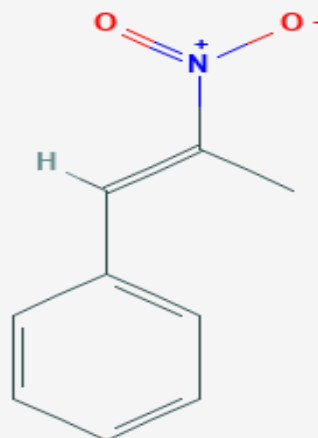
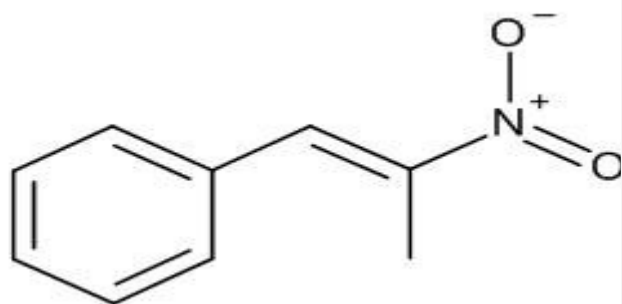


Amphetamine Sulphate Synthesis - 'Reductive Amination using Sodium Borohydride'

> a ketone and an amine > the route via NaBH₄/CuCl₂



THE Work Up-

> **C₉H₉NO₂ MP=63-65°C**

> **NaBH₄** (reducing agent) - **Sodium borohydride** (somebody say it's in excess because it's diprotic. Review the stoichiometry)

> **C₃H₈O IPA – 60,1g/mol – BP:82,5 °C -- d=0.786 g/cm³** solvant polaire miscible dans eau et alcool de faible masse molaire,

> **dH₂O**

> **CuCl₂ -134,45g/mol** - (oxydant) dichlorure de cuivre attention not to > that's way too much CuCl₂ for this reaction, you need a catalytic amount, probably 1 gram for every 10 of NaBH₄ - The value for copper chloride is based on anhydrous copper chloride, so make adjustments based on if you're using the dihydrate. Selon Wiki, on n'utilise le catalyseur que dans une proportion représentant généralement entre 1 et 20 % (en masse ou en équivalent) par rapport au produits de départ (quantité stochiométrique).

> **NaHSO₄ Sodium bisulphate**

> **NaOH hydroxyde de sodium**

> **CH₂Cl₂ DCM 84,93 g/mol** (d=1,33g/cm³ - solvant Non Polaire donc non miscible dans l'eau. Miscible dans acétone, ether et autres solvants)

> **MgSO₄ Sulfate de magnésium 120,37 g/mol**

> **AcOH Acétique acide 60 g/mol**

> **H₂SO₄ Acide sulfurique 98%**

What I do to recrystallize my P2NP -

- > Heat some IPA to boiling (2x the amount of P2NP just in case, use even less)
- > Put the glass with the P2NP on a hot plate and slowly add IPA while mixing well.
- If the solution is not too concentrated (not too much IPA I mean!) the P2NP will start forming its long needles as soon as the boiling stops.
- > After cooling slowly, put the solution in the fridge, then filter the crystals on a Buchner funnel.
- > Repeat cooling filtering to get a second crop.
- > If you want to get all the P2NP out, you can add a little water to make the IPA cloudy. All crystals will fall out immediately...

Can be recommend first filtering the catalyst (can be reused several times btw.), wash catalyst with alcohol, add enough saturated potassium carbonate solution to salt out an amine/alcohol layer, separate phases, distill the alcohol off, then directly water-jet-vacuum-distill the clear colorless product.

By the way, it is in my experience, important to powder the borohydride before really well, this works much better this way.

Also, I usually use a bit more IPA to get everything well into solution, and fast enough too.

add grams substance 1 Mol :)

0.1 M 16.3 gr Nitropropene 163.17

0.75 M 28.35 gr * Sodium borohydride 37.8

0.1 M 13..45 gr CuCl₂ 134.45

> (in 480ml) solvent 1:2 IPA/H₂O > can be *warm*ed *seperately* to 40°C

* can be reduced to 6x mol-eq. / 227 gr NaBH₄ per 163 gr p2np (37,8*6=227)

(strong overhead stirring switched on and not shut down till end)

1-phenyl-2-nitropropene (16.3 g, 0.1M) is added in portions to a stirred mixture of Sodium borohydride (28.35 g, 0.75*M) in 320 ml IPA and 160 ml dH₂O, which increase the temp to 50-60°C.

* Some reports better yield when nitropropene being add in portions and do it fast to increase the process. Comment said : the complete dissolution of the P2NP being of utter importance. The solution has to be completely CLEAR no turbidity. If it does not get clear yor P2NP is dirty.

* Pensez-y. On ajoute de l'eau dans le mélange réactionnel, un mélange d'isopropanol et d'eau, auquel on ajoute le borohydrure. Donc évidemment, si on utilise la forme anhydre, elle ne restera pas anhydre. Cela ne devrait pas avoir d'importance, sauf peut-être de légers effets dus à la modification de la teneur en H₂O, afin d'hydrater et de solvater le CuCl₂.

* IPA > You will want to use 15-20 ml/g of either solvent, enough solvent so the solution turns clear. You should be able to see through the flask and it should not be cloudy. It sounds like a lot of solvent but trust me, your recovery after cooling should be around 90%. If you don't add enough solvent to form a clear solution your crystals will be ugly.

Thereafter a 2M solution of Copper(II) chloride () (50 ml, 0.1M) is added dropwise causing further heat increase.

The reaction is then held at 80°C for 30 min by external heating, then let cool to RT.

Boil ?: someone said as hard as possible to drive all shit out. This removes by steam-distillation all the stuff which steamdistills and is not a salt at this moment like amphetamine ,

The reaction is quenched with NaOH.

When solution is RT > IPA distill off. - Product steamdistilled rather slowly and not pushed at all for that's another key to quality.

(sometimes Sodium carbonate and water can be added until no more fizzing (CO2 formation) because that's much softer on the product and no insane amounts of NaOH needed)

add with stirring 20 ml of 25% NaOH solution to pH 14, and the phases separates (with amine base in the aqueous phase)

now stirring is shut down

The aq phase is extracted 3 x 30 ml DCM.... [patent say IPA], the extracts combined and dried with MgSO4, then filtered (the amine is a liquid that will dissolve in most organic solvent).

Distill off solvent yield amine ca 70% (ca 9.5 grams freebase)

Cristallisation-

1:5 IPA and 96% H2SO4 is measured up, put this mix in freezer 15-25 min.

One mol amine freebase (135 gr) is mixed with 375 ml IPA, also put in freezer.

The freebase solution is now placed in an ice bath with stirring at temp 0-10°C, the acid mix/IPA mix is then slowly dripped in with stirring until pH6, let stir another 30 min, then put in fridge,

filter, re-crystallize with dry acetone to get cleaner product.

9.5 gr freebase. By using the freezer & fridge during crystallization you get 5-10% more final product. 9.5 gr freebase gives 12 gr sulphate.

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Comment :

Quench/Acidify with acetic acid (mixture usually clears up), strip the alcohol properly - until vapor is 100 C and then keep on distilling a while longer. Switch receiving flask to a new one. Let mixture cool off and then basify with an alkali

hydroxide, until strongly basic (oily mess will come out of solution). Perhaps add extra water. Then distill away. When the oil co-distills with the water, inner surface of glass and condensing liquid will take on a milky appearance. When it's not milky any longer, keep distilling (perhaps 30-60 minutes?). Hopefully you will now have a receiving flask with an upper layer of oil. Pour entire contents of receiving flask in a sep funnel. You can separate the distinct oil layer first if you want to, but either way you should extract 3 times with for example iPrOH or toluene. You might want to add some KOH/NaOH and/or brine, if using iPrOH you definitely should do that. The combined organic phases are dried (for example with MgSO_4) and the drying agent filtered off into a dry flask.

Put that flask in the freezer until decently cold. Put it into an ice bath and put it on a stirrer with a stir bar in the flask. Add concentrated sulfuric acid, dropwise with good stirring. Sulfate salt should be very evident within a short amount of time after adding the first few drops. Measure the pH as often as you need *use pre-wetted pH-paper*, but naturally more often as the pH approaches neutrality. *Don't overshoot!* Even just one drop too many of conc. H_2SO_4 can mess it up pretty badly, IME a thick paste can become a loose slurry that is not overly filterable. Filter off your salt, wash with acetone and then a dash of DCM. Let it all dry in a pyrex dish or similar, then crush lumps up and let it dry more. Voilá!

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=FROM Vespiary

I would recommend to get used to the process and take like 5-10ml of the freebase, dissolve that in 10x acetone and add a 10% H_2SO_4 in IPA solution to it, very slowly due to it heating up of course, and and especially slowly in the end when you have almost reached neutrality.

Amphetamine sulfate does not form instantly, so it can happen that you measure a pH of 7, but two minutes later when you measure again, you see it is now acidic and not neutral anymore.

Because of this, be especially slow when you get near this point.

But you should also put a little bit of the freebase in acetone away, to correct the pH if you overshoot on acid and need to readjust to a higher pH.

Amphetamine sulfate is typically precipitated from an alcohol solution of the base using H_2SO_4 . If you save the filtrate and alcohol washings and later distill off the alcohol you'll find you get some amphetamine sulfate which can be purified by A/B extraction and vacuum (or even 1 atm) distillation of the free base. If you used the Leukart reaction on P2P you might find the amphetamine sulfate in the filtrate to have the consistency of peanut butter, but the (first) ppt. is white. After washing with alcohol and then made into a block to dry, a brown coloring forms on it which can be scraped off and the blocks stay white. Also, I know of someone who tried recrystallizing about 30 grams of amphetamine sulfate from water, but after concentrating and cooling the filtrate several times to get more ppt. until there was no water left he found that about 3 grams disappeared which must have evaporated with the water, so he decided to stick with ppt. from alcohol using

H₂SO₄, wash with alcohol, make into blocks, scrape off color after drying and also saving the alcohol filtrate to recover a small amount (5%?) of the amphetamine sulfate.

Also, if you add too much H₂SO₄ to amphetamine base in alcohol the sulfate redissolves, so its best to add small amounts of H₂SO₄ at time, filter, then add H₂SO₄ to the filtrate to get more ppt., filter and repeat until there's no more ppt. Save the alcohol filtrate from several runs to get more amphetamine sulfate as described above.

Abstract-

The workup - is carried out else too, borohydride is quenched with AcOH as usual, IPA distilled off then, base added, so any copper complex is broken and it allows for simple steam distillation then to isolate the amine directly from the reaction flask.

I always re-distill the amine freebase to get ultra clean product +99%, without re-distillation of amine oil you get 90-93% final roduct which has the familiar unpleasant side effects like drier mouth, face tics, jaw clenching, lower sexuality, erection problems etc

The first is using sodium borohydride plus transition metal salts (nickel chloride or copper chloride). Usually, on its own, NaBH₄ will only break the double bond on a nitroalkane, forming dihydro-P2NP or dihydro-nitrostyrene. However, if you do the reaction with the addition of a transition metal salt as a catalyst, the resulting borate (liberated from the borohydride by water) will displace the chloride ion, yielding nickel or copper borate, which increases its oxidative state and therefore acts as an electron donor (thus it can donate more electrons to a substrate present in the solution). In the presence of nitrostyrene or P2NP this means it can completely reduce the whole thing in a one-step, thirty minute reaction to phenethylamine or amphetamine, requiring a relatively very small workup and time investment (compared to zinc/hcl). Yields are also pretty high, up to around 60-70 percent for ring-substituted nitrostyrenes (not as high as the reported 80-95% yields from zinc/hcl but still very good).

This valuable method, especially for nitropropenes. It has been tried by several people on 1-phenyl-2-nitropropene, with high success in 80-90% direct amine yield. Some report yields of +90%. Usually the use of NaBH₄ has been 6 to 6.5 mol-eq.

I can confirm from own experiments, that it is indeed possible to reduce a nitropropene into yields of above 80%.

The reaction is done more harsh, as the nitropropene stands much better to be added fast enough to cause the reaction being at constant reflux during this, and it even proved to increase yield.

<https://www.thevespiary.org/rhodium/Rhodium/Vespiary/talk/index.php/topic%2C3519.0.html>

<https://www.sciencemadness.org/talk/viewthread.php?tid=4965>

Another short workup - from thevespiary forum usefull to see the proportion of our exploited chemicals

"With vigorous stirring, to 5ml of IPA / 1g of P2NP and **0.4 times volume of H2O** in RBF was dumped 6 mol eq. of NaBH4.

Recrystallized P2NP was washed with sodium bisulfite solution***, then with and water and it was then added in small portions to the mixture to minimize frothing. No external cooling applied. After all substrate was added and near-colourless solution is obtained, the solution left stirring for 30 minutes. Claisen adapter was fitted to the rbf with reflux setup and addition funnel. With one portion, 10%-mol equivalent (for the nitroalkene) of CuCl2.2H2O dissolved in IPA was added to the solution via the funnel. It was refluxed for 30 minutes.

After which the remaining NaBH4 was destroyed by adding about **50% acetic acid** untill the fizzing stopped and the solution was fairly acidic.

Lors de la dernière réaction, j'ai ajouté de l'acide acétique pur et après quelques minutes d'agitation, la solution est devenue d'une belle couleur bleu coolaid et a semblé dissoudre le cuivre2. Est-ce que le fait d'en ajouter trop est contre-productif ? I always just add a random amount most Likely very much over kill just so I know everything g is acidic before I strip IPA.

***I wash the nitropropene with sodium bisulfite solution to get rid of unreacted benzaldehyde. Maybe crashing the nitroalkene into bisulfite solution would be better, then recrystallization... Anyway then I rinse the crystals with ice cold water (either by dumping them into water and then filtering or just on the buchner) and suck to somewhat dryness.

With the claisen adapter still attaced, short path distillation setup was rigged and everything below 100°C was stripped. Then the solution was made basic dripping

strong NaOH solution to the reaction flask and steam distilled. Additional very dilute NaOH solution was added through the addition funnel every now and then. The distillate was then made slightly acidic with H₂SO₄ and evaporated. Total yield after recrystallization: 80% mol wise from the P2NP due to very sloppy work"

///// Une autre question que j'ai en ajoutant du h₂so₄ au H₂O et à la base d'amphétamine, je comprends qu'on peut suracidifier et dire que si la base est dans un solvant comme le toluène, le sulfate d'amphétamine se dissoudra à nouveau dans la solution. Est-il possible de le sauver quand cela se produit ? Je pense qu'il suffit d'ajouter du NaOH et de le transformer en base libre à nouveau, mais quand cela se produit, il semble que je perde tout et que je ne puisse pas récupérer l'amph. Encore une fois.

Qu'arrive-t-il à l'amph quand on ajoute trop de h₂so₄ ? Pourquoi se redissout-il ? Comment peut-on sauver l'amph. Quand cela arrive ?

>>> For quenching the borohydride, when done at a 10mmol scale, it only took a few drops of 50% v/v AcOH to quench, there seems to be very little left over, once the borohydride has first reduced the nitroalkene to nitroalkane and then reduced the CuCl₂ to Cu(0).

Quench of the borohydride wasn't violent at all, slight fizzing when adding the AcOH (as either 25 or 50%, forgot which, aqueous) dropwise, just a bit of hydrogen evolution, one can tell when the NaBH₄ is quenched by the way that after it has, adding the acid dropwise, H₂ evolution takes place with each drop, until one drop after the last one required to decompose the remaining NaBH₄ traces, which, as it's all quenched, will not induce any further fizzing or bubbling, since it's all done. Using the bubbling of H₂ during the quench, and dropwise adding 50% or 25% AcOH allows it to be precise, to within a single additional drop of acid.

>>> suggestions sur la façon de réduire en poudre le NaBH₄ ? Il suffit de le mettre au milieu d'un morceau de sac plastique et de l'écraser avec le fond d'une tasse à café ou toute autre surface plate d'un objet qui ne se brisera pas sous la pression, puis d'utiliser le côté de la tasse pour le rouler d'avant en arrière. La stabilité à l'air/sensibilité à l'humidité du NaBH₄ n'est vraiment pas un problème.

Le mien se présente généralement sous une forme plutôt poudreuse, avec tout au plus quelques grumeaux pas très durs, et ceux-ci peuvent très simplement être réduits en poudre en les écrasant avec une spatule ou une cuillère plus grande... pas en une poudre fine, semblable à de la poussière, ce n'est pas nécessaire, il suffit de se débarrasser des grumeaux les plus gros pour moi.

Je sais que l'ancien borohydrure peut former des masses dures avec de gros grumeaux...

Je n'ai encore rien trouvé qui puisse indiquer que ce n'est pas conseillé pour le borohydrure de sodium...

Le sac ici n'est bien sûr pas si important pour être étanche, mais je déconseille le papier car le NaBH₄ est un peu hygroscopique et colle au papier s'il y est laissé trop longtemps, et certainement plus vite s'il est en poudre.

De plus, je m'attendrais à ce que le borohydrure perde un peu de pouvoir réducteur quand il est vraiment fin, je ne sais pas, mais ça n'aurait pas beaucoup d'importance de toute façon.

>>>

>>> It's been done with 500g of P2NP using 10L RBF and refluxing for 2 hours, 90% yield. Adding everything (near) refluxing temperature increase yield. I use 5ml IPA and 2.5ml H₂O for every gram of P2NP I use. After extremely slow addition of P2NP to minimize frothing and after it is all added I let this react with vigorous stirring for 30-45 min.

I'll then attach a reflux condenser and add in all at once/or in 2-3 additions an IPA/H₂O solution of my home made very light blue in color CuCl₂ . (10% mol eq. For P2NP)

So for every gram of P2NP I use .1g of CuCl₂.

When I add this the reaction mix instantly turns black and heats up.

I'll then reflux this for a good 30-45min

After I let reflux I'll add to reaction mixture through reflux condenser (while still hot) Glacial Acetic Acid untill fizzing stops and mixture is very acidic. (Usually I add a large excess of GAA)

After I add GAA, shortly after the reaction mixture takes on a very blue transparent color. Exactly like blue coolaid.

When I stop stirring two blue transparent layers form in the flask and a small amount of black precipitate sits on the bottom.

I'll then set up for distillation and distill off everything below 100c.

Once temp reaches 100c I'll switch the receiving flask. I'll then add very strong NaOH/H₂O (I don't usually make a specific NaOH/h₂O % solution , I basically just add a large amount of NaOH to a beaker and add water and mix untill it dissolves) I then add this to reaction flask.

I then collect H₂O/freebase oil in receiving flask.

Usually the H₂O/freebase mixture in the receiving flask is a milky white solution with small amounts of clear oil floating on top.

Once the water distilling over becomes clear I stop distillation.

I now drip in 2 drops (I don't have any PH paper at the moment) of phenolphthalein solution into the freebase and water mixture turning it purple/pink. Now I'll drip in diluted H₂SO₄ until the pink color disappears and I don't see any freebase oil floating on top of the water.

I now place the solution in a beaker on the hot plate with a stir bar and boil down to dryness with stirring.

I am then usually left with a whitish substance caked to bottom of the beaker to which I'll add acetone and boil with mixing for around 1 minute

Then I filter through a coffee filter and collect amphetamine sulphate and let residual acetone evaporate.

I'm always left with a shitty yeild and don't understand why. I think next time after

distilling off IPA I'll basify the reaction mix and then extract freebase with DCM. I'll then separate/collect DCM, dry with MgSO_4 and then evaporate off DCM leaving me with just the freebase oil. I'll then know exactly how much I have and then I can add the exact amount of H_2SO_4 need to convert it into the sulphate.

Best results regarding yields and (more important in my eyes) quality were achieved by:

- Al into the flask (shavings, 99,9%)
- IPA, acetic acid, water, warmed separately to 40°C and P2NP dissolved. The complete dissolution of the P2NP being of utter importance*. Then HgCl_2 (nitrate will work fine too) added and dissolved. Stirring of course, what else.
- This dumped onto the Al.
- Strong overhead stirring switched on and not shut down till end.

And here we go, refluxed with heating for 6 hours after the wild part was over.

- Water added and alcohol distilled from solution
 - Water distilled from solution - not all but a good part of it. Boil it as hard as possible to drive all shit out.
- This removes by steam-distillation all the stuff which steamdistills and is not a salt at this moment like amphetamine.
- Sodium carbonate and water added until no more fizzing (CO_2 formation)**. NaOH 10% solution (cold) added to pH 14.
 - Product steamdistilled rather slowly and not pushed at all for that's another key to quality. Now stirring is shut down.
 - Collected in petrolether, filtered, separated and solvent switched to IPA (Petrol ether distilled mostly out, IPA added, rest ether and some IPA distilled out) This takes no time at all***.

This removes all water too. Salt formation is superior in alcohols though.

- H_2SO_4 well mixed with the 5-fold amount of IPA is slowly added, fridge, filtration and again until no more clouds form and it starts turning pink. Washed with small amount of dry acetone or better ethylacetate.

* the solution has to be completely CLEAR no turbidity. If it does not get clear yor P2NP is dirty.

** that's much softer on the product and no insane amounts of NaOH or KOH are needed.

*** petrol ether is superior in selectivity on extraction but a beast to make the salt in.

The result is something what does already optically only remotely resemble the pictures of horrible unclean, smeary discolored and revolting smelling shit one sees posted or is told on boards, in pdfs (Oparaya's for example), or at trading places (yellow! yellow P2NP stains declared as a sign of quality! Vomit!).

I know everything about this "almost amph" as it was what was produced in the beginning, in all known (and some new) variations.
Most simply declare dirt to gold and thats that.
A few work on though.

Oh! On the boards EVERYONE is only after highest purity.
That is one of the more funny parts of the game here....
...just dont buy it.

your choice.

/ORG

Is amphetamine soluble in water?

They are weakly basic substances and can exist as either in a free base form or react with various acids to form salts such as amphetamine hydrochloride. The salt forms of the amphetamines are **highly water-soluble** whereas the **free base forms are less so**

Other Experimental Properties

Colored liquid with an amine odor
Volatilizes slowly at room temp

- Crystals > bitter taste; sinters at about 150 °C; decomposes around 300 °C; pH of 10% solution is about 4.6. More soluble in water than amphetamine sulfate; slightly soluble in alcohol; practically insoluble in benzene, chloroform, ether /Amphetamine phosphate/
- Crystals > slightly bitter taste followed by sensation of numbness; MP above 300 °C (decomposes); solution of 1 g/10 mL water has pH 5-6; 1 part dissolves in 8.8 parts water, 515 parts 95% alcohol/amp sulfate

<https://pubchem.ncbi.nlm.nih.gov/compound/Amphetamine>

<https://www.thevespiary.org/rhodium/Rhodium/Vespiary/talk/index.php/topic%2C3701.0.html>

About The Ingredients And Procedures-

CuCl₂

The CuCl₂ + NaBH₄ method is superior, best of all p2np reduction I've ever done, but only up to 20 gr batches, the more you increase Mol per batch the lower theoretical yield you'll have (all people I know say the same., so I would be very surprized if someone have other news to tell) . The sodium bisulphite washings of the p2np is first time I see someone mention here (THUMBS UP), that superb, very important to have good yield for whatever you're using your p2np. That's the far best way to clean out all benzaldehyde contamination of the p2np, and whatever reaction you wanna do with your p2np the benzaldehyde is a bitch and will interfere and gives lower yield ketone, oxime, nitroalkane or amine. Always wash p2np with sodium bisulphite solution and recrystallize before doing any reaction A/B.

Is CU acidic or basic?

However, copper, and most transition metals, often exist as positively charged ions in solution. Positive ions are electron pair acceptors, and can be considered Lewis acids. To reiterate, copper atoms themselves are not acidic or basic, but copper ions are acidic.

Preparation CuCl₂ solution

Le catalyseur, qui est généralement en quantité bien plus faible que les réactifs, n'est pas consommé et est retrouvé inchangé à la fin de la réaction. S'il est séparable aisément du milieu réactionnel, il pourra être recyclé dans une nouvelle synthèse.

Copper(II) chloride dihydrate is a beautiful green crystalline solid, while its diluted aqueous **solution** has a pale blue **colour**

Molarity = number of moles of solute /volume of solution in litre

Molarity = mass of solute (in grams) /molecular mass x volume of solution in litre

2M = le nb de moles de soluté dissout dans 1L (1dm³).

If 2 moles of a solute are present in one litre of a solution then the molarity of solution will be (2M).

$$C=n/V$$

2M signifie qu'il doit y avoir 2 moles de soluté présent par L de solution soit:

$2 \times 134,5 = 269 \text{g/L}$ (ratio 0,1M=26,9g/L). Il doit y avoir 26,9g/L au total.

on veut réaliser 50ml (sachant que 1 litre = 10dl = 100cl = 1000ml = 1kg = 1000g)

26,9g > 1000mL

x > 50mL

Puis, $x = 50 \times 26,9 / 1000 = 1,345 \text{g de CuCl}_2$ dans une solution de 50 ml.

Explanation:

To make x mL of 2M CuCl₂ solution, you need to make sure that every litre of the solution contains 2.00 mol.

So, 1 L of 2M CuCl₂ solution contains $2 \times (134,5) \text{ g} = 269 \text{g CuCl}_2$

If we mix 40.0 g of NaOH with enough distilled water to make 500 mL, we will get a 2.00 M NaOH solution.

In what volume of the solution?

1 L X 2 M X 13,45 g/mol = x g NaOH in 1 L of 2M solution

To calculate the mass in any other volume, just replace the "1L" in the calculation with any other volume (in L).

Pour une Concentration de 0,1M avec masse molaire de 134,45g/mol

.48 g/mol. 1/10th of 170.48 g is 17.048 g, so this is the amount of the CuCl₂*2H₂O you need for the solution you are tryin

A 100ml 1M CuCl₂ – H₂O SOLUTION

Firstly, 100 ml at 1M is 1/10th of a mole of Copper chloride dihydrate. Knowing this, you must portion out 1/10th of a mole of CuCl₂ *2H₂O and in order to do this, you also need to know its molar mass; it happens to be 170.48 g/mol. 1/10th of 170.48 g is 17.048 g, so this is the amount of the CuCl₂*2H₂O you need for the

solution you are trying to make. Since you need 100 mL of the solution, it is important that you put the $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ in the volumetric flask (technically you can use any container, but volumetric flasks are very accurate) **first** and then pour in the water to the 100 mL line. If you put 100 mL of water in first and then add the $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$, it will result in a solution that is more than 100 mL, which will in turn have a molarity that is not quite 1M. Always add the solute before the solvent. In short, to make x mL at y M for any solution, multiply x by y and multiply that by the molar mass of the solute. Add solvent to that mass of solute until you reach the desired volume

.48 g/mol. 1/10th of 170.48 g is 17.048 g, so this is the amount of the $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ you need for the solution you are tryin

The purpose of this experiment is to acquaint you with the concept of concentration of solutions. Although there are a variety of ways of describing a solution's concentration, we will use just one system in this experiment, that of "molarity," or

"moles per liter," abbreviated mol/L or M. The experiment will also give you a chance to see how differences in concentration affect the behavior of a solution, making it react faster or slower, and making it able to react with more or less of some other substance (for the same volume of solution).

"Prepare a tenth of a liter (0.10 L) of a 1 M solution of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$." What, exactly, does this statement mean? First of all, a tenth of a liter needs to be expressed as a certain number of milliliters, since our laboratory graduated cylinders are marked off in milliliters. You will be making, then, 100 mL of solution.

What does the "1 M" mean? It means one mole of the material that you are going to dissolve – in this case, $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ – would be present in one liter of a solution that has the concentration described as "1 M." (M really says, "moles of dissolved material per liter of solution.") However, we will not need a full liter, so we will make only 0.10 L (100 mL). Question: If you need 170.05 grams (which is one mole of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) to make one liter of 1 M solution, then how much will you need to make just one-tenth as much solution? grams, or moles

Sodium Bisulfate is an acidic salt (acidic solution is produced after salt is dissolved in a solvent) which is produced by partial neutralization of Sulfuric acid (H_2SO_4) with an equivalent amount of sodium base, basically in the form of sodium hydroxide (NaOH) or sodium chloride (NaCl). The anhydrous is being hygroscopic in nature. The solutions of Sodium Bisulfate are acidic in nature with a pH value of 1 for every 1M solution. It is a white and relatively dry granulated compound. It is corrosive to metals and tissues and also is soluble in water.

Physical Properties: Sodium Bisulfate of NaHSO_4 is usually accessible in both **monohydrate** (hydrate comprising of *one mole of water/mole of the compound*), $\text{NaHSO}_4 \cdot \text{H}_2\text{O}$ and anhydrous (dry) forms. Anhydrous NaHSO_4 is a dry granulated white powder, having a density of 2.74 g/ml with a melting point of 315 °C. Whereas, monohydrate is a white colored granulated solid having density of 1.8 g/ml with a melting point of 59 °C. Monohydrate is considered a dry acid which is appropriate for safe storage and shipping. Its water solubility is 28.5 g/100 ml (25 °C) and 100 g/100 ml (100 °C) respectively

DROP IDEAS

Amines and amides are very weak acids and only react with very strong bases.

Comment identifier la phase aqueuse de la phase organique ?

En cas de doute pour **identifier** les **phases**, on peut prélever avec une pipette quelques gouttes de la **phase à identifier** et les ajouter dans un tube à essais contenant un peu d'eau : si elles se mélangent il s'agit de la **phase aqueuse**, si elles forment une nouvelle **phase** il s'agit de la **phase organique**.

On sait que la **masse volumique de l'eau est** : $\rho_{\text{eau}} = 1\,000\text{ g/L} = 1\text{ kg/L}$. Car l'eau est une constante étalon est a une densité de 1.

BASIC THEORY

An acid is a compound that releases H^+ ions into a solution. So that there is more H^+ than HO^- ions.

A base is a compound that can react with H^+ ions to neutralize them and form a salt. Bases come with a huge variety of cations (positively charged ions) and anions (negatively charged ions). In base we can resume there is more HO^- than H^+ .

An example of an *acid* reacting with a base is hydrogen chloride (HCl ; an acid) and *sodium hydroxide* ($NaOH$; a base). When these two react the products are water and sodium chloride.

Common acids are nitric acid, hydrochloric acid (HCl in water), sulfuric acid, formic/acetic/benzoic/other organic acid(s), and phosphoric acid. Of these, sulfuric acid is the strongest.

Acids are also often oxidizing in nature, though not always. Sulfuric acid and nitric acid are both oxidizing acids (nitric is very oxidizing), while the organic acids (excluding formic acid) and phosphoric acid are all very neutral towards redox. HCl is extremely weakly reducing, with the heavier halogen acids (hydrogen iodide and

hydrogen bromide) being more reducing; formic acid, oxalic acid, and sulfurous acid are all quite reducing.

Common **bases** include hydroxides (the cation is fairly irrelevant), (bi)carbonates, and ammonia (and related amines; this is a lewis base and will be covered more in a later lesson).

The strength of bases is limited by the solvent being used. For example, the strongest base that water can support is a hydroxide. This is because water is slightly acidic and any base stronger than a hydroxide will react with the water to form a salt (a hydroxide). Liquid ammonia can handle stronger bases as ammonia is much less acidic than water (water's pka is 14-16 while ammonia is >30).

General bases are **sodium hydroxide**, potassium hydroxide, and calcium hydroxide. These bases are the most common and least reactive, and are non-toxic as salts (to a degree; never ingest a solution that does not have a PH between 4 and 9, your body is not built for that type of abuse). Other weaker bases that are common for general use are (bi)carbonates and phosphate salts. These last bases are useful for making buffer solutions or destroying acids (basically just a buffer solution) more than actually making a solution basic. Stronger bases will need to be made, and the strongest base that you will likely have access to is Grignard reagents (magnesium is cheap enough that using these is not extremely wasteful as long as it is a valuable reaction). It is possible to make bases like N-butyl lithium (!), but it is very dangerous (making and using) while only offering a few advantages.

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C Polar compounds dissolve well with Polar solvents. In the same manner Non Polar compounds dissolve in Non Polar solvents. Therefore if a compound and a solvent have identical properties they dissolve well and are miscible.

If we try to mix NP/P there is no solubility or miscibility. The more dense (d) or thick will leave at the bottom and $d < 1$.

We use solvents in reactions to mix the reagents together and make them react.

Dans une réaction chimique exothermique, l'énergie dégagée par la formation des liaisons chimiques dans les produits de réaction est supérieure à l'énergie requise pour briser les liaisons dans les réactifs.

De nombreuses réactions impliquant des acides ou des bases sont exothermiques.

SOLVANTS

Polar solvant

P2NP Preparation

Après la filtration par système buchner vous obtiendrez peut etre une pate jaune gorgée de solvant. Un bon moyen de **nettoyer celui-ci est de** commencer par le faire sécher puis des les dissoudre à nouveau dans un **faible volume d'alcool isopropylique chauffé**. Et maintenir le tout à haute température au bain marie. Quand tous les cristaux sont dissouts mettre une coupelle ou un film d'aluminium sur l'orifice du récipient peut s'apérer utile pour éviter trop d'évaporation. Remettez le récipient au **freezer et attendez l'apparition des cristaux**. Et repetez l'étape du filtre buchner en nettoyant avec de l'eau pure. Faites secher et vous récuperez des **cristaux jaune pale**

Produits à prévoir

80 mL Toluene (ACS)

40 mL Benzaldehyde (~99%)

30 mL Nitroethane (>99%)

10 mL n-Butylamine (p.a.)

Procédure

Les produits sont tous mélangés dans le toluène dans un ballon de 250ml, des pierres d'ébullition (boiling stones) sont ajoutée et le Dean&Stark est monté (piège de 10ml) ainsi qu'un condenseur. L'objectif est de porter la solution à ébullition pour que tout coule nettement depuis la surface du condenseur.

Après 1h, 2.7ml d'eau se trouvaient dans le piège. Après 6h c'est environ 7.0ml qui s'y trouvent soit environ 100% de l'eau en théorie (cela s'est peut être passé 30min plus tôt, le chimiste faisait une petite sieste). La solution est vidée dans un bécher de 250ml et est refroidi à température ambiante. Quelques cristaux de phenyl-nitroporpène apparaissent. La solution est ensuite refroidie doucement à -10°C au freezer. De beaux cristaux apparaissent et il ne semble pas nécessaire de purifier (35g de cristal récupéré avec un filtre). Il peut sembler nécessaire d'utiliser un filtre buchner pour aider la filtration à la sortie du freezer. Agissez rapidement, quand la température augmentera certains cristaux se dissolvent à nouveau et pourront être perdus à travers le filtre. Le filtrat est concentré sous pression faible et refroidit comme ci-dessus. Les cristaux sales tombent et sont recristallisés avec 50ml de MeOH ou d'alcool isopropylique (6g récupérés) Au total 41g de P2NP sont récupérés (64%).