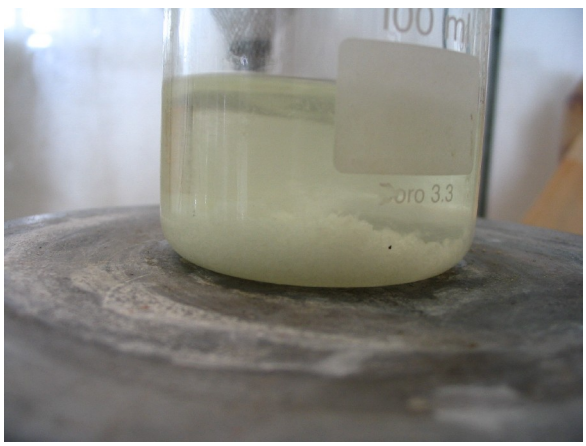
2C-B

Starting from 2,5-dimethoxybenzaldehyde.

First off all the coresponding nitrostyrene needs to be made.
2,5-Dimethoxynitrostyrene.

5,019g 2,5-dimethoxybenzaldehyde and 0,54g ethylenediaminediacetate (EDDA) is poured into ~24ml isopropylalcohol, with gentle heating (~45°C) and stirring it is dissolved in a beaker of 100ml.



Then when everything has dissolved after 5-10min approx., 1,96ml nitromethane is added to the mixture, the mixture turns yellow.
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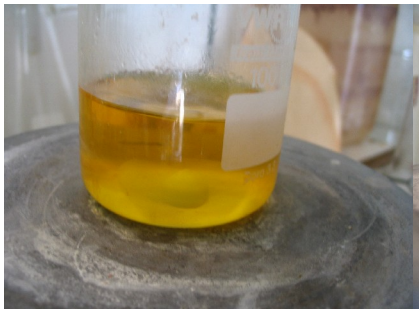

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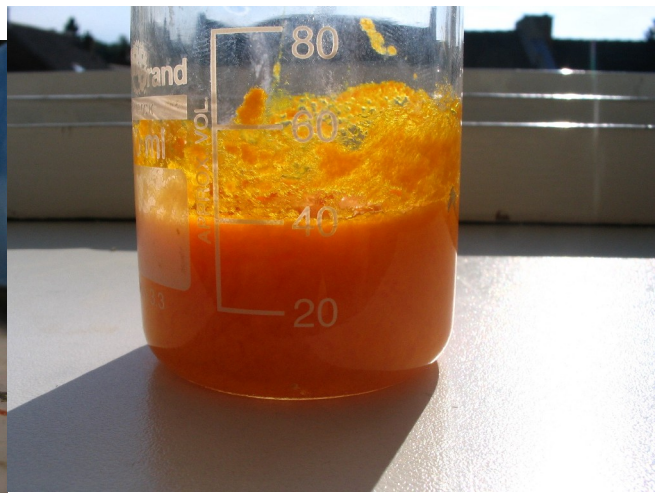
Here you can see pictures taken every 10minutes.

17:02


18:00

Stirring was continued for one hour from now on, the mixture turned a deep orange during the hour and suddenly after 50minutes the pumpkin orange crystals preticipated out. From then on stirring was discontinued and the mixture was left to stand for 48 hours at room temperature.

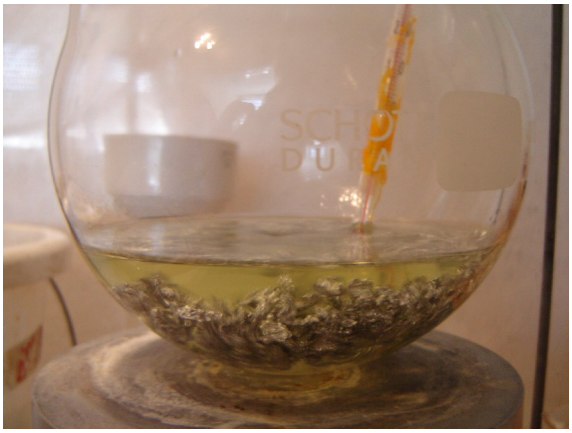
The orange crystalline mass was broken up with a glass stiring rod some water was added since it was really thick and then vacuum filtered until no more liquid came threw the buchner, the crystals were then washed with ice-cold isopropylalcohol (10ml), then sucked as dry as possible in the buchner.

The crystals were put in a dessiscator, until they are dry and crispy. The total mass weighed 5.56g (26.56 mmol, 88.29% yield).

2C-H

The next step is to reduce the nitrogroup on the 2,5-DMNS to an amine group.
Also the double bond needs to be reduced to an single bond, this can be done with an reducing agent like LiAlH4 or NaBH4 but since these are quite hard to get and dangerous to handle with, SWIM'll use Al/Hg.

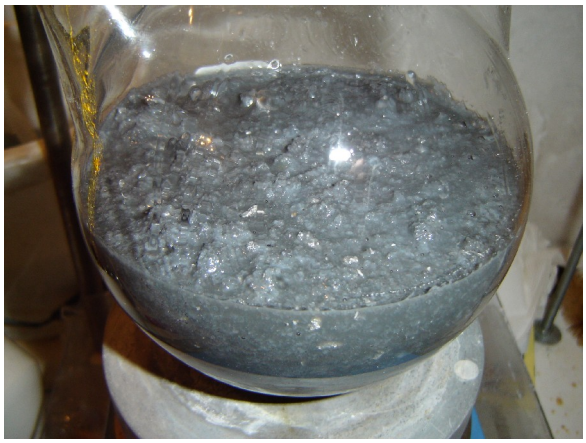
Normally Al has a thin coating of it's oxide Al_2O_3 but when you remove that coating Al is very reactive, so when you add a mercury salt to Al in an solvent like MeOH or EtOH or IPA, the mercury will stick on the surface and prevent new Al_2O_3 to form.
I'll spare you the rest of the story about the reaction, here's what one should do to reduce the nitrostyrene:
Make twice as much Al shreddings as the amount of nitrostyrene you have, in SWIM's case he has got 5g of 2,5-DMNS so he takes 10g of Aluminium foil and puts it in the mixer 3g each time and grinds it for 5-10 seconds.
The shreds will then look as below in the yellow dish. On the right it is seen under MeOH.
Now one needs to add 40mg of $HgCl_2$ solution dissolved in 40ml of water and 40ml MeOH.
After 10-15minutes the aluminium will start to release little bubbles.

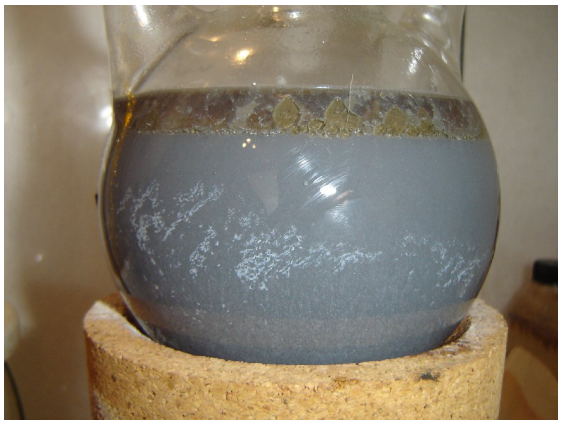
When the bubbles are there add all the nitrostyrene in there all at once it does need to be dissolved in 100ml glacial acetic acid (99-100% acetic acid) and 80ml of isopropylalcohol, you need to heat it to 80°C or so before everything dissolves, it dissolves quite hard.

The flask will heat up quite much, try to get it to a steady reflux by applying heat or using an icebath to cool it down, when it stops reacting one could add more $HgCl_2$ like 20mg.

This is how it looks like after the reaction is finished



Now the remaining aluminium needs to be destroyed, this is done by adding 20% NaOH solution, another vigorous reaction which makes the mixture boil.

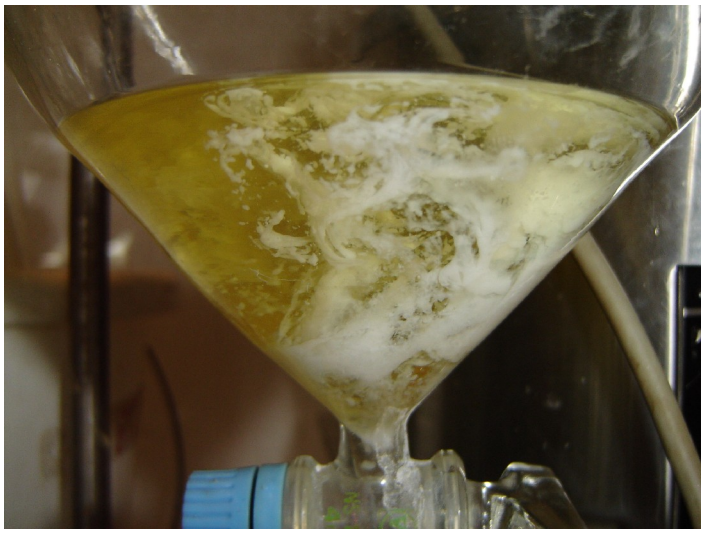


Added 100ml toluene to the mixture and put the magnetic stirrer on maximum, on the right the toluene layer in the seperatory funnel.

Now the toluene has the freebase inside (2C-H).
And as you can see it is not light yellow or clear but red/brown, so an acid/base purification is done.
Now 30% sulfuric acid is added to the toluene.

This happens 2C-H.SO4 crystalizes out but also disapears quickly into the water again.
Now shake very hard so everything becomes the sulfate salt, then add DCM which doesn't mix with water this takes up every crap that shouldn't be in there, now the DCM turns red and repeat this (and throw away the DCM) until the DCM washes remain clear.
Below here a dirty DCM wash can be seen.

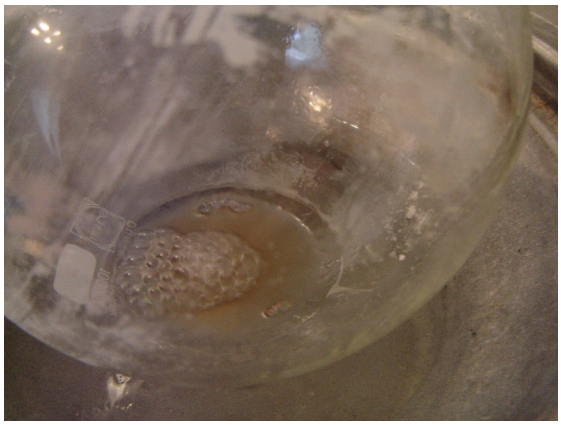
So this is the water layer remaining, which contains relatively pure freebase now, now the freebase has to be unleashed again, this can be done by adding 20% NaOH to this mixture but be carefull only add small amounts until the mixture remains white THEN add toluene to extract the freebase (the white stuff is an emulsion of water and oil (freebase)).
Anyway then after all the freebase went into the toluene more NaOH can be added…



White emulsion, if you would wait quite long you could actually see the white stuff making droplets of oil and floating to the top and forming a top layer of very pure freebase.

Combined the extracts of toluene (since the mixture is extracted 2 times), now this is toluene containing 2C-H freebase, notice the difference in color, slightly yellow now and before dark red/brown.

Then dry the toluene with MgSO4 (anhydrous) for 12 hours and then distill/evaporate/rotavap off the toluene.

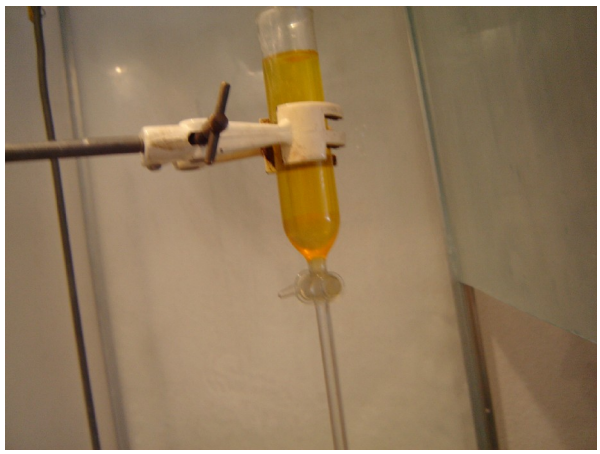


Pure freebase left in the RBF in the rotavap, it already is a bit brown because of the CO2 in the air it really quickly forms 2C-H.CO3 which doesn't matter actualy.

Bromination

Now this 2C-H has to be brominated to yield 4-bromo-2,5-dimethoxyphenetylamine.
The bromine is made in situ and dissolved in DCM.
2,4g of KBr is dissolved in 30ml dH2O and to that 16ml 35% H2SO4 is added to that 8ml of DCM is added and then finally 0,97ml of H2O2 30% concentration is added in portions.

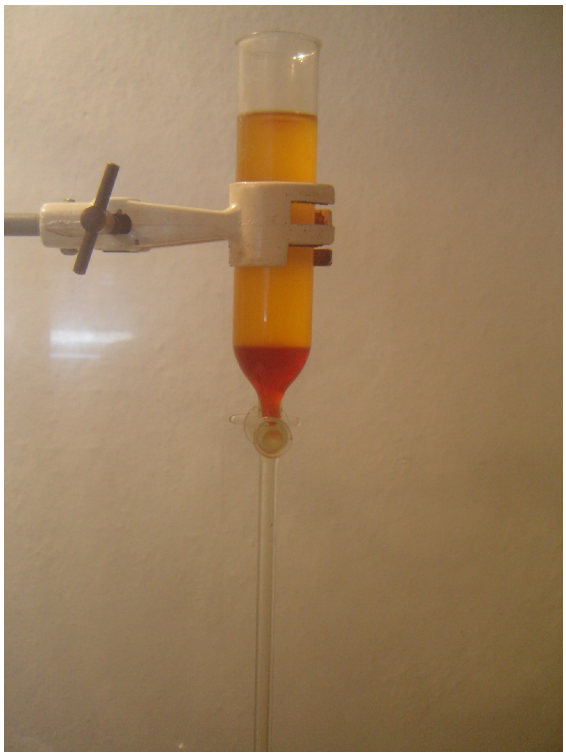
KBr and water



A few drops of H2O2 30% added.

The DCM phase absorbing the bromine.

The DCM layer is added to the freebase in an icebath, the freebase is first dissolved in the same volume of glacial acetic acid.



Overall view of the bromine production.

Now one has formed 4-bromo-2,5-dimethoxyphenetylamine.HBr most of the people want to convert this to the hydrochloride salt, so then first the formed crystals (after 15min light brown crystals formed), are filtrated and washed with ether and ice cold acetic acid to get rid of the bromine excess.
Then NaOH is added to get 4-bromo-2,5-dimethoxyphenetylamine freebase then again this is extracted with toluene, toluene evaporated and a few drops of acetic acid 99% and a few drops of water are added to the pure 4-bromo-2,5-dimethoxyphenetylamine freebase, and then HCl 37% is dripped in now immediatelly or after hard stirring white needle like crystals form. This is the 4-bromo-2,5-dimethoxyphenetylamine.HCl but first put it in the freezer at –20°C and then you will get something like the above picture little crystals captured in the ice, now put the chunk of ice in the buchner filter and let it vacuum filtrate while the ice melts.
And that's actual 2C-B.HCl

Have fun and be safe, Ice.