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So my friends, this will be SWIMethLab's touching-up for several personal reasons. That does not mean that SWIMethLab will not be around to check what's up with the kidz.



This work deals with the solvent extraction of fortified ephedra extract and following reduction to methamphetamine hydrochloride via 57% w/w hydriodic acid p.a. and 50% w/w hypophosphorous acid (H₃PO₂) as recycling agent in a long wet reflux session.

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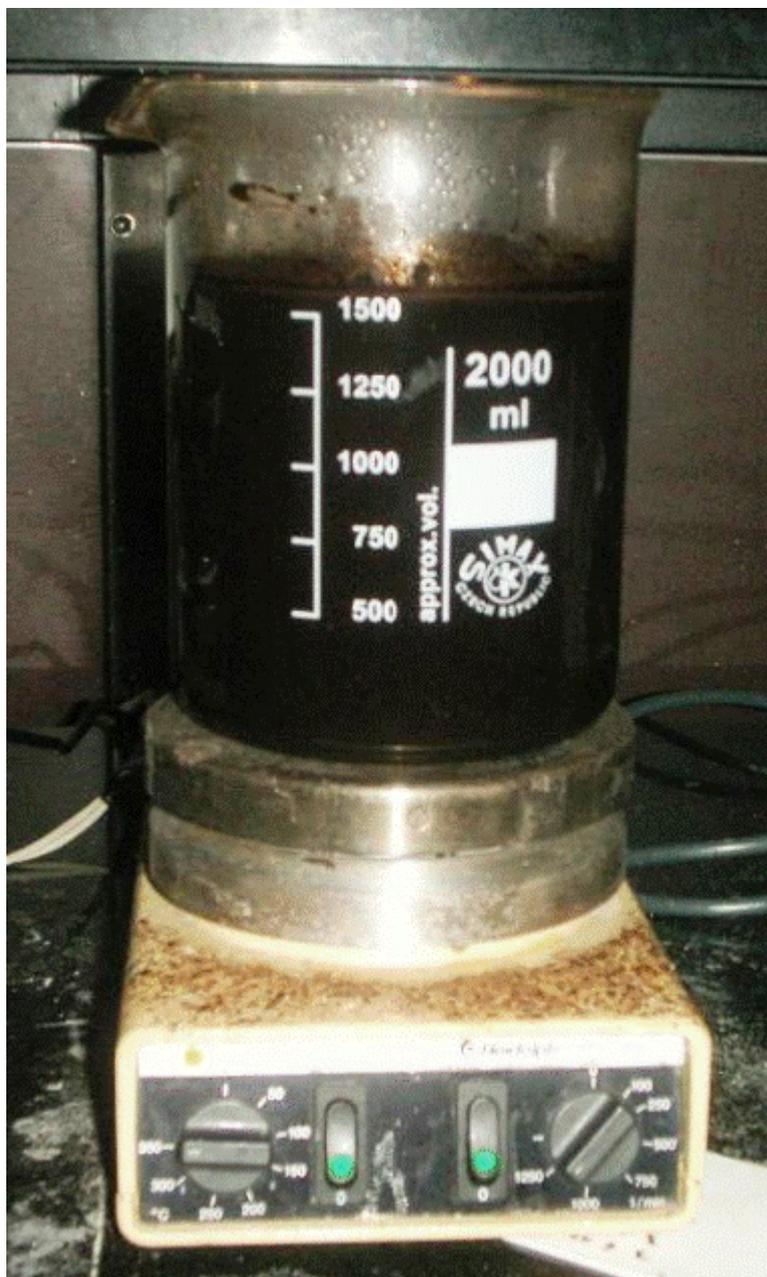
Let's start with the preparation of the alkaline aqueous solution for the extraction. For the extraction of 200g ephedra (which is said to contain 10% alkaloids) there has been prepared a solution of 1200-1250ml water which contain:

- 180g NaOH p.a.
- 60-70g Na₂CO₃
- 100-120g NaCl

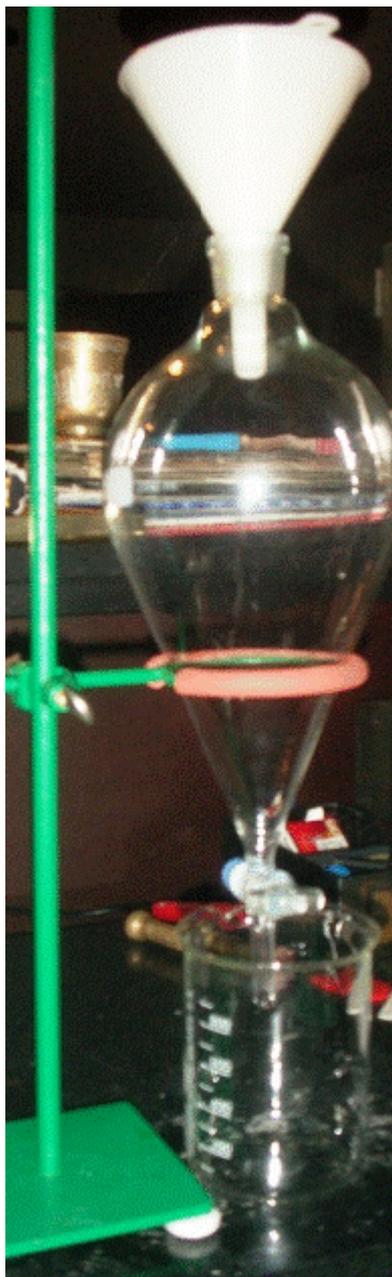
The following pictures show the preparation of this solution on the hotplate with good magnetic stirring:



To this solution 200g of herbal ephedra extract have been added in smaller portions under magnetic stirring. Of course, after the aqueous solution has cleared up:



This mixture has been stirred until it cooled down to room temperature while a separatory funnel of appropriate size has been set up:



Afterwards the ugly mixture has been transferred into the separatory funnel and 250ml of TCM aka chloroform has been added.

Side Note for those having actually no access to chloroform:

"The first industrial process was the reaction of acetone (or ethanol) with sodium hypochlorite or calcium hypochlorite. The chloroform can be removed from the resulting sodium acetate or calcium acetate (or sodium formate or calcium formate if ethanol is the starting material) by distillation. The reaction mechanism is called haloform reaction, and is still used for the production of bromoform and iodoform."
(<http://en.wikipedia.org/wiki/Chloroform>)

Looks like this:



Afterwards the aqueous solution has been extracted twice with TCM. The second pull used 150ml chloroform and the third on additional 100ml. One should think about recycling the solvent after the whole procedure with the help of distillation.

But you need to have patience for this extraction process because it lasts a while until the layers separated totally after shaking the solution not too gentle. This shaking is necessary because freebase ephedrine tends to dissolve in water well.

After all three pulls are finished they should be combined in the sep. funnel again.



Here you see the situation as it looks like after about 60-70ml of a 5-6% w/w Na_2CO_3 solution has been added. This solution is very effective in washing out some of the plant trash. So shake the shit like your life depends on it and don't forget the bottom layer is what you want. This step is going to be repeated once more. You will probably see something like this:



So at this time there should be at least three washes with distilled water each with about 50ml.

After those washes make sure to separate the non polar layer as good as possible from the aqueous layer and put it into a beaker. To this solution one has to add a little bit of dry K_2CO_3 or $MgSO_4$ as drying agent. But the first one is preferred for amines. After you mixed it a little bit you should notice that the solution becomes clear. Good. So wait about 15-20 minutes before you filter this solution by the way of choice.

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The now dry non polar solution is transferred into a beaker or something similar. While the stuff dried you should set up a HCl gas generator of your choice. For SWIMethLab an apparatus working with H_2SO_4 is the first choice. But also HCl with $CaCl_2$ should do the job.

This could look like this:

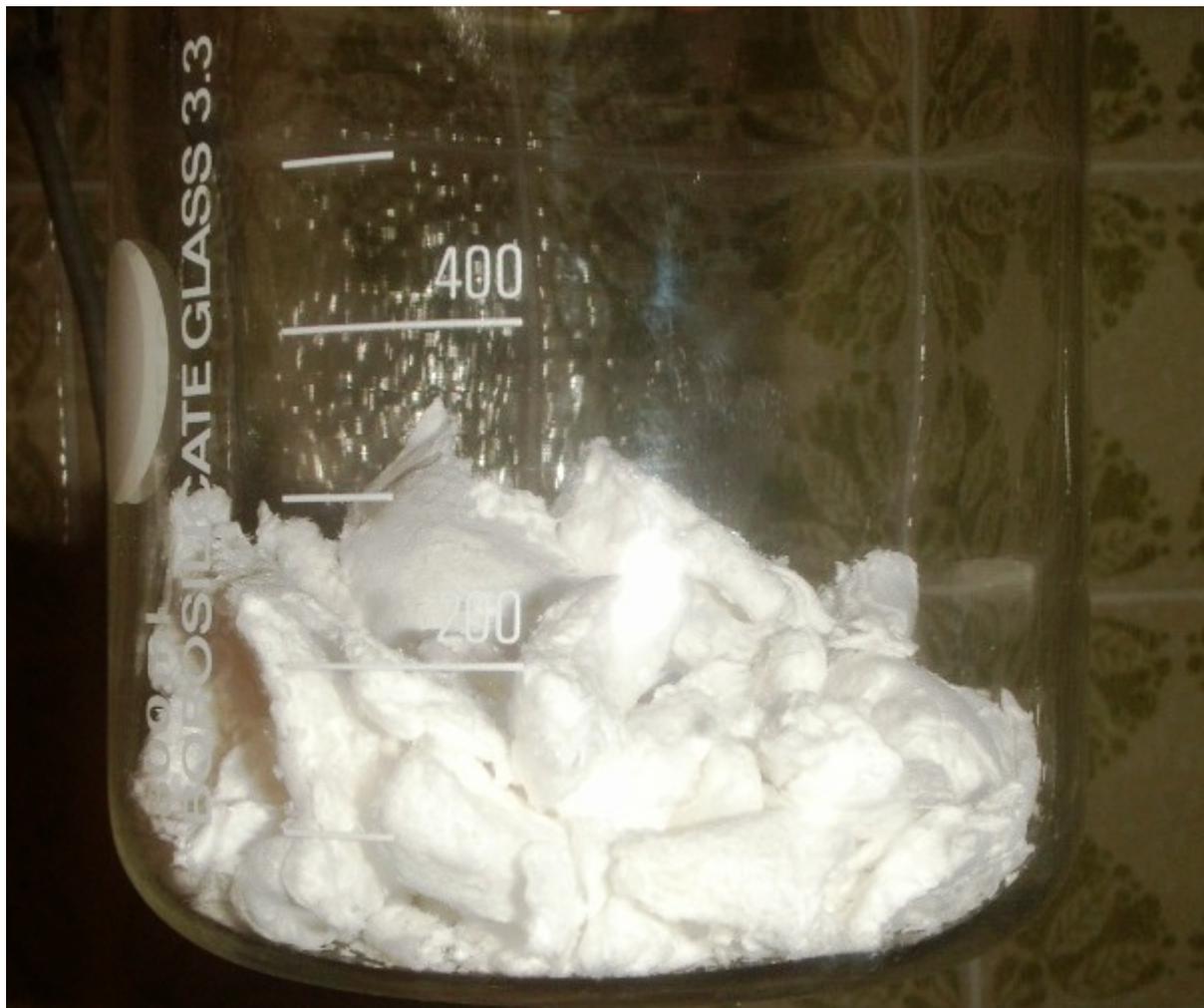


And after one gassed it could look like this:



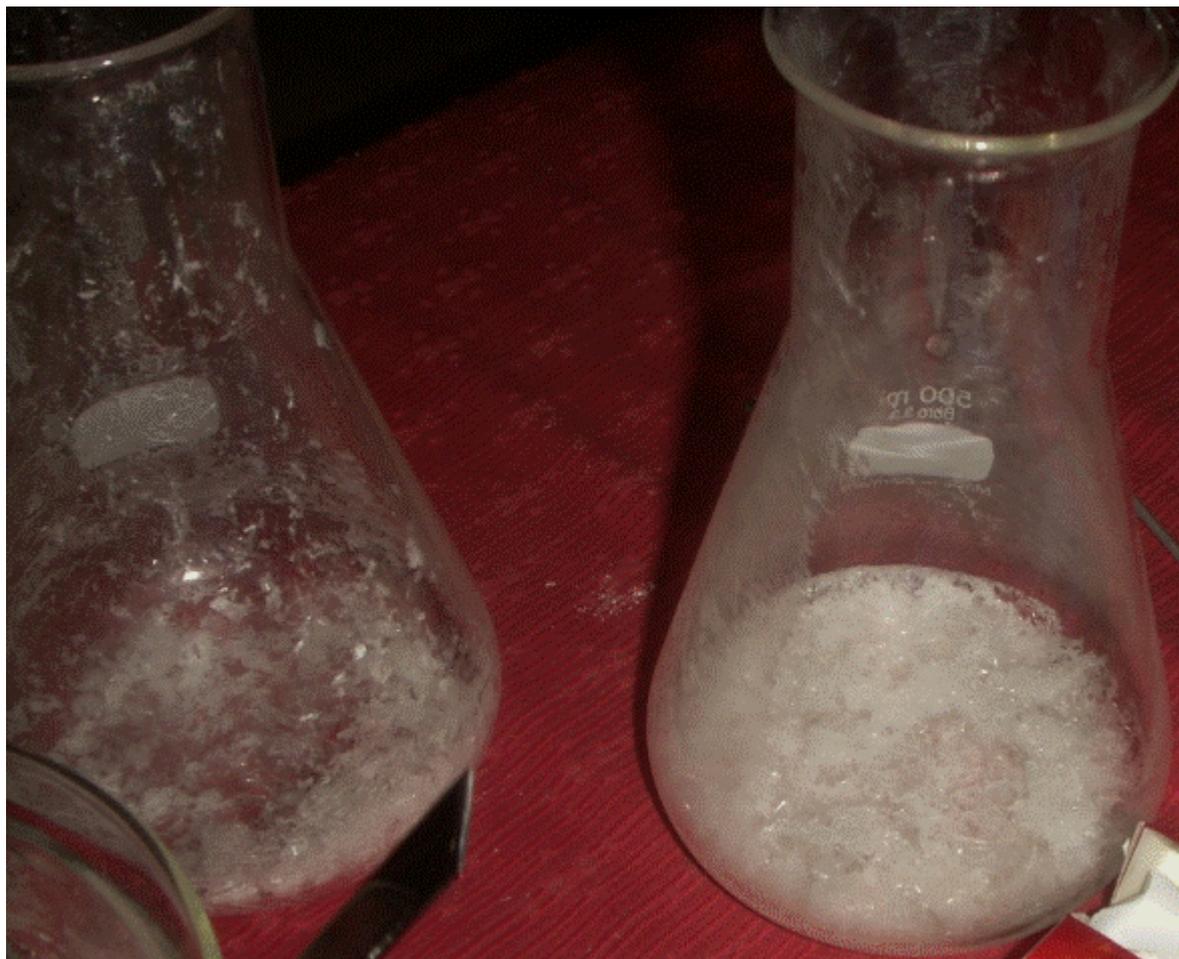
So now it is time to filter again by vakuüm as far as it is available. After the filtration by

suction with a little bit more than one 200g extract one gets something like this:



Ah and don't forget to wash the now extracted alkaloids with fresh & dry acetone while it is still in the Buchner.

This stuff should be recrystallized at least one time in methanol and acetone or ethanol & tone. Afterwards one will see something like this:

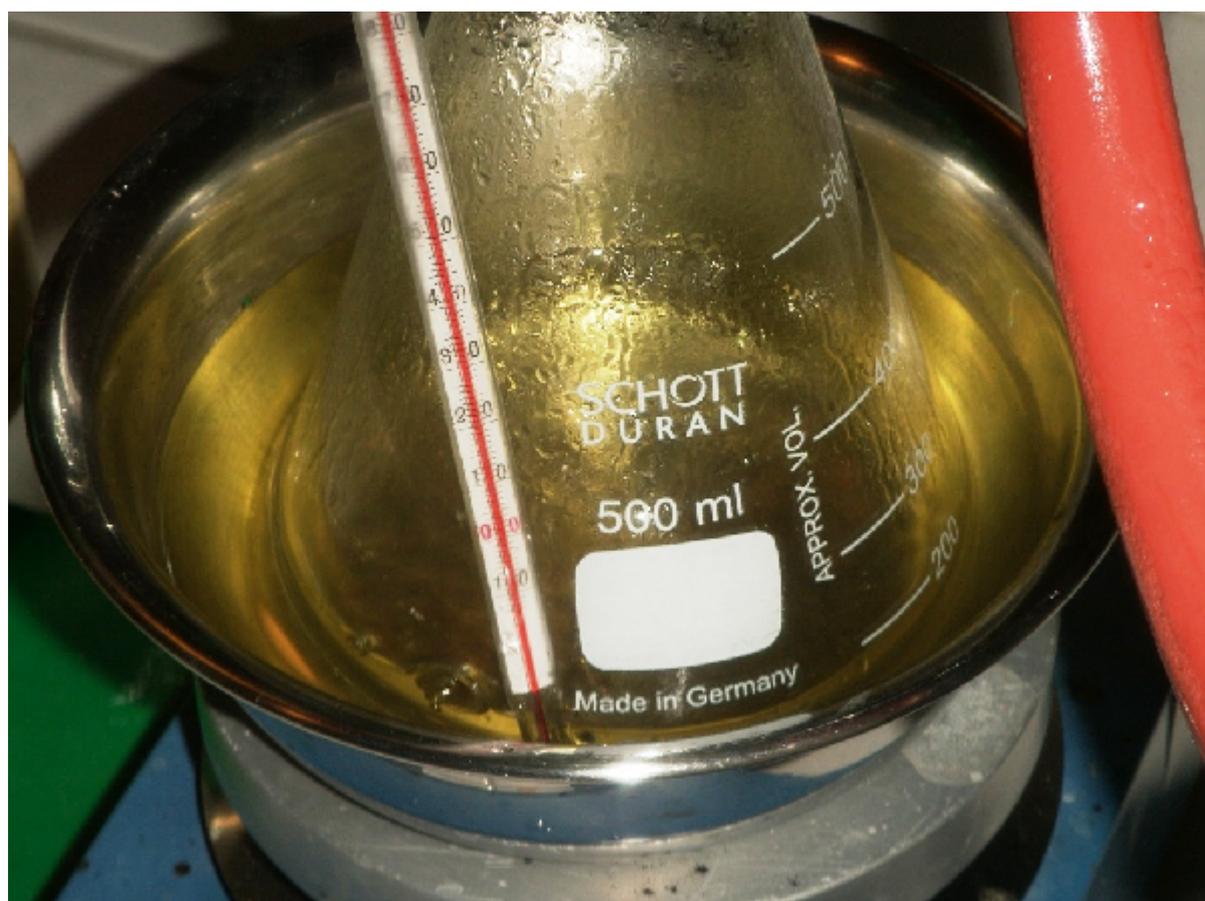


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After a few days of work one ends up perhaps with 45g of ephedrine hydrochloride which cries for reduction. Hmm... So after a few calculations with the Hi spreadsheet which is mentioned in several threads here at the board, one figured out that it is a good idea to use about 90ml of 57% w/w HI and 36,9ml of 50% w/w H₃PO₂ to react that shit in an apparatus that looks like this:



Or a little bit closer:



This is allowed to run at about 120-125°C for 24-36 hours after which this thread is to be continued!

So far, have fun...