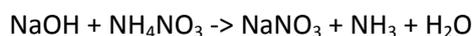


The first major consideration for the shake-and-baker is how much lithium to use. Under ideal conditions two moles of lithium, 13.9g, is sufficient to reduce one mole of pseudoephedrine, 165g of freebase or 201g of the hydrochloride salt. That's 0.10g of lithium, roughly 1/10 of an AA battery, for every 60mgx24pills=1.44g of pills, at least in theory. Using this amount in practice will leave considerable unreacted pseudoephedrine because lithium is consumed by water which is either added to the vessel initially or produced by the reaction:



Any excess lithium that is able to escape oxidation by water or oxygen will be left free to reduce our desired product to the corresponding 1',4'-cyclohexadiene, a less dopaminergic stimulant. We are thus walking a tightrope between merely extracting pseudoephedrine on one side and overcooking it into a less desirable product on the other. The tendency of water-soaked and burning lithium to burrow through the wall of the reaction vessel and/or ignite our pressurized and solvent-soaked nitrate mixture (ie. fertilizer bomb with lye for shrapnel) coerces us somewhat in the direction of using less lithium and having less water. Even so, we will probably have to use at least twice the theoretical amount, ¼ of an AA battery per box of pills, to achieve satisfactory results. Higher yields of sometimes better product are achieved by producing more ammonia to better extract pseudoephedrine then using more lithium to compensate for the additional losses to water. People who end up needing skin grafts often use 10-fold the theoretical amount or more and always see lithium burning in their bottle. I've seen it maybe two or three times over more than a hundred reactions and it's scary enough that I stick with 2-fold.

The amount of lithium is minimized by limiting its interaction with water, which means using less water initially to kick off the production of ammonia then making the best use of ammonia, produced always alongside of water, by venting as little as possible while using excess lye to soak up water and not shaking any more than necessary. Should even this minimal amount still prove excessive the Birch reduction, the conversion of aryl groups to 1,4-cyclohexedienes, can be competitively limited by the addition of a couple ounces of aromatic solvent to the reaction mixture. The reduction of the aryl group, which typically takes 8-12 hours under laboratory conditions, can also be limited by proceeding as quickly as possible to the final filtration; the dehydroxylation reaction is essentially immediate when electrons from lithium are freely available. The reduction of the aryl group is slow enough that complete dehydroxylation of pseudoephedrine can even be achieved in pure xylene or toluene, which brings us to the second major consideration.

Our solvent must be able to dissolve freebase pseudoephedrine but unable to dissolve the hydrochloride salt and must, at a minimum, not react with metallic lithium. Alcohols are thus not appropriate.

Ethers can dissolve so much ammonia as to be then able dissolve lithium even at atmospheric pressure and normal temperatures and they are thus the hands-down winner when conducting dissolving metal reductions with alkalis in laboratory. The improvisational chemist will be concerned by their ability to dissolve not only the hydride of nitrogen but that of oxygen as well. Diethyl ether can dissolve 1.5g of lithium-destroying water per 100ml which is then capable of dissolving even the hydrochloride salt of our product. The ease with which lithium dissolves in ammonia-saturated ether can speed the whole process until the point of gassing including the over-reduction of desired product. For that reason ether should be used with a small amount of aromatic solvent included, which will somewhat limit the solubility of water. Even so, ether should be reused

from batch-to-batch as it is not practically possible to recover all product. The high capacity of ethers for dissolving ammonia means that ammonia can be produced and lithium dissolved in a bottle even while no bubbles and no bronze are visible and it is thus difficult to gauge the progress of the reaction. It also means that the reaction fluid will contain a great deal of ammonia after the reaction and will necessitate either a long wait or a water wash before the product can be recovered by gassing with hydrogen chloride, otherwise our desired amine salt will be contaminated by the simplest amine salt, ammonium chloride. In hot, humid weather evaporating ether will cause water to condense during gassing so that it's often necessary to dry the product before proceeding to an acetone rinse.

Aromatic solvents like toluene and xylene exist at the opposite end of the spectrum from ethers. They are excellent solvents of freebase pseudoephedrine, and also of waxes and other pill crap, that take on very little ammonia or water and are thus poor solvents of lithium that gas well. Lithium that is dissolved in a bottle containing only aromatic solvent will be present only as the floating liquid alloy with ammonia, lithium bronze, while much of the dissolved lithium in a bottle containing only ether will be invisible within the ether itself. In order to reduce pseudoephedrine in a solution of aromatic solvent it is necessary for it to contact the surface of the ammonium/lithium alloy, thus some amount of agitation is necessary to prevent the alloy from coalescing into large blobs. The reduction also proceeds more slowly but with less risk of over-reducing product. The very aryl group that makes aromatics such great solvents also allows them stack neatly together and hide unexpectedly high amounts of solvent in apparently dry product and, what's worse, to slip neatly between the nucleotide bases in DNA and cause cancer. For these reasons it is absolutely essential that a cook conducted in aromatic solvent be followed by an acetone rinse regardless of the apparent necessity.

Light petroleum distillates occupy a middle ground between ethers and aromatics. They take on a good amount of ammonia so that dissolving lithium is not quite so tedious as with an aromatic solvent but also enough water that gassing sometimes doesn't result in complete recovery of product. They are also not as aggressive a solvent of freebase pseudoephedrine and are required in greater volume than either ether or aromatics. Still, they are easily available and can be used with fewer considerations than other options and for these reasons Coleman's is my personal favourite, often with a little aromatic mixed in.

The third major consideration is the methods by which the product will be recovered from the reaction vessel and purified. The reaction fluid may be washed with an equal volume of water before being exposed to an acid to produce an insoluble salt and decanted off or being separated by filtration. The powder produced by gassing can then be rinsed with dry acetone to remove excess solvent, acid and pill impurities or even recrystallized. Steam distillation can be used to separate unreacted pseudoephedrine from the much more volatile product. Often, however, essentially pure material is produced by simply allowing sufficient time for ammonia to escape from the filtered reaction fluid then gassing with hydrogen chloride, decanting the solvent and allowing time for any excess to evaporate from the precipitate.

Having now disposed of the most important considerations, let's look at a workable procedure. Remember that any idiot can dissolve lithium in condensed ammonia under an atmosphere of nitrogen to conduct the same exact reduction as the next guy every time, just as long as he's paid his tuition. The improvisational chemist is required to overcome his material limitations

with what can only be called artistry, his ingenuity resting on his intuition and his outcome evaluated subjectively by the audience. The one-pot reduction of pseudoephedrine is the pinnacle of improvisational chemistry and thus there is incredible artistic licence afforded to those who pursue it. What follows is a rudimentary procedure free from artistic embellishment.

Materials:

Chemicals:

- 2 boxes of 24x60mg pseudoephedrine pills
- lithium from ½ of an AA lithium battery
- 1 ammonium nitrate cold pack (containing ~55g NH_4NO_3)
- 150g lye
- a small amount of water
- 250ml Coleman's fuel
- 50ml of aromatic solvent free from alcohols and acetone
- 100g or so anhydrous calcium chloride
- a small amount of hydrochloric acid
- a small amount of dry acetone (store-bought acetone can be dried over calcium chloride, allow 12 hours)

Apparatuses

- Coffee grinder or mortar and pestle
- Pliers and wirecutters, pipecutter
- 1L Sodastream bottle or PET drink bottle
- A syringe with an attachable tube that can reach to the bottom of the bottle
- Funnels
- Coffee filters
- A shallow dish, ~500ml capacity
- A small, disposable bottle or bag to produce HCl gas in
- A thin, flexible tube or straw to channel HCl gas through
- Gloves and goggles, if you're a pussy

Procedure:

- Grind 2x24x60mg of pseudoephedrine pills to a fine powder

- Measure out lye and open your cold pack, measure out solvent
- Add lye, pill powder and solvent to bottle and shake to mix while keeping ammonium nitrate near at hand (a wide-mouth funnel might be helpful, or just dump dry stuff from a piece of folded paper)
- Using pliers, wirecutters, a pipecutter, curses and any magic you can muster, even your teeth if necessary, open that damned battery. If the lithium strip comes into contact with the steel casing then the battery will start to warm up and might become too hot to hold but I've never had one catch fire. Once open, the lithium is identified as a strip of metallic foil with a steel tab attached at one end while the other strip is black and non-metallic with attached plastic backing. Remove the steel tab from the end of the lithium strip and rip half the strip into fingernail size pieces. Lithium reacts with oxygen fairly slowly but in humid air it is necessary to move quickly if the whole strip is unrolled at once, that or the strip can be unrolled and ripped at a more comfortable pace. The half-strip of lithium not required for this reaction can be stored in oil/fuel or folded up and wrapped tightly in a latex glove or saran wrap. Folded and wrapped it'll last a week or two.
- Having pill and lye well-mixed in a bottle with solvent and lithium, add ammonium nitrate. Be careful to disturb pill powder as little as possible and quickly cap the bottle. If the lye and ammonium nitrate absorbed sufficient moisture from air before going in the bottle then they will react to produce ammonia, at first slowly and then more quickly. In that case you can skip to step 7, otherwise proceed to step 6.
- Using a syringe with a tube attached add 10ml of water to the reaction mixture at the lye/ammonium nitrate interface. Try to minimize the disturbance of pill powder as it will contain crospovidone or other disintegrants which can sequester enough water to prevent intimate contact between lye and ammonium nitrate. Try also to not wet the lithium as it will oxidize and even burn on contact with water. Be ready to cap the bottle quickly as the evolution of ammonia can be quite violent. Using too much water can cause great globs of wet pill crap to float up and meet the lithium while excess water left below dissolves all your ammonia. Such a bottle is maybe one to step away from. Shaking the bottle while liquid water is present will also cause problems.
- Allow time for lye and ammonium nitrate to react producing water and ammonia and turning the 'dry' portions of the mixture to a slurry. Shaking or swirling may help mix ingredients to produce ammonia or may hurt the process if it causes crospovidone and friends to soak up what little water is present. Pressure will climb with the production of ammonia until it reaches the pressure and temperature at which ammonia after which liquid ammonia will be produced without accompanying bubbles or increases in pressure. Using a 1L sodastream bottle and 55g of ammonium nitrate it shouldn't be necessary to vent any pressure until the reaction is complete, though the bottle may become so taut that it's difficult to even slightly indent even the most flexible part of it pushing hard with both thumbs. Vent when you can't find any flex in the most flexible part of the bottle if using a fresh 1L sodastream bottle. Keep in mind that the PET plastic of the bottle's body is susceptible to attack by bases while the plastics of the cap are susceptible to non-polar solvents, meaning that eventually the bottle will fail regardless of internal pressure. If re-using a sodastream bottle or if using a pop bottle then watch for signs of impending failure such as cracks in the plastic or unusual bulges and

listen for escaping pressure. Failure, even catastrophic failure, is seldom more than an inconvenience which often doesn't even damn the batch and has never burned my face off. Structural failure combined with an ignition source, perhaps burning lithium or a cigarette, should be avoided.

- Having now a bottle containing ammonia-saturated water and condensed ammonia mixed with pill crap and excess lye at the bottom, swirl or shake the contents at least well enough to ensure that freebased pseudoephedrine gets a chance to migrate into the solvent. If metallic lithium is dissolving in ammonia to produce bronze at an acceptable rate then you can minimize lithium losses to water by not letting lithium contact the slurry, otherwise shake the shit out of it. Your slurry would need to be really quite wet for the lithium to burn at this point but all the same you should watch that any lithium embedded in the slurry not be left long against the wall of the bottle lest it melt through.
- Depending on lithium losses you are done either when you have no remaining lithium/bronze or when what does remain doesn't seem to be getting consumed. Usually if I have leftover bronze when I think the reaction is complete I'll shake the bottle until all that bronze has reacted with water just to be sure. In any case, when you think you're done it's time to filter the solvent. Opening the bottle will blast ammonia everywhere and might burn your eyes or throat. People have even died just from the ammonia, no fire or nothing. After the initial blast is let off well away from your face pour the solvent through a funnel and coffee filter. The slurry, probably coalesced to lumps of crud at the point, will continue to evolve ammonia for hours as it has a high heat of vaporization and thus evaporates slowly. The crud can be expected to contain still considerable substances of interest and often a second and even third pull is desirable. Circumstances permitting, add additional solvent and recap the bottle or else wait until you've gassed the just-separated solvent to add it back for another extraction. Be aware that the bottle will fail after a few days exposure to lye and ammonia and that the silicone seal of a sodastream bottle will soften with continued exposure to solvent so you'll want to improvise a cap if you want to re-use the bottle.
- Your solvent now separated you need to remove residual ammonia. Time, agitation and ventilation will accomplish this in perhaps 10 or 20 minutes or you can wash it with water (or lye solution or brine but nothing acidic). Washing may additionally remove other contaminants but few of these would precipitate upon gassing anyhow. On the downside it will cause some loss of product which is slightly soluble in water even as a freebase and might also introduce water to your solvent resulting in less precipitation on gassing. Water is essentially insoluble in the aromatics but is nevertheless present as nearly microscopic droplets that take hours to settle after aggressive washing. Separation of residual water after washing can be expedited by heating the solution. Usually I'm happy to just wait for ammonia to evaporate and only wash when I want to control the odour.
- Once the ammonia is gone from the solution it's time to gas. In a bottle or bag you will introduce a few ml of aqueous HCl to maybe 10 or 20g of CaCl_2 so that gaseous HCl evolves as water is sequestered by the salt. The bottle or bag should be sealed around a tube or straw and that tube should reach into a dish containing your solution. Getting a good seal can be a bit of a trick with the cost of failure being burning eyes and lungs and corroding tools and electronics. I like to use rubber bands and balloons but tapes, glues and screw caps

might also be handy. The CaCl_2 and HCl method is slower than methods using sulfuric acid with the upside being that you don't have to deal with sulfuric acid. Dripping HCl into H_2SO_4 is faster and more efficient but requires more care in designing an apparatus and disposing of waste and also introduces a risk of suck-back. Regardless of gassing method, HCl will precipitate the hydrochloride salt of our amine first as clouds in the solvent then as crystals on the walls of the dish and then as crystals floating freely in milky white solution. Gas it until it stinks of HCl then let the solution clear and see if adding more gas results in more precipitate. If it doesn't then you're done gassing. Decant the solvent from the precipitate and filter it to collect floating crystals if necessary. Be aware that HCl reacts with metals which shouldn't be used in this step. If using an aromatic solvent then your gassing vessel must not be made of a susceptible plastic. Glass or ceramic is best for scraping anyhow so use that.

- Your precipitate will be wet with solvent at this point. Keep the dish on an angle as it dries so that solvent can run off. Coleman's contains additives that will leave a residue on the product if too much solvent is allowed to evaporate, as does ether directly from the can. Aromatics can take quite a while to evaporate and are hazardous to health. Any water that was present in solution during gassing will now be left in the product as dilute hydrochloric acid. All of these problems can be solved by rinsing with dry acetone which will likely pick up some water in the process and carry away some product. If you choose to rinse always reserve the acetone and check what's left when it evaporates. Because acetone evaporates so quickly a rinse can be used to expedite the drying of even pure and clean material, but removal of aromatics or excess acid is my main application.
- Test your product by smoking. Hopefully it vaporizes easily and completely leaving little to no residue and without any strange flavours in the smoke. Leftover pseudoephedrine is common and will leave a residue which melts without vaporizing but which burns with a distinct flavour at high temperatures. There's another substance with a distinct flavour that vaporizes at a much lower temperature and occurs when excess water was used for the reaction. CMP is also distinguishable by smoking, as are many other contaminants.
- Purify by hook or by crook, or else accept your material as is. Steam distillation is the obvious winner for removing unreacted pseudoephedrine and should help with everything else as well but I've not tried it. Some contaminants can be more-or-less removed by simply dissolving in water and filtering. Fexofenadine, for example, has fairly low solubility in water. Other times you'll want to recrystallize or even conduct an A/B extraction. The procedure as given produces a material from my local pills which requires no additional purification but, as I said, this is an art. Results will vary according to reaction conditions, materials and their sources, and at the discretion of the fates.

Using more pills is possible but requires more lithium while the additional crospovidone can make it difficult to get the reaction rolling and thus require more water, which requires more lithium and so on until inevitable self-immolation. The simple bulk of the pills is the greatest consideration and using a more concentrated pill will best allow for a larger batch. Modest increases in batch size are also possible by grinding the ammonium nitrate and/or lye. Truly impressive batches require extracting the pseudoephedrine from the pill mass beforehand and raise the question of whether you'd not prefer to condense your ammonia at low temperature over high pressure. It's also possible to use

some combination of the two by ice packing your bottle which can help considerably in dissolving your lithium. Ice packs make it possible to add the pill powder to the bottle after the lithium has dissolved which can also allow for larger batches. Even with ice packs, however, a great deal of ammonia evaporates when the bottle is opened to add the pill powder and ammonia is required as a solvent for pseudoephedrine well as lithium. Heating the reaction might be helpful to ensure more complete extraction of the pills or to initiate the production of ammonia if you've already added a scary amount of water and it's all been sucked up by pill crap. There's also a lot of room for variation in the choice of reaction vessel. I've conducted successful sub-gram reductions of pre-extracted pseudo in a hotel shampoo bottle. Two litre pop bottles are classic but tend to fail (watch for fine cracks at the bottom). Corrugated water bottles are better than you might think in that the corrugation helps control pressure but the caps tend to explode off when attempting to vent. Using more solvent can result in a better extraction but also slows the dissolution of lithium if the solvent isn't ether. I prefer to conduct repeated pulls and reserve the later extractions for a future batch.

A yield of 50% on the first pull is okay and 75% is pretty damn good. Optimizing yield requires consideration of individual conditions and capabilities.

Though normally considered an outdoor sport the reaction can also be conducted indoors provided one is prepared to deal with the inevitable several grams of ammonia as well as the eventualities of bottle failure or fire. Ammonia is very soluble in water and is neutralized by acids. It can be contained by most, but not all, plastic bags.

Disposal of wastes is important. If your coffee filter contains lithium and/or bronze then it may spontaneously ignite after a few minutes. Lithium bronze is composed mostly of ammonia which evolves from it as the lithium reacts with air. The solvent-soaked mixture of lye and nitrates is quite flammable even in water and can be ignited by any leftover lithium (though in practice I've only had it ignite when dumped from the bottle onto the wet ground). Ammonia is toxic to aquatic ecosystems. The bottle can be finger printed. If you're expecting to do a number of reactions then it would be worthwhile to get an air-tight waste container to store your solids along with a bit of solvent. Time spent storing waste now pulls double duty as time spent recovering feedstock and/or product. The vessel must be compatible with lye which rules out PET while strong solvents excludes HDPE from consideration. Ultimate waste disposal will likely mean either burning or dissolving in water and either option requires consideration of individual circumstances. If using waste streams developed for other purposes, like garbage or sewage, be sure to account for worker safety and the integrity of the infrastructure. Check whether your pipes are compatible with your solvents, plan for when your garbage bags get ripped open during handling, et cetera.