Asarone

Calamus oil is some interesting stuff! Its composition depends upon the country the oil comes from. Luckily, most of the oil on the market comes from India. The vast majority of oil from that country contains about 80% B-asarone, although there are reports (see *Journal of Indian Chemical Society*, Volume 16, page 583 1939) that some oils from that country contain around 80% allylasarone.



Allylasarone

Other major sources of commercial calamus oil are Japan and Europe. These oils contain lesser and variable amounts of a-asarone. This is the cis-trans isomer of B-asarone. It differs in that a-asarone is a solid at room temperature, and may precipitate out of oils upon cooling in a freezer. It reacts in an identical manner as B-asarone. Both can be obtained in a pure form from the oil by fractional vacuum distillation.

On the topic of purifying essential oils, it has been proposed by other underground sources that sassafras oil can be purified by putting it in a freezer, allowing the safrole to solidify, and then filtering out the solid safrole. Let me fill you in on the facts of the matter. Sassafras oil is very stable in a supercooled state. You can put a bottle in a freezer for months, and never see a crystal of solid safrole form. Believe me, I've tried it. To get crystals to form, a seed crystal of solid frozen safrole would have to be added to the supercooled sassafras oil. Where do you get this seed crystal to start with? And at 80-90% pure safrole, the oil will then freeze into a virtual solid block, so what would filter out except the safrole that begins melting during the filtering process? This whole line of pursuit is a waste of time. Moreover, the small amount of impurities are actually beneficial if the HBr route is chosen for production of MDA or MDMA from the sassafras oil.

Starting with essential oils, how does one make the desired amphetamine from them? Let's take the conversion of sassafras oil to MDA or MDMA as the example to illustrate the various processes which can be used. If we go to *PIHKAL*, and read the recipe for MDA, you get the old classical procedure. Safrole obtained from sassafras oil is first converted to isosafrole (a propenylbenzene). This is done by putting safrole into a flask, adding some 10% alcoholic KOH, and then warming the mixture up to 243° C for 3 minutes. This isomerization works just fine so long as absolute alcohol is used, and the alcohol is allowed to distill off. You know that you have gotten isomerization, because the boiling point of safrole is 233° C.

The isosafrole is then mixed with acetone, formic acid and hydrogen peroxide to give the glycol mentioned in Chapter Ten. The reaction mixture is evaporated away under a vacuum, then the residue in the flask is heated with sulfuric acid in alcohol solvent to give the phenylacetone. The phenylacetone is then used to make the amphetamine by any of the methods given in this book.

My opinion on this method? It's a lot of work, the yields are on the low side, and that evaporation of the reaction mixture under the vacuum will destroy your aspirator. Peroxyformic acid is rough on metal. Let's use the more direct approach.

The first problem which confronts the chemist in the process of turning sassafras oil into MDA or MDMA is the need to obtain pure safrole from it. In spite of the fact that crude sassafras oil consists of 80-90% safrole, depending on its source, it is a good bet that the impurities will lower the yield of the desired product. The axiom "garbage in, garbage out" was custom made for organic chemistry reactions. It is simplicity itself to turn crude sassafras oil into pure safrole, and well worth the effort of underground chemists bent on MDA production.

Sassafras oil is an orange colored liquid with a smell just like licorice. It is a complex mixture of substances which is easily purified by distilling. To obtain pure safrole from sassafras oil, the glassware is set up as shown in Figure 13 in Chapter Three. The distilling flask is filled about $^2/_3$ full of sassafras oil, along with a few boiling chips, and then vacuum is applied to the system. A little bit of boiling results due to water in the oil, but heat from the buffet range is required to get things moving. Water along with eugenol and related substances distill at the lower temperatures. Then comes the safrole fraction. The safrole fraction is easily spotted because the "oil mixed with water" appearance of the watery forerun is replaced with a clear, homogenous run of safrole. When the safrole

begins distilling, the collecting flask is replaced with a clean new one to receive it. The chemist is mindful that the safrole product is 80-90% of the total volume of the sassafras oil. Under a vacuum, it boils at temperatures similar to phenylacetone and methamphetamine. When all the safrole has distilled, a small residue of dark orange colored liquid remains in the distilling flask. The distilled safrole is watery in appearance, and smells like licorice.

With a liberal supply of safrole obtained by distilling sassafras oil, work can then commence on converting it into 3,4 methylenedioxyphenylacetone. This is done in exactly the same manner as described in Chapter Ten. Any one of the three Wacker oxidations of the allylbenzene (safrole) to the phenylacetone (m-d-phenylacetone) can be used. When the essential oil contains a propenyl benzene, such as the B-asarone in calamus oil, then the electric cell discussed in Chapter Ten and *Practical LSD Manufacture* should be used to get the phenylacetone in high yield.

With the methylenedioxyphenylacetone obtained in this manner, the chemist proceeds to make it into XTC by one of the methods used to turn phenylacetone into meth. Of all the methods to choose from, the most favored one would have to be reductive alkylation using the bomb and platinum catalyst. The free base is converted into crystalline hydrochloride salt in exactly the same manner as for making meth crystals. It is interesting to note here that XTC crystals will grow in the form of little strings in the ether solution as the HCl gas is bubbled through it. Once filtered and dried, it bears a remarkable resemblance to meth crystals. It generally has a faint odor which reminds one of licorice.

To make MDA from the methylenedioxyphenylacetone, one has three good choices. Choice number one is to use the reductive amination method with a bomb, with Raney nickel catalyst and ammonia. See *Journal of the American Chemical Society*, Volume 70, page 2811-12 (1948). Also see *Chemical Abstracts* from 1954, column 2097. This gives a yield around 80% if plenty of Raney nickel is used. The drawback to this method is the need for a shaker device for the bomb, and also a heater.

A complete discussion of these two methods can be found in Chapter Twelve. The only difference is that the substituted phenylacetone is used instead of regular phenylacetone, and a substituted amphetamine is produced as a result. One should also see *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture* for a Convenient Tabletop MDA recipe using a Raney nickel cathode to do the hydrogenation, and also for a convenient method of making ammonia-saturated alcohol. MDA distills at about 150° C at aspirator vacuum of 20 torr, and MDMA will distill at around 160° C under the same vacuum. Poorer vacuum will result in higher boiling temperatures.

Another method for converting methylenedioxyphenylacetone to MDA is the Leuckardt reaction. My experience with mixing formamide with phenylacetone to get amphetamine is that using anything other than 99% formamide is a waste of time. You just get that red tar. Two ways have been found around that. These variations use the much more easily available 98% formamide. See *Chemical Abstracts* from 1953, column 11246, and Austrian patent 174,057. In this variation, 40 ml of methylenedioxyphenylacetone is mixed with 110 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 Five, until a temperature of 150° C is reached. This should take at least 5 hours. The yield is 70%, according to the patent.

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 which is produced is led away in plastic tubing. The formyl amide is then separated, and hydrolyzed by refluxing in a mixture of 60 grams of 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 free base obtained by making the solution strongly alkaline

to litmus by adding lye solution. The free base is then extracted out with some toluene, and distilled.

Most people don't get close to the 70% yield claimed in the patent for this method.

Another choice is to use the European Variation of the Leuckardt reaction, given in Chapter Five. The last I heard from Geert, the heat was closing in on him, but he was going to pass along an XTC recipe that is very popular over there. He says that they do it in an icebox! I haven't heard from him since, and that was nearly 2 years ago. This space is dedicated to him.

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 *PIHKAL* by Dr. Shulgin in the section under MDA, for full cooking instructions.

References

Psychedelics Encyclopedia by Peter Stafford.

The recommended dosage of MDA or XTC is about a tenth of a gram of pure material. TMA-2 is 40 milligrams.

The other good synthetic route of making MDA, MDMA and related psychedelic amphetamines from the substituted allylbenzenes found in essential oils like sassafras oil is a two-step procedure involving first reacting the substituted allylbenzene (e.g., safrole from sassafras oil) with HBr to make the corresponding phenyl-substituted 2-bromopropane. Then this substance is mixed with an alcohol solution containing excess ammonia or methylamine to yield MDA or MDMA from, for example, safrole. Heating is required to get a good yield of product. Details on this procedure are found in the chapter covering the production of meth or benzedrine from benzene and allyl chloride (Chapter Twenty One). The reason why it is in that chapter is because the final step of heating the 2-bromopropane compound with ammonia or methylamine solution is pretty much identical. Some further commentary on this route not found in that chapter is called for.

The addition of HX (HCl, HBr, HI) to a double bond is a general reaction, meaning most all double bonds, other than those found in benzene rings, will add HX. Of these three acids, HBr adds most easily to double bonds. It is also the only one that will add abnormally, meaning that one can get, besides the 2-bromopropane, the 3-bromopropane also. Exposure to strong light or oxidizing substances promotes the abnormal addition, so this reaction shouldn't be done in full sunlight.

The strength of the HBr used in reaction has a great effect upon the yield and speed of the reaction with safrole. The less free water floating around in the acid, the better it reacts with safrole. So dry HBr gas will react best with safrole, followed closely by 70% HBr, while the ACS reagent 48% HBr is practically useless as is.

Another point to be aware of is cleavage of the methylenedioxy ether by HX. HI is much better at cleaving this ether than is HBr, which is better than HCl. It is because of "ether" cleavage that the temperature during this reaction must not be allowed to rise above the stated limits in the procedures given in this book. If your magnetic stirrer gets warm while working, the batch must be insulated from this source of heat.

An obvious variation upon this procedure which would pop into the head of any thinking chemist reading this tract would center around adding dry HCl to safrole by bubbling dry HCl through a toluene solution of sassafras oil to get the 2-chloropropane, and reacting this substance with ammonia or methylamine like the other phenyl-2-chloropropanes listed in the *Journal of the American Chemical Society* article cited in the meth or benzedrine from benzene and allyl chloride chapter in this book. My observations on this route will be useful if someone is contemplating this procedure.

First of all, dry HCl adds only slowly to safrole at room temperature. A toluene solution of sassafras oil literally reeking with HCl, sealed up and kept at an average temperature of 90° F for three weeks, resulted in only about 10% conversion of the safrole to chlorosafrole. No doubt, some further heat must be applied to the mixture to get reasonably complete conversion of the safrole to chlo-

rosafrole. HCl doesn't cleave ethers very well, so this can be considered safe.

How does this observation jibe with the *Journal of the American Chemical Society* article in which they postulate that when allyl chloride adds to benzene or substituted benzene, the 2-chlorophenylpropanes are the result of HCl adding to the double bond of the allylbenzene? Either the theory was mistaken, or iron chloride is a catalyst for adding HCl to the double bond. I haven't yet checked this out personally, but it's worth a try.

Further, once one has chlorosafrole, what good is it? See the above cited *Journal of the American Chemical Society* article. You will note that the yields obtained converting similar phenyl-substituted ether 2-chloropropanes is pretty low, down near 10%. That's why bromosafrole is used to make MDA or MDMA. The bromine atom is much more easily replaced with ammonia than is chlorine. It's termed a better leaving group. The iodine atom is a much better leaving group than is the bromine atom, so even better results should be had reacting iodosafrole with ammonia or methylamine. One would expect that lower temperatures could be used, maybe even room temperature. This would avoid all the tar formed as a byproduct when heating bromosafrole.

Chlorosafrole can be converted to iodosafrole by refluxing one mole of chlorosafrole with 2 moles of sodium iodide in a saturated solution in acetone for about 15 minutes to ½ hour. After cooling this reaction mixture, the sodium chloride that precipitates out of solution is filtered. Then the acetone is taken off under a vacuum. The resulting residue of iodosafrole and NaI crystals is extracted with toluene to remove the product from the NaI crystals, which can be reused. This toluene extract is shaken with water containing some sodium thiosulfate and a little HCl. This destroys iodine formed by decomposition of the NaI. Snorting iodine really sucks. Exposure to light speeds the decomposition of NaI to iodine, especially in solution. Experimenters using this procedure are invited to write in with their results.

A final word needs to be said about the Ritter reaction. Since safrole and related allylbenzenes from essential oils are all allylbenzenes, one would assume that the Ritter reaction would be directly applicable to them. Such is not the case. See *Chemical Abstracts*, Volume 22, page 86, for an article titled "Cleavage of the Methylenedioxy Group." Here they detail how concentrated sulfuric acid quickly cleaves the methylenedioxy group. As a consequence, brave experimenters wishing to use the Ritter reaction to make MDA must use the substitutes for sulfuric acid which are listed in the *Journal of the American Chemical Society* article cited in Chapter Fourteen. Substitutes include methanesulfonic acid and polyphosphoric acid. Directions for how to make the latter from phosphoric acid and P_2O_5 are to be found in the *Merck Index*. A final caveat for those trying to make chlorosafrole is also to be found in that article. The article states that fuming HCl, heated to 100° to 130° C in a sealed tube, is a potent cleaver of the methylenedioxy group. Heating of safrole with dry HCl must be held well below this level.

Know Your Essential Oils

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

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

Indian Dill Seed Oil — contains up to 53% dill apiol (3,4-methylene-dioxy-5,6-dimethoxy-allylbenzene). Boiling point is 296° C with decomposition at normal pressure. Aspirator vacuum will distill 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.

Oil of Bitter Almonds — contains around 95% benzaldehyde. This is a precursor to phenylacetone or amphetamine.

Oil of Cinnamon — contains 80-90% cinnamaldehyde. This can be reduced to allylbenzene with borohydride.

WARNING!! Some wholesale distributors of essential oils are being leaned upon to give up their customer lists. The heat wants to know who is buying sassafras oil, and oil of bitter almonds. They will soon want to know who is getting cinnamon oil, after this book hits the streets. Oils fall under the definition of "mixture" in the chemical division act, and so are not subject to reporting. However, these companies are knuckling under to pressure and threats. Buying retail is still completely safe. Be warned!

References

PIHKAL by Dr. Shulgin
The Essential Oils by Ernest Guenther
Psychedelics Encyclopedia by Peter Stafford

Chapter Nineteen
Ice

At the time of the writing of the second edition, the latest drug craze was the smokable form of methamphetamine called "ice." At the writing of this fifth edition, this material was still popular, with most usage being confined to those with serious drug problems.

I'm not going to endorse or encourage the foolhardy practice of smoking meth. Seeing firsthand what this stuff does to rubber stoppers, corks, and razor blades, I can only imagine what it does to lung tissue. My opinion on this practice is similar to my opinion on injecting the substance. If snorting the hydrochloride salt doesn't get you as wired as you could ever want to get, it is time to give up and find something else to fill your spare time with.

I have never made nor used "ice" as such, but I can tell you how to get smokable forms of meth. Since the godless importers of this stuff have already created a market for it, it's only right that I help American technology catch up.

The regular hydrochloride salt is poorly suited for smoking, as most of the product will get charred during the heating. The free base is quite smokable, but it is a liquid, and as such is not easily sold, as it is unfamiliar. I will cover this matter from two angles: a home technique that works well to base your personal stash for smoking, and a more large-scale procedure for commercial use.

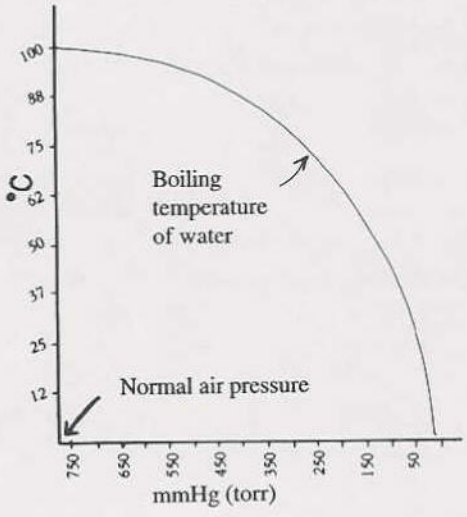
To base your stash and smoke it, mix your stash with an equal amount of bicarb, and then with a dropper drip a little water onto it with stirring to make a paste. Now take some aluminum foil, and with your finger indent a well into it about an inch deep. Into this well put some of the paste, and heat it from underneath with a lighter. Suck up the smoke with a straw.

For making a crystalline yet volatile derivative of meth similar to crack rocks, one first needs meth free base. All of the production methods in this book yield meth free base. Then to this free base, add dry ice. This will convert the free base to the carbonate, which can be chipped and scraped out of the beaker when the dry ice has evaporated. Use of a solvent during this conversion will be helpful.

Chapter Twenty
Calibrating the Vacuum

Before he starts doing the vacuum distillations described in this book, the underground chemist needs to know what kind of vacuum he is able to produce inside his glassware. This is important because the temperature at which a substance distills under vacuum depends directly on how strong the vacuum is. The distillation temperatures given in this book assume a vacuum of about 20 torr for an aspirator and about 5 torr for a vacuum pump. This chapter describes an easy method by which the chemist finds out just how strong his vacuum is. Once he knows how good his vacuum is, he adjusts the temperatures of his distillations accordingly. The better the vacuum, the lower the temperature at which the substance will distill. He keeps in mind that an aspirator will get a better vacuum in winter because the water flowing through it is colder in that season. The vacuum obtained with a vacuum pump may get poorer over time because solvents from the chemicals he is distilling, such as benzene, may dissolve in the pump's oil. If this happens, he changes the oil.

To begin, the chemist sets up the glassware for fractional distillation as shown in Figure 13 in Chapter Three. He uses a 500 ml round bottom flask for the distilling flask, and a 250 ml flask as the collecting flask. He uses the shorter condenser, and puts 3 boiling chips in the distilling flask along with 200 ml of lukewarm water. He lightly greases all the ground glass joints. (This is always done when distilling, because the silicone grease keeps the pieces from getting stuck together, and seals the joint so that it doesn't leak under the vacuum.)



He turns on the vacuum full force and attaches the vacuum hose to the vacuum nipple of the vacuum adapter. The water in the distilling flask should begin boiling immediately. As the water boils away, the temperature shown on the thermometer steadily drops. Finally, the water gets cold enough that it no longer boils. He notes the temperature reading when this happens, or better yet, disconnects the vacuum and takes apart the glassware and takes the temperature of the water in the distilling flask. Using a graph such as the one

above, he reads off the vacuum that goes with the boiling temperature.

If his vacuum is bad, the water will not boil. In that case, he checks to make sure that all the joints are tight, and that the stopper in the claisen adapter fractionating column is not leaking. He also makes sure that his vacuum hose is not collapsed. If, after this, the water still doesn't boil, he has to heat the water. He turns on the buffet range at low heat while continuing the vacuum. In a while the water begins boiling. He checks the temperature reading on the thermometer while it is boiling, and notes the temperature. From the graph he reads off the vacuum that goes with that boiling point.

His vacuum should be 50 torr or lower to be able to make methamphetamine. If his vacuum reading is more than 50 torr, he gets a new aspirator or changes the oil in the vacuum pump.

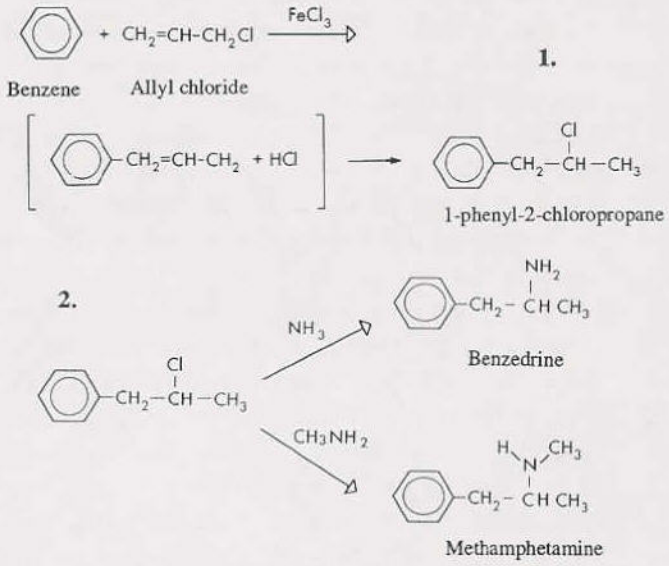
The chemist can use this information to adjust the temperature at which he collects his distilled product. The boiling temperature of phenylacetone is about 105° C at 13 torr, and about 115° C at 20 torr. The boiling temperature of N-methylformamide is about 107° C at 20 torr. The boiling temperature of methamphetamine is about the same as phenylacetone. Phenylacetone and methamphetamine should be collected over a 20-degree range centered on their true boiling points. This makes sure that the chemist gets all of it. The purification scheme he goes through before distilling removes all the impurities with boiling points close to that of his product.

Chapter Twenty One
Production From Allyl Chloride and Benzene

At present there are a few viable options left for large-scale manufacture of meth or benzedrine. Phenylacetic acid, benzyl cyanide, and even benzyl chloride are all history. At the time I was writing the Fourth Edition, I had just heard that benzaldehyde and nitroethane had also just been added to the Chemical Diversion reporting list. Allylbenzene is now toast too, although it was never a big item of commerce anyway. This dwindling selection of raw materials is the result of the Chemical Diversion and Trafficking Act of 1988 discussed in Chapter One. Over the years, a continually lengthening list of chemicals has been subject to reporting requirements when sold. These sales reports then go to the DEA, which sorts through the ever-increasing number of reports to try to develop leads. As you can well imagine, the more chemicals listed, the more chaff there is mixed with the wheat, and the less effective this snoopervision scheme is. It is my lifelong aim to get every chemical under the sun put on this reporting list, with the end result being that *none* of them are on a list.

In keeping with this spirit, there are a couple of good methods left out there that are suitable for scaling up to an industrial level of production, while using materials which are not subject to reporting. Sharpen your pencils, and order fast before they are gone. The reporting list has closely mirrored each of my previous editions, and I expect no change in this pattern.

One of these good remaining methods will be presented in this chapter. For the original report on this quite-versatile synthetic route, see *Journal of the American Chemical Society*, Volume 68, pages 1009-11 (1946). The route is a Friedel-Krafts alkylation of benzene with allyl chloride to yield 1-phenyl-2-chloropropane. Then this is reacted with methylamine or ammonia to give meth or benzedrine, respectively.



The first reaction is your typical Friedel-Krafts alkylation, with the complication that the HCl produced in the reaction then goes on to add to the double bond according to Markonikov's Rules to yield 1-phenyl-2-chloropropane from the intermediate allylbenzene. The yield from this reaction is unavoidably on the low side because of the tendency of the product to then further go on to react with either benzene to give 1,2,-diphenylpropane, or with more allyl chloride to give multiple-ring substituted products. These can be removed by a

fractional distillation, so getting a pure product is no problem, but the yield is going to be only about 35% based upon the allyl chloride used. Unreacted benzene can be recycled back into future batches to cut chemical consumption.

To do the reaction, a suitable glass or stainless-steel reaction vessel is chosen. A 1000 ml round-bottom flask is perfect, but substitutes such as the stainless-steel canister flask depicted in Chapter One will work fine. The size batch given here is right at the upper limit for which magnetic stirring will work. If this procedure is scaled up, only mechanical stirring will work to get the $FeCl_3$ up off the bottom of the flask. Moisture is harmful to this reaction, so be sure that the vessel is dried, and that the reactants are free of water.

Now nestle this vessel into an ice-salt bath, and add 360 ml benzene, then 32 grams anhydrous ferric chloride. When the contents have chilled down to about -20° C (with strong stirring), slowly with stirring add 76 grams (80 ml) of allyl chloride. This addition should take about two hours, and is best done dropwise. After the addition is done, continue stirring for another two hours. The reaction mixture will fume a little HCl, so some ventilation is called for. Don't allow the temperature to climb above -10° C.

Next, pour the reaction mixture into a one-gallon glass jug containing 1 kilo of crushed ice and 100 ml of concentrated hydrochloric acid. Stopper the jug, and shake until the ice has melted. Now separate off the benzene layer floating on top of the water by use of a sep funnel, and wash this benzene layer with some dilute hydrochloric-acid solution, and then with some distilled water.

Next, filter this benzene solution, and dry it over some anhydrous $CaSO_4$. This drying is important because removal of water prior to distillation allows direct recycling of the distilled benzene. If the water was carefully separated off the benzene layer, about 10 grams of $CaSO_4$ in contact with the solution for ½ hour should do the trick.

Now distill this solution through a claisen adapter without glass packing to get a rough separation of the components. The unreacted benzene distills first at a temperature of 80° C or so. When nearly all of the unreacted benzene has distilled, the receiving flask should be changed, and a vacuum applied, slowly at first so as not to cause too vigorous boiling, then at full force. The product, 1-phenyl-2-chloropropane, distills at about 80° C at a vacuum of 10 torr. Tarry gunk remains in the flask, and should be cleaned out with solvent at the end of the distillation.

The crude product should be redistilled through a fractionating column under vacuum to get pure product. The yield is about 50 ml. The recycled benzene should be stored in a sealed bottle until reuse.

This second step of production is also to be found in *Journal of the American Chemical Society*, Volume 68, pages 1009-11 (1946). It is the ammonolyis of the 1-phenyl-2-chloropropane with either methylamine or ammonia to yield meth or benzedrine respectively. This reaction is done in alcohol solution with heating inside a sealed steel pipe. The sealed steel pipe is required because the reaction is done at the temperature above the normal boiling point of the solvent, so a pressure vessel must contain the reactants. The main competing side reactions are further reaction of the product with 1-phenyl-2-chloropropane to give a high molecular weight secondary or tertiary amine. This is suppressed by using a large excess of ammonia or methylamine. Also, the 1-phenyl-2-chloropropane can react with the alcohol solvent to form an ether. It's also possible for the chloropropane to react with water to give the corresponding alcohol. Just plain tar formation is also prevalent. A yield of about 50% and 60% is obtained for benzedrine and meth respectively, based upon the amount of 1-phenyl-2-chloropropane used.

Now, let's vary from the procedure given in the article. In the article they just mixed strong ammonium hydroxide (28% NH_3) into methyl or ethyl alcohol. Then they mixed in the of 1-phenyl-2-chloropropane, and heated the mixture inside a pipe. They said that they got the same yield of meth or benzedrine with this watery reaction mixture as they did with a dry one. That may be the case. However, if phenyl substituted starting materials are used, such as bromosafrole, all one will

get is tar from this watery reaction mixture. Perhaps this presence of water explains why they got such low yields from the other chloropropanes they made in their experiments. It is your Uncle's opinion that a fairly dry reaction mixture should always be used. The best way to get this dry reaction mixture is to add anhydrous ammonia from a cylinder to the alcohol. First cool down the cylinder and the alcohol in a freezer, then invert the cylinder, crack open the valve (strong ventilation!), and add about 100 ml of liquid ammonia in about 400 ml of alcohol solvent for each 50 ml of 1-phenyl-2-chloropropane or bromosafrole used in the reaction. An alternative method of making fairly dry alcohol solutions of ammonia is given in *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture.*

This same method can be used to get alcohol solutions of methylamine, or one could place some methylamine free base in water solution inside a distilling flask, and apply heat to force the vapors up through the ice cold condenser, and then through some tubing into a chilled and stirred beaker of alcohol. See Chapter Four for a diagram of such an apparatus.

Let's further vary from the procedure given in the article. They used methyl or ethyl alcohol as solvent. One of the side reactions is with the alcohol solvent to form an ether with the phenyl-2-chloropropane or bromosafrole. If one substitutes a secondary alcohol such as isopropyl alcohol, this reaction is less likely to occur. Virtually pure and water free isopropyl alcohol is easily available. It is a product called ISOHEET gas line de-icer. Look for it at the gas station or hardware store. This will give better yields than methyl or ethyl alcohol. One would naturally wonder if replacing the alcohol solvent with something inert like toluene wouldn't further improve yields. Maybe, give it a try.

So now we have a solution of 50 ml of the chloro or bromo propane in about 400 ml of solvent just saturated with ammonia or methylamine. Now pour this solution into one or a series of steel pipes. They should be threaded at each end so that the caps may be screwed on at both ends like a pipe bomb. The plumbing section at the hardware store is well stocked with these parts. Screw the cap on tightly when filled.

The reader should be aware that commercial steel pipe and caps are heavily galvanized with zinc. The zinc must be stripped off prior to use in the procedures outlined in this book. Zinc is stripped off by immersing both the pipe and the end cap in 5% hydrochloric acid solution until the violent bubbling slows to a crawl. Then the pipe and caps should be thoroughly rinsed off in clean water and then assembled. A pipe wrench will be required to get the caps on tight enough to prevent leakage while cooking.

This degalvanizing process can lead to some confusion as to when the zinc has been removed. Take for example this conversation I had on the Net:

Posted by piper on March 03, 1998 at 10:04:37:
DE-GALVANIZING
What if someone were stripping the zinc from galvanized pipe, and that went good, but the end caps have been going for two days now, and there is STILL zncl2 coming off them. Someone needs those caps soon! Can the process be accelerated? Just up the strength of the HCl solution? Maybe some DC current? Also, Teflon tape melted @ 130° C. What's up with that? Is it bad?

Posted by Uncle Fester on March 06, 1998 at 17:21:27:
In Reply to: Re: DEGALVANIZING-hypothetically! Posted by metal man on March 06,1998 at 04:59:06:
Metal Man is correct when he states that concentrated nitric will strip plates off steel without attacking the steel. If, however a little bit of water gets in the mixture, the steel will be history. The end caps of pipes have the thickest amount of zinc on the inside. I strip zinc off steel all the time at work. The end caps should have been completely stripped within a couple of hours using 5-7% HCl. That's conc. HCl diluted 6 to 7 times. Under that zinc is mild steel. It too will fizz in HCl solution, but much slower. Using a scrub pad, one can go into that end cap and scrub off the surface layer of gunk that forms while metals are being stripped. Zinc while it dissolves will be black. Exposed iron or mild steel will be a brighter color.

After rinsing, application of some copper sulfate solution will show exposed steel. The old copper displacement reaction, leaving a copper deposit. For a part to take two days to strip in HCl, either such a small amount of HCl solution was used that it has been exhausted by the stripping action, or the dissolution of the underlying iron has been mistaken for more stripping of zinc.

This possible source of confusion can be eliminated by using stainless-steel pipes and caps rather than galvanized steel. These can be used as is, without any treatment.

Now with the reaction mixture inside the pipe, it's time to heat the mixture. For production of meth or benzedrine from 1-phenyl-2-chloropropane, the preferred heating procedure is to heat at 160° C for about 9 hours. For production of MDA or MDMA from bromosafrole, the preferred heating is at about 125° C for 3 to 4 hours. One does this heating by putting a pan with cooking oil on a stove burner, and then immersing the pipes in the cooking oil. The temperature of the oil bath is then held at the desired temperature for the required period of time.

After the cooking period is complete, the pipes are removed from the heating bath, and allowed to cool down. Once they have cooled, they can be opened, and the contents poured into a distilling flask. Most all of the alcoholic ammonia or methylamine should be distilled off. In the case of methylamine, great care should be taken to catch its vapors for reuse. This is done using that apparatus shown in Chapter Four, making a fresh load of methylamine in alcohol for use in the next run. The last portion of alcohol should be removed using a vacuum, down to a volume of about 100 ml.

The residue in this flask should be shaken very vigorously with 10% HCl solution. This converts the amine products into their hydrochlorides, which are water-soluble. The shaking should continue for at least 5-10 minutes to get all of the product extracted out of the gunky tar matrix. Now extract this 10% solution which contains the product with a couple 50 ml portions of toluene. This removes entrained gunk. Finally, make the solution strongly alkaline to litmus with lye. This generates a lot of heat, and should be done cautiously with shaking between adds of lye. When the solution is strongly alkaline, shake vigorously for about 5 minutes more, then check again the pH of the water to make sure it is still quite alkaline. There should be a healthy amphetamine layer floating on top of the water. With the hot water, it will give a strong aroma of amphetamine when sniffed. Cool the solution, and extract with two 50 ml portions of toluene.

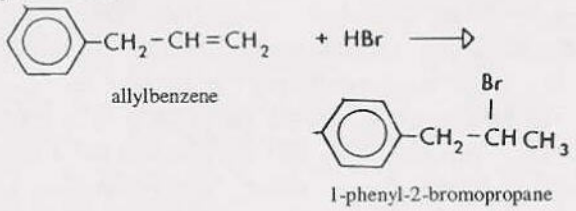
The toluene extracts contain the amphetamine. This should be distilled in the usual manner as described in Chapter Five to yield about 25 ml of amphetamine or meth. This is converted to the hydrochloride salt as also described in Chapter Five, to give about an ounce of pure benzedrine or meth. This procedure can be scaled up as desired. In case you were wondering, the boiling point of benzedrine free base is about 15° C lower than meth.

Distilling must be done when using this method, because there is just no other way to remove the higher amines made as byproducts. If one is making MDA by this method, those higher amines produce a scary and paranoid trip. If one can't distill, this method shouldn't be used.

Making Bromosafrole from Safrole, and 1-phenyl-2-bromopropane from Allylbenzene

To use this "pipe bomb" method to make MDA or MDMA from bromosafrole or amphetamine and meth from 1-phenyl-2-bromopropane, one of course first needs the bromo compound. Good luck finding that stuff. Luckily, it's not a very complicated procedure to cook your own. Let's take, for example, making 1-phenyl-2-bromopropane from allylbenzene. Allylbenzene was covered back in Chapter Nine. It's pretty easily made from cinnamon oil, or cinnamaldehyde. This conversion to the bromo compound and then the "pipe bomb" reaction is the alternative route to the Wacker oxidations to phenylacetone. Both routes are quite practical, and can be scaled up at will. Directions for making 1-phenyl-2-bromopropane can be found in

the *Journal of Biological Chemistry,* Volume 108, pages 622-23, by H.E. Carter. Reaction with hydrobromic acid gives the bromopropane from allylbenzene:



1-phenyl-2-bromopropane

His procedure is to put 200 ml of glacial acetic acid in a bottle along with 200 ml of 48% hydrobromic acid. This mixture is chilled in an ice bath, then 100 ml of allylbenzene is added to the bottle. A stopper is wired in place on the bottle, and the mixture is slowly allowed to come to room temperature with occasional shaking. After 10 to 12 hours, the original two layers merge into a clear red solution. After 24 hours, the contents of the bottle are poured onto crushed ice.

When the ice has melted, the 1-phenyl-2-bromopropane will have formed an oily dark liquid layer separate from the acid-water solution. It may be at the bottom of the beaker if lots of crushed ice was used, or it may be floating on the top if less was used. This crude product should be separated from the acid-water using a sep funnel. Then the acid-water should be extracted with about 100 ml of toluene. This extract should be added to the crude product. The combined extract and crude product are then washed with water, and then with bicarb solution. Fizzing from the bicarb solution will be produced as it neutralizes acid in the crude product, so beware of pressure building up in the sep funnel. Always wear eye protection so that mists of this stuff don't end up in your eyes.

Then when all the acid has been neutralized, as shown by lack of fizzing when put in contact with fresh bicarb solution, the toluene-bromopropane solution should be placed in a distilling flask, and fractionally distilled to remove the toluene-water azeotrope, and then the remaining toluene solvent. When the toluene has mostly distilled away, a vacuum should be applied and the 1-phenyl-2-bromopropane distilled. It will boil at a temperature similar to phenylacetone, roughly at 120° C under a good aspirator vacuum of around 20 torr. Less efficient vacuum will result in higher boiling points. The yield is around 235 grams (180 ml).

So that method using 48% HBr apparently works fine with allylbenzene. When using substituted allylbenzenes such as safrole, however, it is quite useless. There is just too much water in the reaction mixture (48% HBr is 52% water), and the HBr in the mixture just refuses to add to the double bond to give bromosafrole. The general method, which works equally well with allylbenzene or the safrole found in sassafras oil, can be found in the *Journal of the American Chemical Society.* The paper dates to 1946, Volume 68, pages 1805-6.

In this general procedure, the chemists mixed 100 ml of allylbenzene with 250 ml of glacial acetic acid. One could simply add 100 ml sassafras oil with 250 ml of acetic acid and get basically the same solution. Then to this solution with rapid stirring, they bubbled a rapid stream of anhydrous HBr gas from a cylinder for a period of about two hours, while keeping the mixture cooled with an ice bath. The fumes of HBr are injurious, so ventilation out a window or working outside is recommended. The bubbling of HBr produced the formation of two layers in the reaction mixture. Then they added 200 ml more glacial acetic acid to make a homogenous solution, and kept the mixture cold overnight.

In the morning, they poured this reaction mixture onto ice, and recovered the bromo compound in exactly the same way as in the first example. This general method is good, but that HBr in a cylinder isn't that easy to come by, and the dangerous fumes from the bubbling are something one would want to avoid. There is a way around that.

Thirty-six percent HBr in glacial acetic acid is commercially available. This makes doing the reaction much simpler. One just mixes one volume of sassafras oil or allylbenzene with two or three volumes of ice cold 36% HBr in acetic acid, and then stirs the mixture with cooling for about a day. Then upon pouring the mixture onto ice, the bromosafrole or 1-phenyl-2-bromopropane is recov-

ered just as in the first example. Bromosafrole smells a lot like phenylacetone, and its boiling point is about the same as m-d-phenylacetone, around 150-160° C under a vacuum of about 20 torr.

So how much water can you have in the acid, and still get bromosafrole from sassafras oil? See *Chem. Abstracts* 1961, column 14350.

Unfortunately, it uses 70% HBr, a quite uncommon reagent. To do this procedure, a beaker containing 100 ml of 70% HBr is chilled to 0° C in an ice bath. Then, with stirring, 50 ml of sassafras oil is added dropwise. The stirring and cooling is continued for an additional 14 hours, then the mixture is poured onto a few hundred grams of crushed ice. This mixture is stirred or shaken until the ice melts, then the product is extracted with ether or toluene. The organic layer should then be separated, and washed with some water, followed by some bicarb solution, to remove traces of acid. The solvent is then evaporated away, either by distillation or under a vacuum to give a virtually 100% yield of the product, bromosafrole. Its aroma is similar to phenylacetone.

Let's suppose that all one can get is the standard ACS reagent 48% HBr in water. How can one get bromosafrole using this stuff? If one looks in the *Journal of the Alabama Academy of Science,* Volume 64, pages 34-48 (1993), one can find a claimed method. It uses the standard ACS reagent HBr, which is 48% strength. In this variation, 50 ml of sassafras oil, 250 ml of 48% HBr, and a magnetic stirring bar at least one inch in length are placed in a 500 ml volumetric flask. The top is stoppered to keep the nasty vapors inside (HBr is bad to breathe), and fast stirring is continued for one week at room temperature. A layer of cardboard between the stirrer and the flask will help keep the stirrer from warming up the solution. Within a few hours, the reaction mixture takes on the color of a cheap burgundy. A homogenous mixture never results. When stirring stops, the product just floats on top of the acid. At the end of the week of stirring, the bromosafrole is isolated just as in the above method. 250 grams of crushed ice are used. It is best if the product is distilled to recover unreacted safrole, and remove the colored matter. Bromosafrole distills at about 160° C with aspirator vacuum. Note here that the Alabama article chemists used their crude bromosafrole without distilling it, and got quite pure XTC as the product. I and others have found that 48% HBr is quite ineffective in reaction with safrole.

An alternative procedure using 48% HBr gives superior results. 48% HBr can be dehydrated to HBr gas upon contact with phosphorus pentoxide, P_2O_5. Sulfuric acid can't be used as the dehydrator because it breaks the HBr down to bromine gas. To use this variation, a gas bubbler is set up as shown in Figure 19. In the round bottom flask, place a bed of phosphorus pentoxide. Into the sep funnel or dropping funnel, place about 210 ml of 48% HBr. Lead the bent glass tubing into a beaker containing about 160 ml of sassafras oil. It is best to dilute the safrole with a couple of volumes of solvent such as toluene or glacial acetic acid, because this will help to catch and hold the HBr gas as it is bubbled into solution.

Now chill down the sassafras oil solution in ice, and begin dripping 48% HBr onto the bed of phosphorus pentoxide. When it hits the P_2O_5, the water in the 48% HBr reacts with the phosphorus pentoxide to make phosphoric acid, and the HBr puffs off as a gas which escapes down the glass tubing into the sassafras oil solution. All joints and plugs on the gas bubbler must be tight so that the HBr is forced into the sassafras oil solution. It is also a good idea to magnetically stir the sassafras oil solution so that the HBr bubbles are more likely to either react immediately or go into solution, rather than escaping. HBr gas is very foul and dangerous to breathe, so good ventilation must be provided.

As the approximately 210 ml of 48% HBr drips onto the bed of P_2O_5, pay attention to its reaction when it hits the P_2O_5. If the rate of gas generation goes down, the bed of P_2O_5 may need some sloshing around or stirring up with a glass rod, or even the addition of some more P_2O_5.

When the bubbling of HBr is completed, the sassafras oil solution should be poured into a stoppered bottle, and kept cold overnight to complete the reaction to bromosafrole. It is then poured onto

crushed ice as in the other procedures, separated, washed, and distilled to yield about 100% bromosafrole.

One might want to consider doing this drying of the 48% HBr the other way around. By cautiously adding P_2O_5 to a stirred mixture of 48% HBr and glacial acid, one should be able to dehydrate it enough to react with safrole. How much would one have to add? I don't know. One mole of P_2O_5 reacts with three moles of water, and the phosphoric acid formed then exerts a further drying action by associating with water molecules. One could only find out by trying.

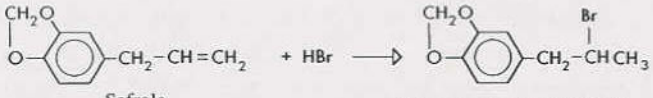
Another way of drying 48% HBr is one I came up with a few years ago. I got this procedure to work on the first try, so I considered it a slam dunk gimme. Apparently, other people have had some trouble doing it, so let me give more detail.

To get good yields of bromosafrole from 48% HBr and sassafras oil, mix one part sassafras oil with one part glacial acetic acid and two parts 48% HBr in a nearly full Erlenmeyer flask. Chill this mixture down in ice, then with strong magnetic stirring pass a stream of dry HCl gas into the solution for about an hour. See Chapter 5 for the dry-HCl gas-generator. How much HCl to pass into solution? Well, for a batch using 50 ml of sassafras oil, the amount of dry HCl generated by dripping 75 to 100 ml of sulfuric acid onto a half-full 500 ml flask of salt-hydrochloric-acid paste is about right. A little bit more wouldn't hurt. Good ventilation is required!

A nearly full Erlenmeyer is used to give maximum column depth for the bubbles of HCl to rise up through. The drying is a surface phenomena. An Erlenmeyer is used because the inward sloping walls slow up the rising bubbles. A plug of glass wool stuck in the neck of the flask down into the solution would slow them up some more.

As the dry HCl passes into the solution, it dehydrates the 48% HBr, causing it to react with the safrole. The dehydration and the reaction both generate a good deal of heat, so fresh ice will periodically have to be put into the bath around the reaction flask. The temperature of the reaction shouldn't be allowed to rise above 10-15° C. The reaction mixture first turns green then blue, then purple, and finally burgundy. When the bubbling with dry HCl is finished, stopper the flask and continue stirring in the cold for two days. Sometime around a day into this stirring, no separation of phases can be seen when stirring ceases.

I would say that the maximum temperature seen by the reaction mixture during this time was roughly 20° C. Keeping things cold in Wisconsin is easy.



Safrole

3,4-methylenedioxyphenyl-2-bromopropane
a.k.a. – bromosafrole

The amount of dry HCl 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 HCl, 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 the organics clinging to the glass. If it still smells like the candyshop fragrance of sassafras oil, an additional bubbling with dry HCl 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 HCl one is getting. If the aroma has changed to something more chemical and fruity, yes, just like phenylacetone, sufficient HCl has been added.

When two days of stirring are completed, the batch is poured onto crushed ice, as in the other methods. When the ice has melted, a little bit of toluene is added (a volume about equal to the amount of sassafras oil used), and the water-bromosafrole mixture shaken. Prior to adding toluene, the bromosafrole will likely be on the bottom

of the container, but after adding toluene and shaking, it should be floating on top. It's still burgundy-colored. Separate the bromosafrole layer with a sep funnel, and then wash it with about 3 volumes of water. Add bicarb slowly until the fizzing stops. This will knock out the carried-over HBr, HCl and acetic acid. Shake some more, then add a little more bicarb to make sure all the acid has been neutralized.

Separate the toluene-bromosafrole, and place it in a distilling flask. Distill off the toluene at normal pressure, then vacuum-distill the remaining bromosafrole. A vacuum that distills safrole at 110° C will distill bromosafrole at about 140-145° C. Some chlorosafrole distills at about 125° C. It can be used as is, or the chlorosafrole can be converted to iodosafarole according to the directions found in Chapter Eighteen in this book. The yield is about 66-75% conversion to bromosafrole, with the remainder being unconverted safrole and chlorosafrole. Bromosafrole smells a lot like phenylacetone. It may turn pink on standing, and should be stored in a freezer until used.

Last, but certainly not least, check out the Pugsley Bromosafrole Recipe in *Advanced Techniques of Clandestine Psychedelic & Amphetamine Manufacture*. People have been getting very good results using this procedure. Essentially, it involves reacting sulfuric acid with sodium or potassium bromide in ice cold DMSO solvent to give anhydrous HBr solution. Sassafras oil is then added to the reaction mixture to give virtually 100% yields of bromosafrole.

Chapter Twenty Two
Phenylacetone From Benzene and Acetone

This procedure makes use of the simple and common solvents, benzene and acetone, and links them together to form phenylacetone. Back when I was cooking phenylacetone, I often fantasized about how this could be done. Little did I know that it had been accomplished by a couple of Russians a few years before my cooking began.

This isn't a procedure to get overly excited about, as the yields are low (36% based upon the manganese III acetate used), and a quite dilute solution is required. This procedure is most suited to someone willing to do large-scale cooking, not the typical basement experimenter.

The interested reader should see *Chemical Abstracts*, Volume 77, column number 151620 (1972), and *Journal of the American Chemical Society*, Volume 93, pages 524 to 527 (1971), and *Bulletin of the Academy of Science of the USSR*, Volume 21, number 7, page 1626 (1972).

In a large glass pot, as for instance one could get from people who sell milk pipeline equipment to dairy farmers, place 20 moles of acetone (900 ml). Hardware store acetone can be used by drying it with $^{1}/_{10}$ volume of calcium chloride, also available at the hardware store as ice melt. Then add 5 moles of benzene (340 ml), and 1000 ml of glacial acetic acid, and one mole of manganese III acetate (268 grams; price about $500). Equip the flask with a reflux condenser.

These ingredients are mixed together, and then heated at 70° C until the brown color of Mn^{+3} disappears (about 2 to 3 hours). Then the contents are poured into a large stainless-steel distillation set up as described in Chapter One of this book, and the acetone, benzene, and part of the acetic acid are distilled off under reduced pressure. The weak vacuum produced by cheapie vacuum pumps, or low powered aspirators, is about right for this vacuum distillation. By chilling the receiving flask in ice, the unused acetone and benzene can be recovered for reuse.

Then the residue, which consists of phenylacetone and other organic products dissolved in acetic acid along with Mn(II) acetate, should next be diluted with several volumes of water. The product phenylacetone can then be extracted from this watery mixture, with toluene. The extracts should next be washed with dilute sodium hydroxide solution, and then the toluene-phenylacetone solution can be distilled to yield around 25 ml phenylacetone.

To get decent results from this reaction, the amount of water in the reaction mixture should be held to under 1%, and preferably under ½%. Improperly dried acetone is a prime culprit when tracking down sources of water in the reaction mixture. Water introduced from Mn(II) acetate can be removed by distilling off the water-acetic acid azeotrope at 76° C.

The other main problem with this reaction, besides the large dilution used is the need for Mn(III) acetate. This expensive material isn't something one can find on the hardware shelves. Mn(II) acetate, on the other hand, is a very common industrial

chemical, used as a mordant in dying, and as a drier for paints and varnishes. It's also pretty cheap, so long as one doesn't want 99.9% pure chemical.

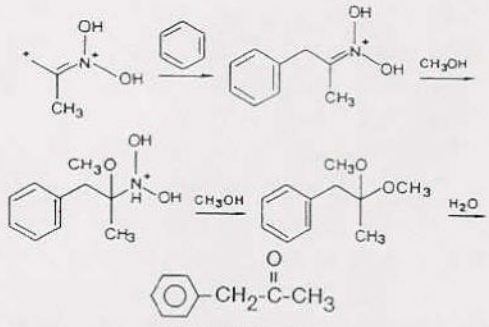
To get Mn(III) acetate from Mn(II) acetate, we return to a recurring theme in industrial chemistry — the electric generation of Mn(III) from Mn(II). We saw one example of this kind of conversion back in Chapter Nine in the benzaldehyde recipe. For this next one see *Acta Chemica Scandinavica* B 33 (1979), pages 208-212. At a graphite or platinum anode in a simple, undivided cell, using a cathode much smaller than the anode to minimize reduction of the Mn(III) formed, the chemists produce Mn(III) acetate from Mn(II) in glacial acetic acid solvent.

One mole of Mn(II) acetate is dissolved in one liter of glacial acetic acid. A little bit of sodium lithium fluoroborate (a few grams) is added as current carrier to the solution. One could also try sodium or potassium acetate as current carrier; it may not interfere in this reaction. The fairly large graphite or platinum anode is placed in the solution, along with the smaller cathode. The mixture is warmed, and then with stirring, DC current is made to flow through the cell. One should apply 4 milliamps of DC current for each square cm of anode surface facing the cathode. This is a one-electron oxidation, and one can count on getting around 66% efficiency in the oxidation. So one should pass about 1.5 faradays of current. One faraday is 96,500 amp seconds, so if for example one is passing one amp through the solution, the electrolysis to Mn(III) will take 40 hours. At four amps, it will take 10 hours, and so on.

At the end of the electrolysis, one has the Mn(III) acetate solution in acetic acid. Then to this solution, one can add the benzene and acetone with stirring, and react as usual. It's a lot of work to get 30 ml of phenylacetone, but those chemicals certainly are low profile, cheap, and easily available.

For another example of electric generation of Mn(III), see US Patent 4,560,775. One can also use permanganate in glacial acetic acid to oxidize the Mn(II) acetate to Mn(III). For an example of this procedure, see *Journal of the American Chemical Society*, Volume 96, pages 7977-7981 (1974). On a smaller scale, this procedure is preferable to the electric oxidation. The phenylacetone synthesis can be made to work, and it is just plain unstoppable from a policing point of view. It won't be long now, and every chemical under the sun will be on the "watched list."

A kind of related reaction can be found in *Journal of Organic Chemistry*, Volume 54, pages 733-34 (1989). Here phenylacetone is made in 85% yield. Just two problems with this reaction. It runs in a pretty dilute solution, and it uses 2-nitropropene as a reactant. A quick look through my chemical catalogs doesn't turn it up for sale anywhere. I would imagine it isn't too hard to make one's own 2-nitropropene though. The reaction proceeds as follows:



PHENYLACETONE

The strong acid trifluoromethanesulfonic acid protonates 2-nitropropene, and this intermediate then links up with benzene. After pouring the reaction mixture into dry methanol, and then adding water, the product, phenylacetone, is formed in 85% yield.

In one of their typical examples, the chemists mix 3 grams of 2-nitropropene in 45 ml of benzene. In another container they have a solution of 31 ml of trifluoromethanesulfonic acid in 50 ml of benzene along with an unspecified amount of the methylene chloride co-solvent. The co-solvent acts as antifreeze for the mixture, so 50 ml of methylene chloride is probably about right. They cool this solution down to -40° C with a dry ice-acetone bath. Then with vigorous stirring, they add the nitropropene in benzene solution to the trifluoromethanesulfonic acid in benzene and methylene

chloride solution. They allow this mixture to react for one minute.

After the reaction time, they then pour this reaction mixture into 1000 ml of dry methanol cooled to –78° C (dry ice-acetone bath) with vigorous stirring. This reaction mixture is then allowed to warm to room temperature. The yellow colored mixture is then diluted with 1500 ml of water.

To recover the product, they start by adding bicarb to this reaction mixture until it is neutralized. This is when the added bicarb no longer causes fizzing. They next added salt until the solution could dissolve no more. Then they extracted out the phenylacetone with methylene chloride. One could also use toluene. Distilling this extract then gave them pure phenylacetone, around 5 ml.

As is, this isn't a clandestine suitable process. It just uses way too much solvent to get such small amounts of product. If one could reduce the amount of benzene used from the thirty fold excess relative to the 2-nitropropene down to around 10 fold, and if then one could also reduce the amount of methanol used, this method would have some promise. Good luck, and happy cooking!

For another somewhat related reaction, I have only the abstract. The research was done in 1959 by Robert Levine. It gives a 34% yield of phenylacetone. To liquid ammonia, one first adds sodamide and acetone. This forms a sodio derivative of acetone. Next, bromobenzene is added, and the mixture allowed to react for 10 minutes. Then the reaction mixture is quenched by adding ammonium chloride. After the ammonia evaporates away, the residue is extracted with toluene. This extract is washed with some dilute hydrochloric acid to remove aniline and diphenylamine formed as byproducts. Then the toluene extract is distilled to get pure phenylacetone. Dibenzyl ketone is formed as a byproduct also.

Chapter Twenty Three
Last Resort — Extracting l-methamphetamine From Vick's Inhalers

By popular demand, this method of last resort will be covered in this edition. The Vick's Vapor Inhaler is available off the shelf at your local grocery or drug store in the cold- or allergy-remedy section. It contains 50 mg of the free base of the weaker isomer of meth, along with the "Vick's vapors" which are bornyl acetate, camphor, lavender oil, and menthol.

Of the above ingredients, only the meth free base (1-desoxyephedrine) has a basic nitrogen, so separation is possible. To extract and separate the 1-meth from the other ingredients, we first disassemble the inhaler to get at the cotton-like wadding that contains ingredients. This wadding should be immediately soaked in 10 ml of the 10% hydrochloric acid. The hardware-store brands of hydrochloric acid are about 20% strength, so dilute accordingly. Using surgical gloves, squish up this wadding repeatedly to get the HCl into contact with the meth free base and convert it to the hydrochloride, which is water soluble. After a good thorough squishing, pour the hydrochloric acid into a sep funnel. If solids are floating around, filter the solution. Now add another 10 ml of plain water to the wadding, squish it around again to rinse out more product, and pour this too into the sep funnel.

Now extract out the entrained vapors with a couple of 20 ml portions of toluene. Throw away the toluene, and keep the hydrochloric acid solution. Now make this hydrochloric acid solution strongly basic to pH papers by adding some lye or lye solution, with strong shaking between adds of lye.

The meth has now been free based, and is freed of most of the Vick's vapors. Extract out the meth free base with about 20 ml of toluene. Separate off the toluene, and bubble dry HCl gas through it as described in Chapter Five. The crystals of 1-meth hydrochloride should be spread out to dry after filtering, and their aroma noted, once they are free of toluene. If they still smell like the Vick's vapors, one should first try drying them under a vacuum for an hour or so. If this still doesn't render them odor-free, they can be recrystallized by first dissolving them in a minimum amount of alcohol (91% isopropyl from the drug store shelves), and then adding toluene with shaking until about 10 volumes of toluene have been added. After some standing in the cold to get complete precipitation, the crystals can be filtered out. At this point the smell of Vick's vapors should be gone.

I have heard an unconfirmed report from a correspondent named Tammy that new versions of this inhaler don't respond to HCl extraction so well. The wonders of polymer science. If this is the case, the first extraction should be with 91% isopropyl alcohol. After two extractions with isopropyl alcohol, add a couple of drops of HCl and then this extract should be evaporated under a vacuum, or barring this, just mix with 20 ml of toluene.

Now extract this toluene solution with two 20 ml portions of 10% hydrochloric acid. From here, proceed as with the 10% hydrochloric acid solution.

Chapter Twenty Four
Keeping Out of Trouble

Making methamphetamine, it should be remembered, could be a dangerous activity. But, in addition to any dangers inherent in the activity, underground chemists making methamphetamine face dangers of another sort. The source of these other dangers are the agents of the various law enforcement agencies. This chapter will discuss some of the dangers and how underground chemists avoid them.

How then does the underground chemist minimize his risks? The first and most important thing is to use hit-and-run tactics. He makes a lot of product at a time, and then closes up shop. It is much safer to spend a week or so on steady work and make a supply of product that will last for a while than to keep setting up and supplying a lab every few weeks to make smaller amounts. This cuts the chemist's exposure to a minimum. Secondly, all the chemicals to make methamphetamine are only brought together when the chemist is ready to begin production. Having all the chemicals together could result in a conspiracy charge. For example, having phenylacetic acid, acetic anhydride and pyridine together could result in a charge of conspiracy to manufacture phenylacetone, if the knuckle-heads at the state crime lab are aware of this method of making phenylacetone. To avoid this, phenylacetic acid and methylamine are kept at one location, and the other chemicals and glassware at another. After the chemist is done making his supply of methamphetamine, he washes all the glassware in hot, soapy water, rinses them a couple of times with hot water and then with rubbing alcohol. He lets the glassware drip dry, and then bakes the glassware in the oven at 400° F for an hour or so. This removes all traces of product from his glassware. The empty glass jugs of chemicals are rinsed out with water and the labels scraped off. Then they are broken and the pieces taken to a far away dumpster.

A very important precaution for the underground chemist is to keep his mouth shut. While his friends may mean him no harm, they would tell their friends and eventually the wrong ears would hear about it. The streets are crawling with snitches who keep themselves out of jail by reporting what they hear. Without a snitch, police agencies are incapable of detecting a cockroach crawling across a loaf of bread.

The people to whom the chemist sells his products have no business knowing where it comes from. In fact, he is constantly on guard against his customers, because they are his main source of danger. If one of them should foul up, he may very well try to set up the chemist to get out of his own problems. This is the way that Johnny Law makes his busts, so the underground chemist is on guard. If one of his customers has a newfound buddy who wants to buy from him, he starts babbling crazy nonsense or claims ignorance. He decides how to deal with them later.

As long as the chemist does not deal with strangers, the only way that the narcs can get at him is to have one of his customers make what is

called a "controlled" buy on him. This is when they send his customer in to make a purchase from him while they wait and watch outside.

The underground chemist protects himself by only making deliveries to his customer's home. He never does business out of his own home, or at bars, parks, parking lots or any other place suggested by his customers. He knows his dealers well, and knows their schedules. His dealer does not know exactly when he will be showing up with the next shipment; he just shows up unannounced and makes the delivery. A street-legal dirt bike is a good delivery vehicle. If the narcs try to jump the chemist at his customer's home, he takes off cross-country, leaving a cloud of methamphetamine powder behind him. He can melt the baggie on his tail pipe. If the narcs eventually catch him, he says they looked like a sleazy gang of hit men. He never lets his customers talk him into meeting at a bar, park or other public place where Johnny Law can watch and make a controlled buy. He ignores excuses such as not wanting a roommate to know about the shipment.

If the underground chemist must store significant quantities of methamphetamine in his home, there is a good way to keep it undetected. He dissolves the uncut material in 190 proof grain alcohol. He uses uncut methamphetamine because alcohol dissolves it better than cut. Alcohol dissolves a surprisingly large amount of methamphetamine. He records the exact amount dissolved per hundred ml of alcohol. He pours the alcohol into a dark whiskey bottle and adds it to his liquor collection. It smells just like any other booze. It will go undetected in a search.

When he is ready to sell undissolved methamphetamine, he measures out the required amount of alcohol and pours it into a filtering flask along with a couple of boiling chips. He stoppers the flask and attaches the vacuum hose to the vacuum nipple. He boils off the alcohol under a vacuum. He can heat the flask with hot water to speed the process, but does not use any stronger heat. In a little while, the alcohol is gone, leaving the crystals in the flask. He scrapes them out and chops them up. He can now add the cut to the crystals. The filtering flask can be rinsed clean with hot water.

The maker of methamphetamine, like the user, may be subjected to urine testing, and so he is aware of the following information. A single dose of methamphetamine can be detected in the urine for three days after taking it. When repeated doses are taken over an extended period of time, it builds up in the cerebral spinal fluid, lymph, and other noncirculating bodily fluids. As a result, it is detectable for considerably longer than three days.

The three day period mentioned above assumes a normal fluid intake. Since the kidneys are the main way the body has to get rid of methamphetamine, the process can be considerably sped up by putting the kidneys on overtime. I can think of no more enjoyable way to do this than to drink a lot of beer over a period of a few days. This process can be sped up even more by increasing the efficiency of the kidneys. This is done by drinking a lot of cranberry juice. This increases the acidity of the urine, and shifts the partition coefficient in the nephrone of the kidney in favor of excreting the methamphetamine more rapidly.

A last-ditch method to avoid detection is to get some Snowy Bleach or other similar powdered bleach, put it on the fingertip and under the fingernail, and rinse it off into the urine sample. The bleach attacks the methamphetamine by oxidation, converting it to a harmless set of fragments. This technique works better with THC and other more easily oxidized drugs, but it works satisfactorily with methamphetamine if the urine is dilute. In order to avoid wasting the limited oxidizing power of the powdered bleach on the other normally occurring compounds in the urine, a lot of water is drunk before giving the urine sample. And, in order to keep the concentration of bleach high enough to ensure the destruction of the methamphetamine, as small a sample as possible is given.

Remember: *The information in this book is intended for informational and research purposes only! If you do anything illegal and get caught, you will have to be prepared to face the consequences! This is a letter that I recently received from one of my readers:*

Dear Uncle Fester:

I am the subject of the first methcathinone (Cat) case in the Fifth Circuit (Texas, Louisiana, Mississippi, Alabama, etc.). I was one of many whom a dude named Bill Killion "fingered" to avoid prosecution in Wichita, Kansas. You may know him; his correct name is William Killion.

I still await sentencing sometime in early January, 1996. And many of the investigations for my defense involve research of ephedrine white-cross and methcathinone, which I thought might interest you. Since I didn't discover your book, ***Secrets of Methamphetamine Manufacture***, until after my arrest, I learned of cat through its American patent (2,802,865). There is a German patent, and one other foreign patent with R+ and S- methcathinone types. I wish I knew how to obtain those patents, because it may be argued that all of these types are not covered by statute, and the yields from the ephedrine may be helpful in establishing "drug-quantity" guidelines for my sentencing.

Currently, under federal laws, once an individual has been found guilty of manufacturing any amount of any drug, he may then be sentenced according to any amounts that can be established by a light standard of proof. The sentencing in all federal drug offenses (except simple possession) is governed by the drug weights involved. In my case, where no drug seizure was made, the drug quantity involved is estimated based on the lab seized or the established amount of precursor seized.

In my case, Killion fingered me as having ordered mini-thins from two distribution companies (T&M and Olympus), and stated that I was in possession of a methcathinone lab. The first thing the Kansas DEA did was call and subpoena the companies' records, which showed that 39,000 mini-thins had been shipped to my parents' and sister's addresses (both living next door to me). Then the DEA simply awaited the next order and shipment.

I was found guilty, based largely on the DEA's surveillance of me picking up 3,000 mini-thins at the post office. Soon, after agents followed me to my sister's home, my wife was observed going to buy a can of Red Devil lye and delivering it to me. A warrant was sought, and seven hours later the DEA rushed in to find me in a detached garage on my sister's property, where two five-gallon paint buckets containing toluene paint thinner were discovered, along with some Batman drinking glasses (one with a trace amount of chrome salt), a jug of HCl, a container of water mixed with sodium hydroxide, and one broken-up, duct-taped 3,000 ml flask.

My point is that on a bust where these white-cross mini-thins are involved, the distribution company's records (and any other records that the law can obtain) are used to establish the amount of precursor that had been involved in the laboratory manufacture, and from this the total quantity of drugs involved is determined, which in turn sets the length of the sentence to be imposed. The same thing has been done with methamphetamine precursors for years, but the yields of cat from ephedrine have not yet been established.

In early 1993, two cases of cat manufacture arose in Marquette, Michigan. One is published: *US vs Baker*, 852 F. Supp. 609 (W.D. Mic 1994). Affirmed on appeal in the Sixth Circuit, this case established that 50% of the weight of the ephedrine pills will reasonably equal the weight of the cat which could be produced from it. This amount was established by a government-employed chemist who testified at the trials in these cases. The prosecutor in my case is using *Baker* to calculate my sentence. I do not feel that 50% is unreasonable, unless I could find proof that it is. I am now arguing that insufficient proof exists that the 39,000 white-crosses were actually dropped into a cooking pot! They were ordered COD over a four-month period in 3,000-lot batches. And white-crosses have been sold for many years themselves as a "speed" on the black markets. The FDA, which is now blasting against these pills, is a good source for this information.

I wrote the FDA for information about white-cross mini-thins being illegally abused and sold as speed and lookalike-speed. The FDA sent me a copy of their proposed laws or regulations on

ephedrine under published law, from the *Federal Register*, part 310 & 341, Volume 70, October, 1994 or 1995, I believe (I sent it to my lawyer, so I'm going by memory). It's a section on bronchial medications which are sold over-the-counter (OTC). In the FDA's proposed regulations, they mean to designate all ephedrine, norephedrine, and racephedrine products as prescription drugs. I do not believe that the DEA will succeed in its proposal, but they also propose (and have moved forward considerably) to remove all single-ingredient ephedrine products from OTC sales. They focus on the white-cross thins (which companies are selling now as stimulants) being sold as bronchial-aid products. Well, it's obvious that the distributors of white-crosses that sold them as stimulants before they were outlawed by the FDA have now relabeled them as asthma medicine, and continued their sales. Stimulants were prescription drugs at the time of those former sales, and that's why white-crosses were commonly sold as speed on the black markets years ago. There have been official discussions about outlawing OTC sales of the white-cross bronchial products, as recently as November 14, 1995. Also noticeable is that the FDA has mentioned how the DEA has moved in to dictate distribution-company regulations and notification rules on these single-ingredient ephedrine sales until they can persuade the FDA to outlaw the OTC sales. The DEA recognizes the manufacturing implications, and claims that the FDA is hampering their attempts to control precursor chemicals.

I have also noticed that the feds have added benzaldehyde and nitroethane to their list of "hot" chemicals, under 21 USC 801 in 1995. However, I do recognize that the grocery-store "extract" oil of bitter almond has distillation potential for quantities of benzaldehyde. I am not aware of where the nitroethane is commonly sold. But I like this recipe the best of any in ***Secrets of Methamphetamine Manufacture***, because it is so easy to get the ingredients. I ran across your book when my lawyer gave it to me as part of the government's evidence against me. They never used it at my trial, though.

Secrets of Methamphetamine Manufacture was found in Killion's abandoned car after I had run him off from my Texas home because he was getting too crazy on drugs. He went a short distance towards Kansas before his car conked out, and then he abandoned it, leaving a copy of your book and others to be found by the DEA. When Killion got back to Kansas, he started manufacturing cat again in that state, but then got popped and started fingering a lot of folks. I had to settle for a crappy court-appointed attorney who lost the case. If the 39,000 white-crosses are sufficiently proven to be involved, my sentence will be from 10 to 12½ years without parole. I've been kept in solitary for my protection, due to blindness in one eye (medical), for over a year now, while fighting this case. Please, if you think of something that might help me, let me know. I'd be greatly obliged and most appreciative.

I thought you might be interested in taking a look at the *Baker* case and the *Federal Register*, part 310, at your local law library. They are very interesting. I will send you a copy of them if you like. I'd send them now, but my attorney is currently reviewing them.

Sincerely, a fan and friend,

Mr. X

P.S.: I plan to leave the USA and do my own thing with chemistry in a better country, after I win my appeal and get out!

Posted by Uncle Fester on February 24, 1998 at 22:35:20: A Story for Hive Bee
You asked for a story, so...

I'll tell you a bedtime story, and I'll make it a scary one because I know that the scary ones make you hot.

Once upon a time there was a happy little cooker. He loved to cook, and between consum-

ing the product himself and some dealing he produced a few pounds of product over the years.

Our happy little cooker thought he was pretty safe because he never kept much of the goodies he cooked around his place, and he always cleaned up his glassware when finished cooking.

But our happy little cooker had been making some tragic mistakes. Our cooker loved his meth, and so did his friends. He ordered lots of ephedrine, and then pseudoephedrine pills, from various mail-order companies. He paid for them with his credit card. This credit card had his real name!

He also bought chemicals with this credit card. Things like iodine and red P. Also a couple of quarts of sassafras oil, $PdCl_2$, ammonium chloride, and formaldehyde.

One day while checking over reports of mail-order pill purchases, the evil narcoswine became interested in our happy little cooker. They decided to check him out. Just the purchase of large amounts of pills is enough to get a search warrant.

In the happy little cooker's house, they found about a gram of stash, and just a little trace of meth on some glassware. Our happy little cooker was now not so happy, but he thought he'd get off lightly because he only had a gram around the house.

Oh, he was so wrong! Using his credit card records, the evil federales were able to reconstruct all of our shell shocked cooker's past activity. That includes the X he made last year!

Our now very unhappy little cooker was portrayed as a menace to society at trial and lost. In the federal system, sentence is based upon how much they can show you made. A very flimsy standard of proof suffices. Using those credit card records, they were able to give our tragic little cooker 15 years in the Big House. He doesn't like it there, but at least now he has gotten rid of that American Express Card.

The end.

Posted by flaskjockey on March 06, 1998 at 21:10:55:

In reply to: A story for Hive Bee, posted by Uncle Fester on February 24, 1998 at 22:35:20:

The state system doesn't care how much or how complete, which is scarier. California has an amusing way of convicting you, called "Intent to Manufacture." All they need now is extracted ephedrine (any amount or trace) OR any reducing agent, even if the agent doesn't reduce ephedrine. These police state bastards can get away with anything. The only solution is to shoot them.

Chapter Twenty Five
Legitimate Uses of Some Chemicals

Acetic Anhydride — commonly used in the chemical industry, especially for making dyes.★★★

Benzene and ether — common solvents, but they are sometimes used for free basing coke.★★

Formic acid — used for taxidermy and tanning leather.★

Hydrochloric acid and sulfuric acid — the two most common mineral acids with too many uses to list. When buying them, underground chemists say they want them for electroplating.★

You can pick up these two at the hardware store. Hydrochloric will often be labeled as muriatic acid, and will generally be 30% HCl. This is good enough for most uses. I found an industrial grade concentrated sulfuric at my local hardware store in the plumbing section. It was a product called Liquid Fire by Amazing Products. It sells for around $6 a pint, and is used as drain opener. This is plenty good enough for dripping on salt to make HCl gas.

Methylamine — used in photography, as an additive to racing fuels, and as an ingredient in rocket fuel and tanning solutions.★★★★

Phenylacetic acid — used in perfume to produce the smell of honey, and added to the nutrient broth of penicillin mold to increase the yield of penicillin.★★★★

Platinum and Rainy nickel — catalysts used in all hydrogenations.★★

Pyridine — a common but expensive solvent and reagent.★★

All other List I chemicals ★★★★
All other List II chemicals ★★★

One should obtain toluene and acetone at the hardware store in the paint section. There it carries zero stars, so long as you don't buy so much at one place that you get them wondering.

★ Least suspicious to purchase
★★★★ Most suspicious to purchase

Chapter Twenty Six
Web Sites

Learning about clandestine chemistry is a lot of fun, as you well know after finishing this book. It's also a lot of fun to follow along with conversations and "posts" that people put on the Internet. There are a few web sites exclusively devoted to such conversations and postings. They are great places to drop in on and spend hours keeping up with the latest news or reading about clandestine processes and equipment.

Before you eagerly dive into the Internet, let me give you a few necessary caveats. To start with, go ahead and read, but keep your mouth shut. If you are doing any cooking, you certainly don't want to draw attention to yourself by posting questions or mentioning any results you are getting. These bulletin boards require registration before you are allowed to post. The heat trolls on these web sites, either by asking questions that giving an answer to would involve you in their "conspiracy," or by checking out the e-mail addresses of posters who seem like they are actively cooking. By keeping quiet and just reading, no one will know you are there.

Caveat number two is a corollary to the first one. Since no one who is actively cooking would be stupid enough to wave around their e-mail address for everyone to see, the people doing the posting will fall into two classes: retired cookers who still love to talk about the excitement of their glory days and relate war stories, and simple students of the field, ranging from rank amateurs to the fairly advanced. The problem is to discern the two classes, and separate the wheat from the chaff in the huge amount of material posted on these boards.

Making this differentiation is a difficult task for the beginner. The second class tends to be skilled at sophistry, and in the process they give birth to misconceptions that carry onward in time with the tenacity of urban legends. Let me give you some help in this sifting process; a good source posting on the net will cite references for the readers to follow up on rather than just making claims. This is the same style your Uncle uses in this book and all my other ones. It is the only legitimate style to use when making posts as well. Suspect sophistry and underlying agendas when references aren't mentioned during chemical discussions.

Now that you have been sufficiently warned, let me pass along two web sites that will get you started on the Internet world of clandestine chemistry. Place number one to visit can be found at DejaNews (www.Dejanews.com). The particular bulletin board is alt.drugs.chemistry. This is a good beginner's site, a place where stupid questions are routinely addressed. For more advanced chemistry, go to www.lycaeum.org/~strike. This will get you into "the hive." Several boards are found at this site dealing with various topics in clandestine chemistry. Enjoy, and keep your mouth shut if you are cooking!

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☐ **85186 OPIUM FOR THE MASSES: A Practical Guide to Growing Poppies and Making Opium, *by Jim Hogshire.*** Everything you want to know about the beloved poppy and its amazing properties, including: • What does the opium high feel like? • The stunning similarities between opium and your body's natural endorphins • Morphine and its derivatives • How to grow opium poppies • Sources for fertile poppy seeds • How to harvest the opium from a crop of poppies • How to make poppy tea • Other ways of making and ingesting opium • And much more! Also includes rare photographs and detailed illustrations that bring this magnificent plant to life. *Sold for informational purposes only.* ***1994, 5½ x 8½, 112 pp, illustrated, soft cover.*** **$14.95.**

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