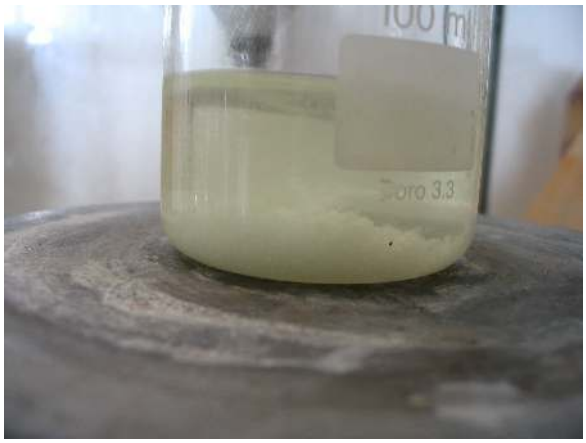
2C-B

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.

16:08

16:18

16:28

16:38

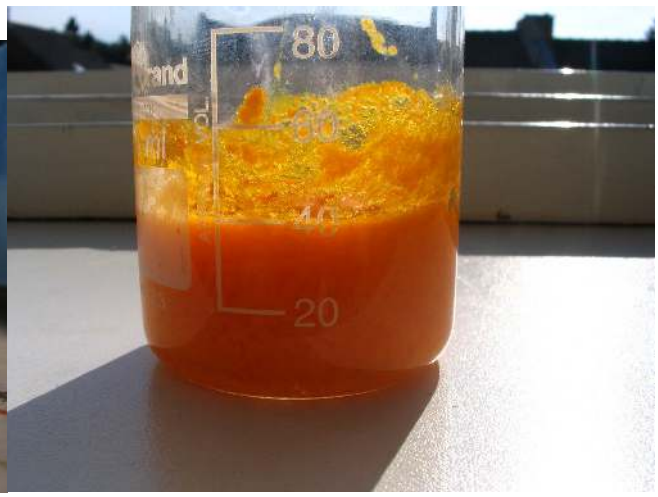
16:48

16:58

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.

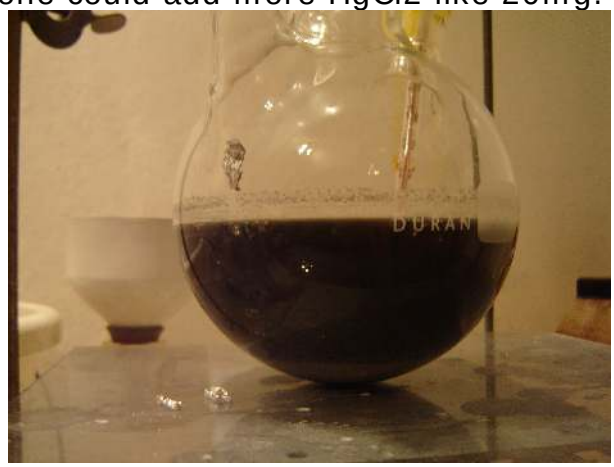
The crystals were put in a dessiscator, until they are dry and crispy. The total mass weighed 5.56g (26.56 mmol, 88.29% yield).

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 Al2O3 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 Al2O3 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 HgCl2 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 HgCl2 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.

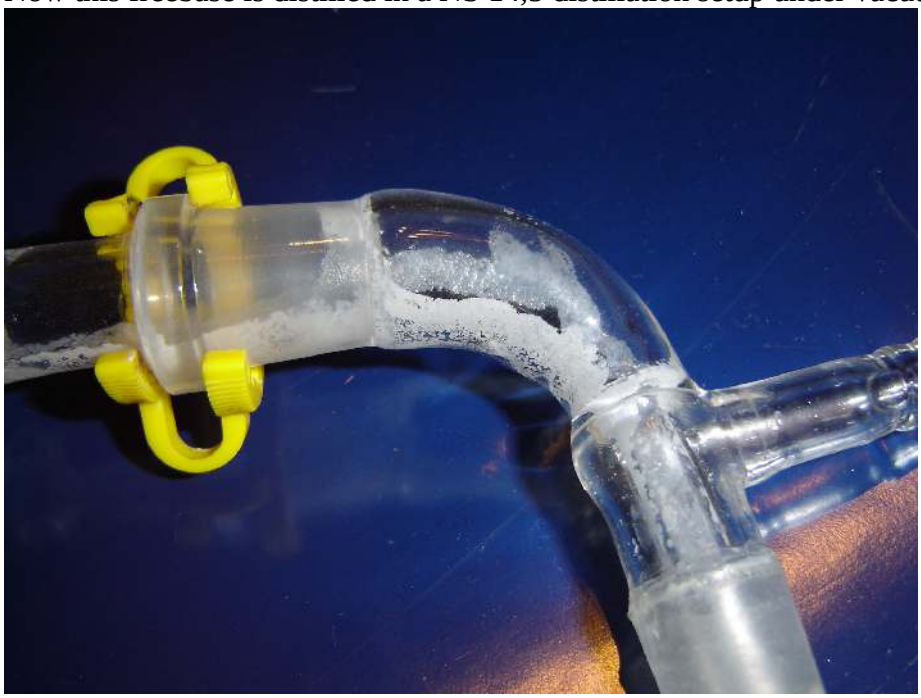


Added 100ml toluene to the mixture and put the magnetic stirrer on maximum, on the right the toluene layer in the seperatory funnel.

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


Impure 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.

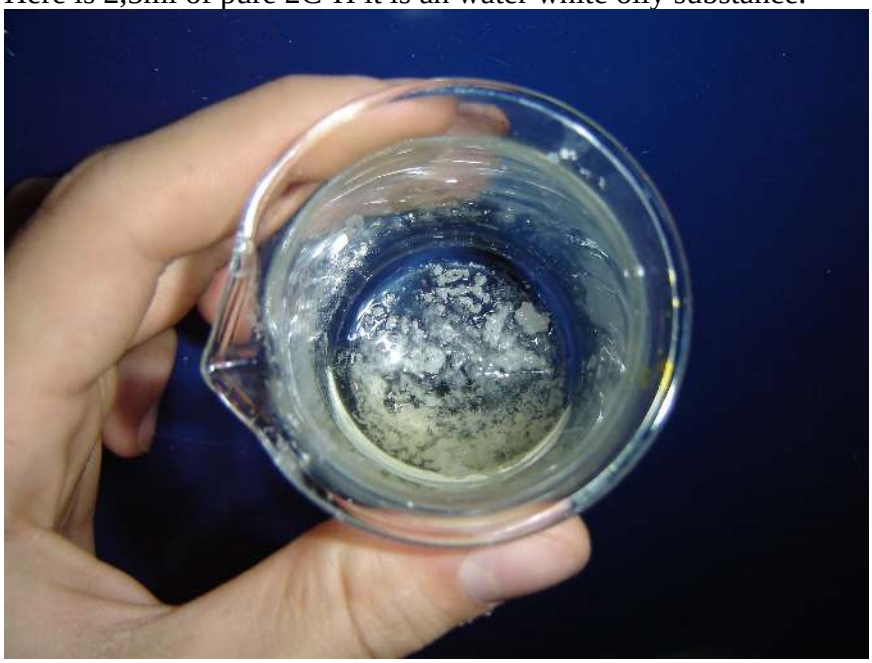
Now this freebase is distilled in a NS 14,5 distillation setup under vacuum.


Notice after the distillation that there is a white solid this is 2C-H.CO3

Bromination

Now this 2C-H has to be brominated to yield 4-bromo-2,5-dimethoxyphenetylamine.

Here is 2,5ml of pure 2C-H it is an water white oily substance.



Now the 2C-H needs to be dissolved in glacial acetic acid (2,5ml 2C-H needs 4ml glacial acetic acid) , this produces A LOT of heat and a white preticipate which is 2C-H.Ac.
After some stirring this dissolves, then bromine is taken out of the freezer and measured out. MIND that bromine has a density of 3,1111g/ml so if one needs 3,1g then 1 ml is measured out. 2,5g bromine is measured out. (For the people who pay close attention to the picture

4,5ml is measured out this is because the bromine is dissolved in 2ml glacial acetic acid.

2,5g (0,8ml) bromine in ~4ml glacial acetic acid.

2,5g 2C-H dissolved 2,5ml glacial acetic acid.

2C-H dissolved in glacial acetic acid mixed with 2,5g bromine dissolved in glacial acetic acid. This is immediately after mixing the two, some heat and some hydrobromic gas is evolved, the beaker is covered with Al foil to keep the fumes inside.


After approximately 1minute there was the crystalization of 2C-B.HBr
After filtration it was washed twice with cold glacial acetic acid and twice with ether.
Then recrystalized from isopropylalcohol and gave off-white a bit pink crystals.
The glacial acetic washes and ether washes are important since they remove excess bromine and by products from bromine which are toxic.

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 are mixed with NaOH 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.