2C-B Synthesis (With Pictures)

Phenethylamine Chemistry Discussion of the Synthesis of amphetamine and related compounds

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10-01-2005, 06:55 AM #1

DrugPhreak
Silver Member

Join Date: Jan 2005
Location: Cook Islands
Age: 64
Posts: 769

I don't see Icecool coming around here anymore so I thought it would bee a good idea to post his excellentwrite-up, which was posted on another forum that most people here probablynever go to. I'm sure this will bee reallyhelpful to members that don't have much experience with chemistry who are interested in this substance.

2C-B

Starting from 2,5-dimethoxybenzaldehyde.

First off all the coresponding 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 (~45ºC) 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

The orange crystalline mass was broken up with a glass stiring rod some water was added since it was really thick and then vacuum filtered until no more liquid came threw the buchner, the crystals were then washed with ice-cold isopropylalcohol (10ml), then sucked as dry as possible in the buchner.

The crystals were put in a dessiscator, until they are dry and crispy. The total mass weighed 5.56g (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 an single bond, this can be done with an reducing agent like $LiAlH_4$ or $NaBH_4$ but since these are quite hard to get and dangerous to handle with, SWIM'll use Al/Hg.

Normally Al has a thin coating of it's oxide Al2O3 but when you remove that coating Al is very reactive, so when you add a mercury salt to Al in an solvent like MeOH or EtOH or IPA, the mercury will stick on the surface and prevent new Al2O3 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 Al shreddings 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 MeOH.
Now one needs to add 40mg of HgCl2 solution dissolved in 40ml of water and 40ml 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°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 HgCl2 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% NaOH solution, another vigorous reaction which makes the mixture boil.

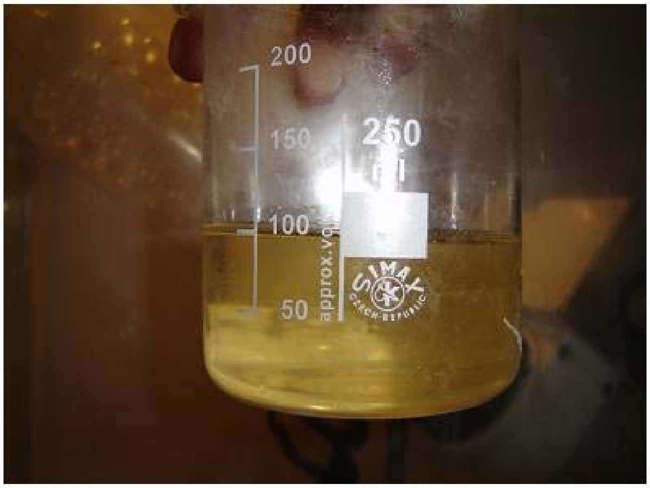
Added 100ml toluene to the mixture and put the magnetic stirrer on maximum, on the right the toluene layer in the seperatory funnel.

Now the toluene has the freebase inside (2C-H).
And as you can see it is not light yellow or clear but red/brown, so an acid/base purification is done.
Now 30% sulfuric acid is added to the toluene.

This happens 2C-H.SO4 crystalizes out but also disapears quickly into the water again.
Now shake very hard so everything becomes the sulfate salt, then add DCM which doesn't mix with water this takes up every crap that shouldn't be in there, now the DCM turns red and repeat this (and throw away the DCM) until the DCM washes remain clear.
Below here a dirty DCM wash can be seen.

So this is the water layer remaining, which contains relatively pure freebase now, now the freebase has to be unleashed again, this can be done by adding 20% NaOH to this mixture but be carefull only add small amounts until the mixture remains white THEN add toluene to extract the freebase (the white stuff is an emulsion of water and oil (freebase)).
Anyway then after all the freebase went into the toluene more NaOH can be added...

White emulsion, if you would wait quite long you could actually see the white stuff making droplets of oil and floating to the top and forming a top layer of very pure freebase.



Combined the extracts of toluene (since the mixture is extracted 2 times), now this is toluene containing 2C-H freebase, notice the difference in color, slightly yellow now and before dark red/brown.

Then dry the toluene with MgSO4 (anhydrous) for 12 hours and then distill/evaporate/rotavap off the toluene.

Pure freebase left in the RBF in the rotavap, it already is a bit brown because of the CO2 in the air it really quickly forms 2C-H.CO3 which doesn't matter actualy.

Bromination

Now this 2C-H has to be brominated to yield 4-bromo-2,5-dimethoxyphenetylamine.
The bromine is made in situ and dissolved in DCM.
2,4g of KBr is dissolved in 30ml dH2O and to that 16ml 35% H2SO4 is added to that 8ml of DCM is added and then finally 0,97ml of H2O2 30% concentration is added in portions.

KBr and water

A few drops of H2O2 30% added.

The DCM phase absorbing the bromine.

The DCM layer is added to the freebase in an icebath, the freebase is first dissolved in the same volume of glacial acetic acid.

Overall view of the bromine production.

Now one has formed 4-bromo-2,5-dimethoxyphenetylamine.HBr most of the people want to convert this to the hydrochloride salt, so then first the formed crystals (after 15min light brown crystals formed), are filtrated and washed with ether and ice cold acetic acid to get rid of the bromine excess.
Then NaOH is added to get 4-bromo-2,5-dimethoxyphenetylamine 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-dimethoxyphenetylamine freebase, and then HCl 37% is dripped in now immediatelly or after hard stirring white needle like crystals form. This is the 4-bromo-2,5-dimethoxyphenetylamine.HCl but first put it in the freezer at 20°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.

____
No thing is without poison. Dosage makes it a poison or a remedy.

-Paracelsus

Quote

10-01-2005, 09:56 AM

#**2**

Operacya_Y
Donating Gold Member

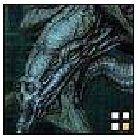
Join Date: Aug 2005
Location: Cook Islands
Posts: 247
🟩🟩🟩🟩🟩

Very nice sythese.Could you put it in to some link that we can download it like ebook PDF format?

Quote

10-01-2005, 03:13 PM

#**3**



behemuthzero
Donating Silver Member

Join Date: Sep 2005
Location: USA
Posts: 49
🟩🟩

HOLY CRAPAMOLY!!!!

Extremly nice post....Thank you very much...nice lab gear too ~.o

The pictures were a tremendous help...

Quote

10-01-2005, 07:23 PM

#**4**

DrugPhreak
Silver Member

Join Date: Jan 2005
Location: Cook Islands
Age: 64
Posts: 769
🟩🟩🟩

http://members.home.nl/phenyl/2C-B.pdf

The PDF pages where extracted from that and converted to JPEG format to reduce the file size, which allowed for posting, but it did make the non-bold font look kind of crappy.

____
No thing is without poison. Dosage makes it a poison or a remedy.

-Paracelsus

10-01-2005, 07:48 PM

#5

Operacya_Y

Donating Gold Member

Join Date: Aug 2005
Location: Cook Islands
Posts: 247
🟩🟩🟩🟩🟩

2,5 dimethoxebenzaldehyde...can SWIM make it from benzaldehyde?

10-02-2005, 11:00 PM

#6

Kemikaru_Tenshu

Platinum Member

Join Date: Jul 2004
Location: USA
Age: 26
Posts: 450
🟩🟩🟩

Quote:

> Originally Posted by **RasputiN**
> *2,5 dimethoxebenzaldehyde...can SWIM make it from benzaldehyde?*

Not easily, if at all.

~KT

____
"If you can't explain it simply, you don't understand it well enough."
- Albert Einstein

10-02-2005, 11:19 PM

#7

poozjurry

Silver Member

Join Date: Jun 2005
Location: Neutral Zone
Age: 41
Posts: 158
🟥

realy nice! Itsnice too, for DOB-Dreamers.You must only use nitroethane for nitromethane.

10-04-2005, 03:50 AM

#8

Eirias

Platinum Member

Join Date: Jan 2005
Location: United States
Posts: 485
🟩

You rawk DP!!!

So just to clarify-- he adds the Br in the DCM directly to the

2C-H base dissolved in GAA? The DCM does not fuck up the
rxn? Or is it distilled off (or otherwise removed)? Are the Br
and the freebase so much more soluble in GAA that none of it goes into
the DCM? What happened then to that lil' bit of DCM?

SWIM was dreaming of trying to dream of oxidizing
the KBr without any DCM, and then just pipetting up the Br and
putting it directly into some GAA. Then obviously the Br in GAA would be combined w/ the
freebase in GAA.

SWIM's concern(s) were that 1)
distilling the DCM/Br mix to remove the DCM would produce lotsa nasty-ass Br fumes, and
2) that if he added the Br while still in the DCM, it might fuck up
the rxn (this is not the case I presume?). He knows that Br is probably more soluble in GAA than in
DCM, and that an immiscible DCM/GAA mix would form two phases
obviously, but still the DCM in the rxn issue was still unresolved.

Thank you so much IceCool and DP! Please, though, if you
can answer SWIM's question(s), or maybe explain that part of the rxn a
little more, he would
be most grateful.

He apologizes for his ignorance and lack of comprehension in
advance, and he tells me that he just wants his dreams to be good and
proper...

OH- and I assume that since this write-up uses 2.4 grams of KBr,
that the yield of freebase post Al/Hg was about 2 grams, or around
40%? Just curious, since yields for the nitrostyrene are stated,
but not for the freebase amine.

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:31 PM.

Quote

10-04-2005, 09:05 AM

#**9**

daeron
The Platinum Post Whore
Platinum Member

Join Date: Dec 2004
Location: Slovenia
Posts: 1,184
🟩🟩🟩🟩🟩🟩

if i remeber his yields are never um..glorious...but may mistaken

____
...perche io so io, e voi non siete un cazzo!

Quote

10-04-2005, 09:28 AM

#**10**

Eirias
Platinum Member

Join Date: Jan 2005
Location: United States
Posts: 485
🟩

Yeah if I recall correctly, MegaLab 420 or someone else may have
mentioned that the Al/Hg had somewhat poorer yields, versus the LAH or
NaBH4 routes. But product is product, and a successful synth is

still a success even if the yields are less then glorious.

Swim is fantasizing of making KBr as per above, but with no DCM, and just pipetting the liquid elemental Br up and then immediately putting it into GAA, in prep for the addition to the base in GAA. Swim may also cool the mix prior to pipetting in order to make the Br less soluble in the small amount of water in the mix (it's only slightly soluble in water anyway) and also to minimize any Br fumes. Swim will probably make a slight excess of the Br to compensate for slight losses and the small amount being dissolved in the water layer. He would then proceed ala the "large scale" bromination with pics as per Eurotopian and Salvinorin as described Rhodium, keeping the product as the HBr salt and work-up with EtOAc. Swim likes that the HBr is more water soluble than the HCl.

I'm still curious what happens to the DCM in IceCool's procedure... where does it go? Does it affect anything? Does it hold on to/dissolve any of the freebase or Br? Inquiring minds want to know...

But props to IceCool nonetheless for the write-up. With a bit more detail here and there, this would be Rhodium Archive-worthy IMHO.

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:32 PM.

Quote

10-13-2005, 12:33 PM

#**11**

Eirias

Platinum Member

Join Date: Jan 2005
Location: United States
Posts: 485

Bump.

And an update.

Swim told me he tried to brominate ~5g of base with Br2 in DCM (prepared from KBr as described above) in GAA. An orange colour formed, but absolutely no precipitate! WTF?!? Swim is not sure what is going on, but he is considering trying to heat the mix up to drive off the bit of DCM in the mix (DCM and GAA are in fact miscible, Swim found out to his surprise), and then cool it down to see if any precipitate then forms. Another possibility would be to basify the mix with NaOH sol'n and see if any base oil falls out, extract the oil with toluene, and attempt to xtallize it as the HCl salt according to SWIIceCool's above procedure.

If neither of these attempts succeed in producing any yield, Swim is considering trying another hypothetical bromination by producing a few grams of Br2 from KBr (with H2O2 and H2SO4 or with manganese oxide), and then carefully (FUMES!!) evaporating off the DCM to get pure liquid Br2, which he will quickly put into sol'n in some GAA (in prep for addition to the freebase amine in GAA). This is a bit more sketchy for Swim since heating up a sol'n of Br2 doesn't sound too fun, but Swim is seriously wondering if the DCM is fucking up his dreams.

Can anyone here help Swim have nice dreams??? He can't seem to figure out what is going wrong here!

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:32 PM.

10-14-2005, 03:32 AM

#**12**

DrugPhreak
Silver Member

Join Date: Jan 2005
Location: Cook Islands
Age: 64
Posts: 769
🟩🟩🟩

Hhhmmm... SWIDPwould have to say thatsomehow enough water got in thereto dissolve allthe2C-B.HBr. The DCM shouldn't be an issue since we are now dealing with2C-H as the Acetate.Why not just brominate the 2C-H HCl and thenA/B? The yield would bee better too.

____
No thing is without poison. Dosage makes it a poison or a remedy.

-Paracelsus

10-14-2005, 07:31 AM

#**13**

Eirias
Platinum Member

Join Date: Jan 2005
Location: United States
Posts: 485
🟩

Although the HBr salt is much more H2O-soluble than the HCl, Swim isn't sure where water would have come from, since he added the Br2 in DCM to the 2C-H in GAA. The 2C-H acetate did precipitate before going back into sol'n, so the acetate was clearly formed. Where would the H2O have come from?

So since Swim already presumably brominated the 2C-H (although no precip formed after addition and stirring, an orange colour did develop), are you suggesting that he now basify the mix (NaOH sol'n), extract with an NP (toluene?) and then precip the HCL with muriatic as per SWIIceCool?

Also, would it be prudent for Swim to attempt to drive off the DCM (and possibly also any H2O that might be present) before doing the A/B, and also possibly to first see if any precipitate then forms? Swim thinks it wouldn't hurt...

I appreciate your observations, DP. Lemme know what you think.

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:33 PM.

10-15-2005, 04:24 AM

#**14**

DrugPhreak
Silver Member

Join Date: Jan 2005
Location: Cook Islands
Age: 64
Posts: 769
🟩🟩🟩

SWIDP would put it in the freezer for a while and see if anything precipitates. If not, then they would continue with anA/B.

____
No thing is without poison. Dosage makes it a poison or a remedy.

-Paracelsus

Quote

10-15-2005, 07:18 AM

#**15**

DrugPhreak
Silver Member

Join Date: Jan 2005
Location: Cook Islands
Age: 64
Posts: 769
🟩🟩🟩

Some info from the Hive backup for brominating the salt directly...

About 0.55g of grayish-white (not the cleanest stuff you ever saw) 2CH HCl was placed in a small beaker and swirled with about 2mL glacial HOAc. Not everything dissolved at first. About 1mL of glacial HOAc with about 0.5g Br2 dissolved in it was then added, and all remaining solids dissolved. The beaker was covered with foil and swirled, the contents began to heat up noticeably. Within about 10 minutes, everything was a tan slush.

The contents of the beaker were allowed to return to room temp, and the foil was removed as SWIM coughed and choked on HBr fumes.... The contents were then triturated with anhydrous ethyl acetate until all color disappeared. SWIM once read a long time ago that somebody thought ethyl acetate was an ideal solvent for cleaning up the tan 2CB HBr formed from the bromination, and *they were VERY right!* Without any extensive cleaning procedures, only trituration with anhydrous ethyl acetate, SWIM managed to get about 0.60g of pure white, fluffy 2CB HBr.

Of course thiswill give some 2C-B HCl also.

____
No thing is without poison. Dosage makes it a poison or a remedy.

-Paracelsus

Last edited by Alfa : 08-26-2006 at 04:33 PM.

Quote

10-16-2005, 10:36 AM

#**16**

Eirias
Platinum Member

Join Date: Jan 2005
Location: United States
Posts: 485
🟩

Yep, the freezer was the 1st idea Swim had-- alas no precip.

Interesting, the Hive notes above basically follow the procedure on Rhod for the "Large Scale Bromination". Swim wonders if this makes a difference in terms of yield.

Swim is very grateful for your feedback and your note-sharing, SWIDP. He will let you know what becomes of his lil' dream...

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:34 PM.

Quote

10-16-2005, 11:07 PM

#**17**

DrugPhreak
Silver Member

Join Date: Jan 2005
Location: Cook Islands
Age: 64
Posts: 769

Rhodium statedthat the¹H NMR datareceived showed that this procedure gave the purest product...

http://www.erowid.org/archive/rhodium/chemistry/2cb.brominat ion.html**Edited by: DrugPhreak**

____
No thing is without poison. Dosage makes it a poison or a remedy.

-Paracelsus

Quote

10-16-2005, 11:23 PM

#**18**

daeron
The Platinum Post Whore
Platinum Member

Join Date: Dec 2004
Location: Slovenia
Posts: 1,184

in the past swim used the non insitu bromination method, and had the luck to test it via HNMR,the result was less than 1% of impurities,mainly 2ch.swim already stated that hes very against the insitu method elsewhere.furthermore the elemental bromination produced a product of high purity(after the washes,of course) very little discoloration,lazyass swim never used recryst,except once w ipa.eirias swim wishes swiy the best of luck but swim really recommends the elemental Br next time...as for ices way..well w no disrespect to him or anything but swim did advise the elemental Br to him as well and if swim remembers well he didnt actually have the best of the yields w his work now did he?

____
...perche io so io, e voi non siete un cazzo!

Quote

10-17-2005, 07:43 AM

#**19**

Eirias
Platinum Member

Join Date: Jan 2005
Location: United States
Posts: 485

Thanks SWIDaeron and SWIDP. Yeah Swim removed the
DCM/H2O with heat (water bath w/ stirring) and stuck it in the
freezer-- he was excited at first as some non-GAA looking crysts seemed
to have formed, but alas they quickly dissolved back into sol'n.

So now Swim is thinkin' he's gonna bite the fuckin' bullet and
oxidize a bunch of KBr, then strip off the DCM (unlike SWI-IceCool
stated, performing the KBr oxidization under acidic conditions without DCM does
not allow the pure Br2 to just "drop out" of sol'n on the bottom of the
mix--
Swim tried it and NOTHING came out, just an assload of Br fumes -cough
(!!!)- and some bromine water that seemed to be getting rather
saturated... and Swim thought that Br2 was only sparingly soluble
in H2O-- WTF??) He will then CAREFULLY strip off the DCM to leave pure Br2,
and hopefully even under heat (which makes the already-volatile Br2
even more fucking volatile and fuming! Fun!!), after the DCM is
all gone Swim'll be left with a hypothetically useable amount of pure
liquid elemental Br2. It'll be smelly, but at least then he won't
be dealing with any unknown variables this time.

And as for the stuff Swim imagined he already tried to brominate
ala SWI-IceCool, he supposes an A/B work-up is in order, even though no precip ever appeard at
room temp! This will (hypothetically) bee done as the HCL.

(fingers crossed and gasmasks on...)

Thanks so much for all your observations! Please continue to put your 2 cents in on the matter!

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:35 PM.

Quote

10-17-2005, 08:40 AM #20

pharmapsyche
AKA Miss Methylene
Silver Member

Join Date: Oct 2005
Age: 20
Posts: 375

That is an amazing post. It's very much appericated. You must be quite intellegant....AND what a wonderful substance.

Quote

10-17-2005, 05:56 PM #21

daeron
The Platinum Post Whore
Platinum Member

Join Date: Dec 2004
Location: Slovenia
Posts: 1,184

just a thought-maybe this path really,really,really....really sucks after all, depending on the amount of h2o2 used this may be expected as well:

(1) H2O2 + 2KBr + 2H+ -->Br2 + 2K+ + 2H2O (plus the Ac-,and theres your water)

and if theres extra H2O2:

(2) 3Br2 + 3H2O2 --> 6HBrO

and than maybe even:

(3) 3HBrO --> 2HBr + HBrO3

(4) 2HBrO--> 2HBr + O2

okay swim hasnt done a redox rxn on paper in eons so he hopes he hasnt made some embarrassing mistake buthes pretty sure that the (2) rxn is possible under the rxn conditions(something of a lingering memory)

swim would recommend to read up a bit on a KBr oxidation to Br2 via MnO2 and H2SO4,swim has done that in ancient past(1st yr uni,so dont expect any detail,sorry).and again water is formed so swiy will basically end up w a Br saturated, slightly acidic water.**Edited by: daeron**

____
...perche io so io, e voi non siete un cazzo!

Quote

10-18-2005, 01:17 AM

#22

Join Date: Jan 2005
Location: United States
Posts: 485

Eirias
Platinum Member

Yeah Swim is seriously considering looking at the manganese oxide route, since using ether instead of DCM the Br moves to the ether layer and forms a sort of pool floating on the ether.
Ether is bitch for Swim to get...

SWI-Daeron, do you remember in the redox with MnO2 if you got straight elemental Br2 somehow, or if you used Et2O, and if so did the bromine float on the ether's surface.

There's definitely some factors that are unknown in the KBr redox with H2O2 and H2SO4-- Swim just dreamed of trying it scaled up 10x from what IceCool suggested above, and there's no question that some of the Br went into the water, but with vigorous agitation, the DCM layer took on a dark red colour, indicating obviously it had absorbed a good deal of bromine.

Swim is rather nervous about driving off the DCM to get the remaining elemental Br2, especially since the fumes from the redox were bad enough, and Swim has no closed system vac distillation set-up at present to contain the fumes. He is thinking he'll use a water

bath and a fan set on 'high' in a well-ventilated area with something
to cover his face. Swim purposely made enough Br2 in the DCM so
that even if almost half of the Br is lost from the heat, that enough
will remain for his hypothetical experiments.

If this dream is an utter disaster, Swim will pretend to attempt something with the MnO2.

Swim appreciates all the feedback he's getting, and hopes that
his silly little dream adventures will serve to help other researchers
in the future whom might be interested in this particular route.

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:36 PM.

Quote

10-19-2005, 01:18 AM

#**23**

Join Date: Jan 2005
Location: United States
Posts: 485

Eirias
Platinum Member

UPDATE:

It is of Swim's opinion that the KBr redox with DCM is a horrible
way to produce bromine. There is no way to get the bromine out of
the DCM, as it (Br2) is so fucking volatile that any attmepts to
distill the DCM off to leave pure Br2 are entirely futile.

Swim is very frustrated at how oversimplied this procedure has
been portrayed. There are many discrepancies and what not
throughout the process, and especially in regard to the KBr oxidation,
many presumptions were made which are simply inaccurate. This has
been very very time-consuming for Swim, but he is still happy that at
the very least his discoveries may help others not waste time.

To clarify:
GAA and DCM are miscible-- i.e.they mix, they do not form two seperate layers.

If no DCM is used in the KBr redox, the elemental Br will not simply "drop out" of sol'n.
Rather, one will end up with an aqueous layer supersaturated with
bromine that will give off choking vapors until it stabilizes.

If one attempts to reduce the DCM/Br2 layer in order to be left with just elemental Br2, this will
not work.
The volatility of the Br2 will prevent it from being able to be
isolated, and it will vaporize at the same rate as the DCM
evaporates. Swim tried this with a 25 gram sol'n of bromine in
200 ml of DCM, and was left with NOTHING in the end. (This was
done with low heat under a water bath that never even got close to
boiling hot.)

Finally, it is questionable if via following the procecdure
illustrated above of in the bromination step ANY xtals will form.
Swim is not suggesting that this process will not allow the 2C-H to be
brominated at the 4-position, but he is speaking from experience when
he points the the xtal issue.

Swim's last recourse with the material he dreamed of processing

according to the above method is to perform an A/B and see if he gets
any yield as the HCl. He is rather skeptical at this point, but
for the sake of dream science he will continue and report back his
imaginary findings.

Swim hopes this will help anyone whom might be having 2C-B dreams anytime soon.

____
\'tis an il wind that blows no minds

Last edited by Alfa : 08-26-2006 at 04:37 PM.

10-19-2005, 01:42 AM

#**24**

daeron

The Platinum Post Whore
Platinum Member

Join Date: Dec 2004
Location: Slovenia
Posts: 1,184

ahh where would swim bee if it wasnt for his smallbut paranoid habit to periodically backup hismulti GB archive(swiE can find the MnO2 ox data here):2005-10-19_014145_bromine.rar

____
...perche io so io, e voi non siete un cazzo!

10-19-2005, 02:19 AM

#**25**

Prince_Charles

Banned

Join Date: Oct 2005
Posts: 20

stratosphere
(Hive Bee)

12-20-03 17:27
No 477998

bromine **prep reaction**

I noticed in the inorganics chemical index there is no procedure for the prep of liquid bromine, so I thought I would contribute the method I have developed and “perfected” for possible inclusion in the index.

liquid bromine from KBr]
Add 85ml 20% H202 (0.5mols h202) to 100ml of dH20.

To this add 60g KBr (0.5mols) and stir till dissolved.

(Will require a lot of stirring. Since KBr is hygroscopic you may need to oven dry it beforehand to ensure proper weight)

once the KBr is dissolved in the H20/H202 slowly add 80ml 12M (30%) HCl (1mol HCl).
At this point Br2 will form and the solution will turn reddish, some (mildly irritating) Br2 fumes will evolve so be cautious.

Other
concentrations of H202 and HCl will work as long as the correct number

of moles are used, although I suspect very dilute forms would give a
poor yield.

At this point setup for distillation, I have attempted to extract the bromine from the water with DCM but this was not very effective.

Distill off the bromine by holding the temp of mixture at ~85C for about an hour until the red Br2 can no be seen condensing. The water will probably have a slightly
yellow turbid appereance even after most of the bromine is removed. I recommend chilling the reciever in an ice bath while distilling to reduce fuming.

To dry the condensed bromine pour it in a small graduated cylinder. Use a pipette to add about 1 ml
of conc. H2S04, mix gently with pipette tip. When H2S04/H20 de-emulsify
and float to top use pipette to remove them.

Yield: 10ml Br2 = 31g Br2 = 0.4mols Br
Percentage Yield = 80% relative to Bromine

Rhodium
(Chief Bee)

12-20-03 17:51
No 478003
BrCl ?

I would **strongly** suggest to chill the recieving flask in an ice-bath while distilling.

Also, as you acidify with HCl rather than H2SO4, what prevents the formation of BrCl - thermodynamics?

The Hive - Clandestine Chemists Without Borders

stratosphere
(Hive Bee)

12-20-03 18:02
No 478006

actually i did chill the reciever while ...
actually i did chill the reciever while distilling, will edit accordinly.

the yield was 80% so maybe some of the missing portion was BrCl which boils at 5C.

ive tried h2SO4 but noticed it was catalyzing the decompostion of h202. whether this
was due to impurities in it (it was tech grade), or is intrinsic i don't know.

so i guess it would be neccasary to hold the Br2 at a little over 5C to allow the boil off of BrCl, or to use H2S04 and
deal with whatever decomp it causes.

Last edited by Alfa : 08-26-2006 at 04:44 PM.

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