Wed, Mar. 10th, 2010, 02:27 am
Easy, High Yield MDA

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Exerpt from something I heard somewhere.....

The Finkelstein reaction is the word for the day! Bromosafrole in acetone treated with KI to form the iodide (iodine is a much better leaving group). Iodosafrole forms the amine with NH3 alone at normal pressure. Start to finish MDA in the +90%!

Or, as per twodogs:

Quote:The following reactions make use of a commercially available product that goes under various trade names eg Helional, Floramelon.

The aldoxime of this aldehyde undergoes the Beckman rearrangement with Nickel Acetate to give the amide that is then easily transformed to the amine by the Hoffman reaction. The Hoffman reaction can be achieved with 4.2% NaOCl sold in supermarkets as bleach.
I don't have any references as this was done a while ago. The aldoxime and Hoffman reactions are pretty standard. The Beckman rearrangement of this particular product using Nickel Acetate is my own work.
It doesn't get any easier that this.

The aldoxime.
*100 grms of the aldehyde is placed in 200 mls of ETOH with 50-60 grms of Hydroxylamine Hydrochloride * in 100 mls of H2O. 40 grms of Sodium Carbonate in 100 mls of H2O is slowly added with stirring. Once addition is complete the mixture is stirred overnight then filtered to give about 100 grms of the aldoxime. If the aldoxime doesn't solidify add more hydroxylamine and base.*

The amide.
*100 grms of the aldoxime is mixed with 300 mls of xylene and 2 grms of Nickel Acetate ** is added and refluxed for 5 hours. About 100 mls of xylene is distilled off and the mixture is left to cool. About 70 grms of the amide separates and is filtered and dried,. Can be recrystallized with xylene.*

The amine.
To a solution of 10 grms NaOH in 200 mls H2O in a 500 mls flask is added 20 grms of the amide and 250 grms of 4.2 % NaOCl . The

mixture is then heated to 80C with stirring. At about 50 to 60C the mixture can become white as the carbonate forms. At 70 – 80C an oil separates. (This is crude MDA). The solution was cooled and extracted with xylene. The solution was filtered and the filtrate washed with xylene and the xylene portions added together. About 100 mls of conc HCL was added to the xylene shaken and separated. The aqueous solution was extracted with xylene and the xylene separated. The amine was then liberated from the aqueous portion with NaOH and worked up in the usual way to give MDA

** Hydroxylamine HCL*
200 mls nitromethane, 300 mls Acetic acid and 300 mls HCL are heated to just below reflux and held there for 2 hours then refluxed for 12 hours. The solution is reduced to half gone the cooled in fridge overnight and filtered.

***Nickel Acetate*
20 grms of nickel oxide (black) is dissolved in Acetic acid and heated with stirring until the mixture turns pale green. The excess Acetic is drained off , the rest is dried and powdered.

Making Hydroxylamine HCl
http://psychonaut.com/post-35899.html?f=43

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Thu, Sep. 19th, 2013, 05:26 pm
***(Anonymous)*: 3 Stupid questions**

1) You said the amine was worked up in the usual way to give MDA: Can you clarify what that way might be?

2) I have been checking some thread on this method and they mentioned bleach and lye which are not found here. Have you tried this recipe yourself?

3) Please clarify this portion, i am confused with all the xylene extractions and additions:
"The solution was cooled and extracted with xylene. The solution was filtered and the filtrate washed with xylene and the xylene portions added together. About 100 mls of conc HCL was added to the xylene shaken and separated. The aqueous solution was extracted with xylene and the xylene separated."

Otherwise very good post. :)

Link Thread - Reply

Wed, Oct. 9th, 2013, 01:48 am
***(Anonymous)*: Re: 3 stupid questions**

Hi buddy, have heard alittle about this from threads that hve gone around.. was wondering if this actually is a true receipe? Have you tried doing this?

Cheers

Link Parent - Thread - Reply

Sun, May. 18th, 2014, 02:03 am
***(Anonymous)*: Re: 3 stupid questions**

Yes it works. I'm having trouble with separating the salt from the mda in the end? I don't understand the same thing as work up as usual? Can someone explain? I tested what I do have, black and smokes instantly.

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