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29/12/2008 21:47 pyroarchy: yea i have 2 friends wit degrees having hard time finding jobs :/
29/12/2008 21:42 Samadhi: i have no money because of school... which is supposed to get me money... irony sucks like no other
29/12/2008 21:33 pyroarchy: heheh i saw fargo on few channels back
29/12/2008 21:33 pyroarchy: living check to check is no fun at all
29/12/2008 21:32 pyroarchy: indeed samadhi

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25-09-2008, 13:46 #1

headstrong
Silver Member

Join Date: 02-12-2007
Location: earth
Posts: 306

Further about Methampetamine via H3PO3

He really impressed by **H3PO3** route to Meth, H3 is cheap-OTC as a salt, tame reaction, and after run some rxn he found that it's provide a high purity yield. Low percentage of unreacted PSE and Iodo-Meth, very low if doing right. (Thanks to SwiX and SwiBF for assisting).

He wanna try to dig deeper, since just a little bit info is already available, some missing links about rxn equi, how to maximalize the yield, posh reaction,....

So, he wanna share what he find, and hope any Swimster would give advices, answers, assisting, many thanks for that.

He start by flask temperature,
To avoid confusing;
about flask temperature during rxn, endothermic-exo-final phase (thread #18 in "Regarding Methampetamine via **H3PO3**"). Cause those phases ever talked few times in newbie chem forum, he want to remind;
The exothermic was happened in certain condition;
- when HI made, he heated by a low temperature; 70 c oil bath, a lot of H3, it's take couple hours.
- the ratio of PSE : I2 : H3 = 1 : 1,25 : 1,45
- And 0,7 ml dH2O every g of **H3PO3**
It was a very thick solution, that mean it has a high boiling point, and (may be) dehydration of PSE will become easier-lower yield.

In best ratio of PSE : I2 : H3 = 1 : 1,2 : 1,25 - 1,5 (technical grade I2, H3);
1. after flask temperature reach certain degree c, temp will be relatively constant although oil bath temp is heated up. That's the BOILING POINT of the rxn solution. If boiling point=126 c; when flask temp = 126 c, oil bath should be around 141 c. Flask temp will stay at 126-127 c although oil temp raise to 160 c. So, the completion phase (all PSE has been converted to Meth, flask temp = oil temp that was happened in his first rxn mentioned above) won't happen in regular (best) ratio of PSE : I2 : H3.
SwiXtaldoc ever promise to dig about it, but if he don't it because SwiHS's fool, his data base on uncommon ratio.

2. flask temperature can use as a tool to determine what's wrong in pre-rxn and to decide what should do during rxn.
- He found that 126 c is the boiling point for 1 PSE : 1,2 I2 : 1,3 **H3PO3** (crystal form, trace water).
- if rxn solution has boiling point 121 c, precise amount of each prec, right ratio, big possibility it cause too much water contained in **H3PO3**.
- if too much amount of d-water is the problem, flask temp=boiling point will be under 126 c. He found that 123 c is ok, rxn can continue - better if additional rxn time is added, but if it's temp 121 c or below better stop the rxn and add some **H3PO3**, this work.

3. when rxn is complete, he found 2 things;
- flask temp/boiling point is slightly decrease, 0,5 c max. He think it cause some water is created (by PSE reduction)
- "white mass" volume (in flask bottom) is increase, can make bumping. He'll post latter about this 'white mass' (question, opinion).

4. boiling point can increase 1 c (126c when oil temp=141 c to 127 c when oil temp=160), because rxn solution absorb energy from the excess temp to evap, it's make more water change to gas and the rxn solution become thicker=higher boiling point. He don't know how many degree c (the difference between flask and oil temp) is the best, reminding of PSE dehydration-azziridine and if it's has a contribution in complete conversion.

Quote

25-09-2008, 18:55 #2

Spyder
Palladium Member

Join Date: 08-04-2008
Location: USA - Eastern
Posts: 448

Re: Further about Methampetamine via H3PO3

Just a few observations of swiS's **H3PO3** Rxn:

1. He uses a ratio of : 1 PSE / 1.5 I2 / 1.75 H3 / .75dH20
This ratio seems to make a larger "Excess" of HI. Only issue is during the post rxn, this 'excess' takes more to neutralize/base , the rxn fluid is much more acidic.

2. SwiS ALWAYS gets that 'white mass' - First he thought it was KCl from the H3 Extraction? (Doubtful, as extraction was very precise). Maybe some type of Iodine salt formed w/ intermediate of the H3 rxn ? KI? (If some K is 'hiding' in the H3)

*Question: Does swiHS get the Mass if 'lab grade' **H3PO3** (NOT extracted) used?

SwiS is never able to 'test' the white mass - when the rxn. is diluted w/ dH2O after rxn, the "white mass" goes into the solution. Maybe filter first????

3. SwiS rxn temp is always 125-130C INSIDE RXN TEMP. Bath temp. 160C (+ / -).
SwiS saw only very very little change in temperature (inside) throughout the rxn. His rxn held temperature very stable during 10-12 Hours.

SwiS is interested to hear opinions about the white mass? Also, what swiY think about swiS's ratios?

S

Quote

27-09-2008, 13:35 #3

headstrong
Silver Member

Join Date: 02-12-2007
Location: earth
Posts: 306

Re: Further about Methampetamine via H3PO3

Quote:

> 1. He uses a ratio of : 1 PSE / 1.5 I2 / 1.75 H3 / .75dH20
> This ratio seems to make a larger "Excess" of HI. Only issue is during the post rxn, this 'excess' takes more to neutralize/base , the rxn fluid is much more acidic.

For what? faster, fully converting or...?

Quote:

> *Question: Does swiHS get the Mass if 'lab grade' **H3PO3** (NOT extracted) used?

he sure YES.

Quote:

> 2. SwiS ALWAYS gets that 'white mass' - First he thought it was KCl from the H3 Extraction? (Doubtful, as extraction was very precise). Maybe some type of Iodine salt formed w/ intermediate of the H3 rxn ? KI? (If some K is 'hiding' in the H3)

For now he just can say that it's not KI, much KI will make rxn bumping since when rxn boiled. He plan to post about it and PI3(? in posh rxn)

Quote

27-09-2008, 17:16 #4

Spyder
Palladium Member

Join Date: 08-04-2008
Location: USA - Eastern
Posts: 448

Re: Further about Methampetamine via H3PO3

"Why Excess?" Honestly, the only reason is that swiS started doing it that way, and just stuck to it since it worked well. He originally just wanted to be positive of the HI creation since all of the chems were extracted (No lab grade chems used). He was also a bit worried about the water content in the **H3PO3**. He did get a 'waxy solid' but it only became waxy after apx. 1hr in the freezer. Prior to that it was a very very viscous liquid. He believes it is only a very very small amount of water, probably could be dried easily w/ a simple IPA wash, but as the old guys say, "If it ain't broke, don't fix it" SwiS rxn works ok, so no changes for now. (It is a bit wasteful on the H3, so possibly he can lessen a bit?)

Next time, he will try to get some of the mass out prior to diluting the rxn fluid....

Best regards....Sweet Dreams....S

Quote

02-11-2008, 12:33 #5

headstrong
Silver Member

Join Date: 02-12-2007
Location: earth
Posts: 306

Re: Further about Methampetamine via H3PO3

FURTHER ABOUT H3PO3 EXTRACTION-CONVERTING
He just add infos and experiments report, Basically is the same with extraction in this threads, ; http://www.drugs-forum.com/forum/showthread.php?t=58683
Since phosphite fungicide is inexpensive, the main issue isn't high yielded. High purity and fast are. Of course wasting anything useless is avoiding. A 'recycle bottle is needed, wash by water the glass ware, filter, etc and pour to this bottle, then SwiY can re extract it or convert to Potassium salt again (can use as a drying agent for IPA and EtOH, reuseable drying agent).

Here the extraction;
1. Purifying of potassium phosphite;
to remove some water and all inactives impurities (that have a lower solubility than p phos)
- 1 l of fungicide (he use the blue one, contains 400g potassium phospite / 1l fungicide), boil till saturated.
- IPA wash; when the solution is saturated (in room temperature), it won't dissolve in dry IPA. If not, some of this solution will dissolve in, and swiY will loose it.
-in saturated solution, all impurities that has a lower water solubility than p phosphite will percipitate out, it's make IPA can wash it perfectly.
-if swiYou fungicide contains other impurities than just (p phosphite and water); *wash by IPA properly when it still a salt, in his case he wash it till this solution water clear.* If swiY wash it when has been converted to **H3PO3**, will loose many of H3, since it has a great solubility in water, hard to make a saturated H3-water solution. Not saturated make IPA able to chew few water, and in this few water many H3 is contained. But it still needed to do.
-dry IPA is preferable, if water contained IPA is used make sure that this solution is over saturated, some p phos will percipitate out in room temp and act as a drying agent.
- filter if look foggy
2. Converting to H3PO3;
-heat/boil this p phos solution till saturated (in hot temp).
-add HCl to this solution, stir well till all p phos is converted to H3, many KCl will percipitate out.
-HCl volume needed to convert 400 g p phos;
a. determine by check the pH, HCl pH = 1, **H3PO3** pH=2, so add HCl till pH very very 1, give enough time and stirring for that, reminding that this reaction isn't as vigorous as acid + base reaction. Re check after several minutes to ensure.
b. phosphite fungicide come as a mix of KH2PO3 and K2HPO3 with unknown ratio. If it contains KH2PO3 only, for 1 l fungicide (400g p phos) need 370 ml of 32% HCl. If K2HPO3 only; 560 ml 32% HCl. So, *HCl (32%) volume needed is 370 ml min to 560 ml max.*

headstrong added 1344 Minutes and 52 Seconds later...

3. purifying of H3PO3;
to remove all water, all KCl, and remaining impurities (that have a greater solubility than p phospite), so if impurities that have lower and greater solubility than potassium phosphite are removed, **it's mean a reagent great H3PO3 is obtained**.
- lets p phos solution cool and bring to freezer for 1 hour or more, filter,
- heat it till some KCl percipitate out, lets cool, bring to freezer again, filter, repeat this step till no more KCl percipitate out. Will look like a viscous syrup.
note; heating H3 should be under 100 c to avoid oxydizing (by air) and do not ever heat it in 180 c coz H3 will decompose to deadly poisonous gas Phosphine!
- if the H3-water solution is **saturated** and no KCl percipitate out, it's mean this solution is free from KCl. But H3 has a **great solubility in water** (freely soluble) and has a low melting point

74 c (165 f), that are make almost impossible to get saturated H3 only by heating under 100 c. After a viscous syrup is reach, no more water can evaporate, no water condensed at the glass, this trick ("alcoholising out "the KCl) can do; add little IPA (around 1/4 volume), stir well till this solution is soluble in IPA, let sit, if no KCl perc out then repeat this step till some white mass is perc out. He calls white mass because it may be KCl or H3 (determine by take it a little bit, and try to dissolve (stir) in saturated KCl-water solution, KCl won't dissolved, H3 will dissolved easily). If it's KCl, bring to fridge for 1 h, test again, repeat till H3 is perc out, bring to freezer 15-30 minutes each time if needed. Filter, **KCl FREE!**

He add; 1. H3-IPA solution is easy to filter than a viscous syrup H3.
2. After freezing H3PO4 will left in IPA since H3PO4 soluble in alcohol.

Last edited by headstrong; 20-11-2008 at 14:19.. Reason: Adding info

Quote

07-11-2008, 11:28 #6

headstrong
Silver Member

Join Date: 02-12-2007
Location: earth
Posts: 306

Re: Further about Methampetamine via H3PO3

- **removing water from H3;**
> freeze that solution over night, longer and cooler are better.
> H3 crystal will sink, discard solution.
> wash by dry IPA (this time H3 won't soluble)
> heat to evap remaining IPA, remove from heat source, quickly close this beaker glass by plastic bag/cling wrap.
> let's cool, **DRY SOLID H3.**

Quote

17-11-2008, 13:08 #7

headstrong
Silver Member

Join Date: 02-12-2007
Location: earth
Posts: 306

Re: Further about Methampetamine via H3PO3

Other way to get dry H3 (from solution in thread #6);
1. add Xylene, stir, add MEK , stir well. Freeze, H3 crystallized.
2. using a drying agent; add few baked Na2SO4, shake well, repeat till some H3 perc out, decant solution to another plastic bottle. Repeat this step, unhydrous H3 perc out. Decant/filter solution, heat H3 to remove IPA, heat till 105 c, filter by plastic filter.

Quote

21-11-2008, 11:01 #8

headstrong
Silver Member

Join Date: 02-12-2007
Location: earth
Posts: 306

Re: Further about Methampetamine via H3PO3

H3PO3 / I2 rxn
What he write below is basic on personal research, some advices, infos are needed.

Rxn Equilibrium :

H3 route is "half" of RP route, very simple as below. This tame reaction is much faster then RP route, he guess because H3 rxn solution much more homogeneous.

H3PO3 + I2 + 2 H2O -> 2 HI + H3PO4
HI + E-OH -> E-I + H2O (or ; 2 HI + E-OH --> E-I + H2O + HI)
E-I + HI -> M + I2

headstrong added 1191 Minutes and 41 Seconds later...

rxn 'recycling' equi ;
I2 that was produced during rxn will be recycled by H3 as like above ;
H3PO3 + I2 + 2 H2O -> 2 HI + H3PO4

rxn 'minor' equi ;
H2O stripped by HI, HI will prefer to strip OH ring from PSE/E since it's more reactive/unstable.

Some conclusions from this equi ;
For simplify let's divide this rxn become 3 ; first run (make HI), second run (PSE reduction) and posh rxn.

1. First run (**H3PO3** + I2 + 2H2O --> H3PO4 + 2HI); some water is used up to make HI = 14,17% x I2 weight. Eg; if I2 = 12 g, **H3PO3** = 15 g and H2O = 15 ml, then 1,7 ml H2O is used up. The presence of HI as a solute will make rxn solution become thicker, then the "white mass" will percipitate out when rxn solution cooling down. The **'white mass'** that appear after HI is made is **H3PO3** more to H3 than H3PO4 since the melting point of H3PO4 is 21 c and H3 is 74 c.
2. Just opinion; for one mol of PSE/E need 2 mol of HI >>> meth, say give 3 mol HI(aq) for every mol PSE without Phosphorous, run for certain hours and in certain degree c. Meth should be produced. He ever read E/PSE + I2 + RP in 50 c for 2 week, some is converted to meth.
3. basic reduction rxn is ; E-OH + HI --> E-I + H20 then E-I + HI --> M + I2.
Overall ; E-OH + 2HI --> M + I2 + H2O. It's mean 2 mole of HI is needed to convert 1 mole E/PSE.HCl to one mole of Meth. In weight ; HI needed = 78,9 % PSE weight. Eg ; 789 mg of HI is needed for 1 g of PSE. HCl. E amount of HI needed. I2 needed = 0,783 % PSE weight. So by ratio I2 : PSE = 1,2 : 1 , amount of HI (from first run) actually has been enough for total conversion, without HI from I2 recycling (in the second run). So, recycling process (I2 + **H3PO3** + 2H20 --> 2HI + H3PO4) functions are not to provides enough HI but for ; #to decrease rxn time needed (more hydrogen available). # avoid too much water in rxn solution (by ; use H2O that's produced when E-OH convert to intermediate E-I). # and sure as a cleaning service, keep rxn solution free from insoluble substance I2.
4. water, like mentioned above; in the first run H20 used up = 14,17%(xI2 weight). Second run (PSE reduction) 7,085%(xI2 weight) will be produced and 14,17%(xI2 weight), so **water amount in 2nd run is deficit 7,085%(xI2 weight)**. Water will use up total (1st run + 2nd run) = 21,255%(xI2 weight). It's also mean if more I2 given more water used up, rxn solution

become thicker if water amount stay on the ratio.
5. **H3PO3**, in the 1st run 1 mole of H3 is needed for 1 mole of I2, H3 needed = 32,23%(xI2 weight). In the 2nd run equal weight is needed = 32,23%(xI2 weight). Total H3 needed = 64,46%(xI2 weight). So, ratio I2 : PSE : H3 : H2O = 1,2 : 1 : 1,2 : 1,2 has provide enough H3, since H3 ratio needed (base on equilibria) = 64,46% x 1,2(I2 ratio) = 0.775, **no reason to upgrade the H3 ratio**. More I2 >> more H3 needed >> more water needed, then **why in H3 route the amount of water is depend on H3 amount instead on the I2 amount?** Hope it's not coz H3 is cheaper..., then I2 : PSE ratio is made static, H3 amount used as a key to play the amount of water. How if H3 stay at 1,2 and the "key prec" in H3 route is replace by I2 as how it should be?
6. H3PO4 is produced, total = 1,195 x H3 (used up) weight or 1,195 x 64,46%(xI2 weight) = 77,03 %(xI2 weight). After some rxn, H3 can be recycled/extracted.
7. during rxn, by time ; amount of water decrease, H3 decrease but H3PO4 increase, HI static, solution become thicker, a substance must be percipitate out-sink on flask bottom-make several bumping-suspicious as the enemy.... guess what is it (try to remind the melting point of each prec) ha ha ha ***they are definitely the sign of completion, the king of rxn, Meth it self!!***

Last edited by headstrong; 27-11-2008 at 13:51.. Reason: Automerged Doublepost

Quote

27-11-2008, 14:58 #9

headstrong
Silver Member

Join Date: 02-12-2007
Location: earth
Posts: 306

Re: Further about Methampetamine via H3PO3

8. during rxn some HCl are replaced by HI; M.HCl + HI --> M.HI, it's happen to E.HCl and E-I.HCl as well. Unexpected, since it's decrease HI amount. Fortunately these amines are a weak base so they prefer to HCl than HI, may be just 20% is replaced. The HI salt can be observed in posh rxn, the reddish oil float above the solution. if swiY add some lye, stir, add again, stir,...let sit, then this oil mass will much increase.
9. The enemy of this route is the same with I2/RP route, dehydration/ azziridines route. Rxn is more sensitive to the amount of water than to temperature. He ever tried use less water, twice. First as he posted in 'regarding meth via H3' thread, much unreacted PSE and IodoE, by low temp, flask temp 121 c max. Then he just tried an rxn of 33 g (PSE and E around the same weight), ratio PSE : I2 : H3 = 1 : 1,2 : 1,5 and 0,8 ml water every 1 g H3. Flask temp 127 to 130 c, 12 hrs. The yield is worse than before. So, he thinks this rxn is more sensitive to 'less water' than 'too much'.

OPINIONS ABOUT HOW TO INCREASE THE YIELD

1. 1 ml water every g of H3, is a min amount. This rxn is more sensitive to water amount then temperature, and *less water used* is more sensitive then *more water used* (base on common ratio).
2. oil level should be the same with solution level or slightly under. If not, in flask; between oil level and solution level will be the hottest and driest space, some E/PSE will go to dehydration route.
3. for small scale/under 30 g PSE, a 30 cm jacket long Liebig Condenser is sufficient enough, since this rxn is tame compare to I2/rp route, so other like Graham one may decrease the yield because of dehydration especially for under 10 g PSE rxn.

4. the ratio should be PSE : H3 : I2 = 1 : 1,2 : 1,2 - ? and 1 ml of water every g of I2. Play with I2, and may be for every g of I2 more than 1 ml water is needed. Considering ; - the enemy is the same with I2/RP route that is dehydration (azziridine route) - I2/RP route use little water but HI/RP use much water since 57 % HI is used. So, a wide range of water ratio is available, and rxn will still work. He believe H3 route can provide a 80% yield if the ratio is proven.

Last edited by headstrong; 02-12-2008 at 15:19.. Reason: to continue

Quote

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