U N I V E R S I T Y *of* L O U I S V I L L E

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Laboratory Safety

General Considerations

1) Lab goggles/ glasses **MUST** be worn at all times while working in the lab! This is extremely important because even things that seem pretty common and safe (e.g., using the rotovap) involve placing glassware under reduced pressure, which can lead to implosions.

Note: See attached (at this document's end) account from Dr. K. Barry Sharpless concerning the damage an exploding NMR tube can cause to a person when they forget to wear proper safety glasses.

2) Do not wear gloves or your lab coat at your desk or in the group room.

3) Gloves can and should be reused if they are not contaminated. Just carefully remove them and place on your bench for reuse. Unless you are using highly toxic reagents (in which case you should throw out gloves after any chance of contamination – see toxicity hazards section), you should not have to use more than 2-3 pairs of gloves a day. Do not wash gloves with organic solvents (latex and nitrile gloves are permeable to acetone).

4) Know where all eyewashes are located in the lab.

5) Know where all safety showers are located in the lab.

6) Know where all fire extinguishers are located in the lab, what kind they are, and what they can be used for.

7) Nothing should be stored on the lab floors! Keep the floors free of anything other than lab stools, wooden platform boxes, etc.

8) Try not to work alone (especially late at night) in the lab (computer work is okay). This is particulary true if you are doing a large scale-up, running reactions with very reactive materials (i.e., strong oxidants or reductants, Grignard reagents, lithium reagents, etc), carrying out reactions requiring high pressure, or when running a reaction for the very first time. If you do end up working alone, always leave the door open so that someone can get in if there is a problem. Avoid quenching or dispensing large quantities of highly reactive chemicals when no one else is around.

9) The last person out of the lab should turn all lights out and lock all doors.

10) EMERGENCY PROCEDURE: Dial 911 on campus phones – this will connect you to the campus emergency center. Be sure to tell them exactly what happened and what you need(e.g., ambulance, fire trucks, police, etc) and where you are. Otherwise, they will send the campus police over to find this out first which takes lots of time.

Reaction Safety

1) LABEL, LABEL, LABEL all your reactions clearly! This is not only for your safety, but for everyone else's as well.

2) Reactions under high pressure (e.g., with condensed gases or in super-heated solvents) are explosion hazards and should be treated with extreme caution. A blast shield should be placed in front any system larger than an NMR tube under pressure. NMR tube reactions should also be treated with extreme caution – the hood sash should always be lowered when NMR tubes are under pressure.

Note: See attached (at this documents end) account from Dr. K. Barry Sharpless concerning the damage an exploding NMR tube can cause to a person when they forget to wear proper safety glasses.

3) Water condenser hoses should be fastened with Cu wire, and water flow should be turned as low as possible at night (water pressure increases at night).

4) Although water aspirators are used all the time in the lab (for filtration, etc), you should keep in mind that these involve reduced pressures and therefore pose a significant implosion hazard. Use caution when evacuating any flask [especially large round bottoms and large filter flasks (>500 mL)] and check glassware regularly for cracks.

5) Exercise caution in pulling tubing off Schlenk ware! If it is too difficult to remove the tubing, carefully cut the tubing away with a razor blade. Excessive jerking and pulling will snap the glass stopcock off, which may result in a cut to your hand. Remember rubber tubing is cheap, and it is meant to be cut when necessary.

Common Explosion Hazards

1) Oxidants (e.g., bleach, CrVI and MnVII salts, hypervalent iodine reagents, H_2O_2, etc) should be placed in separate waste from organic reagents/solvents. The oxidation of organics with these reagents can lead to violent exotherms/explosions.

2) Oxidizing acids (e.g., nitric acid and aqua regia) can react extremely violently with organics (especially acetone), and the resulting explosions/release of corrosive solutions can lead to serious injury. Acids should always be stored in a **separate location** from organic chemicals. Additionally, waste bottles for acids should be clearly marked and placed in a **separate location** from organic waste. This will prevent mistakenly pouring acid waste in with organics (which is the most common cause of this type of explosion).

3) Perchlorate salts can explode without warning, especially when concentrated in the presence of organics (once again, ClO_4 – is a strong oxidant!). Always use a blast shield when concentrating mixtures containing these salts and avoid the use of the ClO_4 – counter anion whenever possible.

4) Metallic lithium should **never** be placed in N_2 filled dry boxes or under a nitrogen atmosphere on your line. A violent and highly exothermic reaction will result from spontaneous "Li_3N" formation.

5) Remember that something as common as flash chromatography columns are run under high pressure and can crack/explode unexpectedly.

6) The condensation of liquid O_2, liquid N_2 and solid Ar in traps on your vacuum line can lead to explosions. See the vacuum line safety section for further details.

Toxicity Hazards

- Thallium salts (e.g., TlOEt).
- Alkyl mercury salts (e.g., $HgMe_2$).
- Tin reagents (especially tetra-alkyl or tri-alkyl aryl Sn compounds).
- Alkylating agents (e.g., MeI).

1) Exercise extreme caution when using these reagents!! Clean up spills in your hood and in public areas (balances, dry boxes, etc) immediately using appropriate procedures, and dispose of cleaning supplies/gloves in solid waste containers beneath the hood (to avoid fume inhalation).

2) Dispose of gloves (in solid waste container beneath the hood) whenever you may have come in contact with these reagents.

3) If any of these compounds are used in the dry box, be sure to (i) use a secondary pair of gloves so as not to contaminate the main gloves, (ii) dispose of all contaminated waste in a separate Ziploc bag before removing it from the box, and (iii) purge the box after the use of these compounds (and before opening the antechamber).

4) For specific instructions on how to wash glassware that has contacted these reagents, speak with Dr. Burns directly.

A cautionary tale from the past

K. Barry Sharpless

The following essay was written some time ago, but continues to pop up on laboratory doors around MIT, most recently at the Ceramics Processing Research Laboratory where it was read by a Tech Talk reporter. It is reprinted here with Dr. Sharpless' permission in an effort to reach everyone in the MIT community. Dr. Sharpless was a long-time member of the MIT faculty, last holding the Arthur C. Cope Professorship in Chemistry. He is now at the Scripps Research Institute in California.

Many of you may know that I was blinded in one eye during a lab accident in 1970, shortly after I arrived at MIT as an assistant professor. I always wore glasses whenever I was at my bench, and while I felt I conscientiously observed safety measures, my experience proves one can't be too cautious about wearing safety glasses.

As I prepared to go home from the lab during the early hours of the morning of the accident, I looked in the bays to see what my co-workers were doing, and then returned to my own bench, removed my safety glasses, and put on my parka. As I was walking to the door, I passed the bench where a first-year graduate student was flame-sealing an NMR tube. I asked how it was going, and he replied, "Good, I've got it sealed."

He was sealing off the tube at atmospheric pressure under a flow of nitrogen gas while cooling the tube in a liquid nitrogen bath, a technique neither of us had performed before. Nor, I regret to say, had we looked up the procedure, which we subsequently discovered to be incorrect.

I stopped by his bench, picked up the tube from the bath, and held it to the light. The tube immediately frosted over, and, as I wiped it to better see the contents, I noticed that the solvent level was exceedingly high. Suddenly the solvent level dropped several inches. Though I instantly realized condensed oxygen had been sealed in the NMR tube, I was quite literally unable to move a muscle before it exploded. Glass fragments shredded my cornea, penetrated the iris, and cause the partial collapse of one eye. My only other injuries were superficial face cuts.

My first two weeks at Mass Eye & Ear were spent totally immobilized and with *both* eyes bandaged. The pain was terrific, but my fear was even greater: I had been warned that when my eyes were uncovered there was a small chance I might blind in *both* eyes due to "sympathetic ophthalmia." Because eyes are walled off from the rest of the body *in utero*, eye protein driven into the blood stream can raise in immune response that leads to the "killing" of the uninjured eye. My disappointment at having no functional vision in my injured eye was, needless to say, surpassed by my joy at retaining full vision in my good eye.

The lesson to be learned from my experience is straightforward: there's simply never an adequate excuse for not wearing safety glasses in the laboratory *at all times*.

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