

# 2C-B

Starting from 2,5-dimethoxybenzaldehyde.

First off all the corresponding 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 10 minutes.

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 50 minutes the pumpkin orange crystals precipitated 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 stirring rod some water was added since it was really thick and then vacuum filtered until no more liquid came through the Buchner, the crystals were then washed with ice-cold isopropyl alcohol (10 ml), then sucked as dry as possible in the Buchner.



The crystals were put in a desiccator, until they are dry and crispy. The total mass weighed 5.56 g (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 a single bond, this can be done with a reducing agent like  $\text{LiAlH}_4$  or  $\text{NaBH}_4$  but since these are quite hard to get and dangerous to handle with, SWIM'll use  $\text{Al}/\text{Hg}$ .

Normally  $\text{Al}$  has a thin coating of its oxide  $\text{Al}_2\text{O}_3$  but when you remove that coating  $\text{Al}$  is very reactive, so when you add a mercury salt to  $\text{Al}$  in a solvent like  $\text{MeOH}$  or  $\text{EtOH}$  or  $\text{IPA}$ , the mercury will stick on the surface and prevent new  $\text{Al}_2\text{O}_3$  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  $\text{Al}$  shreadings 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  $\text{MeOH}$ . Now one needs to add 40mg of  $\text{HgCl}_2$  solution dissolved in 40ml of water and 40ml  $\text{MeOH}$ . 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  $80^\circ\text{C}$  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  $\text{HgCl}_2$  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%  $\text{NaOH}$  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 separatory funnel.

Then dry the toluene with  $\text{MgSO}_4$  (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  $\text{CO}_2$  in the air it really quickly forms  $2\text{C-H.CO}_3$  which doesn't matter actually.

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



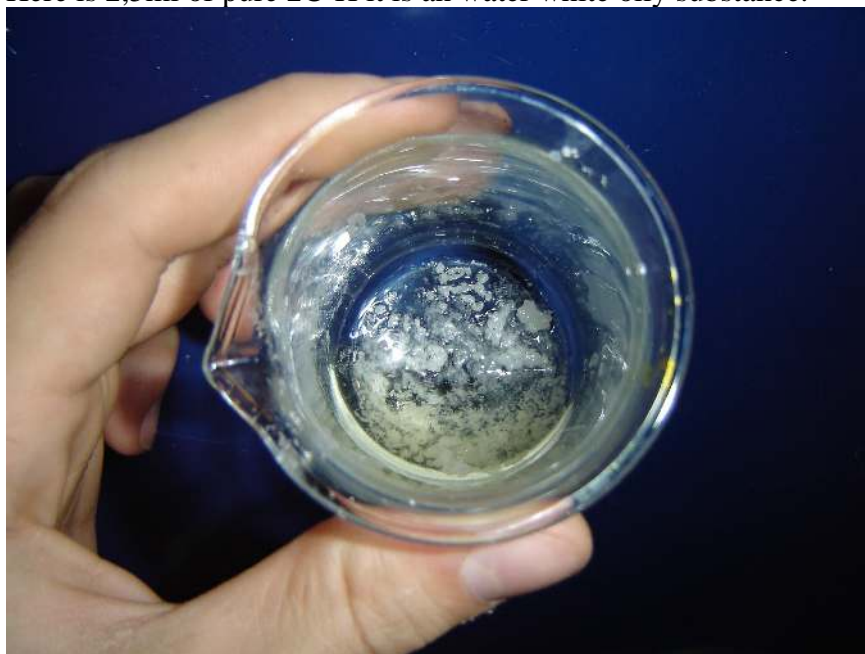
Notice after the distillation that there is a white solid this is  $2\text{C-H.CO}_3$

## Bromination

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



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 precipitate 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.

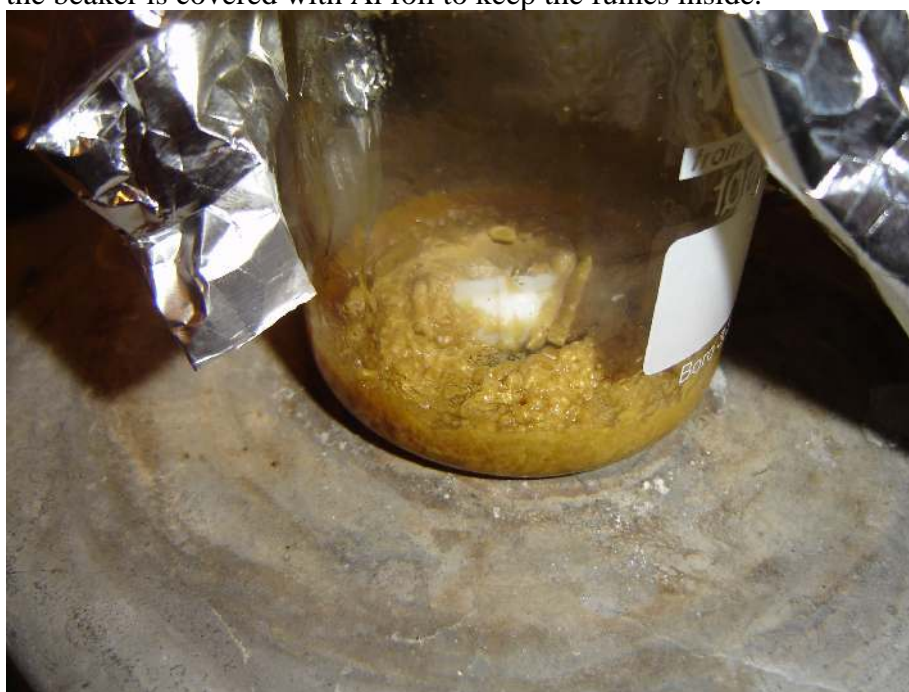


2,5g 2C-H dissolved 2,5ml glacial acetic acid.





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 1 minute there was the crystallization of 2C-B.HBr. After filtration it was washed twice with cold glacial acetic acid and twice with ether. Then recrystallized 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-dimethoxyphenethylamine.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-dimethoxyphenethylamine 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-dimethoxyphenethylamine freebase, and then HCl 37% is dripped in now immediately or after hard stirring white needle like crystals form.

This is the 4-bromo-2,5-dimethoxyphenethylamine.HCl but first put it in the freezer at  $-20^{\circ}\text{C}$  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.