

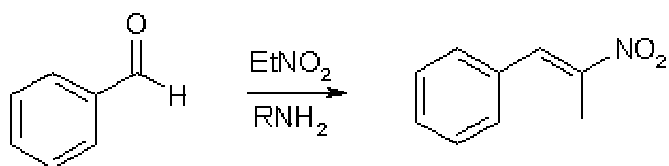
Complete amphetamine synthese.



This is everything you need for making P2NP, from left to right:
Pipette-aparatus, several pipettes, dean&stark trap, PTFE greas, toluene, measuring cylinder 100ml, nitroethane, benzaldehyde, n-butylamine, above the brown flask a heating mantle and a 250ml RB-flask.

The ratios you should use are 1mol benzaldehyde and 1 mol nitroethane and 0,1mol R-NH₂.
Converting mols to ml s you will find out that you will need:
SWIM didn t use 1 mol right away but first wanted to experiment with smaller amounts, so he used:

17ml benzaldehyde
13,3ml nitroethane
3,3ml n-butylamine
33ml toluene



Be sure that none of the products you use will contain water since when performing the reaction water will evolve which will be taken out of the reaction with the dean&stark trap because it prevents a good yield of P2NP.



All the chemicals mixed together.



An overall view



After heating it a bit to get it at 100* C aprox.

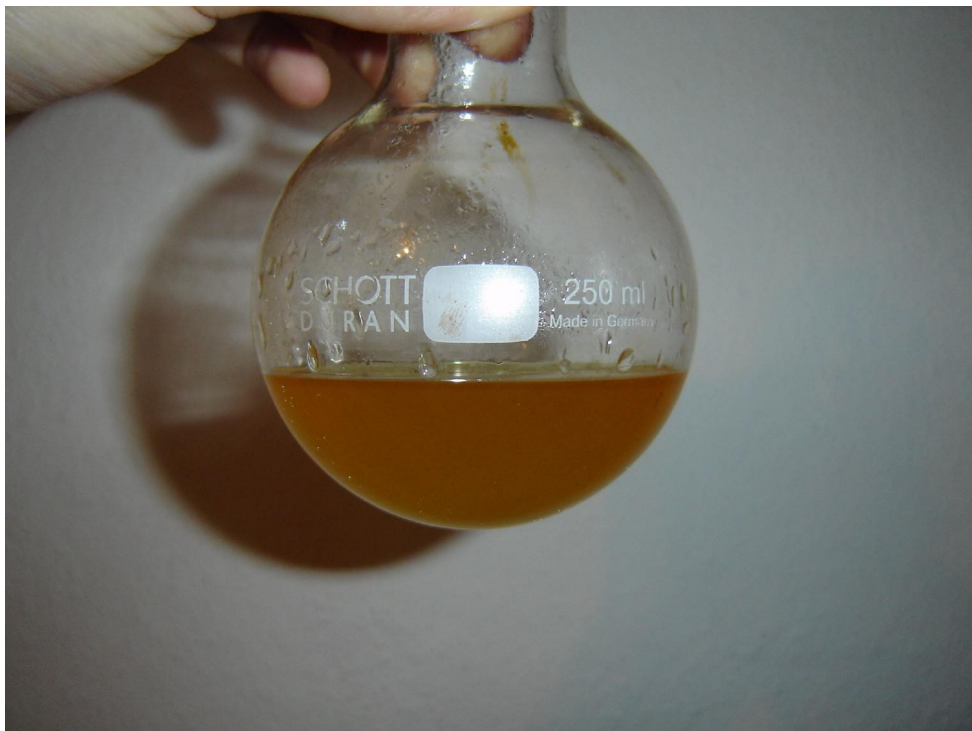


A vague picture of how it looks like after 3 hours of refluxing, if the color will get orange to dark orange before 6 hours you should stop refluxing at that time.

But don't reflux it longer than 6 hours, sometimes you only need 3 hours though.

To improve yield you can also add 1ml of glacial acetic acid to buffer the n-butylamine.

For every mole of benzaldehyde one mole of water will form and since SWIM used 17ml of benzaldehyde which is ($17\text{ml} \times 1,05\text{g/ml} = 17,85\text{g benzaldehyde} / 106,12\text{g/mol} =$) 0,17mol benzaldehyde. Thus 0,17 mol of water will form which is ($0,17 \times 18,01 =$) 3,03ml of water. So after the 6 hours or earlier you should have 3,03ml of water in your trap, it will likely be more since your toluene will contain a bit of water too in SWIMS reaction he got 4ml of water.



The color after 6 hours of refluxing and collecting 4ml of water in the trap. Now SWIM puts the round bottom flask in the freezer at -18°C because the P2NP is now dissolved in the toluene but when you will make the toluene very cold the P2NP won't dissolve in the toluene anymore or at least very bad.

7 hours later scratched the walls of the RB flask to initiate crystal forming. 10 hours later a lot of crystals in the RB flask,



The crop of crystals in the bottom of the flask.



Crystals formed on the sides.



Now quickly break up the mass with a glass stir rod or a thermometer and throw the mixture through a buchner filter (filtration under vacuum). The toluene will get back to room temperature and thus the crystals will dissolve back into the toluene that is why you need to be so quick and necessarily do need to use a buchner filter.



Just a picture of the buchner filter how it looks like.
Put the crystals on a paper to air-dry.

As you can see the crystals are yellow coloured so they need purification or else your next product will be crap and yellow too.
The first crop weighed 6,02 gram when completely dry.
The second crop was 8,01gram SWIM evaporated off the half of the liquid and put it back in the fridge.

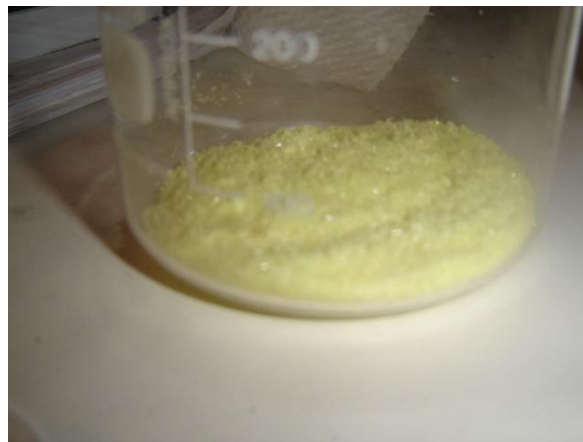


Evaporating off the half of the toluene.

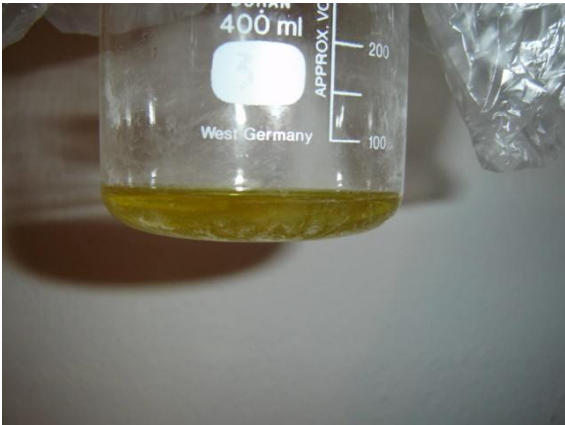


Toluene and water seperated.

Now the crystals were purified by dissolving them in a minimal amount of boiling isopropylalcohol which is standing in a beaker with water, when they have all dissolved put a watch glass over the top of the erlenmyer and let them cool to room temperature; then put them in the deep freezer and vacuum filtrate them over a buchner filter and let them dry again.



Beaker with IPA standing in beaker with water. On the right the P2NP crystals.



P2NP dissolved in boiling IPA.



Cooling down, you can see crystals forming.



Here you can see the crystals forming while cooling down, in the buchner filter the crystals were washed with water.

From the 14,03g of P2NP which SWIM started with 13,7g of P2NP was left over after the recrystallization/purification step.

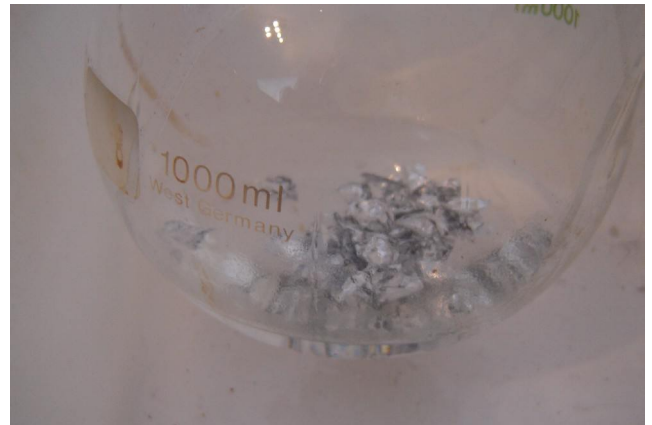
Now it is time (by the mean time the P2NP is dry again ;-)) to do the aminated reduction of the P2NP to amphetamine.freebase.

This was first tested with 3,5g of P2NP:

You will need 5g Al foil cut it into 1 (2,5cm) squares and then fold them up to nuggets and you should wrap them up so tight that you can only still press them a little with your fingers.



This is the reflux setup that SWIM will be using. had 70mg of HgCl_2 dissolved in it.



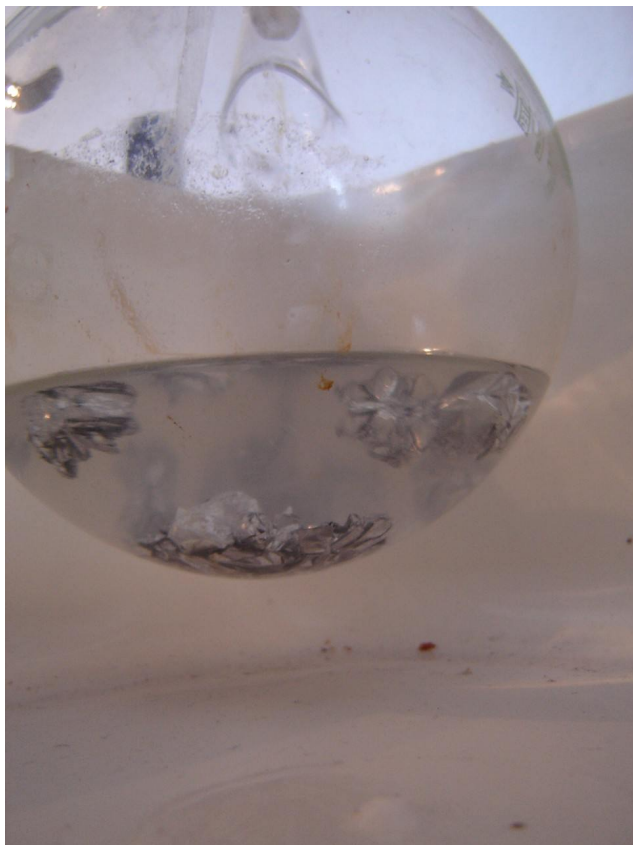
Close up and methanol added which

As sayd above at the picture you will need a solvent like methanol and you dissolve HgCl_2 inside the solvent and add it to the Aluminium make sure the aluminium is not too fine else a very heavy reaction will occur with the possibilty that mercury and hydrochloric acid, boiling methanol and hot aluminium will cover you like a vulcano.

The HgCl_2 can also be substituted by other Hg salts like $\text{Hg}(\text{NO}_3)_2$.

After 10min average the Al will start to evolve hydrogen gas, depending on thickness of the foil.

If you use thin foil wrap it up 3-4 layers before cutting squares out of it else it will react too heavily.

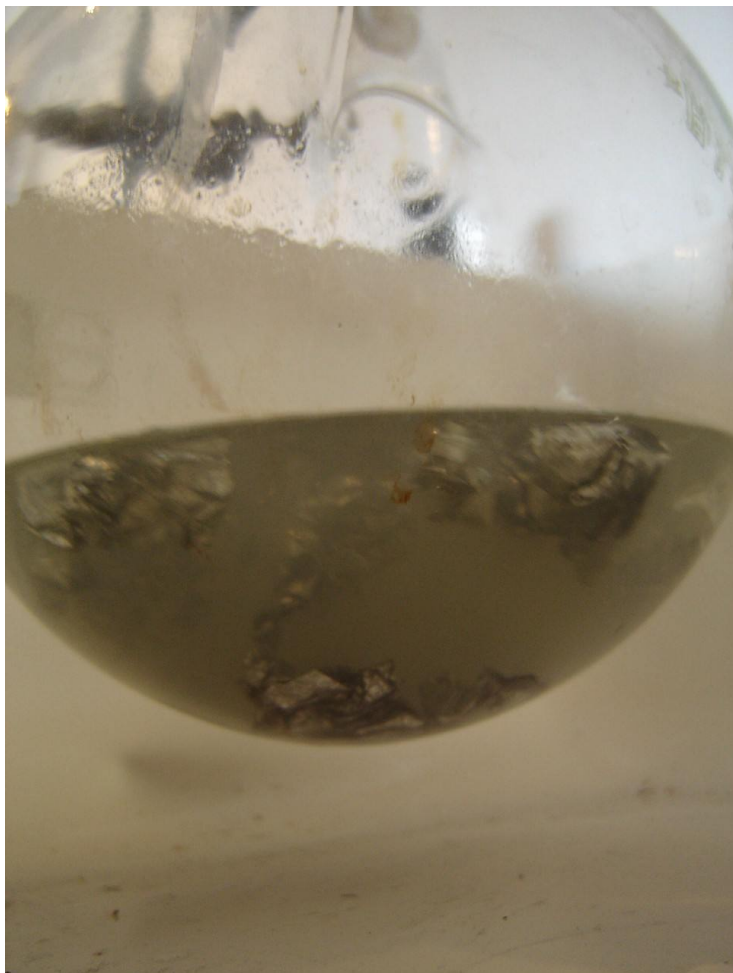


The methanol will become gray/blue, and the aluminium will become less shiny.

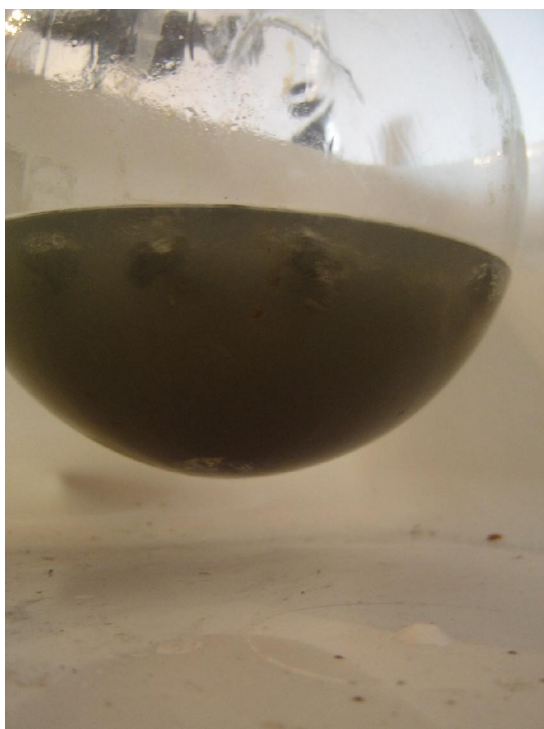
On for hand you should make a solution of your P2NP that will be added to the mixture later on.

The solvent used was isopropylalcohol and glacial acetic acid, in a ratio:
40ml IPA; 60ml GAA; 3,5g P2NP.

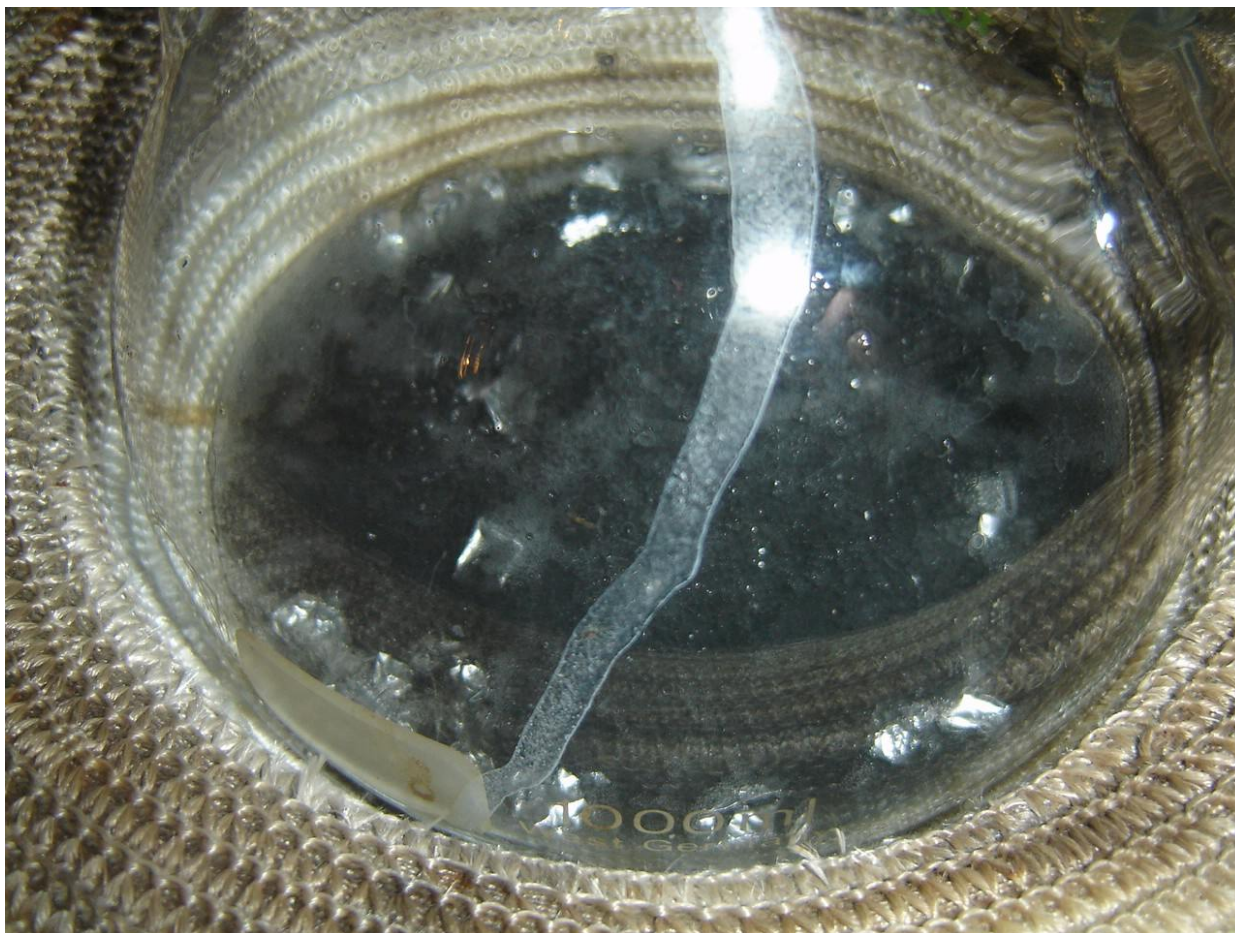
You might need to heat it up because the P2NP will not dissolve easily at room temperature.



The reaction will heat up and when there are dark spots on the Al and there is hydrogen gas bubbling of it you can add all the P2NP solution at once to the Al+ MeOH+ HgCl₂.



A little later in the progress, the mixture gets darker and heats up even more to a mild reflux.



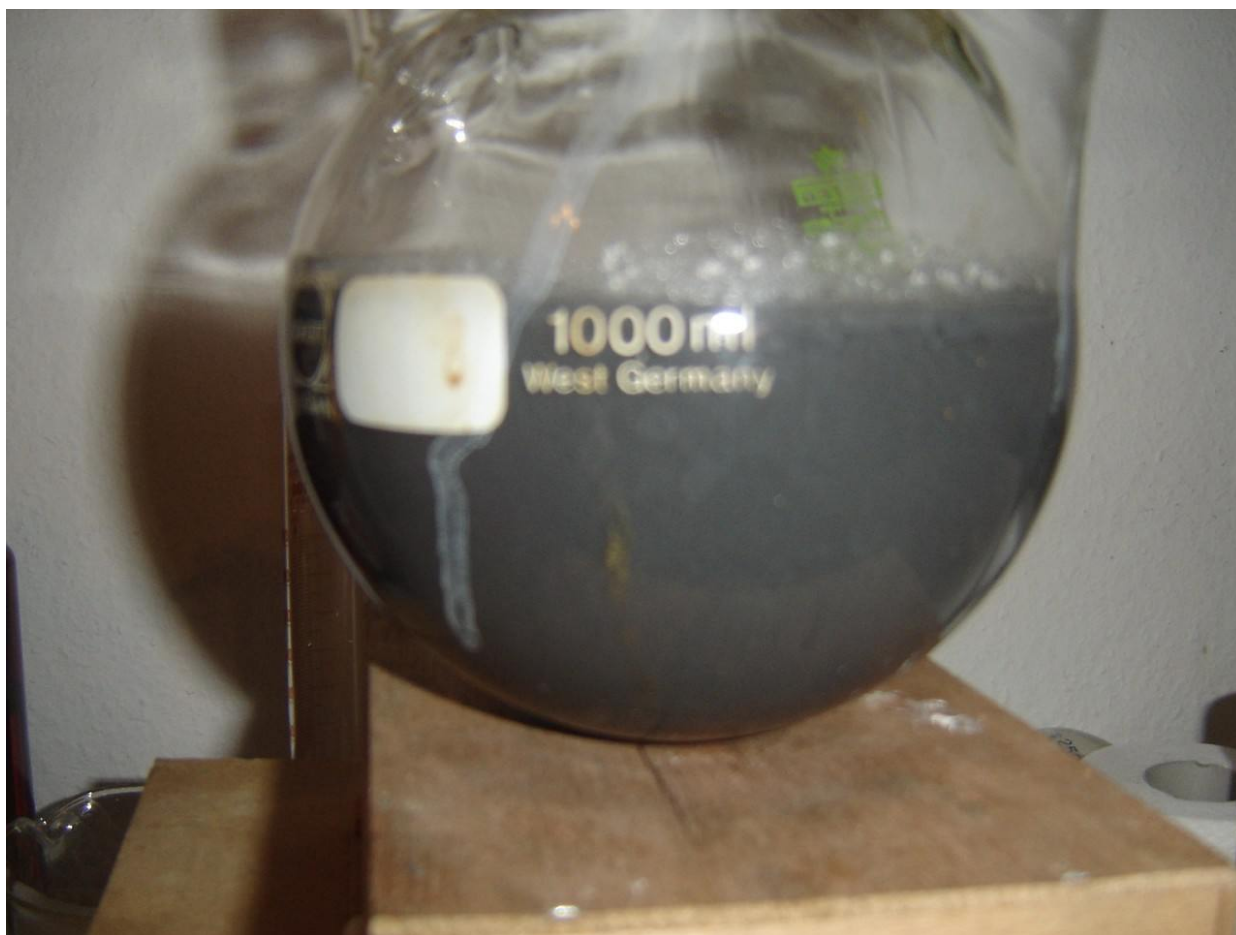
By now the mixture is dark and the aluminium is dissolving slowly, you can notice small shiny pieces of aluminium floating around, extra heat is applied here because the Al was so big that it reacted not fast enough, but hey better slow reaction than a volcano, right.



Now when almost all the Al has dissolved add 200ml of cold water to the mixture to cool it down.

Then basify it to pH 12 with 50g of NaOH dissolved in 150ml water.

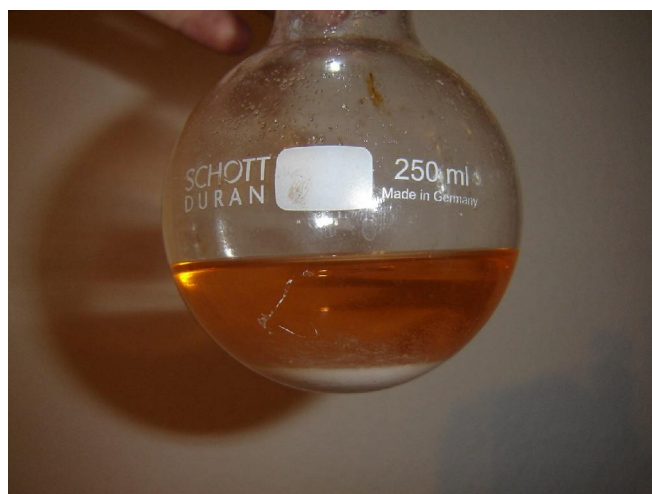
All the remaining Al will now react with the NaOH and form hydrogen gas and the mixture will heat up but not to a boil, that wouldn't matter though.



After an hour stirring there had been some foam and after an hour of stirring the mixture was still quite hot and all the Al dissolved now
Time to extract your product.
SWIM used ether since it has a very low boiling point so that comes in handy later on in the progress.



SWIM doesn't own a huge separatory funnel so he put the mixture in the separatory funnel in portions and added 30ml ether at a time approx. and the ether is the upper clear layer (your amphetamine.freebase is dissolved in the ether now).



After all the washed this was collected in total. Anhydrous magnesium sulfate was added to suck up the water in their molecules and thus drying the ether and your product.

Now you only have to wait 12 hours to let the $MgSO_4$ do its work, filtrate it and then you can let the ether evaporate off at room temperature or with the help of a warm lamp.

NOT AN OPEN FLAME!!!

Ether is very inflammable.

All the ether was evaporated off and 50ml anhydrous IPA was added to the amphetamine freebase.

Then to anhydrous IPA 98% sulfuric acid was added calculate how many mol amphetamine freebase you've got and then take 90% of that and calculate how many mols of H_2SO_4 that represents and add that to 15ml of anhydrous IPA.

Now drip H_2SO_4 +IPA mix into the freebase which is dissolved in the IPA.

Here you can see the crystals forming after a few drops have been added.





Glass full of crystals.
It wasn't that much though but it looks like it is full of crystals.



Filtrate it.



And finally pure amphetamine sulfate has been obtained this was around 180mg.
When only starting with 3,5g P2NP, when you lose 1g in the filter that is a lot, with bigger amounts yields are better anyway.