A Compilation of Documents

Contents:

Part 1: Safrole Distillation, followed by notes

Part 2: Isomerization, followed by second example

Part 3: Peracetic Oxidation, followed by second example and ketone distillation

Part 4: Al/Hg with Nitromethane, followed by streamlined version and notes

Part 5: Crystalization, followed by notes

Part 6: General Notes

**Part 7: Boiling Points** 

**Warning/Disclaimer:** Never use the given information without having the theoretical background, the practical experience, the proper equipment, and a valid permission. Any person caught breaking the law is subject to prosecution to the fullest extent of the law. This book was written for the enlightenment of the general public or at least the open minded and curious percentage. It is to be used for informational purposes only. It is not meant to be a production manual. All the syntheses are geared to demonstrate how the underground chemist might proceed in "secretly" manufacturing MDMA. None of these writings have been tried, and are not guaranteed to work in real life, or even theoretical situations... YOU'VE BEEN WARNED!!!

# Part 1: Safrole Distillation:

Always put a little (vacuum grease) on the ground glass joints - this way they won't stick when you try to take them apart (and helps hold vacuum). Start turning up the heat slowly! SLOWLY! The slower you do it, the better/purer your safrole will be. At normal pressure safrole boils at  $232^{\circ}$ C - but under your vacuum, it may boil at anywhere from 110-160°C. Whatever temp it starts to come over at - make a note of it. And if the temp is higher than 160°C - check your seals on the tubing and glassware (more than likely there is a little leak). At the end of the distillation, you should have a water white oil that really refracts light, has a lovely smell (a little like potpourri).

The Peanut Oil: A bowl with a flat bottom rests on the Hotplate. It is filled with Peanut Oil. The distillation flask sits in the bowl but not touching the bottom, so that the Hotplate heats the bowl, the bowl heats the Peanut Oil, the Peanut Oil heats the distillation flask.

\*Hints On Distilling Sassafras Oil Into Safrole:

1) high stirring

- 2) big stirbar
- 3) make sure there are no leaks
- 4) raise C slowly
- 5) insulate all up to condensor

\* Thus maintaining a big difference in vapour pressures between your boiling flask which has a high vapour pressure (at high temp) and the condenser plus receiving flask which has a lower vapour pressure (at low temp), thus forming a sort of heat pump mechanism for vapour molecules (thus cool the receiving flask with ice, and condensor with water). A glass or SSteel (vacuum tested) valve with grounded joints on top of your receiving flask, so you can close this valve before you take off the receiver flask to discard impurities or just empty your product because the flask gets full.

# Step 1. (4 hours)

# 1. Distillation of your essential oil to obtain pure safrole.

Written by Chromic, with thanx going out to Bright Star for the document that this is liberally based on.

# Materials required for this step:

- 1. Essential oil that contains safrole
- 2. Complete ground-glass jointed vacuum fractional distillation setup, including stand, clamps and thermometer
- 3. Heating source, including pot, oil and cooking oil thermometer
- 4. Vacuum source
- 5. Cooling water source
- 6. Column insulation

# **Procedure:**

Set up for a fractional vacuum distillation (**see notes unless you've already performed this technique!**) by getting out your hotplate stirrer putting on an aluminum pot (copper with heating-rings engraved works better) (big enough to mostly submerge your 500mL flask), and filling it with oil.

Put no more than 300ml of our fragrant essential oil into the 500mL round bottom flask with a 1" egg (or octagonal) stirbar and set it to spin like mad. Setup your column, then the 3-way adapter, then the condenser, then the vacuum adapter, and put a 250mL RB flask as the receiving flask. Put on your thermometer adapter and your thermometer last. Get your vacuum ready, but not connected or turned on.

During this run, you're going to have to insulate your fractionating column. Glass wool is the best heat resistant insulator. If you have it use it, if not cotton cloth (such pieces of a towel or sock), wrapped around the column will suffice. Use wire to keep it on. The cotton may lightly brown, but won't char. You need this insulation, otherwise too much safrole will fall back into the distillation pot as reflux. You should be using a candy thermometer to monitor the temperature of your oil bath, so you don't vastly overshoot the mark where safrole is going to come over.

Next heat the oil bath up quickly to the temperature I expect my safrole to come over, in my most recent case it's 145C. Or sometimes I'll just set it on low, and go off to grab something to eat as the oil warms up. Now, when the cooking oil is at 145C... turn down the heat, and apply the vacuum. Nothing usually comes over. Now, continue heating, but this time slowly, to about 30C above that temperature... nothing usually comes over. If you have got something to come over, and it isn't your main fraction, remove the heat, disconnect your vacuum, and empty the garbage fraction that's in your flask, then replace it and continue... All of a sudden you're going to see the temperature on your thermometer sky rocket and hit that temperature, say 145C, and you will hold it there... do not rush this step of the distillation... and now just slowly collect the oil (1-2 drops/second) until no more oil starts to come over, and you've shot at least 5C past that temperature. So in my case I collect 145-150C. With more experience, you can narrow that range to get even more pure safrole. A good visual indicator of a different fraction starting to come over, is there will be little "swirlies" in the receiving flask as the new liquid drips in. This is because the index of refraction of the next fraction is different.

Make a note of the temperature that your safrole comes over at.

If the temp that your safrole comes over is higher than 160C check your seals on the tubing and glassware .. more than likely there is a little leak. If you're using an aspirator, and if you don't have good water pressure, you won't be able to get a good vacuum. Also, remember the Vaseline! Use an automotive vacuum guage setup on a T-connector of your vacuum tubing if you suspect a leak, this will be able to quickly let you know if you're reaching near full vacuum each time. You should be getting at a minimum, 28" of vacuum.

At the end of the distillation, you should have a water white oil that really refracts light.... and has a lovely smell.... a little like potpourri..... expect a yield of 75-90% safrole.

Now you have the fun job of cleaning the dishes. The best way is to use acetone to remove all the oils then wash with soapy water (if you feel it necessary). If there is any charring in your distilling flask, rinse with acetone, then let it soak in household ammonia for a few hours, then use a brush to easily scrub it out.

# STEPS ON BUILDING CONFIDENCE BEFORE YOU JUMP IN

**Step 1:** Once you get your distillation apparatus, set up your distillation apparatus your bed before you try to put it together on the stand. You will get a good idea about how the pieces go together, and become familiar with the fragility of the whole thing.

**Step 2:** Now you're ready to move it to the stand. Remember, this thing is build like a house.. bottom up. The bossheads connect to the 3/8" or 1/2" steel rods that stand is made of and keep the clamps in place, the clamps will attrached on both of the round bottom flasks, and perhaps on the top of the column as well. Read Zubrick for more advice.

**Step 3:** Get practice distilling regular tap water using an oil bath. Make sure your condenser water is reliable whether is comes from a recirculating fountain pump or from the tap, do not try to siphon water thru the condenser instead of using a pump or tap, although this technique works, it's too inconvenient and requires too much attention. Once you've done this, setup for regular fractional distillation and proceed.

The oil bath idea is simple... a metal bowl (not steel if you're using magnetic stirring) with a flat bottom rests on the hotplate. It is filled with peanut or saflower oil. The distillation flask sits in the bowl but not touching the bottom, so that the hotplate heats the bowl, the bowl heats the oil, the oil heats the distillation flask. This is very effective, and although it has a bit of an oil smell, it's tolerable. And will be perfect for all your distillation needs... most especially if you do it under vacuum. You want even heating, especially if you're not using magnetic stirring.

The water should come across at 100C, otherwise this indicates that your thermometer is off or the atmospheric pressure is significantly off in your area.

Step 4: Try again with water, but now under vacuum, and note the temperature it comes over at.

Always put a little Vaseline on the ground glass joints when doing a vacuum distillation ... this way they won't stick when you try to take them apart and will also let you know if you have a leak (air bubbles will be seen travelling thru the vaseline if you do have one).

This will indicate the strength of your vacuum (see vacuum distillation notes at the end of this document) Once you know what your vacuum source can pull, then you can cross-reference it to when safrole should come over to know exactly what to keep and what to throw out when you do the distillation.

Remember, the flask is never directly heated in vacuum distillation, it can be heated with an "air bath" for regular distillation but it is far too erratic for vacuum distillation.

**Step 5:** You're now ready for sasssfras! Go up and continue reading from the top. If you **DON'T** think you're ready for sassafras, go buy some cheaper oil of star anise, and practice with it. It is a very similar oil and will come across at almost the same temperature as safrole. If you want to experiment more with the technique of fractional distillation,

try distilling vodka or wine.

#### **Other notes:**

#### If you do not have a vigreux column:

Although it is preferable to use a short Vigreux column, distillation without a column will suffice as so long as you have magnetic stirring.

#### If you do not have the magnetic stirring:

If you do not have magnetic stirring, it will be much harder to do this distillation, but it is still possible. You must use a column or a Claisen adapter so that if you experience any heavy bumping, that the liquid will not spill over into the receiving flask. If you do not use magnetic stirring, add about 5g of very porous boiling stones made by breaking up pumice stone.

**Figure 1: Example of a fractional distillation setup.** You can use room temperature water for your condenser. Note that four clamps are used in this setup, one at the distilling flask, one to secure the fractionating column, one to secure the condenser, one to secure the receiving flask. It is a good idea to use at least 3 clamps to hold this setup, any less is unstudy. In this setup there is no thermometer adapter, as the three way adapter has one built in. Note the position of the thermometer bulb, this is where you want to place your thermometer. In this example boiling stones are shown being used, however it is more likely that you will be using magnetic stirring.

**More Notes:** You will find out that if this kind of setup is used also, for example, in a performic procedure to obtain MDP2P (Methylenedioxyphenyl-2-propanone, one of our favourite precursor ketones), and furthermore for the distilling of your freebases after the aminations of your prefered ketones, you will need to add 2 important upgrades:

1. A cooling bath with icewater around the receiver flask, to prevent your collected product to start boiling in the receiver flask and thus let those vapours contaminate your vacuum source, and lower your yields, especially when an oil-filled vacuumpump is used. A simple plastic or aluminium pan will do the job.

2. A glass or SSteel (vacuum tested) valve with grounded joints on top of your receiving flask, so you can close this valve before you take off the receiver flask to discard impurities or just empty your product because the flask gets full.

This is very important and lacking this thingy is probably the cause of so many people reporting strange colors when distilling ketones or freebases, and getting lower yields than expected, compared to official references.

The reason for the colorisations is the oxidation of the snot-hot oils in your boiling flask, when air is let in when removing receiver flasks. The reason for lowered yields is also a partly oxidation of your hot oils, every time you remove your distilling flask and let air in under atmospheric pressure.

The ideal setup is a receiver flask with a vacuum valve on top with a grounded connection to attach the receiver flask, and a vacuum valve at the bottom.

This lets you keep the rest of your system under vacuum as far as to that top valve on top of the receiver flask, and opening the bottom valve facilitates the draining of your receiver flask.

Don't worry, air will first bubble into your receiver flask, untill the pressure inside is equal to atmospheric pressure, then the collected product will start streaming out into the beaker you put under it.

Do not forget to bring your receiver flask again under full vacuum first, with the help of a 3way valve with a length of vacuum tubing which you connect to the bottom valve after draining the receiver flask, place that 3way valve inbetween your vacuum tubing from pump to vacuum alonge, before you close that bottom valve, set the 3way valve straight again, and open the top valve above the receiver flask again.

That way no air at all can ever enter your distillation setup while collecting distillation products, which air would directly finely solute again in your boiling flask oil and let the oil there foam like hell when you apply new vacuum again, when you did NOT install these valves.

Especially while distilling high boiling ketones and freebases will this be of high importance.

I never understood why the glassware industry didn't saw this weak point in their produced vacuum alonges.

Notes:

A. For your oilbath, the ideal pan is a full copper restaurant frying pan sold by restaurant supplier stores, and buy then also an aluminium frying pan for cooling your receiver flask, where the receiver flask + vacuum bottom tapvalve easily fits in. Just remove that aluminium pan during tapping off of your distillation products. The full copper pans have the advantage of having a thick, perfectly flat bottom, so even a 10 liter roundbottom glass setup with oilbath copper-pan will be evenly heated on a stirrer/hotplate which is smaller than your copper pan. Pure copper is a very good heat collecting metal, and does not interfere with your magnetic stirring.

B. This whole setup can be easily made totally from stainless steel, where the vigreux column is fabricated by drilling downward holes in a 40 cm stainless steel piece of pipe, and then welding ss-nails under a downward angle in the drilled holes, and make sure it's all vacuumtight welded.

A good welder/machinist can make the same standard "grounded" joints as used for glassware.

SS pots to use as boiling and receiver flasks can be bought at the same restaurant supplier stores.

*Especially the clumsy kind of lab assistants are famous for breaking ANY piece of expensive glassware they can get their hands on*, so this will in the end safe you a lot of headaches when opting for a corporate scale-up of your activities, heheh.

*Only* the receiving flasks can be used in glassware, so you can at least see at what kind of colour/texture/rate your products come over. The joints between the SS and the glass there will not differ much in temperature, so the expanding coefficient differences will not break the glass joint.

If a stubborn researcher thinks he can outsmart me by using his expensive 10 or 20 liter boiling flask with standard grounded joint connected to a SS joint from the vigreux column, so he can see what's going on in his distillation flask, he will end up within 10 minutes with a split glassjoint caused by the faster expanding SS joint.

C. It is a good idea to make a little mixing device from a small electric motor and a shaft with a little propellor to submerge into your heating bath oil. This will keep the overall temperature of your oilbath everywhere the same when its stirring capacity is strong enough.

#### **Continued Notes:**

1) Can add high-oleic safflower oil (all natural, no preservatives) to sas when distilling so as to get the very last drop of safrole w/o scorching flask and burning it dry.

2) Always use a teflon coated stir bar to eliminate bumping - use a strong magnet applied to the bottom of the flask to retain stir magnet when transferring contents to another container.

3) Use a vac that pulls at least 27".

4) Wrap column in insulation or a number of layers of foil (wrap claisen adapter also) - plumer's insulation (~1" fiberglass insulation coupled with a layer of foil) is far superior in its insulation properties than just plain aluminum foil alone.

5) Thermometer bulb (top of it) 1/4"-1/2" below bottom of side arm of dist. setup leading to conndensor.

6) Always wash receiving flask with a couple of squirts of dried acetone if discarding first distillate fraction then using the same receiving flask to collect the 2nd or 3rd distillate portions (the goods).

7) Always use ROUND bottom flask when vac distilling - flat bottom may implode with vacuum

8) Disconnect hose from aspirator (when removing vacuum) instead of just turning h20 off to stop vac. Doing this will ensure no h20 suckback into the receiving flask (or install valve).

9) Oil bath will need to be ~30C higher than distillate coming over for successful distillation.

10) Always increase temperature VERY SLOWLY!

11) When using an oil bath, expect to experience a lot of thermal lag.

12) When distilling 1-2 drops per second ~100ml an hour.

13) Should take between 4-7 hours to distill sassafras to safrole.

14) Sassafras oil is usually between 80-95% safrole

15) Can collect portion coming over right under safrole's bp and reprocess with a later batch to get the safrole out of that fraction.

16) Stir bar must be spinning fast to prevent bumping. Slow spinning stir bars will evidently still allow bumping to occur.

17) When distilling sas., safrole in receiving flask will be clear to slight yellow tint.

18) Need to get oil bath oil level to come up to the level of the liquid/oil in distillation flask if possible.

19) Is good to have a stir bar in both distilling flask (a must) AND in the oil bath just below the center of the distilling flask (can be a large paper clip).

20) Cold water through condensor - but will cause need for real good insulation up to claisen adapter because a cool condenser transferrs its coolness down through the distillation apparatus (may use air via aquarium pump).

21) Distill with tube leading from the aspirator down past U curve under kitchen sink (to rid air of distillate smells).22) 27" vac., safrole comes over at less than 130C

VAC (mm Hg) SAFROLE BP

90 159 C

80 155 C

- 70 152 C
- 60 148 C
- 50 143 C
- 40 137 C
- 30 129 C
- 20 120 C

10 106 C

23) Always use high-vac joint grease when distilling.

24) Do not use any type of mineral oil in place of high vac joint grease - always use high vac joint grease.

25) To distill sassafras oil you want 5-10 drops a minute when distilling all fractions.

26) Distilling flask should be no more then 1/2 - 1/3 full at beginning of distillation.

27) Boil over occurs if vac suddenly varies - could be shitty vac source or a change in water temp/pressure going through aspirator.

28) There is at least a 10 min. delay in increasing hot plate dial temp and seing a temp rise on the thermometer placed just before the condensor.

29) Color of safrole is water-white - this indication is better than using thermometer temp reading to discern when the safrole is coming over.

30) Insulate setup all the way up to the take off where the condensor begins.

31) To insulate, do this: vac grease on joint surfaces and teflon tape wrapped around outside of joint seams

32) Use stainless steel bowl for hot oil bath mixture. If glass is used for this oil bath bowl and the glass breaks, or cracks, oil will hit the heating mantle and immediatly a large lab fire ensures...so use stainless steel bowl for oil bath

that won't crack like glass might.

33) Usually everything coming over before bp of safrole is reached is considered forerun.

34) Need hot plate at least 500 w for distillations.

35) Insulate neck and riser assemblies with foil then insulate the whole thing again in a wrap around of foil.36) Don't distill to dryness - BLACK CRAP WILL NOT COME OUT !!!! - use high-oleic safflower oil as a distillation buffer.

37) When distilling sassafras oil, 5% is of lower bp (water?) and 15% of the stuff is of higher bp...collect the middle 80%.

38) When distilling sass oil, distill without vacuum (using a condenser) to like 130C or something just to make sure all the water and other low boiling shit is gone from the sass oil. After this is done, cap the sass oil and let it cool back down to room temperature before applying vacuum and distilling. It must cool down before restarting because if vacuum is applied to hot sass oil it might start coming right over as soon as the low vacuum is reached...or it might start boiling (bumping) violently even with good stirring.

39) Bee says to keep all your yellow leftovers (whats still in the distillation flask) from distilling sassafras oil and combine it to later redistill and a good amount of sasfrole will come over.

# Part 2: Isomerization with KOH/CaO:

If calcium oxide is added to a mixture of potassium hydroxide and safrole, the reaction becomes less water-sensitive as the CaO reacts with any water present to form Ca(OH)2. The reaction is also very fast. (Perhaps is best to decant liquid, then wash goo left by the CaO with DCM to avoid a decrease in yieldl; also, may be able to distill isosafrole without removing CaO, though glass will get caked; best to clean with HCL, then acetone).

# Experimental:

100 grams of safrole was placed in a 250 ml RB flask fitted with a water-cooled Allihn reflux condenser, and was heated with good stirring. When the solution started to boil, 1 gram of KOH pellets was added through the condenser, and the safrole immediately took on a brown color. Next, 15 grams of CaO was also added through the condenser, and the solution was allowed to reflux for 15 minutes, and the heat was turned off, and the solution was poured into a beaker and was allowed to cool. The dark brown suspension was vacuum filtered through a pad of celite, and the filter cake was washed with 2x50 ml of CH2Cl2. The CH2Cl2 was removed through distillation at atmospherical pressure, and unreacted safrole was also distilled off at 240°C (bath temperature), and amounted to about 1 ml. The residue was distilled at aspirator vacuum (~25 mmHg), and the isosafrole came over at 120-130°C as a water-white oil, weighing 70 grams (70%).

# Another Example:

Vacuum distill sassafras oil to obtain relativle pure, clear colorless safrole. Weigh 15-16 g of CaO. Set aside, weigh 2 g NaOH, set aside. In a flask capable of holding a

condenser begin heating the 100 g safrole req'd fro this rxn. Heat it to just below its bp, 230 deg c...add the NaOH carefully in small portions, never allowing the saffy to cool down to much, and when it is all n and the temp climbs some, add the CaO...now crank the heat and pay attention.

It will begin to heat up and something EXCEPTIONALLY strange begins to happen. Safrole begins to go PAST its boiling point and creep slowly upward, stopping occasionally. Sometimes it will quit rising and stick at , say 238 and just stay there for awhile. This is safrole in the process of isomerizing to what is called cis-isosafrole (dont ask) and THAT becomes further converted into it's more stable isomer, trans isosafrole. You may find yourself increasing the heat and adding small amounts of **NaOH** to further 'drive' the reaction forward. If you are patient and the electronic gods of isomerization smile down, eventually you will see the temp hit 254 c and hold at a full reflux. Hold it here for ten min. Allow to cool, and then decant (pour off) all the iso you can while leaving any solids in the boiling flask. Try to get it all. Vac distill isosafrole @60\_70% yield. the rest of the yield is in the solids, wash the solids with dcm and vacuum filter and distill, to get the rest out

Isomerization of DISTILLED 1-alkene with 1% KOH CAN be done, according to SWIM with >90% yields in under thirty minutes @ 243°C.

**Experimental:** Dissolve using heat and good stirring 1 gram of KOH in ethanol of methanol. Once dissolved, add 100 grams of DISTILLED 1-alkene to mixture and increase heat to distill off the alcohol, which can be reused. Place thermometer in flask and continue to heat and stir until temperature reaches 243°C (it will hit 232°C first, linger awhile, but will soon ascend). Hold for 5 minutes, the turn off heat and let cool. Next, vacuum distill to get desired product.

If dirty 1-alkene is used, considerable tar will form, covering up the KOH (making it inactive). Also, the stirring will be slowed immensely. Up to 500g of dirty 1-alkene was used. Stirrer stopped at one point but continued heating and refluxing ensured relatively complete conversion into the 2-alkene. For odor control, a dampened paper towel was lightly stuffed into the opening of the flask. Keep in mind that the cis-isomer of the alkene in question boils at 245°C at normal pressure. The trans boils at 250°C or so.

# Part 3: Peracetic acid Oxidation:

An added improvement would be to neutralize the sulfuric acid used to catalyze the formation of the peracetic acid with 25 grams of sodium acetate (28 grams sodium bicarbonate) before adding the isosafrole. Performic acid forms very easily just by mixing formic acid with hydrogen peroxide, but with acetic acid, it requires a strong acid catalyst and 12-16 hours standing.

\*<u>Note:</u> Adding carbonate to formic/peroxide first would surely lead to a nasty volcano and would not help any. Carbon Dioxide is emitted as performic is slowly added to alkene/carbonate solution. As far as advances are concerned a few things have been realized. Extremely vigourous stirring, such as that produced by a mechanical stirring rig, seems to increase yields. If a considerable amount of isoalkene is recovered during vacuum distillation an easy solution is to add more performic acid mix to rxn. This is usually an indicator that the peroxide used has degraded somewhat below 30%.

Peracid: 1500ml of Glacial Acetic Acid 325ml of 30% H2O2 5ml of H2SO4 Olefin: 500ml Acetone <100ml acetone per 65 grams isostuffrole> 325g of Isosafrole Solvents: 7500ml of Water 1500ml of DCM 500ml of MeOH H2SO4 (15%): 368g (200ml) of concentrated H2SO4 2300ml of Water Wash(s): 1250ml of Water 1250ml of NaOH 5% solution Drying: 50g of Anhydrous MgSO4 Distillation: 100ml of Cooking oil to prevent scorching

1) Mix the peracetic acid chemicals together in an appropriate sized flask (3L), swirl or stir solution until thouroughly mixed and let stand for 12 hours.

2) Combine the olefin chemicals and place into the freezer to chill.

3) Bring the peracid solution down to  $5^{\circ}$ C with the help of an ice/salt/water bath. While magnetically stirring this solution sitting in the ice bath, add the now chilled olefin solution to the peracid solution at such a rate that the mixture stays below 40°C. After all of the olefin has been added, let the solution stir for 16 hours, letting the ice bath melt away and the solution come to room temperature on it's own.

4) After the 16 hours of stirring has elapsed, pour the solution into 7500ml cold water, mix thouroughly, and extract this mixture with 3x500ml DCM. The extractions are combined and the solvent removed via distillation to yeild a thick, dark red syrup.

5) This syrup is taken up in 500ml methanol and added to the 2500ml 15% H2SO4 solution and lightly refluxed for two hours, after which time the flask is cooled under running water and/or the use of an ice bath, do not proceed until solution has reached room temperature or lower.

6) Using a large sepratory funnel or by simply pouring off the acid layer collect the dark bottom layer BEFORE extracting this mixture with 3x400ml DCM (you did reuse the DCM from the first extraction, didn't you?). Combine the extracts with the dark layer which was first collected and proceed.

7) Wash this dark mixture of ketone/solvent with 1250ml of water, then with the 1250ml of the 5% NaOH solution which was made previously. Please note that the separation with the second wash will be difficult to see, therefore the use of a flashlight is favorable to determine the different layers.

8) Dry organic phase over 50g of anhydrous MgSO4 (made by baking epsom salts at 300°C for a couple of hours, let cool, then grinding into a powder) making sure to filter off the MgSO4, and washing the filter cake with a small amount of DCM. The solvent is removed and kept for a later run, the resulting residue is combined with 100ml of vegetable cooking oil and distilled under vacuum to yield ketone (>200 grams) [That is a 56%+ yield].

When adding GAA and H2O2, and the H2O2 slowly to the GAA, which already has 1% sulfuric acid catalyst.

# Another Example:

OK, dreaming of the Semtex Enigma process at a 1/20th scale. In the dream, the smooth solution is clear reddish-orange at the 3 hour mark.

Other than the scale down the only difference so far is that the reactants were cooled a bit more before the addition and were not put into the ice until the temp reached 35C. This didnt really take very long at all considering that the acid started at -5C instead of 5C as Semtex specifies. The ice bath quickly stablized the temp which dropped slowly down down to about 15C. Color seems to change more slowly when it is cold. Considering pulling it out to see if it will go back above room temp on its own...

Color seems to change more slowly when it is cold. Considering pulling it out [of cold bath] to see if it will go back above room temp on its own...

Dreamed that it was pulled out of the ice bath and off the stirrer and it came up to room temp. The temp then climbed to 2C above room temp but got no warmer. Checked with 2 different thermometers, same result. Seems to be holding steady at this temp. Perhaps air cooled by the room? If so the rxn must be proceeding very slowly. Color changed much more quickly once it was taken out of cold bath. Now it is fully that same clear red color that you get with most other techniques (performic, O2 Wacker, HBr, etc..) The color is not an indication of success, just a common color that you get when you destroy a little (iso)safrole by whatever technique. Therefore, I cannot say if the epoxidation goes quicker at higher temps but it does appear that one of the side reactions (the one that causes the red) does go faster at higher temps.

It is now 5 hours since the dream began, the bath no longer contains any ice, just cold water. Since solution is smooth (no layers) and temp is not hard to control, the stirrer is not really that important and its use will be discontinued. Back into the cold water for the rxn flask.

The original reason that I dreamed that the stirrer was removed was that I wanted to make sure that the +2C over ambient temp was not being caused by the slight warmth generated by the stirring plate. It was not. In my dream, I thought about it a little more and decided that stirring wasn't really needed if there was no separation of layers. The atmosphere, or exposure to it, doesn't really play a part in this reaction, right? It isn't like a O2 wacker where it is important that the liquid contact the atmosphere. Also, there isnt really evolution of gas because no hydrogen is being replace or whatnot...an oxygen is adding to a double bond...so there is no issue of freeing bubbles of gas from the liquid to pull the equation to the right.

My dream guess is that the only purpose for stirring is to ensure even cooling so that localized temps do not go above above the criticle level. At 1/20th scale even the air (and perhaps evaporation of acetone) provides enough cooling to keep even the center of the rxn mixture well below the dangerous levels.

This is not to imply that stirring was not required during the addition of the not **isosafrole**, it was. Just noting that the evolution of heat is much less now at the seven hour mark, that it was within the first 30-60 mins. Because the total volume is only 1/20 of the amount in your procedure, the thermal enertia of this rxn is also much much less. The only reason I can think of to continue stirring would be to prevent the concentration of potentially explosive expoxides of acetone. I don't know if such expoxides are indeed formed in this procedure but if they are, it would again be more important to prevent a big explosion than a little one.

What is the lowest temp that this thing should be run at?

I woke up before the dream could procede. The next night I dreamed that the 1/20th experiment was continued but that the solution had been sitting for 24 hours instead of 16. Flooding with water at this point leaves a lot of orangish yellow opaque aqueous layer sitting above a clear red organic layer. So far so good.

Dreamed that it sat in the water for a few hours, water turned more pinkish and less orangish. Extracted with DCM. Evap solvent. Mmmmm, smells right, time for the boiling acid. Dreamed 5 hours in a boiling bath. Aqua layer was amazingly free of color. Started out darker than it finished. Strange. Organic layer much darker as expected. Yield looks about right. Blacklight action looks good. Awoke before the distillation. Perhaps in later dreams. refluxed with 15% H2SO4 & methanol for 5 hours.

# Yet Another Example:

Yields decrease when peracetic is allowed to stand any longer than 12 hours or so (as to say yields have consistently been lower with 16, 20, and 24 hour standing time.) The peracetic acid smells different the longer it stands, which SWIM originally thought was peracetic formation, but now believes to be the decomposition of the peracetic. B)SWIMS yields have been lower, and never have been successfully aminated when using DCM as a solvent, but acetone has produced yields in excess of 50%, which have aminated beautifully. Also, external cooling during the addition (ice water bath) has increased yields by as much as 20%.

All isosafrole used was created via osmiums KOH/vacuum reflux, and known to be of high quality, while SWIMS acetic acid is only 90%, and H2O2 is 25%, with a few carefully calculated and measured milliliters of battery acid. All of SWIMS reagents are OTC.

A)I was refering to the iso addition, using the ice bath. The product quickly takes a red color (indicating the decomposition of iso) when left to it's own devices, and yields have been terrible. When an ice bath is used, the mix maintains a pretty orange color. The monoacetyl(??????)glycol was extracted by flooding the mix with water, and extracting with DCM. Swims latest, greatest, and untested idea is using a carefully controlled vac reflux during the addition of the iso/acetone into the peracetic, which will be attempted whenever SWIM gets around to buying a 24/40 pressure equalized vacuum proof addition funnel. Also, peracetic formation always took place at approximately 20-30 degrees celcius.

B)Swim has never noticed any acetone peroxide at any point in the process. SWIM believes that to be an urban legend, like ecstasy containing heroin, etc...

Hydrolysis was always done with battery acid (check the specific gravity to determine %) diluted about 45/55 to produce 15% sulfuric, and GENTLY heated for 2 hours, and ketone distilled at ~29"hg with swims BEAST of an aspirator station. As soon as I get a digital camera, I'll post a picture.

All aminations were of the al/hg variety, using methanol as a solvent (isopropanol was never used successfully), with ~.5 gram blobs of hg dropped into I2 tincture, allowed to react for a few days, and then used to amalgamate.

Swim recorded an actual amalgamtion time, it took 17 minutes(.5g mercury blob, 30g Al). The basic procedure is to put a plastic dish on the old balance, zero it, and poke at a mercury blob until about .3-.5 grams is weighed out, then dump it into some 2% I2 tincture, the kind you buy at walmart. I can't verify that HgI2 is formed, but I have successfully amalgamated using this procedure:

300ml methanol and 300ml of water are placed in an erlenmeyer flask with 20-40g of aluminum. The tincture is dumped on top of it, and the amalgamation proceeds for about 30 minutes (I use 1" inch squares, thrown in the coffee grinder). After the formation of the grey precipitate and so on, the solvent is decanted, and the al washed with water and then methanol. next comes the MeAm, the solvent, some naoh, and the ketone is dripped in SLOWLY.

This works very well, and is VERY OTC. Also, it is important to use good magnetic stirring during the amination.

Are you refering to the peracetic writeup? If so, that is the one I've had the most success with, and all of the chems are easily OTC. The only thing I skip, is the MgSO4 drying, which seems to be a waste of time to me.

About the first 7-10% of the distillate is camphor, phellandrene, and pinene. The next~85% is safrole, and the deep red shit in the boiling flask is eugenol. No salt in the icebath, just ice and water, remove from the icebath and continue stirring once the temp stabilizes at room temperature. No cooking oil, SWIM reuses the first and last 10% of the iso for reuse to isomerize another batch. Aluminum foil wrapped around the still head and the column is sufficient for insulation. 6 hours is probably OK, but SWIM always lets reflux for 10-12 hours (~125C).

# Ketone Distillation:

1) This time we are going to collect a particular fraction in the flask - there is DCM (BP 40°C), water (BP 100°C), DMF (153°C), safrole (232°C), ketone (BP est. ~290°C), and polymerized crap (BP ~300°C+)

Safrole distillation temp plus approximately 25°C is the temp that the ketone will come over under vacuum. For example, if your safrole comes over at 150°C, then the ketone will come over at 175°C.

2) WEIGH the receiving flask! Write the weight on a piece of tape and tape it to the flask!

Start your vacuum distillation by first turning on your vacuum - if you remembered your boiling stones, then it will begin to boil immediately. This is the DCM coming off first. The boiling may be very vigorous, So watch it, and be prepared to vary the pressure so it won't fly into your receiving flask. Turn on the heat (Hotplate) SLOWLY! and let the temp climb to just over the temp at which safrole came over (SLOWLY: it should take at least 2 hours to reach that temperature - if you do it in under 2 hours you are going WAY to fast).

3) You are going to have to change the flask, when the temp gets to above the safrole temp. This is a bit tricky, because you are going to have to release the vacuum. Release the vacuum at the pump/aspirator and change the flask quickly - you may just dump it out, rinse it once with acetone, or IPA (IsoPropylAlcohol) - and put it back. Start the vacuum immediately, but be careful here, because the Organic layer that you are distilling might jump out of the flask and into the receiving flask - so if you can - vary the vacuum so that the vacuum comes on GRADUALLY! (IE with an aspirator, turn on the water slowly.) The ketone oil is a clear white/yellow hint of green oil. With an aspirator you can vary the vacuum when you are turning it on. You simply turn on the water slowly. IE

Increase water pressure slowly. By turning it off, you can make water jump into the receiving flask because there is a 'vacuum' in the distillation apparatus and it will suck water out of the aspirator.

# Part 4: Al/Hg with Nitromethane

Materials: 27.5 g Reynolds Wrap Heavy Duty Aluminum Foil (0.3 mm thick aluminum flashing soft) 25.0 g MDP2P ketone 20 ml MeNO2 (Nitromethane) of 99%+ purity 750 ml MeOH + 50 ml more 400 mg HgCl2 2L 2 neck FLAT bottom 24/40 reaction flask 400 mm reflux condensor

250 or 500 ml addition or separatory funnel

262.5g NaOH (make soln. by adding solid NaOH to distilled H20 ice cubes (pre-weighted))

31.45% muriatic acid (HCl) for gassing

cooling setup: bucket, water pump, tubing, large bag of ice, tub o-ring clamps, salt

# Method:

1) Cut 27.5g Reynolds Wrap Heavy Duty Aluminum Foil into 1" x .75" squares (1.5 - 2 hrs by hand with scissors). 2) Fill coffee grinder loosely (2/3 full) and grind foil for ~10 seconds. Will take 4-5 loads to d all the foil. When done correctly (don't stuff coffee grinder too much or grind for too long), the foil should be "gnarled little nuggets about the size of long grain rice grains and should look really tight and small." The smaller the better for efficient stirring.

3) Place 3" stirbar into 2L FLAT bottom flask and only stirplate

- add foil nuggets to flask then setup the rest of the glass

- if using 1 head 2 L Flat bottom, then glass = claisen adapter, addition funnel, reflux condensor, and cooling apparatus.

4) Carefully add 750 ml MeOH first, then 400 mg HgCl2 second to a tightly sealable bottle. Shake to dissolve then set aside (heat up to boiling, then let temp reach 55C before addition for "instant amalgamation").

5) Combine the 25 g MDP2P, 20 ml 99% MeNO2, and 50 ml MeOH in a beaker then add this mixture into the addition funnel. Rinse beaker with a tiny bit of additional bit of MeOH to get residual ketone and add this to the ketone / MeNO2 / MeOH solution already in the addition funnel

6) Very slowly and carefully (gloves, glasses, long sleves) pour (using a large funnel) pour the HgCl2 / MeOH solution from step 4 down the condensor. - wrinse condensor down into reaction flask with some MeOH...not much though.

7) Turn the stirring on full blast for a 5 second burst to immediatly mix the solution and the foil. Then give it 3 more 5 second full blast stirrs over the next 3 minutes (helps to facilitate amalgamation of Hg onto the Aluminum). 8) After ~ 5 minutes or so, one would begin to see bubbles popping up on the surface of the MeOH solution. First tiny champagne-like bubbles, then larger like ones seen in boiling water. Around this same time that the appearance of the Aluminum will change from shiny silver to starting to take on a dull gray look, accompanies by a gray cloudy look that begins forming in the MeOH. This is the magic moment to begin dripping in the MDP2P / MeNO2 / MeOH mixture from the addition funnel. Set a drip rate at (3 drops per second until peak is reached. Then back off for a minute, and finish addition). The drip rate can be increased later to accelerate the reaction if so desired. 9) Place ~ 3 pounds of ice into a bucket. When exothermic heat can be felt by feeling the outside of the reaction flask, quickly add ~ 2.5 L h2o to the bucket and plug in the pump to begin circulating ice cold water through the condensor to be colder.

10) While monitoring the growing intensity of the bubbling amalgamation, turn on/off the stirring intermittently as done earlier. This is to ensure even distribution of the ketone / nitromethane into the reaction flask but also because the amalgamation seems to gain its vital momentum more effectively if given some significant blocks of time (meaning  $\sim$  30 seconds at a time) in between stirring bursts. When the reaction is clearly starting to get vigorous and hot, crank the stirring to 10 and leave it on (others suggest not to do this, but stir constantly).

11) As the reaction progresses a few minutes post addition, the aluminum should be breaking up fairly rapidly. This is good as long as the ketone / nitromethane mixture is dripping in at a good rate of ~2 drops / second. Be careful with the addition rate at this point because a rate of greater than 2 drops per second will cause the reaction to kick into unwanted over- drive...not good. The reflux condensor should be unnerving vigorous as the amalgamation really starts to pick up speed: with the MeOH dripping from the condensor really quickly. This is good. At this point, sludge can be observed already starting to settle at the bottom and forming a ring on the inside of the glass around the top surface of the spinning mess. Consistency will get thicker by the minute. Add more ice to the cooling bucket as needed.

12) At this point, the reaction can be slowed or sped up by controlling the addition rate. If the reaction is already barreling along then no increase in the addition rate is needed. The concept is: the addition of the ketone / nitromethane mix should be paced neck-and-neck with the breakdown of the foil as it amalgamates and gets turned into sludge. Adjust the addition so that the 2 processes progress at the same rate. Might want to keep some type of apparatus ready to cool the outside of the reaction flask if it looks like the reaction is getting out of control and the reflux condensor won't be able to handle the escaping gasses. Could be as simple as a high speed fan blowing on the

reaction flask while simultaneously slowing the addition rate...to as aggressive as some type of luke cool water bath to dip up and around the bottom of the reaction flask.

13) Addition should take between 40-45 (some suggest 30 or less) minutes in total and when finished the state of the aluminum should be ~ 95% broken down. 45 minutes past initial addition, the reaction should look like a really thick, steely-gray chowder with only minor silver slivers of undissolved aluminum visible if any at all. Probably will need to add 20-30 ml MeOH at this point (or even before) to help it keep stirring effectively. Preheat MeOH before adding though so as not to slow down the reflux upon addition. Or, if reaction is going to rapidly, adding room temperature MeOH would be a good way to quickly directly cool the reaction mixture.

\*NOTE: on color: 30 minutes post initial addition, the reaction takes on a color that is "light steely gray with blue overtones." Light shade of gray akin to the color of common gray sweat pants, with a slight suggestion of blue. This color indicates a good reaction with properly prepared aluminum foil. Dark metallic gray with green overtones suggests too thick aluminum foil and not enough HgCl2. Green color comes from unreacted ketone.

\*NOTE: Also, on the timing of the addition against the breakdown of the aluminum: There is a definite spike curve to the amalgamation reaction which is easily observed by watching the reflux rate. This build up to peak and subsequent slow down occurs over approximatly 25 minutes or so -- very fast. So at only 20-25 minutes after the

amalgamation was first felt to be heating up, the reflux will have slowed to ~2 drops per second after having reached a peak where the reflux was a stream re-entering the reaction flask and not drops. At 1hr 15min after the addition was begun the reflux will be a slow 1 drop per 2 seconds or so.

Finally, when...

a) the reflux has slowed to almost no reflux at all

b) if no small bubbles can be seen anymore when the stirring is stopped and c) no "uneaten" aluminum is visible and the solution is a thick, uniform gray soup... then the reaction is essentially finished.

\* It will reach this state ~ 1hr 45min to 2hours after the initial addition was begun

\* At this point, add a bit of external heat where the addition has finished and the reflux slows down dramatically \* Leave the reaction stirring for a total of 3 hours AFTER the addition of the ketone / nitromethane solution IS COMPLETED.

- Total reaction time is ~3hrs 45 min.

13) Turn off the heat and allow everything to cool (reflux condensor still cooling) for ~ 30 min.

14) Make a solution of 35% NaOH by adding 750 ml dh2o and 262.5g NaOH. Let this

solution cool back down to room temperature.

15) In a separatory funnel no smaller than 2000ml, pour the cooled gray reaction mixture. Use a magnet outside of the flask to keep the stir bar from falling into the sep funnel which of course would break it. If the gray mixture is really thick, MeOH can be added in small amounts to thin it to a pourable consistency. Wash the residue out of the reaction flask with a few mls of MeOH and add to the separatory funnel also.

16) Slowly pour the 35% NaOH solution into the separatory funnel (glasses and gloves). Basifying should be a gentle process so pour slowly. Solution in the funnel will warm a bit as the last of the aluminum foil (if any) dissolves.

\* Swirl this solution in the separatory funnel a bit and give it ~ 10 minutes or so to return to a temperature closer to ambient...

NOTE - MeAm will smell here. MeAm smell = successful reaction though.

17) When the mixture in the separatory funnel has cooled, extract it once with 400 ml toluene followed by 100 ml toluene. Shake at least 3 minutes (long and hard) for these extractions (while venting).

\* Toluene product layer will be on the top

\* Give layers at least 15 minutes to separate

- \* Separation is complete when interface of small toluene bubbles finally resolves and a nice clean line can be -seen between the layers.
- \* Finish off with a 3rd extraction of toluene using 50-60 mls

\* Keep in mind a vibrator can help during this separation and any done with these procedures.

NOTE: (actually a whole couple of added steps): The combined toluene extracts will

inevitably contain a tiny bit of base / metal / garbage from the bottom layer.

To separate this shit out:

1) chill the entire solution in the freezer for  $\sim 30$  minutes

- when cold the garbage is a lot less mobile

2) decant the toluene / product layer being vigilent around the last 50 mls. or so.

18) Now drain the garbage layer out of the previously used separatory funnel and wash the garbage residue out of the separatory funnel with water.

19) Wash the toluene / product 4 times (or more) in the separatory funnel with 400-500 ml dh2o and a final time with 500 ml dh2o saturated NaCl solution to remove any traces of solvated HgCl2.

20) Dry the toluene / product solution with 30 g of MgSO4 (epsom salts) in an acetone cleaned heat dried bottle or flask for no less than 45 minutes to 1 hr. Shake a few to 5 times during this drying period lasting up to an hour.

21) Filter the solution of the MgSO4 and gas it with that good ol' HCl bubbler setup.

\* (Some choose 2x bicarbonate, 2x brine, 2x water purification technique).

#### Another Example:

55g Al foil, ground in the usual fashion, went by sound, not by time.

50g ketone (~43mL) (from a 1x MM BenzoWack that i thought originally failed)

40mL MeNO2, ACS grade

~200mg HgCl2, guessed... was a small scoop, possible as much as 300mg. new scale is on the way.

basically this was a pure MM AlHg Nitro Amin, 2x of everything except the HgCl2 and the additional MeOH in the addition funnel.

- 1500mL MeOH (hardware store grade) was put in 3L 3N FB flask with ~200mg HgCl2 and mixed for 10m - addition was started +25m after Al was added to flask. stirring was with 3" octagonal bar on my dilapidated thermolyne 1000.

- addition was varried to avoid eruption.

- addition was stopped after +70m., +5m later another 50mL MeOH was added to reduce viscosity.

- 1100mL of (fresh) toluene was used

- the reaction results were split in two (glassware restrictions), each was extracted 1x 450mL toluene, 1x 100mL toluene

- the pooled toluene extracts were washed only 3x with 1000mL dH2O, and 1x sat. NaCl

- recovered ~1060mL toluene after all washes

- dried 10min in 58g (oven dried) MgSO4

- gassed in the usual manner, H2SO4 over NaCl, with cotton and indicating dri-rite inline in a stiff section of white tubing

# Short Note version of Al-Hg :

1) 3" stirbar + 27.5g Al foil nuggets -> into a 2L FLAT BOTTOM 24/40 flask

2) Add first 750ml MeOH then 400mg HgCl2 shaken to dissolve in a sealed flask / container

3) 25g ketone + 20ml MeNO2 + 50ml MeOH -> into an addition funnel

4) SLOWLY pour 750ml MeOH + 400mg HgCl2 (dissolved) down the condensor to the 2L RB flask previously loaded with stirbar and Al foil

5) After short bursts of intermittent stirring (5-7 mins. later), Al is amalgamated -> not begin the addition of ketone + MeNO2 + MeOH mixture @  $\sim$  2-3 drops per second

6) Add the ketone solution as quickly as the condensor allows

7) Add up to 100ml preheated MeOH soln. to rxn. flask to facilitate stirring

8) When reflux slows, add mild heat and reflux for 3 MORE hours

9) After 3 hrs., stop heat then let soln. cool to room temperature

10) Transfer cooled rxn. mix -> 2000ml sep funnel and add SLOWLY cooled soln. of 35% NaOH (750ml dh2o + 262.5g NaOH)

11) Swirl well and let cool back to room temperature

12) Extract with TOLUENE 3x using 400ml, 100ml, then 100ml

13) Cool sealed and combined toluene extracts in freezer - then decant toluene from slower residual trash layer -

add 100ml more toluene to trash - rechill - re-decant - then combine.

14) Wash toluene / product 4x with 400-500ml dh2o

15) Wash toluene / product 1x with 500ml saturated solution of NaCl in dh2o

16) Dry toluene / product with 30g MgSO4 - filter then seal tightly

17) Now gas this solution directly with anhydrous HCl gas for xtalization

# Notes on Animation:

1) After you add the NaOH to your solution (to rid of unreacted Al) it will heat up. Let it cool before going on, about 1 hour. not sure if longer = bad yeild

Adding the nonpolar first also reduces the extraction time and ensures that you get more of your goods. Especially as it sits for a an hour and the oil is forced to the top into the solvent. All this can be sped up and made easier if you boil off the alcohol before doing anything then replacing it with base solution and solvent for the oil.
 Constant stirring is best so that when it starts to get thick it has some momentum to keep it spinning. You'll find that a low torque magnetic stirrer will not start again once it gets too thick. And you can always add some more alcohol to thin it some.

4) Don't put the nach solution in the fridge too long or let it bee too cool before adding to the nitro mix(which here is let cool for an hour) it will fuck up the yield. When adding the nonpolar before basing always resulted in discolored toluene!

5) Boiling off the meoh, always helps, esp. if you have a smaller reaction flask and sep. ie less volume.

using a heavy duty hp/stirrer is always a good idea. Never cut the stirring on and off here and always got 75-80% 6) Make sure to add more MeOH through whichever neck if the reaction begins to get thick and doesn't stir as well.

Also, the flask may be picked up and stirred by hand occasionally if this helps to get the mag stir bar going.

7) If reflux starts to get out of hand, slow down the addition rate of the ketone / nitromethane solution.

8) Add as much MeOH as it takes to keep the aluminum covered during the addition cycle... Much MeOH is lost due to evaporation even with the condensor circulating ice cold h2o.

9) When the addition is done, try to break up the aluminum as much as possible (by hand swirling?)

10) Cool the condensor to ice cold before the addition is begun to ensure higher reaction yields.

11) During extractions, shake as long as you can for as long as you can.

12) The reaction is refluxing too quickly when bubbles can be seen forming and rising inside the condensor.

13) Methyl Man says - to add incrimentally enough MeOH during the reaction so as to keep it stirring well at all times.

14) Methyl Man says - "you HAVE to make that puppy run HOT"

15) Get a high output water pump for condensor water circulation here to cool this hot reaction down sufficiently.

"Reaction should be really humming when you've dropped in 20% of the nitromethane / ketone / MeOH mixture. Flask too hot to touch for 1/2 second. Should still be this hot when the last of the nitromethane / ketone / MeOH is dropped in.

16) Bizwax says - it wouldn't hurt to let the reaction run 6-8 hours to make sure it is completely done.

17) Water IS needed in this reaction so do not dry MeOH before using.

18) Use at least a 150 gph water pump for condensor water.

20) The condensor water should always be super cold.

21) Methyl Man says - "watch the vortex, if no vortex is noted then add incrimentally up to

~ 100 ml MeOH to help stirring commence again."

22) Methyl Man says - use a 400 mm Allihn for reflux for this reaction.

23) High-oleic safflower smokes when heated to a high C the second time, but its ok to use the first time.

24) To avoid MeAm smell during the reaction, run a tube or hose from the top of the condensor to a 3 N solution of HCl. This will turn any gaseous MeAm into its HCl salt and eliminate any aroma.

25) When adding extra MeOH to help keep the reaction stirring, make sure to pre-heat the MeOH to  $\sim$  55-60 C before introducing to the reaction flask so as not to cool the reaction contents too much.

26) Osmium says all the aluminum sludge must be removed before the ketone workup (a/b washes / extractions) is done. If ANY aluminum sludge is left over it will TOTALLY fuck up the a/b washes. If not sure that ALL the sludge is removed, then filter until ALL THE ALUMINUM SLUDGE IS REMOVED WITHOUT QUESTION!

27) NEVER, EVER, EVER allow too much solvent (MeOH) to evaporate without adding more during this reaction so that improper stirring ever occurs. A guaranteed DECREASE in yield will result if PROPER STIRRING is not MAINTAINED at ALL TIMES!

28) Add 2-3ml dh2o to rxn. flask solution after the aluminum has initially amalgamated.

38) Reply to a pm to baalchemist - he says he gets better yield in the mm al-hg by speeding up the addition rate. He says that mm has said (of his own original tek) that with the slower addition of the ketone / nitromethane / methanol mix (30-45 min) that the aluminum foil is too far broken down by the end of the addition...thus resulting in less product yield %. Baalchemist says he increased the size of the rxn a little and increased the addition rate and now gets 31g salt from 31g ketone. Says he always worried about the volcano factor during this reaction, but has found that as long as the condensor is super cold and if one is using a rxn flask with plenty of headroom that faster addition can be accomplished with no worries.

39) Extraction method:

- keep it stirring while cooling, then add 400ml toluene, then slowly add base solution (300g NaOH in 825ml H2O).

- stir this for 30 minutes
- remove toluene
- add another 200ml toluene
- stir for 20 minutes, remove toluene
- extract with prolonged stirring with a last aloquat of 100 ml toluene
- combine toluene extracts and dump in 35g dried Epsom Salts
- stir for 1 min. then let sit for 2 minutes
- then gas for 31-33g yield
- faster addition + less HgCl2 = higher yields
- also make sure you have a damn efficient condensor

- put a wad of paper towel in the top of your condensor to keep hold of stray vapors

40) Baalchemist says - he "carefully pours nitro, MeOH, then ketone into addition funnel when making the mixture." these layers of chems sometimes separate so this may be a fairly important step he's mentioning here.
41) Make sure that the NaOH is a little below room temperature before putting it in and that a condensor is placed on top of the vessel just after the NaOH solution is added. Methyl Man says about this step..."if basification is not done cautiously and slowly, the goods may get all pouty and decide not to cooperate later during crystalization." SOOOOO the lesson here is, again, to ad the NaOH solution slowly.

42) When doing the Al-Hg addition step, have a high speed fan pointing at setup at all times. This is to be used if rxn. starts to get out of hand and needs to be cooled quickly. If fan is used a wet paper towell or piece of cloth should be placed over the condensor so the air current won't blow any uncondensed vapors out of the condensor. Make sure rxn. setup is well braced though so as not to be blown over.

43) MM says "at the 55g aluminum scale, the al-hg reaction, one 400mm Allihn reflux condensor ISN'T enough." 44) "One key factor - and Baal and I are in agreement on this - is that there is a 'spike' in the middle of the amalgamation process ... a place where it gets hottest based on how fast the ketone had been added up to that point. Assuming a decent rate of addition in the beginning, like around 2 drops per second, with experience one can take the reaction to the brink of volcano-ness --- the spike ---- and then stop the addition for a minute, let it calm down, and then from that point forward it is less subject to go nuts and one can speed up the addition significantly (with caution of course). Yields are ABSOLUTLY LINKED to the rate the ketone goes in verses the rate of amalgamation. In other words, if the aluminum looks mostly reacted, and you still have half your ketone in your addition funnel, you will get a shitty yield. This is not news but it bears repeating because it makes all the difference

in the world ! "

45) When doing the addition part of the al-hg, make sure the glassware is setup so that the heat / stirring source can be removed. This is so that if the reaction starts to erupt due to the addition being done too quickly, the stir source can be removed out from under the glassware (which stays suspended) and a pan of cold ice-water can be brought up underneath the reaction flask to cool it off quickly. One of those platforms that crank up and down would be an ideal platform to set the heat source on. This would allow the heat plate / stirrer to be cranked down and removed quickly.

46) "SWIM uses no more than 100mg of HgCl2 on reactions as high as 60 grams (al?) in a 2 L flask. SWIM cut the HgCl2 amount back because it slowed the reaction pace (amal.) down and the intensity to a more workable pace, thus doing larger scale reactions (60g) in that same 2L flask that was used for the 25g reactions. SWIM has used as little as 40mg HgCl2 in a 60g reaction, but it takes much longer for the initial amal. to kick in, but finishes like a champ with no probs. Definatly not one for the newbees" Take from this that maybe 100mg HgCl2 should be could be a good starting point

47) Baalchemist's exerpt from an al-hg thread "SWIM uses a lot less HgCl2 than MM, about 100mg. Try 2-3 drops in the beginning until it refluxes hard and is about to blow, then stop for a minute or so, then start addition again as fast as possible SWIM ran one the other day with 30g+ ketone in, 34 out. That size reaction seems to produce the highest yield % here.

48) Stuffing coarse steel wool into a reflux condenser makes it work much more efficiently during the initial addition and the subsequent refluxing that takes place in the al-hg rxn.

49) Use a ruler or other straight edge on top of the foil leaving an inch exposed. Pull the foil starting from the corner furthest from you and pull against the straightedge. You'll tear a perfectly straight strip (well, mostly after you get the hang of it). This cuts the foil prep time down by at least 70% and you don't have to fuck up a pair of scissors doing it. Another idea is to use a long straight edge (long metal ruler) but use an exacto knife all on top of glass to cut the strips...then put the strips together and use a pair of scissors to cut them into 1" squares.

50) Red water after the al-hg reaction is a sign that the ketone used in the rxn. was dirty.

51) noj says that stuffing steel wool in a condenser DEFINATLY helps to contain a reflux.

# Part 5: Crystalization:

This first step can be done ahead of time. Like, during a distillation or reaction:

1) Get a fresh bottle of 91% Isopropyl Alcohol, and pour out approximately 100mL. Now get some of your DRY Magnesium Sulphate (Epsom Salts) (about 100mL worth) and dump it into the 91% IPA. There might be a little heat evolution, but not to worry .... Shake it up and then let it sit till the MgSO4 settles out. Pour off the IPA, and then put more fresh MgSO4 into it (about 100mL worth).

2) Now shake the bottle and let it sit for 15 minutes. You must do this at least three times - It really is much better to do it four times - Why fuck it up now right? After three times you will have 'Dry' IPA. You can actually do this while you are distilling or waiting for a reaction to proceed.

3) Make a mixture of 100mL 'Dry' IPA and 150mL Xylene. Pour it into the flask that contains the MDMA oil, and drop the stirbar in too. Stir so that the whole thing is mixed up real well - Now drip in Muriatic Acid slowly. Test with pH paper every 5 drops or so - keep adding the Muriatic Acid until the pH of the solution is 5-6 - or just barely acidic.

4) Set up for vacuum distillation, and distill the solution, distill this with the stirbar in instead of the boiling stones - IE When the solution has reached a pH of 5-6 - Start distilling. Be careful not to let the solution get above  $120^{\circ}$ C - When a lot of the solution has boiled away, crystals will spring to life in the flask. Under vacuum, the solution should never get above  $70^{\circ}$ C.

5) Filter these with a coffee filter, suspended over a jar - This gets the excess IPA/Xylene out. Scrape the crystals on to a plate and let the crystals dry, by letting the IPA/Xylene (mostly Xylene at this point) evaporate - this might take several hours - a 60 Watt lamp 6 inches (20cm) away from the crystals shining brightly helps. Stir the crystals to promote even heating.

6) The crystals will be a brown-yellow color. Now scrape the crystals into a jar, and pour ~20mL of acetone onto them. Swirl the mixture. The crystals won't dissolve - but a lot of the brown will. The brown-acetone is poured off, and the acetone wash is repeated.

Chems:

Non-iodized NaCl (table salt)

HCl acid (muriatic acid at pool stores is 31.45%)

Concentrated H2SO4 sulfuric acid must be at least 93% but 98% is much better

(if h2so4 is less than fuming, heat it until all water steams off and acid begins to fume...then and only then is it conc. fuming sulfuric acid)

Drying agent to bubble gas through (MgSO4 or preferrably conc. fuming sulfuric acid h2so4)

Equipment:

Small separatory funnel / addition funnel

Side arm Erlemeyer flask (makes airtight junction with sep / addition funnel)

Aquarium tubing

12" long glass tube to be placed @ end of aquarium tubing

\* Drying agent in-line before glass tube and drying agent holding apparatus suck back container to contain any product liquid which might get sucked back into the aquarium tubing if HCl gas slows and a neg. pressure is created in the system.

#### Method:

1) Might want to use rock salt instead of non-iodized table salt.

2) Heat H2SO4 to drive off all h2o to fuming before using it.

3) Do ALL of this reaction outside as HCL gas causes EVERYTHING to rust or at the VERY LEAST, for health reasons, do it all under a fume hood that will carry ANY AND ALL excess HCL gas fumes away

4) Layer salt ~1-2 inches in flask

5) Make sure to place cover (with inlet hole for glass tube) over container to be gassed to help reduce the amount of atmospheric humidity entering the dried solution

6) Barely dampen NaCl with muriatic acid - just make sure all salt grains are wet...no more

7) Slowly drip sulfuric acid in from addition funnel above

How to know when to stop gassing:

- a) stop gassing when pH is between 5 and 7
- b) filter off crystals which will make the pH rise again
- c) re-gas until pH again decreases to between 5 and 7
- d) Colour change is a sign of over gassing when your toulene goes from yellow to red.

#### Theory.

Muriatic acid, a mixture of water and HCl, is slowly dripped into a stirred flask containing sulfuric acid. Sulfuric acid has a strong affinity for water. The sulfuric acid dehydrates the muriatic acid, absorbing water and liberating HCl(g). The liberated HCl is then bubbled through a smaller quantity of sulfuric acid to remove any trace of water vapor, and then the dry gas is absorbed in stirred, cold IPA. By weighing the IPA before and after gas absorption, the exact production of HCl can be determined. This exact amount of HCl is very useful in subsequent reactions, since stoichiometric amounts of HCl can be dosed into base/solvent mixtures, in contrast to direct gassing of such mixtures, which is a "hit or miss" and exothermic operation. The HCl/IPA mix is stable and can be stored for future use, unlike HCl/acetone mix, which quickly degrades. IPA is not hygroscopic as is acetone, another important consideration. (Bees may note that this information is in contrast to several major Hive write-ups where HCl loading into acetone and/or direct gassing is recommended.)

#### Practical application.

#### Use 2 parts by volume of sulfuric acid/drai

n cleaner for every 1 part of muriatic acid. Put another way: use 2 bottles of drain cleaner for every 1 bottle of muriatic acid. (This will provide an excess of sulfuric acid necessary for proper dehydration of the muriatic.) The most important consideration is to constantly stir the sulfuric acid/drain cleaner while muriatic acid is being dripped. If the sulfuric acid is not stirred, a layer of muriatic acid will form on top of the sulfuric acid and when the flask is moved for any reason the two layers will mix with violent release of HCl(g). THEREFORE always stir the sulfuric acid while adding muriatic acid, this stirring cannot be avoided without disasterous results--so the magnetic stirrer is essential. Repeating again, ALWAYS stir the sulfuric acid while adding muriatic--do not shake by hand--stir with a magnetic stirrer and telflon coated stirbar. The gas bubbler is another important part of the set-up because it allows visual evidence of how much gas is being

produced and it removes any water vapor that might come over with the HCl(g). The goal is to produce anhydrous HCl(g), since water vapor is detrimental to most hydrochloride crystallizations for which one would use HCl/IPA. Since HCl(g) is completely absorbed by cold IPA to a loading of 25% or greater, there will be no bubbles coming out of the receiver flask after the initial air is driven out of the gas generator set-up, therefore the bubbler provides the only visual indication as to whether the gas generator is going full blast or has stopped or has sprung a leak (although in the case of a leak, the characteristic mist of HCl(g) and the smell will be evident).

#### Suck-back.

Suck-back occurs when the amount of gas being generated cannot overcome the rate of absorption. A trap must be placed between the gas generator and the receiver as a safety precaution to collect any IPA sucked-back. To control suck-back, a stopcock placed before the bubbler and before the receiver will allow the operator to relieve line pressure.

#### Additional tips.

Do not use a dispersion tube while absorbing HCl(g) into IPA, as has been recommended in other posts. The dispersion tube is not needed since HCl(g) is immediately soluble in cold IPA. The dispersion tube could increase line pressure and cause a joint or flask to pop. 3/8" ID polyethylene tubing such as can be purchased at the (big orange) hardware store is sufficient for both the bubbler and the receiver. Stainless steel tubing is not recommend--this is in contrast to what has been suggested in another major Hive write-up--since HCl will corrode stainless steel and chromium/nickel/iron chlorides will go into solution and possibly contaminate the final product. Telfon hose is recommended for glass joint to hose connections; however, the clear vinyl hose found at the (big orange) hardware store can be used once. All hose connections must be fastened with hose clamps (obtained from any hardware store).

With proper fittings this apparatus will not leak and will not smell, but working under a fume hood is always a good general practice.

Yield.

Yield is close to theoretical. Over 300g of HCl(g) will be produced from a 900ml bottle of muriatic acid in about 2 hours of slow dripping. Once the gas production begins to diminish, the sulfuric acid in the reactor can be heated and additional HCl(g) recovered (this is where the stirrer-hotplate combo comes in handy). If more than 10kg of HCl is needed in one day, then acid addition will have to be much more rapid, requiring larger size flasks and larger bore tubing to accomodate the increased reaction rate, but the novice should not need more than a few kilos of gas in one day, which can be generated with a 5L reactor.

# Note on Crystallization:

1) MUST MAKE SURE THE SOLUTION ONE IS GASSING IS ABSOLUTLY DRY BEFORE ATTEMPTING TO GAS. IF THE SOLN. IS NOT DRY THEN ALL SALT WILL FALL OUT IN THE FORM OF AN OIL AND NOTHING WILL PRECIPITATE...MUCH WILL BE LOST

1) Make sure the container one is gassing into has a large opening as the salted out product must be scraped out by hand.

2) ALL SOLUTIONS MUST BE DRY (including the toluene the product is in and the HCL gas).

3) Make sure salt used is baked and dried before using

4) No puddles should ever be present in NaCl layer...no puddles of HCL especially and also no puddles of the h2so4 being dripped in.

5) Epsom salts can be cooked @ 550F for hours and still not be dry. But then it'll be rock fucking solid. Find a medium where (highest temp.) the salts can be baked for  $\sim 2$  hours and not be rock solid and still be usable.

6) Make sure to put an empty wash bottle in-line just before glass tube to save product if suckback occurs

7) To dry gas, one must bubble HCl (g) through concentrated h2so4 to do it effectively.

8) Suckback is caused by too slow a drip rate of h2so4 into NaCl dampened with HCl.

9) NEVER GAS IN DCM...ONLY TOLUENE

10) Yellow droplets collecting on inside of gassed vessel indicates non-anhydrous conditions - probably coming from HCl...bubble HCl through concentrated H2SO4 before it hits the toluene / product layer.

11) Have a beaker / glass full of water standing by (saturated with baking soda) when HCl gassing to receive the remnants of reactant gas (HCl) when xtalizing is finished.

12) When gassing procedure is done and all the white is filtered out...tightly (air-tight) cap the remaining solution and put in the freezer overnight to cool the solution. This will give some more hcl crystals after the solution has cooled down for a while.

13) Might want to wash filter cake (MDMA\*HCl) with freezer cold ANHYDROUS acetone to remove more discoloring impurities. Shoud lose  $\sim 10\%$  weight doing this step.

14) Acetonitrile - is a great solvent for crystalization if you can get it. Might be good for later on if can get this solvent. Note to do more research on this solvent later on.

15) HCl gas EATS THROUGH VINYL hose (the clear stuff at Home Depot), but DOES NOT eat through RUBBER hoses. Sooo...use rubber hoses to pipe HCl gas with instead of vinyl hoses.

16) Felix says - add the H2SO4 SLOWLY!

17) Bizwax says - When gassing - only put enough HCl acid in salt so that all the salt is wet / not dry, and NO MORE....DEFINATLY NO PUDDLES !

18) One poster says he stirrs freebase / toluene solution FULL SPEED when gasing with glass HCL (g) tube 2 cm below the surface of the stirring solution.

19) When gassing, when HCl flow slows down enough (from the end of the pipette) so that the end of the gass tube begins to get clogged with \*HCl salt, remove the tube from the toluene / product liquid layer until HCl (g) production rate increases. When gas slows and salt forms in the glass tubing, this means suckback is just around the corner so remove the tube from the product to reduce the chance of this happening

20) Also, same file as #20 above, if / when any h2so4 'puddles' form in the salt layer when dripping h2so4 onto mixture of hcl (l) and NaCl (s) when gassing, it is ok / good to swirl the beaker around a bit to redistribute the h2so4 puddle.

21) From improv\_chem:...When gassing is complete and no more white stuff is forming, filter out white stuff and add a little distilled water (~5ml per 100ml solvent) and shake the shit out of it for a while until the solvent gets clearer. Then separate off the water and pour it onto a plate on your hotplate on low with a fan blowing on it. When all the water has evaporated, scrape off the remaining product. This helps when hcl gas crystalizing all kinds of amines.

22) After gassing, ALWAYS put filtered gassed solution in the FREEZER OVERNIGHT and a couple more grams will crash out.

23) "as long as your mixture, sulfuric + rock salt, doesn't get too hot, you won't have a problem with your gas line spewing moisture...if this happens you're screwed. You can avoid this by varying your gasline diameter and using longer lengths. If you see moisture start to climb, back it off. Also, if your oil has water or other garbage in the bottom, separate that stuff out before gassing...with it in there one is sure to have problems when gassing."

24) It DEFINATLY IS possible to overgas and bring the pH down too low. Because of this, do one of two things...1) fractionally separate out salt product at different periods during the gassing process to save what has already percipitated out...or...2) find a way to measure the pH of the solution being gassed and check it every so often during the gassing process.

25) If too much water is introduced during gassing, after / during hcl gassing a tiny water layer will form in the beaker being gassed below the non-polar layer and a lot of product will be solvated in that small layer of water.26) Note: evidently HCl can be found in canisters and is used by electricians somehow or another.

27) When finished gassing (DONE OUTSIDE) stopper the rxn. vessel immediatly (the vessel the gas was created in) and remove stopper only when whole apparatus is UNDER WATER (possibly a bucket of water).

28) One guy has found that it is better to gas two container with 3-4" of liquid than one larger container with 6-8" of liquid.

29) Probably need to distill store bought H2SO4 before using it to gas with as it probably isn't CONCENTRATED Sulphuric Acid (93%). Heat it up until it smokes...this means that most all the water has been dirven off.

30) Putting undried mdma.hcl crystals in a round bottom flask and evacuating the air with a vacuum is a good way to speed up the drying process.

31) Chromic says that in crystalization and recrystalization, leaving the anhydrous solution in the freezer for 2 hours is necessary to get all the crystals out. At room temperature, all the mdma will not crystalize out no matter how much the conditions are anhydrous and no matter how much gassing is done...2 hour trip to the freezer is definatly necessary.

32) No matter how pure H2SO4 is before using it for crystalization, make sure to heat it up and fume it (dry it) before using it.

# RECRYSTALIZATION AS PER RHODIUM:

1) Dissolve crystals in minimum amount of boiling IPA (isopropyl alcohol)

2) Let the solution cool to room temperature

3) Add dried acetone with stirring (@ room temperature) until mixture becomes cloudy

4) Cool in freezer (stoppered air tight) for 12 hours.

5) Filter

# Part 6: General Notes:

1) Always use 2-3 wraps of teflon tape on all joints when vacuum distilling from top aspirator threads (where it goes into the sink head) to hoses and nipples. For 24/40 joints use Super Lube found at www.super-lube.com - comes in a silver blue tube...use a match stick head sized bead for each connection...that's all.

2) Use plumber's insulation cut to size for insulation of distilling apparatus. Thick fiberglass cloth.

of the ketone / nitro into the solution / reaction and these reactions had a really hot reaction temperature.

3) An indication the al-hg is going too vigorously is when water bubbles in the condensor and is pushed upward / cracking / falling back into the solution by rising vapors in the condensor.

4) When the Methyl Man al-hg reaction becomes thick - like oatmeal thick – additional methanol was added (pre heated) to get things moving again.

5) To ensure one doesn't over-gas:

- 1) Gas toluene / product layer
- 2) Remove crystals
- 3) Continue gassing and removing crystals incrimentally until further gassing shows no further crystal development

6) The beaker (or whatever) full of H2SO4 (in-line drying agent) should be filled with shattered small pieces of glass to force the HCl gas to run a gauntlet of interfaces before it exits the container.

7) If all the Methyl Man nitromethane al-hg animation results in less than all the foil reacting and poor crystalization follows, this may be a sign that the foil nuggets were ground too tightly.

8) To make a water aspirator more quiet, wrap towels above and below it. A piece of latex hose going down through the "S" trap below the sink drain helps to make it quiet and helps to increase realized vacuum pressure also. NOTE: If using aspirator, run it from the main water line ... this should increase h20 pressure (and vacuum pressure) significantly.

9) Don't fuck up with connecting water hoses to the condensors. Make sure to use hose o-ring clamps so water doesn't end up spraying everywhere fucking shit up.

10) To avoid the smell of MeAm during the MM al-hg, one can lead a tube from the top of the condensor to a glass of 3N HCl to turn MeAm (g) into its HCl salt.

11) Most organic solvents are heavier than water so putting them down the sink will get them trapped in the PVC pipe's low spots eating a hole in the shit so don't do it.

12) Put salt into condensor cooling water (along with ice and h2o of course) - the salt will make the ice liqudize at a lower temperature.

13) Also, keep recirculating condensor cooling water in a STYROFOAM COOLER to help it stay cold ! Especially when doing ketone distillation and Al-Hg addition reactions.

14) WHEN DISTILLING KETONE...NEVER LET IT GET ABOVE 180C...IT WILL CARBONIZE!

15) At least, if a fire extinguisher isn't readily available, have a wet towel always on stand-by when reacting or distilling. And a box of baking soda.

16) Good device to use when transferring / decanting liquids...30-50cc feeding syrings (found at farm stores) coupled with clear vinyl aquarium tubing. Can use to suck up liquids very accuratly. Never never use a turkey baister...they suck and don't hold liquid well.

17) The more expensive metal water aspirators pull 40-50% more vacuum than their cheaper plastic counterparts.

18) Make sure to get an aspirator that has a ball-type valve that doesn't allow suckback if water is turned off.

19) Can reuse DCM (2-3x) a number of times if one takes the time to re-distill it between reactions to purify it on its own.

20) When using an aspirator, make sure to put a water trap in-line with the vacuum hose to make sure the h20 doesn't suck back into distillation receiving flask if h20 is turned off before the vacuum line is removed from the aspirator...ALWAYS REMOVE HOSE FROM WATER FAUCET ASPIRATOR BEFORE TURNING OFF THE WATER TO PREVENT SUCKBACK ONE WAY OR THE OTHER.

21) When washing final product crystals with acetone, make sure to DRY the acetone first or crystals will disolve into the water still in the acetone. Also...make sure the acetone is not only EXTREMELY DRY but also FREEZER COLD before using it to wash crystals.

22) To get rid of emulsions, one might run the whole mess through a buchner funnel to break things up a bit. Afterwards let it sit a while to let the layers separate.

23) When makeing a solution of sodium hydroxide in water, first make distilled h2o ice cubes (as many as possible) and add the solid NaOH directly to the ice (weigh the ice first of course). This is because NaOH in H2O creats boiling heat and must be cooled down before it is used as a wash solution...especially in DCM (bp 40 C).

24) ALWAYS CLAMP ADDITION FUNNELS...ESPECIALLY DURING ADDING H2SO4

TO MURIATIC SALT MIXTURE TO CREATE HCL GAS

25) On drying solutions with MgSO4:

- before dumping dehydrated MgSO4 into a solution to dry it, prewet the MgSO4 with pre-dried solution so that it doesn't absorb much of it (along with product) when it is dumped into solution

- after filtering dried solution of MgSO4, rinse the filtered MgSO4 with pre-dried solution to remove any product that may be sticking to it.

26) All stopcocks must be greased or they will stick in their joint. A trick to get one unstuck is to soak it completely in coke for 24 hours and this should help to get it unstuck.

27) ChemReack warns against getting a diaphram pump...says rotary vane pumps work much better and then one could graduate up to compressors Osmium says 'it depends on what you want to do...a diaphram pump is more versatile.'

28) GENERAL SAFETY NOTES AND NOTICES:

a) if an acid has splashed onto skin, fill sink with water, dump sodium bicarb (baking

soda) in sink full of water and dunk affected area in sink water.

b) if a base burn occurs, dump vinegar directly on skin

29) "Concentrated H2SO4 is like syrup." So if available h2so4 is liquidy and not syrupy, then carefully distill (heat it) until it fumes...then its concentrated.

30) Water is crucial to amine formation... (MeOH instead of isopropyl, may need to add water).

#### **Plans for Aspirator Station:**

SWIM uses a 3/4 hp jet type pump(~\$200, pool store), in conjunction with an ~\$8 nalgene aspirator. The piping is all metal, and is fed from an 8 gallon rectangular trash can. No holes in the can, the inlet piping goes up and over the top, and down into the can. For this to work, you have to first fill the pump resevoir, put the lid on it, rotate the inlet piping so that the inlet hole is facing up, fill that with water, and quickly rotate it into the filled trash can. I'll send you a picture as soon as I take one, which should help clarify what I just said. The way the aspirator is connected to the glassware, is a 3" piece of 1/2" ID pneumatic hose (nylon braided rubber/neoprene) is slipped over the aspirator (that hose adapter that comes with it is not used, and be sure to grease the hose), and as luck would have it, SWIMS vacuum inlet adapter is 1/2 OD, so it fits the aspirator and the glass perfectly, without any vacuum loss due to excessive hose length. SWIMS last ketone distillation, with ice in the can, came over at ~145C-150C(and was successfully aminated). SWIMS newest idea, is to add some ethylene glycol aka antifreeze to the water. It would lower the freezing point of the water, allowing colder water to be used, and raise the bp, allowing higher ultimate vacuum(in theory). The outlet piping on the water pump goes up and over the edge too, with just enough water in the resevoir to cover some of the discharge tubing.

Inches of mercury refers to a gauge measurement usually seen on automotive gauges. It is relative to the atmospheric pressure, where:

0"Hg = 1 atm = 760 torr = 760 mmHg = 0 psig 29.02"Hg = 0 atm = 0 torr = 0 mmHg = -14.7psig.

# Part 7: Boiling Points

MmHg InchHg Safrole Is			Isosafrole	Water	DCM	MDP2P
760	0"	234.5	255.0	100.0	40	~290
700	2.3"	231.0	251.2	97.7		
600	6.2"	224.7	244.6	93.5		
500	10.0"	217.3	236.7	88.7		
400	13.8"	208.4	227.6	83.0		
300	17.7"	197.7	216.3	75.9		
200	21.5"	183.7	201.8	66.4		
100	25.4"	162.2	179.1	51.6		
90	25.8"	159.2	175.9	49.4		
80	26.1"	155.7	172.2	47.1		
70	26.5"	152.1	168.4	44.5		
60	26.9"	148.0	164.2	41.5		
50	27.3"	143.3	159.2	38.1		
40	27.7"	137.3	153.0	34.1		
30	28.1"	130.0	145.2	29.0		
20	28.4"	120.8	135.6	22.1		
15	28.4"	114.4	129.0	17.5		
10	28.6"	106.4	120.5	11.2		
8	28.7	102.0	116.0	7.9		
6	28.8"	96.6	110.2	3.8		
5	28.8"	93.2	106.6	1.2		

\*Check boiling point at STP when distilling, then allow to cool, and distill fraction.