The Vespiary

Main Topics => Drug Synthesis & Extraction => Topic started by: ApeMaia on November 17, 2020, 09:55:43 AM

Title: **From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **November 17, 2020, 09:55:43 AM**

Okay. i was a little bi**h and i've deleted this post because i've believed it was just a noob's boasting, but i now understand it might be usefull to some, so here it is, with some corrections:

3,4,5 Trimethoxy-benzaldehyde to 3,4,5 beta-nitro-trimethoxystyrene

(Ref: Carl's procedure in the 3,4,5 TMNS in MeOH instead of AcOH etc Thread https://www.thevespiary.org/talk/index.php?topic=17616.msg54192627#msg54192627)

In a 500ml Round Bottom Flask there was added 50gr of 3,4,5 Trimethoxy-benzaldehyde, together with 60ml/68gr fresh MeNO2 and 20ml Glacial Acetic Acid. When the Benzaldehyde was totally dissolved, there was added 5ml stinky EtNH2 70%, and the Reaction Mixture was heated to 50-55*C for 100min. The liquid solution was then transferred to a Erlenmayer flask on a Magnetic stirrer, and with strong stirring, a line of cold h2O was poured in, resulting in the separation of a heavy bright yellow (Tweetie the canary) mass of crystals, which was then vacuum filtered as dry as possible. The resulting crystal mass was dissolved in 15Ml/gr warm MeOH, and slowly cooled and then freezed, resulting in the deposition of intense yellow towers/shards of 3,4,5 beta-nitro-trimethoxystyrene. The re-X was done twice, and the MeNO2 was then double Rotovaped to recover it and dried with 4A molecular sieves for a future use. The resulting 2-3cm shards were air dried and then stored in a covered becker with a tiny bit of Nitrogen until their day came:

3,4,5 beta-nitro-trimethoxystyrene to Mescaline 2:1 Sulfate

(Ref: Bubbles procedure in the NaBH4 reduction Thread https://www.thevespiary.org/talk/index.php?topic=15090.msg54192015#msg54192015)

In a 2000ml 3 neck Round Bottom Flask, with a Hg Thermometer and a vertically mounted Alihn condenser, there was added an acqueus solution of 575ml clean/fresh Isopropanol and 230ml h2O with a large stir bar.

To this solution there was added 45,4gr of NaBH4, with was allowed to dissolve with good stiring for 5-8 min; the RBF was placed in an ice bath with took the internal temperature to 20*C, and subsequently there was added 47,84gr 3,4,5 beta-nitro-Trimethoxystyrene (previously re-crystallized 2 times from MeOH, slowly cooled and then freezed, resulting in 2-3 cm long towers/shards of the above) slowly but continuously, making sure the temperature never exceed 30*C.

When the soft yellow solution turned white, there was added a stirred solution of 5,11gr CuCl2 in 50% 100ml IPA/h2O, slowly but continuously, with turned the Reaction Mixture to a black color, with a "strong but not too strong" reaction and 10 second of fumes and fizzing emanating from the Reaction Mixture. (remember to let the NaBH4-styrene mix do it's thing for good before adding CuCL2)

Observation: The yield and quality of the crude greatly improved when the NaBH4 and beta-styrene (added in small portions slowly) solution was allowed to react under good stirring for about 25-30 minutes, when the bubbling was greatly reduced and the the CuCL2 solution was introduced all at once, causing a vigorous reaction but avoiding run-away.

The ice bath was changed with a Pyrex crystalizing dish containing water, and heating was commenced till the RM reached 82-83*C, then the Reflux rate was maintained to 2-3 drops per second for 90 minutes, with good but not strong stirring (50% on my stirrer). (Temperature of condenser water 8*C)
After 90 minutes, heating was tuned off, the warm h2O bath removed, and stirrer changed with another cold one, but stirring was continued until the Reaction Mixture reached 30-33*C (approx 1hour but in my lab you freeze your ass).

Observation: better results where obtained when the reflux rate was maintained even slower, at around 0.5-1 drop per second, resulting in a light tan end Reaction Mixture and avoiding polymerization that might occur above 80-81*C) (gotta buy a better hg thermometer)

Subsequently, the Alihn Condenser was removed, stirring stopped, which resulted in the CuCl2 powder/small lumps depositing on the bottom; the RBF with it's contents was decanted first in a larger Buchner Funnel with filter paper, to remove most of the black component, then subsequently vacuum filtered on a Schott-Duran G4 glass fritt, to remove all the remaining smaller particles. (This is faster than just dumping everything in a single Buchner and waiting 1h to filter everything)

The filtered Reaction Mixture was then basified with a 10% NaoH solution (20-22gr in 200ml h2O) and the 2 Phases separated in a Sep. Funnel. The Isopropanol phase was kept aside, and the h2O phase re-extracted twice with 60ml Isopropanol in the same Sep. Funnel.
Alcoholic Phases were combined in a large Becker, in which was poured a good 8-10 lab spoons of anhydrous MgSO4, and left to themselves for 1h, in which the MgSO4 clumped to harder pieces and other MgSO4 was free-flowing in a "snowstorm" effect.

Observation: Better keeping a lower NaOH solution (10-15%) concentration as the RM is already on the basic side, and filter everything above room temperature to avoid Borate Salts crash out that clog everything from the fritts and sep. funnel.

This was then vacuum filtered on the G4 glass fritt, and the resulting light-beer/champagne IPA solution was acidified with a 20% H2SO4 solution, resulting in pearlescent fine particles crashing out from the solution after a very short stirring. When the solution reached a PH of 7-8, it was placed in the freezer for 3h, then vacuum filtered, washed briefly with 2 portions of 10ml cold acetone, resulting in a thick, Pearlescent/Glistening white "cream" of crystals/flakes. Yield, 39,8gr of the pearleascent flakes.

The above white Mescaline 2:1 sulfate was mixed with 5ml/gr(tried even 10ml/gr) h2O at 75*c, then when the temperature reached 55-60*C, 1:3 of Acetone (eyeballed) was added, the Becker covered with 2 warm towels (Damn that Vogel's Organic chemistry 1455 pages learned me something :D), left to slowly cool, then put in the freezer (not fridge), resulting in spectacular pure Pearl Needlepoint crystals crashing out, which where the vacuum filtered, maintaining their gorgeous Pearl white color.
A point of observation is needed here because maybe I got something wrong, because

my yield from re-X were very poor, with 20-25gr of the above crude Mescaline, resulting in only 6gr of perfectly Needles xtals from the first crop, and another 1,3gr from a second crop the day after(seed crystals were added to the second crop)

I will try next a vacuum distillation and see if I have success in making the Hydrochlorid Salt.

A note to readers, I am by no means a chemist (or maybe I am now?! :)))and this was the first ever chemical reaction conducted in my entire life, aside from throwing some Sodium metal in some water in high school lol, and it took me 3 years of reading and learning from all the great gents on here before even trying to do so. Obviously my re-X yield is still very shitty (25-30% on the crude) but I hope I will have some answers and help from the Pro's.

Also, in 2 trying procedure's i encountered a problem while filtering off the black copper residue, with an unknown mass of a Salt like xtals deposting itself in the glass filter and it's tip, and on the bottom of the filtration flask. Very hard rock consistency, but it dissolved upon the addition of a bit of the hot base solution, then it was mixed together with everything else in the basifing step, any ideas on what it could be ? i will attach a pic of the above shitty hard stuff in the glass buchner.

All reagents used were HPLC/puriss. grade from Sigma Aldrich, Across Organics and Fluka.

Hope it doesn't look too bad, hope you understand it's my first doing anything "chemistry"

||Don't follow too much the re-x part because i didn't do it the correct way, better add the smallest amount of hot water (95-100*C) to the crude sulphate, then fill up with anhydrous acetone, let it crash out, filter, put again the mother liquor in the freezer at -18 for another small crop.

Coming in the following days, an improved work up on the beta-nitro-styrene preparation with porno pics

Sorry again for deleting the original thread, this will stay here, (save it on your pc's in any case !)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **November 17, 2020, 10:45:04 AM**

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needles crashing out from re-x solution

https://imgur.com/a/A6dNvf8

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **bubbles** on **November 17, 2020, 01:47:22 PM**

Be sure to dry your IPA very good before acidifying, there should still be free-flowing MgSO4.
After acidifying and isolating your crude yield, a taste test can tell you if you have a lot of salt contamination (sodium sulfate, sodium metaborate and possibly others idk). If you

do, either reX from IPA and filter out the salts (they dissolve less in hot IPA than mescaline sulfate does). Or re-A/B (add base, extract with non polar solvent, and salt from the non polar solvent.

You definitely have not recovered everything in your reX, mescaline sulfate and the other salts should crash out if you add enough acetone. Place in freezer not fridge.
Probably most of your yield is still in the reX filtrate (I hope you didn't throw it out yet).

To recap: the reX should not result in a lot of weight loss, unless you filtered out a lot of salt contam before cooling down the liquid. It serves to regrow the crystals with less impurities in them than when they grew from the reaction mixture. It removes organic contaminants that stay dissolved in the acetone or alcohol, but these do not make up a lot of the weight.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **November 17, 2020, 02:48:42 PM**

Yeah! That's what I call a well described synthesis.
There was no reason to delete it, even in you're chemistry noob. It's abundant in details and easily accessable (no need to look for it in 69pages- long thread).
As the first synthesis, it went super good for you. I'm still struggling with the same reduction on 2,5 dimethoxynitrostyrene...

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Corrosive Joeseph** on **November 17, 2020, 03:30:45 PM**

Excellent work..... I do love a happy ending! :)

/CJ

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **November 17, 2020, 07:39:07 PM**

Quote from: bubbles on November 17, 2020, 01:47:22 PM

> Be sure to dry your IPA very good before acidifying, there should still be free-flowing MgSO4.
> After acidifying and isolating your crude yield, a taste test can tell you if you have a lot of salt contamination (sodium sulfate, sodium metaborate and possibly others idk). If you do, either reX from IPA and filter out the salts (they dissolve less in hot IPA than mescaline sulfate does). Or re-A/B (add base, extract with non polar solvent, and salt from the non polar solvent.
>
> You definitely have not recovered everything in your reX, mescaline sulfate and the other salts should crash out if you add enough acetone. Place in freezer not fridge.
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> To recap: the reX should not result in a lot of weight loss, unless you filtered out a lot of salt contam before cooling down the liquid. It serves to regrow the crystals with less impurities in them than when they grew from the reaction mixture. It removes organic contaminants that stay dissolved in the acetone or alcohol, but these do not make up a lot of the weight.

Yes sir, snowstorm effect always when adding the MgSO4, till all clumping is gone ! plus i

use only fresh from the bottle IPA, 99.98% reagent grade (at least it says so, damn expensive for some IPA but what's a man gotta do).

As for the lower yield on the first try i think i did the following errors: too much water and not warm enough, and too less acetone, not perfectly anhydrous. But yeah, did not throw away the re-x filtrate and just put it back for another 24h at -18 in the freezer, recovered another smaller crop. Also, i think the first time i conducted the reaction a bit too hot and that resulted in a much lower yield, the crude was fine particles and not silvery flakes, and i overshoot the ph, supposedly bringing shit from the Sulphuric Acid along the way. Will try the IPA re-x next time and document.
Taste was bitter only on the second time with 40.5gr crude yield of perfect Pearlescent flakies, not saltiness like the first time. Also the post reaction mixture was perfectly champagne clear like Blade_runner said, and no polymerization, glad i bought a calibrated Fischer Scientific hg thermometer, the first one i used was showing a good 5-10*C discrepancy, so i was running too hot. Yes the Thermco hg thermometer cost a damn fortune but hey, problem solved for the polymerization :)

Your help has been invaluable Bubbles, you, Carl, loft,blade, and mackolol and the others who chimed in have been the reason this went well :)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **November 17, 2020, 07:42:17 PM**

Quote from: mackolol on November 17, 2020, 02:48:42 PM

> Yeah! That's what I call a well described synthesis.
> There was no reason to delete it, even in you're chemistry noob. It's abundant in details and easily accessable (no need to look for it in 69pages- long thread).
> As the first synthesis, it went super good for you. I'm still struggling with the same reduction on 2,5 dimethoxynitrostyrene...

Re-xing some 2,5 dmns right now from Ethyl Acetate, double re. Tomorrow i will try Carl's work up with DCM, mixing it with that from Orange, and let's see where we get, will look into making the oxalate :)

By the way, how does one go from the oxalate, can you react the salt directly with Br2 or it needs the NBS route ? Might do a thread after i do a short one for the beta-nitro-styrene's workup with a pictorial

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Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **November 17, 2020, 07:52:32 PM**

You're supposed to freebase the oxalate further and extract the freebase. Then you can brominate the freebase, whatever way you want to, or convert it to HCl and brominate. The oxalic acid is just to precipitate clear white salt and purify it. That's the magic ability of the oxalic acid as Carl would say :D

I wouldn't recommend bromination of oxalate though, because we don't know how will the oxalate behave in such situation. You can try it, but even though, oxalate isn't the best way of ingestion, if you're to consume your drug and you definitly are 8)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging**

return)
Post by: **ApeMaia** on **November 17, 2020, 08:00:48 PM**

Yeah, freebasing it was also what i 80% believed needs to be done, with dcm.

Hoped that it would have worked from the salt like it does from HCL with NBS,altough on low yields with that workup.

Freebase everything it is then !

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **November 17, 2020, 08:06:14 PM**

Quote from: Corrosive Joeseph on November 17, 2020, 03:30:45 PM

> Excellent work..... I do love a happy ending! :)
>
> /CJ

That's what i always say at the Chinese massage shop, got a fidelty card by now ;)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **November 17, 2020, 08:52:11 PM**

I believe that one doesn't necessarily need NBS to brominate the 2CH HCl. There is some thread, old but it states that 2CH HCl can be brominated with elemental bromine too: https://www.thevespiary.org/talk/index.php?topic=9037.msg39392500#msg39392500

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **November 17, 2020, 09:22:39 PM**

Very good thread, again, good post dear Biene Maja! :)
Thank you for putting it up again.
It is really worthwhile :)

@mackolol, well I'm surprised you can brominate 2C-H HCl with bromine and don't need the freebase for this.
But you still end up with the lousy HBr salt which you can't smoke.
I am not so sure you can not brominate the oxalate directly as well.
I think its possible.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **November 17, 2020, 09:24:30 PM**

Quote from: mackolol on November 17, 2020, 08:52:11 PM

> I believe that one doesn't necessarily need NBS to brominate the 2CH HCl. There is some thread, old but it states that 2CH HCl can be brominated with elemental bromine too: https://www.thevespiary.org/talk/index.php?topic=9037.msg39392500#msg39392500

We shall try then direct from the hcl/oxalate then, can't wait to get bromine fumes everywhere, damn satanic ritual.

attached a rare pic of Carl and me after i deleted the original thread

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Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **November 17, 2020, 09:29:33 PM**

Hahaha :D
Nothing is ever worthless as a post, I sometimes, when reading my old posts, think as well "oh damn thats horrible, the world would be better without it", but no, someone maybe finds it helpful :)

Quote from: ApeMaia on November 17, 2020, 08:06:14 PM

> Quote from: Corrosive Joeseph on November 17, 2020, 03:30:45 PM
>
> > Excellent work..... I do love a happy ending! :)
> >
> > /CJ
>
> That's what i always say at the Chinese massage shop, got a fidelty card by now ;)

Whaaat, chinese? Here they are vietnamese, and in my hometown thai... didn't expect the chinese to do it as well :P

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **November 17, 2020, 09:41:14 PM**

Quote from: carl on November 17, 2020, 09:22:39 PM

> @mackolol, well I'm surprised you can brominate 2C-H HCl with bromine and don't need the freebase for this. But you still end up with the lousy HBr salt which you can't smoke.

Would I? Does bromine displace Cl in HCl salt?, I think it's other way round.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **December 27, 2020, 10:39:03 PM**

New year i'm sending a sample to test out of this stuff, both crude and Re-x needles. Any bets on what purity it could be ? What is theoretical maxium purity of needles of mescaline sulphate ?

I know for mdma hcl was 84% because of the structure thing of the salt or something like that, Lsd will almost always have a iso part because of the 9:1 equilibrium part, but what about Mesc ?

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **chingchong** on **January 09, 2021, 10:54:08 PM**

Quote from: ApeMaia on December 27, 2020, 10:39:03 PM

> New year i'm sending a sample to test out of this stuff, both crude and Re-x needles. Any bets on what purity it could be ? What is theoretical maxium purity of needles of mescaline sulphate ?
>
> I know for mdma hcl was 84% because of the structure thing of the salt or something like that, Lsd will almost always have a iso part because of the 9:1 equilibrium part, but what about Mesc ?

I'm pretty sure the 84% thing is just because of molecular weight, i.e 100% pure MDMA.HCL contains 84% mdma and 16% hcl, I think it's a dutch thing. That's also why almost all speed advertised on the deep web is "74% PURE UNCUT AMPHETAMINE SULPHATE"(even though it's mostly caffeine and methanol)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Sawdust and Honey** on **January 09, 2021, 11:00:19 PM**

Quote from: chingchong on January 09, 2021, 10:54:08 PM

> "74% PURE UNCUT AMPHETAMINE SULPHATE"(even though it's mostly caffeine and **methanol**)

Methanol?

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **embezzler** on **January 09, 2021, 11:41:00 PM**

Mannitol

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **chingchong** on **January 10, 2021, 02:12:09 AM**

Quote from: Sawdust and Honey on January 09, 2021, 11:00:19 PM

> Quote from: chingchong on January 09, 2021, 10:54:08 PM
>
> > "74% PURE UNCUT AMPHETAMINE SULPHATE"(even though it's mostly caffeine and **methanol**)
>
> Methanol?

Quote from: embezzler on January 09, 2021, 11:41:00 PM

> Mannitol

It's usually paste and usually loses well over 50% of it's weight after drying out. I assume it's mostly methanol left over when they don't bother drying it out after forming the salt, could have water added to increase weight as well though. I've read in a couple places that 1 litre of freebase amphetamine makes around 3kg of amphephetamine sulfate "paste" before it's even cut with caffeine. Nasty stuff that doesn't make any sense to me

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Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **January 10, 2021, 01:47:27 PM**

Here is the topic about the paste shit phenomenon. It may come handy for you:
https://www.thevespiary.org/talk/index.php?topic=18285.msg54199094#msg54199094

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Helios** on **January 29, 2021, 11:02:11 AM**

Hi there mate ;D

I have a far superior method to the nitrostyrene. Tried all those in literature over about 6 months, all were messy and low yeilding.

My method takes about 4-6 hours, ~60 deg C, and no GAA.
Massive ulta pure shards crystallise directly from the reaction mixture, no need for any recrystallisation. Yeild near quantitive, something like 92% molar.

I had some godly images of such but seem to have lost the location of the encrypted files :'(

I'll see what I can do, maybe need to re-run reaction.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Sawdust and Honey** on **January 29, 2021, 11:12:24 AM**

We'd be thrilled to hear about your method, then. :o

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **January 29, 2021, 11:47:41 AM**

Helios! :)
Damn, seeing you reminds me of forgetting to answer at least one of your mails lately, I think... sorry!
Too much in my inbox I have to answer on, too little time, etc... :/
Nothing personal of course, its way too many people else that are still waiting too...
I try to catch up, but I barely get more than a few days of messages answered, and until I have time again for my mails, it is usually again two weeks worth of mails of which I might get a quart done... and so on, almost sisyphean work, I try though but it is inevitable that a portion will fall under the desk.

92% yield of TMNS? Sounds alright.
My method with ethylamine acetate in GAA gave consistently(well, on both trials :P) a whooping 96% for TMNS, and 96-97% for DMNS(first number is from a friend not me).

But no GAA? What do you use else?
Nitrostyrenes like, opposed to nitropropenes, GAA as reaction solvent, or at least MeNO2, the latter being much more precious in europe than GAA is.
With sufficient GAA you can reduce the nitromethane quantity to 1,5eq. or even less.

And what catalyst have you used?
I have a feeling it was not an amine but an alkali hydroxide, those are said to be able to give such yields as well, if done right.
Am I correct?

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Helios** on **January 29, 2021, 02:26:40 PM**

Quote from: carl on January 29, 2021, 11:47:41 AM

> Helios! :)
> Damn, seeing you reminds me of forgetting to answer at least one of your mails lately, I think... sorry!
> Too much in my inbox I have to answer on, too little time, etc... :/
> Nothing personal of course, its way too many people else that are still waiting too...
> I try to catch up, but I barely get more than a few days of messages answered, and until I have time again for my mails, it is usually again two weeks worth of mails of which I might get a quart done... and so on, almost sisyphean work, I try though but it is inevitable that a portion will fall under the desk.
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> With sufficient GAA you can reduce the nitromethane quantity to 1,5eq. or even less.
>
> And what catalyst have you used?
> I have a feeling it was not an amine but an alkali hydroxide, those are said to be able to give such yields as well, if done right.
> Am I correct?

Hey buddy, was hoping you were still around. Don't be sorry, I was gone for like 9 months lol really I should have at least dropped in to say hello.

Great news though! Found most of the files I had stored. A few images I cannot locate (probably the one corrupted archive, knowing my luck). Even found the write-up which is brilliant because I barely remember. Looking back has me thinking "did I really write this?" Really need to get back on my script so I can function adequately. If only I could afford to see the specialist lmao

Give me an hour or so, I'll post it now.

Catalyst is beta-phenylethylamine, solvent is isopropyl alcohol (+ tiny bit of excess nitromethane)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Helios** on **January 30, 2021, 11:10:13 AM**

As promised - https://www.thevespiary.org/talk/index.php?topic=18605.new#new

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **January 31, 2021, 07:05:59 PM**

I found out based on several trials with Carl's method, that it works flawlesly everytime, but i did cut his GAA quantity to half, and this way the styrene comes out way cleaner and faster, sometimes even in 15-20 minutes, 6h seems an unnecessary complication ! But i'll check your method too, fortunately nitromethane is something i can get 24h 7/7

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **January 31, 2021, 07:11:19 PM**

> Quote from: carl on January 29, 2021, 11:47:41 AM
>
> > Helios! :)
> > Damn, seeing you reminds me of forgetting to answer at least one of your mails lately, I think... sorry!

Yeah Carl, like that mail i've sent you in far away 2020 :P
You must be a supermodel, wanted by all girls, never have time for an email

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **January 31, 2021, 09:19:50 PM**

> Quote from: ApeMaia on January 31, 2021, 07:11:19 PM
>
> > Yeah Carl, like that mail i've sent you in far away 2020 :P
> > You must be a supermodel, wanted by all girls, never have time for an email

"Girls", yeah... :D
But maybe that would increase my reply rate?
Send tits, get reply in 20min or the ~~money~~ tits back! ;D

Sorry man, if I had a dozen grams of a good stimulant and a month without any obligations, including the need to sustain and relieve myself, I might be able to get that all sorted out... ::)
But don't worry, I haven't forgotten you.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **StuffedBee** on **April 07, 2021, 03:27:29 PM**

I am experiencing some extremely steep yield losses when doing a proper 15ml/g recrystallization of nitrostyrene in methanol. It's like 3g that are lost in 150ml. Does this mean there are really that much impurities? Or is it just the solubility of nitrostyrene at -20C? I'm a bit lost here...

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **April 07, 2021, 03:53:01 PM**

> Quote from: StuffedBee on April 07, 2021, 03:27:29 PM
>
> > I am experiencing some extremely steep yield losses when doing a proper 15ml/g recrystallization of nitrostyrene in methanol. It's like 3g that are lost in 150ml. Does this mean there are really that much impurities? Or is it just the solubility of nitrostyrene at -20C? I'm a bit lost here...

post a picture of your crude, maybe you ran it too hot like i did the first time, and maybe the benzaldehyde is of a lower purity than what a seller can advertise

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **April 07, 2021, 04:05:20 PM**

15ml/g is a lot, I don't know why everyone tells that this amount is preferred.
I did like 8ml/g + few mls of ethyl acetate on 60gram scale I believe and lost 1/3rd of my styrene. Screw this, I'm never gonna use that much...

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **April 07, 2021, 04:18:44 PM**

Quote from: mackolol on April 07, 2021, 04:05:20 PM

> 15ml/g is a lot, I don't know why everyone tells that this amount is preferred.

Once and for all, you just need to read Pihkal, finally... ::)

But I agree, thats too much.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **April 07, 2021, 04:28:30 PM**

Shulgin didn't give a fuck whether half of his product is going to stay in the recrystallisation solvent or not. He just wanted to obtain the substance and make some tests and probably have fun as well. He wouldn't improve his workups from the same reason, that's why I hardly ever use his synthetic workups.
I do give a fuck, because I'm not sponsored ;'(

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **April 07, 2021, 04:37:02 PM**

I don't disagree with you as I am of the same opinion, I just wanted to explain where this comes from ;)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **blade_runner** on **April 07, 2021, 04:55:20 PM**

Quote from: mackolol on April 07, 2021, 04:05:20 PM

> 15ml/g is a lot, I don't know why everyone tells that this amount is preferred.
> I did like 8ml/g + few mls of ethyl acetate on 60gram scale I believe and lost 1/3rd of my styrene. Screw this, I'm never gonna use that much...

I've experienced 20-30 ml/g with 91% IP or methanol and never had issue with low recovery, always 85% or more. I assume the remaining 15% was impurity...

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **April 07, 2021, 05:23:35 PM**

Then it's the matter of ethyl acetate/ not completely pure MeOH then.
Thanks Carl for explaining, it's very interesting how close people tend to follow Shulgin, although it's probably my fault on that one. Well as for now I don't have a need to figure it out :D

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **April 07, 2021, 05:30:39 PM**

Quote from: blade_runner on April 07, 2021, 04:55:20 PM

> I've experienced 20-30 ml/g with 91% IP or methanol and never had issue with low recovery, always 85% or more. I assume the remaining 15% was impurity...

My thoughts too, aqueous alcohols should perform much better, but you have to figure the ideal water content out.

Which brings me to the topic of denat. ethanol. which I very often use for nitroalkene recrystallisations.
The 4-6% of water in that make it so suitable actually, this is well known and accepted for for example P2NP.

I think making a solution in alcohol and while still hot or even simmering, to add water dropwise(stirred) until a slight turbidity is achieved, not more than 5, maybe 10% at most.
But I would assume that 5% is already more than sufficient.
And then to let it cool down, but somewhat slower than usual, and fridge before you the freezer of course as well.
Water, a tiny bit of it of course, is usually very beneficial in the recrystallisation of nitroalkenes.

Quote from: mackolol on April 07, 2021, 05:23:35 PM

> Thanks Carl for explaining, it's very interesting how close people tend to follow Shulgin, although it's probably my fault on that one. Well as for now I don't have a need to figure it out :D

Rarely followed him to the letter, we usually had it all optimised far beyond his initial research, even twenty years ago.

Only exception is the red-al reduction of indoleglyoxylamides, which I could only realise thanks to Tihkal.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **loft** on **April 07, 2021, 05:52:57 PM**

I hate when numbers are just thrown around. Context matters, a lot. I just recently gave a mL/g number too.

Quote from: loft on April 05, 2021, 05:20:36 PM

> About 17 mL/g anhydrous IPA is needed for 2,5-DMNS.

It should be logic that those numbers only relate to the exact conditions stated. This changes with the solvent (MeOH, EtOH, IPA will all have different solubilites) and the water content presence. So please always be specific when talking about these numbers :)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Sawdust and Honey** on **April 07, 2021, 06:31:09 PM**

> Quote from: loft on April 07, 2021, 05:52:57 PM
>
> I hate when numbers are just thrown around. Context matters, a lot. I just recently gave a mL/g number too.
>
> > Quote from: loft on April 05, 2021, 05:20:36 PM
> >
> > About 17 mL/g anhydrous IPA is needed for 2,5-DMNS.
>
> It should be logic that those numbers only relate to the exact conditions stated. This changes with the solvent (MeOH, EtOH, IPA will all have different solubilites) and the water content presence. So please always be specific when talking about these numbers :)

I found your number to be somewhat accurate, though. Recrystallized around 5g of the nitrostyrene from 80ml of IPA with success.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **April 07, 2021, 06:39:06 PM**

Doing that with success is easy, just avoiding losses is not.
How much got you out of your 5g's in the end?
Just to get a general idea about that.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Sawdust and Honey** on **April 07, 2021, 08:16:54 PM**

4.4g, everything is described in the 2-HEAA thread.
And there's still nitrostyrene in the filtrate that I tossed in the fridge. Too lazy to filter it though.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **StuffedBee** on **April 07, 2021, 08:40:14 PM**

I tried less methanol before - it always crashes out. With like 10ml/g I got it to crystallize on the second try but it was really quite difficult to get right. At least I kept all the methanol so nothing of value can be lost

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **NeonCortex** on **April 07, 2021, 11:08:18 PM**

Concerning IPA/water recrystallization. Some 2C-B HCl was to be recrystallized in absolute IPA, or so was the plan. More and more IPA was added and the suspension stirred and heated to just below boiling. For every addition of IPA the suspension seemed to be "soon there". But as the volume got larger and larger and no real dissolution happened, it was eventually decided that no more IPA would be added. Instead a *few drops* of water was added to an almost full 250 ml E-flask, and presto! Solids dissolved and the solution was left to cool in RT first, then fridge (perhaps freezer also, can't

recall). Result was a mass of very fine, small needles that when still damp looked a slightly greyish off white. On drying they brightened up, but still not your typical opaque white. I suppose the crystals have some translucency that gives that slight off-white hue. All other 2C-B I've seen seemed like filthy shit in comparison.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **April 07, 2021, 11:40:11 PM**

Quote from: NeonCortex on April 07, 2021, 11:08:18 PM

> Result was a mass of very fine, small needles that when still damp looked a slightly greyish off white. On drying they brightened up, but still not your typical opaque white. I suppose the crystals have some translucency that gives that slight off-white hue. All other 2C-B I've seen seemed like filthy shit in comparison.

I guess the only thing left to do for you is know... to smoke some of these crystals :P

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Johnathan Ferrous** on **April 08, 2021, 01:59:52 AM**

Quote from: NeonCortex on April 07, 2021, 11:08:18 PM

> Concerning IPA/water recrystallization. Some 2C-B HCl was to be recrystallized in absolute IPA, or so was the plan. More and more IPA was added and the suspension stirred and heated to just below boiling. For every addition of IPA the suspension seemed to be "soon there". But as the volume got larger and larger and no real dissolution happened, it was eventually decided that no more IPA would be added. Instead a *few drops* of water was added to an almost full 250 ml E-flask, and presto! Solids dissolved and the solution was left to cool in RT first, then fridge (perhaps freezer also, can't recall). Result was a mass of very fine, small needles that when still damp looked a slightly greyish off white. On drying they brightened up, but still not your typical opaque white. I suppose the crystals have some translucency that gives that slight off-white hue. All other 2C-B I've seen seemed like filthy shit in comparison.

Pictures?
Words can only capture so much!

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **loft** on **April 08, 2021, 01:03:52 PM**

In case you're willing to share pictures @NeonCortex, please consider posting it in The Chemicals Image Gallery (https://www.thevespiary.org/talk/index.php?topic=18741).

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **April 08, 2021, 04:18:10 PM**

Quote from: loft on April 08, 2021, 01:03:52 PM

> In case you're willing to share pictures @NeonCortex, please consider posting it in The Chemicals Image Gallery (https://www.thevespiary.org/talk/index.php?topic=18741).

I will surely contribute with some pic puuuuorn

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging**

return)
Post by: **Johnathan Ferrous** on **April 08, 2021, 05:57:05 PM**

I like referring to beautiful samples as chemical porn

Same as amazing guns, it's gun porn. That ping that an M16 Grand makes: gun porn.

Haha.

That thread actually reminds me of Theodore Grey's book, "The Elements", and subsequent book, "Reactions".

Just a big compilation of amazing samples.

h00ps://theodoregray.com/PeriodicTable/Elements/019/index.s7.html

You can feast your eyes on the elements and the chemicals for hours.
That's actually how I first started chemistry, reading these books.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **NeonCortex** on **April 13, 2021, 02:52:10 AM**

Quote from: loft on April 08, 2021, 01:03:52 PM

> In case you're willing to share pictures @NeonCortex, please consider posting it in The Chemicals Image Gallery (https://www.thevespiary.org/talk/index.php?topic=18741).

Sure, I'll do that ASAP.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **Sawdust and Honey** on **April 16, 2021, 02:33:00 PM**

But this paper talks about aromatic NO2 groups. They can even be reduced by things like dithionite, unlike aliphatic nitrostyrenes and nitropropenes. Of course the nickel/borohydride system works but it's not as simple with aliphatics.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **NeonCortex** on **April 16, 2021, 06:36:24 PM**

Quote from: carl on April 07, 2021, 11:40:11 PM

> Quote from: NeonCortex on April 07, 2021, 11:08:18 PM
>
> > Result was a mass of very fine, small needles that when still damp looked a slightly greyish off white. On drying they brightened up, but still not your typical opaque white. I suppose the crystals have some translucency that gives that slight off-white hue. All other 2C-B I've seen seemed like filthy shit in comparison.
>
> I guess the only thing left to do for you is know... to smoke some of these crystals :P

Naturally I already tried to vaporise just a tiny tiny tiny amount. Too little to get effect really, just wanted to see how it vaporised. Will try as soon as I feel I have possibility to go off grid for a few hours (read your reportings on it, hehe). I love smoking chemicals.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **June 01, 2021, 09:43:24 PM**

So, after some months i ended my Mescaline reserves, so i went on to make some more, all nice and well (maybe mother liquor a bit too on the darker orange side after RXN) untill i went to dry the solution with MgSO4: the motherfucker went black and started to fizz quite well !? After freezer time and 2x Acetone washes, it seems i got Silver/metallic color Mescaline (DAFUQ), and i really don't know where i fucked up.

Might be my MgSO4 that was not dry enough ?

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **June 01, 2021, 09:49:35 PM**

A solution, solution in what?

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **June 01, 2021, 10:00:34 PM**

Quote from: carl on June 01, 2021, 09:49:35 PM

> A solution, solution in what?

Hello Grand Wizard Carl ! How are you ? Hope you're through a better time now that summer is here buddy, sending some krauto love :)

Anyway for solution i meant the filtered IPA Layer of post reaction, the one you dry out with MgSO4, then filter it out, and salt with 20% H2SO4/H20.

As soon as i went to dry it with MgSO4, it started to go abruptly black, and leaving out bubbles (hydrogen perhaps?). When i added H2SO4 at 20%, it still salted out the M, but it's Silver Surfer grey :(

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **aes256** on **June 02, 2021, 03:23:58 AM**

Have you tried re-xtal or washing the silver M in dry, ice-cold acetone?

What has the color of your M been previously?

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **June 02, 2021, 07:42:35 AM**

Maybe some borohydride residues were left in solution, and they reacted with Mg2+ ion

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**

Post by: **ApeMaia** on **June 02, 2021, 09:40:07 AM**

> Quote from: aes256 on June 02, 2021, 03:23:58 AM
>
> Have you tried re-xtal or washing the silver M in dry, ice-cold acetone?
>
> What has the color of your M been previously?

Yesterdayi ranout of clean Acetone, so will have to Rotovap some today, dry it with 4A, and then proceed in this way, it was the first thing that went through my mind.

Before i always got pure pearlescent white, already looking very nice as crude, you can see some pics on first page here :)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **June 02, 2021, 09:42:18 AM**

> Quote from: big mac on June 02, 2021, 07:42:35 AM
>
> Maybe some borohydride residues were left in solution, and they reacted with Mg2+ ion

This is what i believe it happened, btw i was about to write you in priv, but it was too late last night and you were probably wanking ultra hard :D :D

A suspicion arises from the fact the post-Rxn reaction was still a bit on the warm side, 40ish C, so that might make it interact with the Mg ion's right ? it's all learning for me

---

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **June 02, 2021, 02:24:43 PM**

Nah, the temp doesn't matter, but just the black shit and gas evolution seems like it.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **ApeMaia** on **June 03, 2021, 09:14:58 PM**

Together with Mac's help in private (always helpful) we managed to pinpoint the problem to the fact that the post Reaction Mixture, was too warm, hence the NaBH4 was not 100% quenched, so it was interacting with the MgSo4 in a weird way. I forgot that 6 months ago my lab was at 8-10*C usually, while now it's over 30*C during the afternoon. We are back to pure pearlescent white, 47,84gr 3,4,5 TMNS in, 39,80 M out :)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **June 03, 2021, 09:44:34 PM**

> Quote from: big mac on June 02, 2021, 02:24:43 PM
>
> Nah, the temp doesn't matter, but just the black shit and gas evolution seems like it.

That happens when you're rubbing one off? :o
I don't know, but maybe see a doctor once in a while, it sounds, well... unhealthy, to be honest :-X

@Maia, thank you, I'm well, more or less ::)
I just hope you're better off though ;)

(pssht... be careful with mac... he doesn't sound overly healthy with these strange symptoms, don't contract it! :o :P)

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **aes256** on **June 04, 2021, 12:59:15 AM**

Quote from: ApeMaia on June 03, 2021, 09:14:58 PM

> ... the post Reaction Mixture, was too warm, hence the NaBH4 was not 100% quenched, so it was interacting with the MgSo4 in a weird way

Wouldn't the base destroy leftover NaBH4?

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **carl** on **June 04, 2021, 01:14:59 AM**

Base, hydroxides especially, stabilise borohydride solutions, that allows them to get a significant long shelf life of even years!(like in a strong lye solution, 40% or so, very long shelf life this way).

However, you don't even need to quench the borohydride after the reaction at all, like in the original paper.
It could be possible some of it travelled within the solution.

Title: **Re: From 3,4,5 Tmba to Mescaline 2:1 hemisulfate dihydrate (the flogging return)**
Post by: **big mac** on **June 04, 2021, 07:50:36 AM**

Quote from: carl on June 03, 2021, 09:44:34 PM

> That happens when you're rubbing one off? :o
> I don't know, but maybe see a doctor once in a while, it sounds, well... unhealthy, to be honest :-X
>
> (pssht... be careful with mac... he doesn't sound overly healthy with these strange symptoms, don't contract it! :o :P)

All those psychedelics have triggered my 5-HT3 and 5-HT4 gastrointestinal receptors.
Nothing bad, I'll be fine Carl ::)

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