The Vespiary

Main Topics => Drug Synthesis & Extraction => Topic started by: Oerlikon on December 01, 2017, 12:29:44 AM

Title: **P2NP-derivation to the alternative approach!**
Post by: **Oerlikon** on **December 01, 2017, 12:29:44 AM**

After a long time of mastering MDP2P to MDMA and MDA reduction using Al/Hg
I decided to try some Al/Hg reduction again. This time with P2NP!

Now I have benzaldehyde and I can get nitroethane but unfortunately I don't have
any amine/catalyst apart from **Ammonium Acetate** AND **o-Toluidine**
https://erowid.org/archive/rhodium/chemistry/phenyl-2-nitropropene.html
I also have some acetone and hydroxylamine.HCl from which I can make some amine to act as an catalyst,or even bubble dry ammonia slowly trough Benzaldehyde+nitroethane slowly if that would work!

So,my first question is,should I try to use some **o-Toluidine** as basic/amine catalyst for this
type of reaction and how much!? If not,what else to do.

Secondly,I see a lot of threads with dad ends and endles discussions about
P2NP--Al/Hg-->Amphetamine and I really don't want this to happen to me too but
what I do know for sure is that procedure that calls for IPA and GAA
https://erowid.org/archive/rhodium/chemistry/nitrostyrene.reduction.alhg.html
Now, IPA and GAA NEVER EVER BEFORE WORKED FOR ME in Al/Hg reduction,
ONLY relatively BASIC MeOH!!! :(???

I mean,I did Al/HgCl2 reduction at least 100-ish times,tryin every solvent there is and ONLY THE METHANOL WORKED!

I remmember trying few times one variation of the procedure involving GAA
(I thin it was calling for GAA to reduce some oxime,it failed miserably!) and after i got nice grayish amalgation bubbling and going I added GAA and damn thing died in its tracks with some greenish color (if I remmember correctly) and I couldn't reactivate it no matter what!

Same goes for Ethanol and IPA,it simply WON'T work!
And yes,my Al foil is cut perfectly,famous Reynolds type kinda thing.

So,can anyone give me any advice!?
Starting from making P2NP itself (Ammonium Acetate or o-toluinide!?)
to reducing it using the appropriate solvents,reactants and conditions,write up I linked is kinda lackign....
(Some people say it's all about heat/temperature over 40-60*C but I am not very convinced
and kinda worried about runnaway reaction even with my big RBF,but still,I don't believe this will do the trick at all!)

Title: **Re: P2NP-derivation to the alternative approach!**
Post by: **whatever9999** on **December 01, 2017, 02:27:44 PM**

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for the Al/Hg reduction of P2NP to Amphetamine I can con firm that IPA+GAA works well. In a reduction like with MDP2P conditions needs to be different, but I have no experience with MDP2P yet.

For the condensation, many amines work well. Methylamine should be already available to you, old bees and wasps have great results with it. I tried ammonium acetate, triethylamine and ethanolamine, also ionic liquids(ethanolamine+formic acid), last one was very easy and doesn't even need heat. For the ionic liquid, molar ration for BA:Nitroethan:IL should be 1:1:5, no other solvent needed. Stir for 3-4 hours at RT, dilute with cold dH2O, crystals should show up almost instant. Recrystallize with IPA, IL can be reused once water is boiled off. Same IL works also well for 3,4,5-TMBA with Nitroethane to make 3,4,5-TMP2NP. Reduction not done yet, but it should yield fine TMA Also very clean and smooth catalysator is ethanolamin, neutralized with GAA(plus some GAA to keep it acid), with IPA+Butanol(4:1) as solvent for BA+Nitro, P2NP crystals filled the beaker after 2h stirring at RT and cooling in the fridge for a few hours.

Title: **Re: P2NP-derivation to the alternative approach!**
Post by: **whatever9999** on **December 01, 2017, 02:39:44 PM**

The paper for the ionic liquid, have good results with this procedure.

Title: **Re: P2NP-derivation to the alternative approach!**
Post by: **carl** on **August 03, 2018, 10:04:05 PM**

How come this has not gotten more attention yet?
I tried the usage of ethanolamine acetate as ionic liquid, both amine catalyst as well as solvent, for the synthesis of P2NP yet too, and besides an astonishing yield of 92% of P2NP while using only 1:1,1 mol aldehyde to nitroalkane, with still only around 15ml used instead of the advised 5 times eq. I could simply boil it dry from water afterwards and consequently plan to use this wonderful substance again as soon as i have the need for it :)
Left the aldehyde and nitroalkane to react for around 8 hours at RT only, precipitated the produced nitroalkene afterwards with water, and wow!
Wonderful and easy stuff, I advise to use this!

Title: **Re: P2NP-derivation to the alternative approach!**
Post by: **Loki** on **September 08, 2018, 11:23:17 PM**

Quote from: carl on August 03, 2018, 10:04:05 PM

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How sensitive does this method seem to be to impure aldehyde contaminated with oxidation products since there seems to be free ethanolamine available to neutralize the carboxylic acid?

Title: **Re: P2NP-derivation to the alternative approach!**

Post by: **Tsathoggua** on **September 08, 2018, 11:31:08 PM**

Why not go for the ketoxime of acetone and reduction to isopropylamine. Also isopropylamine is used as the counterion in many glyphosate weedkillers.

Title: **Re: P2NP-derivation to the alternative approach!**
Post by: **Loki** on **September 09, 2018, 03:16:41 AM**

Quote from: Tsathoggua on September 08, 2018, 11:31:08 PM

> Why not go for the ketoxime of acetone and reduction to isopropylamine. Also isopropylamine is used as the counterion in many glyphosate weedkillers.

Where are you going with this? Isopropylamine as base catalyst, or use it as a solvent/catalyst?

Title: **Re: P2NP-derivation to the alternative approach!**
Post by: **Tsathoggua** on **September 09, 2018, 03:19:10 AM**

as base cat, make the acetate salt of isopropylamine, just thought of it since he doesn't have access to ideal bases like TETA, from the looks of it, but most anyone can obtain acetone, and he said he had hydroxylamine.

Title: **Re: P2NP-derivation to the alternative approach!**
Post by: **Tsathoggua** on **September 09, 2018, 03:20:23 AM**

Oh, and as for O-toluidine, keep that. Get hold of some bird repellent based on anthranillic acid esters and make methaqualone, better use of the O-toluidine by far!