

Chapter Eighteen

Cooking Your Own Ephedrine

You don't like that recipe? Check out this one taken from "*Chemical Abstracts*", Volume 47, column 3347. Twenty 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.

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.

This original recipe seemed good on the face of it, but it suffered from serious problems. The yield of product was low, as a lot of tar was made in the reaction. The starting material, N-methyl alanine, is not commercially available at a reasonable price, and it used to be not easily made by the methods available then.

These problems have been conquered by advances made with the passage of time. The large amount of tar made in the reaction is prevented if DMSO is used as a solvent in the reaction. DMSO also allows the temperature of the reaction to be lowered to 120 to 130 C. DMSO, or dimethylsulfoxide, has found wide use as a pain reliever for creaky joints. It can easily be had at well supplied veterinary supply stores in the horse section. It is also sold widely on the internet at a very reasonable price. One must avoid the creams, and stick to the pure liquid DMSO solvent.

The introduction of procedures using DMSO solvent led to the discovery that this reaction gives very high yields of the methylendioxy derivative of ephedrine and pseudoephedrine if piperonal is used in the reaction instead of benzaldehyde. This opens up an entirely new way of making MDMA. One could react piperonal with N-methylalanine, and then reduce the product substituted ephedrine with lithium in anhydrous ammonia to MDMA. One could also use the reduction routes working through chloroephedrine and palladium. The reduction methods using HI and red P would not work because HI cleaves ethers on a benzene ring.

Benzaldehyde has been added to the List One of watched chemicals, but check out the "Making Shitloads of your own Benzaldehyde" section in the "Other Methods of Making Phenylacetone" chapter. That leaves the N-methylalanine problem. Only N-methylalanine can be successfully be used in this reaction. Ordinary health food store alanine will react to give 1,2-diphenylethanolamine. N, N-

dimethylalanine will give no reaction. Check out the Journal of the Pharmaceutical Society of Japan Vol. 72, pages 812 to 815 (1952) for a complete discussion of these results.

A great stroke of luck for meth cooks was published in 2007. It is a very easy and very high yielding procedure for making N-methylalanine. It can be found in Tetrahedron Letters, Volume 48, pages 7680-82. The reaction uses formaldehyde and zinc dust to do the methylation in water solvent containing partly neutralized phosphoric acid. Formaldehyde can be picked up on the internet or from other sources at low cost and with no heat attached as of 2009. A little bit goes a long way and the other materials can be picked off shelves at stores.

The following procedure has been found to be an easy and very productive method for making N-methylalanine. It is given on a 10 gr scale because larger amounts are not done easily with a magnetic stirrer. The zinc dust is quite dense, but must be kicked up into solution to react.

To 200 ml water in a beaker or other container, add 16 ml phosphoric acid. The hardware store dairy milkstone remover phosphoric would work if account was taken of its weaker strength, and the detergent extracted. Next add 9.5 gr of NaOH to form the monobasic sodium phosphate. Lye would substitute as would bicarb or washing soda so long as the amount added was adjusted. Stir constantly. One should be able to make a roughly equivalent buffer solution by adding 40 grams of hardware store trisodium phosphate (TSP) to 200 ml of water, and then adding roughly 30 ml of hardware store hydrochloric acid. I haven't tried this variation, but it should work and would be entirely supplied right off the hardware store shelf.

Now to this mixture add 10 gr alanine (.11 mole) from the health food store. It dissolves quite easily, and the solution should be around 30 C from the neutralization of the phosphoric acid solution by lye.

Next add 10 ml (.14 mole) 37% formaldehyde, follow that with 15 gr zinc dust and stir for 45 minutes. I know that is more than the Tetrahedron Letters article specified, but the amount of formaldehyde has been backed down from the 1.5 molar excess stated in that article to around 1.2. Formaldehyde is toxic and no doubt folks would do it in a kitchen. Less is more...so increase reaction time...besides that is the ingredient that one would need to purchase.

One will note a constant fizzing noise from the reaction vessel as the zinc dust reacts and makes hydrogen. The zinc gradually changes from grey to blue, and this is zinc phosphate. Zinc dust is easily had, but one can make one's own by taking a piece of zinc metal such as pennies and grinding on a wheel or with a file until they are reduced to grit and dust.

Now let the zinc settle, then filter the almost clear water solution leaving the zinc on the bottom of the flask. It should filter clear water white through a couple of coffee filters.

Add 50 ml of hot water to the settled blue zinc sludge on the bottom of the flask, and swirl for a bit. Then let that settle. Filter off the water, and add this to the main charge of filtered water.

Now for the important part...add bicarb powder slowly and with stirring until the pH reaches roughly pH 7. One will need a pH meter, and make sure it is calibrated. Tap water should read within a few tenths of pH 7, but buffer solutions are easy to get to calibrate a pH meter.

Once the pH has been adjusted to around 7, let the water clear solution sit in the fridge overnight. Great heavy masses of long needle shaped N- methyl alanine will form. Let the mass grow to its greatest extent. It may take more than 12 hours to get the crystal growth to kick off. If one has a seed crystal, it would be very convenient.

When the crystal mass has stopped growing, filter it off. Then rinse it with some alcohol...at least 70%...and set the crystals on a plate to dry. The alcohol dries the crystals and keeps them from becoming bacteria food as they dry. Use less than 50 ml.

Now the filtered liquid should be boiled down. This is another reason for using less formaldehyde. Boiling the mixture makes the residual formaldehyde become part of the steam given off. The mixture will now be kind of yellow colored. Reduce the liquid volume by half and then add the alcohol rinse from

the first crop of crystals. Then put this boiled down water liquid in the fridge to collect another crop of crystals of N-methyl alanine. Total yield is about 10 gr after the crystals have dried. This would react with benzaldehyde in DMSO solvent to give yields of ephedrine and pseudoephedrine which have not been seen since the mid 90's.

This newer and much better cooking procedure was covered in detail during two conferences sponsored by the Pharmaceutical Society of Japan in 2006 and 2007. The abstracts were published on line as "Proceedings of the Symposium on Progress in Organic Reactions and Syntheses". The two papers in question were presented at the 32nd and 33rd Symposiums by Yokoyama and Tsubaki. The easiest way to google up these abstracts is by searching 33rd symposium or 32nd symposium. To get the full cooking details requires some hands on cooking since they are less than willing to give the text of their proceedings. Go figure on that one!

The preferred procedure is to react one mole of benzaldehyde with two moles of N-methylalanine in DMSO solvent. Let's take a small scale example using 30 ml of benzaldehyde. It would react with 60 grams of N-methylalanine to give roughly 25 to 30 grams of a mixture of ephedrine and pseudoephedrine hydrochloride.

Now place 250 ml of DMSO into a suitable heating vessel such as a round bottom flask with reflux condenser...or a lava lamp if you are ghetto...add a magnetic stir bar...and 60 gr of N-methyl alanine. Then add 10 ml of the 30 ml of the benzaldehyde. It is best to add it in portions to save on the total amount of DMSO solvent used. This makes the clean up at the end of the reaction much more practical.

Heat this mixture to about 130 C if one is using piperonal, but benzaldehyde will react at a lower temperature. Look for the fizzing of CO₂ as your visual clue, and go with the lower temperature. Let it fizz for an hour or so, then add the next 10 ml portion of the 30 ml total benzaldehyde add. Let it similarly fizz for an hour before adding the last 10 ml portion of benzaldehyde.

After the last addition of benzaldehyde, let the mixture heat at the lowest fizzing temperature, and then turn up the heat slowly. The max with benzaldehyde should be at about 130 C. Piperonal would react much more easily, but allow the mixture to fizz to complete reaction. Take no more than 6 hours on this stage of the reaction. Just push up the heat to get it going.

Now for the new part of the reaction (just discovered), "Vinegar", yes the really cheap distilled white vinegar at the grocery store, greatly increases yield by breaking up a dimer complex the product forms. At this scale, add 500 to 600 ml of that cheap grocery store vinegar, and swirl to mix it in. Then add from 50 to 100 ml of toluene or xylene. This solvent serves as a catcher for the BS we don't want.

You will note that after adding the two volumes of vinegar, the toluene or xylene now form a layer on top of the DMSO and product layer. This is important as it is the crap catcher. The mixture now must be heated to reflux boiling temperature for 3 to 6 hours to break up the dimer and give the greater yields one wants. A lava lamp is unsuitable for this purpose.

Heat the mixture to boiling gently for three to six hours. Then let it cool. This has broken up the dimer. Add about a shot glass of hardware store hydrochloric acid to the mixture and mix it in. This will assure that all the ephedrine stays out of the toluene layer.

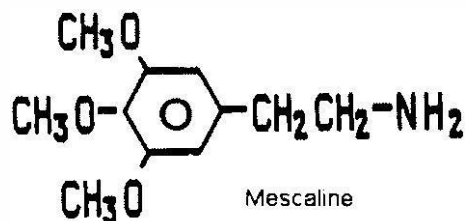
Now put the reaction into a sep funnel and shake it. Let this settle and the toluene layer on top would have crap and unreacted benzaldehyde in it. Put it aside for further work, the water layer underneath has the product.

Next base the DMSO and water solution containing the ephedrine and pseudoephedrine. Add a solution of lye to it with sirring and shaking until the pH is 13 plus. This should not take all that much lye to reach that level. Then add about 100 ml of toluene or xylene to extract the product. Save it. Extract again with 50 ml of toluene or xylene.

Wash the combined extracts with some water to rid them of DMSO, then let them sit to shed water and then transfer to a dry beaker minus the water droplets, and bubble with dry HCL to give about 30 grams of pure pseudoephedrine and ephedrine mixture as hydrochloride crystals. Keep water out of the dry HCl bubbling mixture!

Chapter Nineteen
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 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 subject 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: