SAFETY

When working with a compound or reagent that you never used before, look up its MSDS to learn the proper procedures for handling or working with that substance in a safe manner, and know its physical data (melting point, boiling point, flash point, etc.), toxicity, health effects, first aid, reactivity, storage, disposal, protective equipment, and spill-handling procedures.

Handling air-sensitive reagents

When handling air & water sensitive material (pyrophoric), have a fellow labmate show you proper technique for use and disposal.

There are precautions and proper techniques that you must know when using pyrophoric reagents such as: grignards, borates, organolithium, organozinc, organoaluminum reagents and more.

Here are a few examples of common pyrophoric reagents used in lab:

- MeLi
- n-BuLi
- t-BuLi
- allylMgBr
- MeMgBr
- butylMgCl
- *i*-propylMgCl
- phenylMgBr
- boron trifluoride diethyl etherate ($BF_3 \cdot Et_2O$)
- diisobutylaluminium hydride (DIBALH)
- diethylzinc (DEZn)
- triethylaluminium (TEA)
- Trimethylaluminium (TMA)

These reagents require the use of a needle and syringe to transfer the reagent into the reaction flask. Here are common syringes used in the lab:

Glass syringe w/Teflon plunger

Glass syringe w/glass plunger

Plastic syringe w/plastic plunger

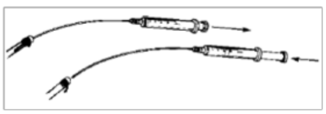
If you ever have to use more than 50 ml of these reagents, **ALWAYS** have someone there with you just in case of an accident does occur. There's a few ways to transfer your reagent depending on the type of syringe you are using.

Glass syringe w/Teflon plunger: It's okay to use a 50 ml syringe or any other smaller volume syringe by adding portions of your reagents.

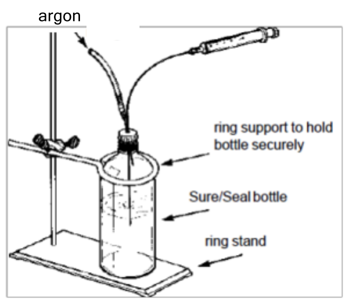
Glass syringe w/glass plunger: **DO NOT USE** to transfer pyrophoric reagents

Plastic syringe w/plastic plunger: **DO NOT USE** a 50 ml syringe; use a smaller volume syringe by adding portions of your reagents.

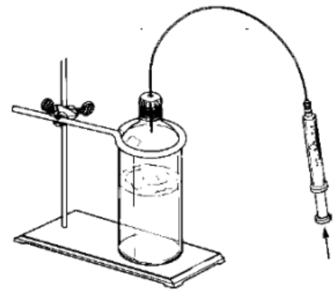
Once you are ready to transfer your reagent into your syringe, follow the diagram below:



Flush your syringe with argon



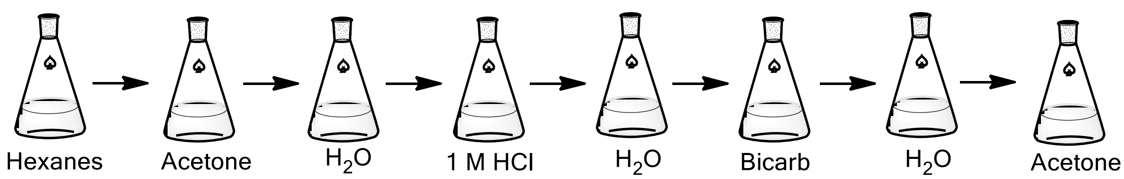
Before filling your syringe, make sure to tap your reagent bottle with argon.



Remove gas bubbles and return any excess reagent to the Sure/Seal bottle **while it's still tapped with argon (not shown)**

Quickly, take out your filled syringe out of the Sure/Seal bottle and add your reagent into your reaction flask. After adding your reagent, you must properly rise and clean your needle and syringe before they are damaged (clogged). The following diagram shows the order of how you must rise and clean your needle and syringe:

Process in Rising and Cleaning a Needle/Syringe After Using Air Sensitive/Pyrophoric Reagents



Do three rinses of each solvent/solution before moving onto the next solvent/solution. Once you're done cleaning your needle/syringe, place your needle in oven so it can dry before you use it again.

For more details and help, look at the following links:

http://ehs.unl.edu/training/Colloquium/PyrophoricsPresentation.pdf

http://www.purdue.edu/rem/hmm/al_techbull_al134.pdf

http://sbrs.cm.utexas.edu/WS/airfree.pdf

Hydride Reagents

Don't take too long weighting out your hydride reagent since they can be air & water sensitive. Always cool your reaction flask to recommended temperature (based on reaction protocol) **BEFORE** adding your reagent. You might need to add your reagent in portions since it can be an exothermic reaction causing your reaction mixture to overflow out of your reaction flask. **BEFORE** quenching your reaction, make sure to cool your reaction flask to the specific temperature that the protocol recommends, since this is also exothermic.

Working with Acids & Bases

Acids and bases are corrosive and will destroy body tissue. The extent of injury depends on factors such as the type and concentration of the chemical, the route of exposure, the type of tissue contacted, and the speed used in applying emergency measures. Acids, especially in concentrated form, are most likely to cause immediate pain upon contact with tissues. Skin contact with strong bases usually goes unnoticed, since pain does not occur immediately.

The eyes are especially susceptible to acids and bases and must be immediately flushed with water for at least 15 minutes if exposure occurs. Inhalating acid fumes and airborne dust and mist from bases irritate the nose, throat, and lungs. Pulmonary edema, a severe irritation of the lungs resulting in fluid production that prevents the transfer of oxygen to the bloodstream, can also occur from intense extreme airborne exposures. Secondary toxic effects may occur if the material is absorbed from the lungs into the bloodstream. The extent of these effects depends on the concentration in air and the duration of exposure. Ingestion causes severe burns of the mucous membranes of the mouth, throat, esophagus, and stomach.

Dilution of acids and bases is exothermic. This is particularly true for sulfuric acid and potassium hydroxide. Concentrated solutions of inorganic acids and bases are not in themselves flammable. Combustion can occur, however, when an oxidizing acid is mixed with other chemicals or with combustible materials. Acids also react with many metals, resulting in the liberation of hydrogen, a highly flammable gas. Bases such as sodium hydroxide will liberate hydrogen gas upon reaction with aluminum, magnesium, tin, and zinc metal. Some acids are strong oxidizing agents and can react destructively and violently when they come in contact with organic or other oxidizable materials. Perchloric acid may form explosive perchlorate crystals, which are shock-sensitive and can detonate. Acids can form toxic reaction products when combined with cyanide or sulfide salts. The corresponding products are hydrogen cyanide and hydrogen sulfide gas.

High concentrations of **hydrofluoric acid (HF)** will cause immediate pain and tissue destruction. These effects may be delayed by several hours with weaker concentrations. Fluoride ion from HF also penetrates the deep tissue layers and can cause bone damage. **When using HF, make sure to have Calgonate® Gel nearby, just in case of a HF burn does occur.** Calgonate® Gel is an effective topical 2.5% calcium gluconate gel that is used in first aid response to HF exposure or contact to the body. Calcium gluconate combines with hydrofluoric acid to neutralize the powerful fluoride ion.

http://www.lbl.gov/ehs/chsp/html/acids_bases.shtml

Solvent Distills

As you may know, many organic reactions are sensitive to air and water. To insure we keep our solvents dry, we distill our solvents over an appropriate desiccant (sodium and benzophenone) under an inert atmosphere and gas (argon). When using these distills, follow these rules:

- **NEVER** leave the tap open, make sure to close it
- **NOTIFY** the person that's in charge of distills if you take more than 200 ml of solvent/base
- **NEVER** let the distills go dry
- **ALWAYS** leave DCM, Et_2O and THF distills ON
- KEEP the MeCN, Et_3N, PhMe, TMSCl and *i*-Pr_2NH distills OFF, unless you are refilling/redistilling these solvents/bases

For more lab safety concerns, visit UCSB's EH&S Laboratory Safety & Chemical Hygiene website:

http://www.ehs.ucsb.edu/units/labsfty/labsafety.html

Or you can contact, *Dave Vandenberg, Laboratory Safety Specialist*:
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