

Weck Group

Guidelines and Procedures

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1. Laboratory Safety

1.1. General Considerations

- 1.1.1. 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.
- 1.1.2. Do not wear gloves or your lab coat at your desk or in the group room.
- 1.1.3. 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).
- 1.1.4. Know where all eyewashes are located in each lab.
- 1.1.5. Know where all safety showers are located in each lab.
- 1.1.6. Know where all fire extinguishers are located in the lab, what kind they are, and what they can be used for.
- 1.1.7. Nothing should be stored on the lab floors! Keep the floors free of anything other than lab stools and the 10 L LN₂ dewar.
- 1.1.8. Do not work alone (especially late at night) in the lab (computer work is okay). This is particularly 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. Avoid quenching or dispensing large quantities of highly reactive chemicals when no one else is close by.
- 1.1.9. The last person out of the lab should turn all lights out and lock all doors.
- 1.1.10. EMERGENCY PROCEDURE: Dial 8-2222 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. For fire call 911.

1.2. Reaction Safety

- 1.2.1. LABEL, LABEL, LABEL all your reactions clearly! This is not only for your safety, but for everyone else's as well. Do not write on flasks, use 'detachable' labels!
 - 1.2.2. 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.
 - 1.2.3. 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).
 - 1.2.4. Although water aspirators are used all the time in the lab (for filtration, running the rotovaps, 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.
 - 1.2.5. 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.
- 1.3. Common Explosion Hazards
- 1.3.1. Oxidants (*e.g.*, bleach, Cr^{VI} and Mn^{VII} salts, hypervalent iodine reagents, H₂O₂, etc) should be placed in separate waste from organic reagents/solvents. The oxidation of organics with these reagents can lead to violent exotherms/explosions.
 - 1.3.2. 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).
 - 1.3.3. Perchlorate salts can explode without warning, especially when concentrated in the presence of organics (once again, ClO₄⁻ is a strong oxidant!). Always use a blast shield when concentrating mixtures containing these salts and avoid the use of the ClO₄⁻ counter anion whenever possible.
 - 1.3.4. Metallic lithium should **never** be placed in N₂ filled dry boxes or under a nitrogen atmosphere on your line. A violent and highly exothermic reaction will result from spontaneous "Li₃N" formation.
 - 1.3.5. Remember that something as common as flash chromatography columns are run under high pressure and can crack/explode unexpectedly.
 - 1.3.6. The condensation of liquid O₂, liquid N₂ and solid Ar in traps on your vacuum line can lead to explosions. *See the vacuum line safety section for further details.*
- 1.4. Toxicity Hazards
- Thallium salts (*e.g.*, TlOEt).
 - Alkyl mercury salts (*e.g.*, HgMe₂).
 - Tin reagents (especially tetra-alkyl or tri-alkyl aryl Sn compounds).
 - Alkylating agents (*e.g.*, MeI).
- 1.4.1. 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).

- 1.4.2. Dispose of gloves (in solid waste container beneath the hood) whenever you may have come in contact with these reagents.
- 1.4.3. 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).
- 1.4.4. For specific instructions on how to wash glassware that has contacted these reagents, speak with Prof. Weck or a Postdoc directly.

2. Vacuum Line Procedure—Using the Vacuum Line

You will receive thorough training on vacuum line technique by a trained group member before using your line. However, remember that many of the techniques involved can be confusing, and the consequences of making a mistake can be very dangerous as well as costly. Therefore, if you are ever in doubt about how to do something, please be sure to ask.

- 2.1. The following are references that contain a lot of useful information on almost all aspects for Schlenk and high vacuum technique (i) **Experimental Organometallic Chemistry**, Andrea L. Wayda and Marcetta Y. Darensbourg, Eds., American Chemical Society: Washington DC, 1987. (ii) **The Manipulation of Air Sensitive Compounds**, D. F. Shriver, Robert E. Krieger Publishing House: Malabar, FL, 1982.
- 2.2. Argon is a solid and N₂ is a liquid at LN₂ temperature (−195°C). Therefore, it is extremely dangerous to place LN₂ cooled flasks/traps under Ar or N₂ as significant quantities of these gases can condense. The huge pressure increase as the condensed Ar/N₂ warms and moves to gas phase can produce extremely violent explosions. As such, **never** backfill a flask a LN₂-cooled flask with N₂/Ar and/or leave it under a flow of N₂/Ar.
- 2.3. O₂ condenses as a bluish liquid at LN₂ temperature (−195 °C). LO₂ can condense in traps if the line is opened to air for any period of time and can spontaneously explode when co-condensed with organics. As such, **ALWAYS** evacuate traps before placing them in LN₂ (to remove air) and **ALWAYS** remove LN₂ before venting traps to air. Also, carefully monitor your vacuum pressure gauge to ensure that there are not serious air leaks that would allow condensation of LO₂ in the traps.
- 2.4. Exercise caution when evacuating **any** flask on the vacuum line. Large round bottoms and solvent bulbs (>500 mL) are especially significant implosion hazards and should be evacuated with the hood sash down. Also, regularly check glassware for cracks and remove and clearly label defective glassware. We can get it fixed (ask Mike).
- 2.5. Using solvent pots/flasks on vacuum line: Attach flask/solvent pot to line via rubber tubing. Be sure to use grease where appropriate. Evacuate head space (to below 10 mbar on gauge) and refill with N₂. Repeat this cycle three times. At this point all of the air should be out of your system and the flask/solvent pot can be opened to N₂ flow. **Note:** If the system won't pump down below 10 mbar, there is likely a leak, and all joints should be checked and re-greased if necessary. Do not simply proceed – contamination will cause problems!
- 2.6. Remember that all reactions/flasks that are open to N₂ can and do “see” and cross-contaminate each other. As such, only “compatible” reactions should be open to N₂ or Ar simultaneously, and care should be taken to avoid this situation if possible.

- 2.7. **A critical point:** When using common solvent pots, ALWAYS close off other reactions from the N₂ and thoroughly flush your Schlenk line with N₂ before exposing the pot to the N₂ atmosphere. If necessary, use the solvent pot on someone else's line rather than risking contamination.
- 2.8. The check valves are in place so that you avoid sucking oil and air into your line when you backfill evacuated flasks with N₂. However, they require a significant amount of N₂ pressure to reopen (after they are exposed to vacuum). Therefore, you generally need to turn up the flow through your bubbler when carrying out evacuate/refill cycles.
- 2.9. Traps
- 2.9.1. The trap closest to the Schlenk line should be cooled with LN₂ **whenever** you are using the vacuum part of the manifold.
- 2.9.2. The trap closest to the pump should be cooled with LN₂ any time you are removing more than 1 mL of solvent under vacuum.
- 2.9.3. If you are leaving something on the vacuum line overnight, be sure to fill the traps right before you leave and right when you arrive in the morning. Generally, the LN₂ will only last ~12 hr.
- 2.9.4. If not being used, the traps should be taken down at the end of the day, and the remaining LN₂ should be returned to the group 10 L dewar.
- 2.9.5. Before putting traps back up, be sure that they are completely free of solvent (if necessary place them in the oven for 15-30 minutes before proceeding).
- 2.10. Pumps and Pump Oil
- 2.10.1. Pump oil should be changed AT LEAST four times a year. This will typically coincide with group clean up days (*see group clean up section*). It is your responsibility to keep your pump clean (by avoiding contamination with solvents) and to change your pump oil on a regular basis. Remember, a clean pump will work smoother, longer, and most importantly it will pump down faster.
- 2.10.2. For problems with your pump (poor pump performance, leaking, strange noises, etc), immediately shut it down and talk to Mike Kahn in order to diagnose the problem.
- 2.10.3. Familiarize yourself with your pump by reading the operating manual. This will be extremely helpful when it comes time to change your pump oil.

3. Cleaning Glassware

Note: *Although it may not seem that important, cleaning glassware is one of the most important tasks that you will do in lab – contaminated glassware (along with contaminated solvents) are the two biggest causes of reactions going bad!*

- 3.1. General Group Glassware
- 3.1.1. Rinse out flask into organic waste to remove organic material by washing with a H₂O-miscible organic solvent like acetone, MeOH, or THF, depending on solubility.
- 3.1.2. Thoroughly clean grease off of all joints with hexanes and a Kimwipe.
- 3.1.3. Scrub both the interior and exterior of the flask vigorously with a washing brush and soap/warm water to remove salts and remaining residues.
- 3.1.4. Glass and Teflon stopcocks should be removed from joints before cleaning. They are easily damaged by small particles such as salts and the stopcock bore tends to hold up liquids.

- 3.1.5. Rinse flask with warm water (at least 2-3 times) and with distilled water (at least 2-3 times) to remove all soap/residues.
- 3.1.6. Finally, rinse with a small amount of acetone and place on the drying racks.
- 3.1.7. If glassware remains visibly dirty after this procedure **DO NOT** leave it on the drying rack for someone else to take and use!! ASK a senior group member about the best way to get it clean – this will usually entail either placing it in the base bath and/or washing with strong acid (e.g. conc. H_2SO_4 , HNO_3) to remove residual metal salts.

3.2. Frits

- 3.2.1. Rinse your frit with solvents in which the solids on it are soluble. Typically this would involve MeOH followed by acetone then EtOAc then CH_2Cl_2 . Then, turn the frit upside-down and rinse with these solvents a second time.
- 3.2.2. Note that aqueous washes (*i.e.*, those with bleach/water/acid/etc) are sometimes necessary to remove toxic reagents like Sn and/or other reagents that are soluble in these media. However, these washings need to be separated from the organic washings, and disposed of separately (*see waste section*). Also, washes with H_2O and/or aqueous solutions should be followed by copious rinsing with MeOH before the introduction of immiscible organics like CH_2Cl_2 or hexanes.
- 3.2.3. If residue remains (especially metal-based residue) it can often be removed by placing 50% conc. HCl and 50% MeOH in the frit and allowing it to drip through slowly, followed by rinses with HCl, H_2O and MeOH.
- 3.2.4. If *any* particulate matter remains on the frit and/or it is not completely white, you should place it in one of the buckets for cleaning with piranha solution (conc. $\text{H}_2\text{SO}_4/\text{H}_2\text{O}_2$) or aqua regia ($\text{HCl}/\text{H}_2\text{SO}_4$). However, YOU MUST COMPLETELY REMOVE ORGANIC SOLVENTS from the frit before subjecting it to piranha solution or aqua regia (highly oxidizing!). So, rinse the frits with MeOH followed by copious water before placing them in the bucket

3.3. NMR Tubes

- 3.3.1. Rinse the contents of your NMR tubes into organic (or aqueous) waste (depending on the contents of the tube).
- 3.3.2. Rinse tubes at least one to two more times with a wash bottle into your waste before using the NMR tube cleaner. These steps are important to avoid excessive contamination of the NMR tube cleaner with everyone's samples.
- 3.3.3. Note that you should never stick the tip of a wash bottle into an NMR tube to wash it out. This will inevitably lead to breaking the end off the tube. Instead, always hold the bottle several cm away from the end of the tube to spray the solvent in.
- 3.3.4. If solids/precipitated metals remain in the tube at this point, clean it out with some solvent (typically acetone) and a pipe cleaner.
- 3.3.5. Use the NMR tube washer to finish cleaning the tube. Typical solvent rinses might involve MeOH followed by acetone, then EtOAc then CH_2Cl_2 .
- 3.3.6. Note again that aqueous washings (*i.e.*, those with bleach/water/acid/etc) are sometimes necessary to remove toxic reagents like Sn and/or when the reagents used in NMR experiments are soluble in these media. However, these washings need to be separated from the organic washings, and disposed of appropriately (*see waste section*). Also, washes with H_2O and/or aqueous solutions should be followed by copious rinsing with MeOH before the introduction of immiscible organics like CH_2Cl_2 or hexanes.
- 3.3.7. Place NMR tubes flat in the oven to dry. However *do not* leave them in the oven for more than ~6-8 hrs (after which they should be placed in a desiccator for storage). Leaving the NMR

tubes in the oven for longer than this can lead to warping, which may cause problems with spinning, shimming and/or result in breakage in the NMR instruments.

3.4. Syringes/Needles

- 3.4.1. **ALL** syringes need to be cleaned directly after use! This prevents them seizing up or clogging (often irreversibly) with dried out residues. Additionally, these expensive pieces of glassware are in limited supply and are shared between many co-workers.
- 3.4.2. Clean gas-tight syringes by rinsing them 2-3 times with 3-4 different solvents. Typically this would include MeOH, acetone, EtOAc, and CH₂Cl₂.
- 3.4.3. Gas tight syringes should be placed in the oven after cleaning *without their plungers* for 3-4 hrs. Longer times in the oven can lead to cracking and/or damage to the syringe. They should then be placed in a dessicator. Plungers should be wiped off and placed directly into a dessicator after cleaning. This prevents irreversible expansion/contraction of the plunger from repeated heating/cooling cycles.
- 3.4.4. Non-disposable needles should be rinsed thoroughly using the aspirator vacuum needle cleaner with appropriate solvents (typically MeOH followed by acetone then EtOAc then CH₂Cl₂).
- 3.4.5. Once again, note that aqueous washing of both gas tight syringes and needles (*i.e.*, those with bleach/water/acid/etc) are sometimes necessary to remove toxic reagents like Sn and/or when the reagents used are soluble in these media. However, these washings need to be separated from the organic washings, and disposed of appropriately (*see waste section*). Additionally, washes with H₂O and/or aqueous solutions should be followed by rinsing with copious MeOH before the introduction of immiscible organics like CH₂Cl₂ or hexanes.

4. Glove Box

4.1. General instructions

- 4.1.1. You must be checked out by the person in charge of the glove box before using this piece of equipment.
- 4.1.2. *Always* sign in the logbook when using the glove box. Please write your initials, which chamber you used, time in/out, any solvents or reactive chemicals used, and whether or not you purged.
- 4.1.3. *Always* turn off the circulator before and purge the glove box after using more than 1 mL of solvent or other volatile liquids in the box. More details about purging are in a separate section below.
- 4.1.4. Flasks being brought into the glove box are subjected to high vacuum, and therefore need to be appropriately sealed to prevent them from bursting open in the antechamber. This can be especially dangerous if you are bringing in a reagent that is pyrophoric and/or will cause damage to the pump. When bringing a sealed flask into the dry box, ***ensure that it has been completely evacuated on your line***. All of the joints should be well greased, and the top should be covered with a greased stopper (*not* a septum). Always keep an eye on the pressure in the antechamber when pumping in reactive chemicals.
- 4.1.5. Paper products (paper towels, Kimwipes, etc) are filled with water and should not be brought into the box unless properly treated. Kimwipes must be placed in the oven for 6-8 hours (be sure to cut off the plastic part of the Kimwipes package or it will melt in the oven). They are then to be placed in the antechamber while hot and left in the chamber under vacuum overnight (for 12 hours) before being brought in.
- 4.1.6. Cork rings are also filled with air and water and should *never* be brought into the box.

- 4.1.7. *Clean up after yourself when using the box!* This means using the dustpan if you spill *and* placing your non-solvent contaminated trash in the waste bag before exiting.
- 4.1.8. Pipettes that are contaminated with any solvents should **not** be placed in the waste bag in the box. The solvents will slowly evaporate and contaminate the closed atmosphere of the box, and this essentially negates the effects of purging the box after solvent use. Therefore, **always** remove all pipettes that have been used for dispensing solvent when you exit the box.

4.2. Using the small antechamber

- 4.2.1. Turn the manual valve below the chamber towards “refill”, and refill to atmospheric pressure (0 mbar on the vacuum gauge) with N₂. Make sure that chamber is completely refilled with N₂ before opening doors – if it is still under vacuum when you try to open it *you will break the door!*
- 4.2.2. **Set valve to “closed” before opening outer door.** (Note: if you fail to close the valve before opening the outer door, then the interior of the box will be directly exposed to the outside atmosphere!)
- 4.2.3. Open outer door and place items in the chamber.
- 4.2.4. Turn valve to “evacuate”, and evacuate chamber (slowly and carefully, especially if there are any powders involved) to –1 mbar (usually takes ~ 30 s to 1 min). Then refill with N₂ to atmospheric pressure (0 mbar).
- 4.2.5. Repeat evacuate/refill cycle 3 times.
- 4.2.6. On last refill, refill completely to atmospheric pressure (if the chamber is under vacuum, the door will break if you try to open it). Open inner door and take your things into the box.
- 4.2.7. When you are done working in the box, place your items in the chamber and close the inner door. Again, make sure that the refill valve is **closed** before opening the outer door.
- 4.2.8. Close the outer door and place the chamber under dynamic vacuum when finished.

4.3. Using the large antechamber:

- 4.3.1. Use keypad to refill to atmospheric pressure. Make sure that chamber is completely refilled with N₂ before opening – if it is still under vacuum when you try to open it *you will break the door!*
- 4.3.2. **Turn off refill function.** (Once again, if you fail to turn off the refill function before opening the chamber to air, then the interior of the box will be directly exposed to the outside atmosphere!)
- 4.3.3. Open outer door, place items in the chamber and close door.
- 4.3.4. Evacuate chamber using keypad (slowly and carefully, especially if there are any powders involved) and leave under vacuum for 10 minutes. Refill with N₂ to –0.5 mbar on pressure gauge (about halfway to atmospheric pressure).
- 4.3.5. Repeat evacuate/refill cycle 3 times.
- 4.3.6. On last refill, refill completely to atmospheric pressure (if the chamber is under vacuum, the door will break if you try to open it). Open inner door and take your things in.
- 4.3.7. When you are done working in the box, place your items in the chamber, close the inner door, then open the outer door and take your things out.
- 4.3.8. Close the outer door and evacuate the chamber to –1 mbar on gauge. Turn off vacuum with keypad when chamber is completely evacuated (leaving it under static vacuum).

4.4. Dealing with solvents in the dry box:

- 4.4.1. **You should always turn off the circulator before using more than 1 mL of any volatile chemical or solvent in the box.** This prevents solvents from getting into the circulator and destroying the catalyst that cleans the atmosphere of the box. Failure to turn off circulator and

to purge after using solvents will ruin the atmosphere of the box and increase the frequency with which the catalyst must be replaced (an extremely costly, labor-intensive, and generally unpleasant task that should be avoided as much as possible).

- 4.4.2. Remember that the box is a closed system. Therefore, any solvent that you open enters the atmosphere and will contaminate any solvent that you open subsequently (until you purge to clear everything out). *As such, solvents are easily contaminated if you don't exercise caution.* You should always open and use deuterated solvents **before** using non-deuterated solvents, so that your NMR samples don't get contaminated. Also, always open solvents in order of non-polarity/reactivity (*e.g.*, pentane before toluene before ether before THF before CH₂Cl₂ before acetone, CH₃CN, DMSO, DMF, phosphines) so that the more reactive solvents do not contaminate the less reactive ones.
- 4.4.3. Also, do not open the freezer after you have used solvents in the box. The solvents will condense in the freezer, leading to contamination of the seals and samples.
- 4.4.4. When you are done using solvents, remove all pipettes that have touched the solvent along with your flasks/NMR tubes etc from the box.
- 4.4.5. Turn up the pressure.
- 4.4.6. Open the purge valve on the top of the box and purge for an appropriate amount of time. Typically for polar/reactive solvents: (THF, CH₂Cl₂, CHCl₃, dioxane, CH₃CH, acetone, volatile phosphines, thiols) as well as large (>50 mL quantities of pentane, toluene, etc) you should purge for ~20-30 min. For small quantities (<50 mL) of toluene, pentane, 10-15 min of purging should be sufficient. When using MeOH, always purge for at least 40 min.
- 4.4.7. When purge is complete, **turn circulator back on** and lower the pressure to min = 0.5 and max = 4.

5. Dri-Solv System

5.1. Dispensing solvent into solvent bulbs:

- 5.1.1. Attach flask to your vacuum line (**use only Teflon – Krytox – grease**). Evacuate bulb completely (<10 mbar) and refill with N₂. Repeat 3 times. On final cycle leave the flask under vacuum, and close Teflon stopcock.
- 5.1.2. Attach bulb to solvent system. Evacuate and refill the head space (between Teflon stopcock and 24/40 adapter) 3 times (by turning the valve to “evacuate” followed by “refill”). You should evacuate for ~ 30 s per cycle. On final cycle leave head space under vacuum and turn valve to “closed” position.
- 5.1.3. To dispense solvent, open solvent valve (upper valve) to “open” position
- 5.1.4. Use the metering valve (the one that turns) to dispense solvent carefully into flask.
- 5.1.5. When complete, close metering valve and solvent valve, and then close Teflon stopcock on your flask.
- 5.1.6. Use “refill” valve to refill line with N₂ and remove your closed flask.
- 5.1.7. Flush line with N₂ for ~ 1 minute to remove most residual solvent.
- 5.1.8. Cap the line with a yellow plug.
- 5.1.9. Fill the trap (on the left of the system) with dry ice/i-PrOH, and evacuate using “evacuate” valve.
- 5.1.10. After 5 min of evacuation turn “evacuate” valve to “closed”.

5.1.11. If any solvent has condensed in the trap, close off the pump and vent the system (using the three-way valve hanging next to the pump). Allow trap to warm and then empty contents. Then place system under vacuum again.

5.2 Dispensing solvent into round bottom flasks/reaction vessels:

5.2.1 If your reaction requires dry solvent, but is not extremely sensitive, you can dispense directly into the round bottom flask. In this case, simply place the flask below the spigot, and turn on the solvent flow.

5.2.2 When solvent dispensing is complete, purge out the line with N₂ (using "refill" valve) for ~ 1 min. Then carry out steps 5.1.7 to 5.1.11 above.

6. Instruments

6.1. Everyone must be trained by the person in charge of the instrument before using this instrument.

7. AcbH `y Cleanup

7.1 We will have a clean up once a month. I expect EVERYBODY to help out and be on time.

8. Lab Notebooks

8.1. Maintaining a clear, well organized, and up-to-date lab notebook is critical for (a) keeping track of your experiments for your thesis, (b) any publications/ patents that you will write and (c) enabling future generations of students to reproduce your work.

8.2. General instructions for keeping a lab notebook are as follows.

8.3. Skip 3-4 pages in the beginning for the Table of Contents and update the TOC regularly (monthly, at least).

8.4. Use only non-erasable ink in your notebook.

8.5. Write the reaction/experiment clearly at the top of each page. If you are following a published procedure, indicate the reference from which the procedure was obtained.

8.6. Make a table including each reagent, its molecular weight, the measured quantity – g (or mL), mol, and eq – used in the reaction, and the commercial source/purity of the reagent.

8.7. Write a detailed experimental, including the rate/order/time/temperature of addition of each reagent and solvent, and, where appropriate, any color changes that take place during the reaction. Also, detail all work up procedures and TLC data (where appropriate) for the reaction.

8.8. Be sure to weigh the product and determine the % yield for all reactions!!

8.9. NMR spectra should be saved and labeled according to the notebook number, page, and sample they refer to. For example, 1mw23.007 would refer to Marcus Weck notebook #1, p. 23, sample #7.

8.10. Everyone is responsible for backing up their data on CD's and to put their data onto the server.

8.11. Sign and date your notebook at least once every four weeks. Have a witness (your lab partner) co-sign it.

9. General

9.1. Please notify Prof. Weck and Darin Dolfi if you will be out of town for one working day or more. Also provide an emergency contact number.

- 9.2. Weekly group meetings will be held at 6:00 pm on Wednesday nights – please notify Prof. Weck if you cannot attend for any reason.
- 9.3. It is important to keep up on the current literature in organic and organometallic chemistry – particularly as it relates to your project.
The following are journals that you should read each week:

J. Am. Chem. Soc.
Macromolecules
Angew. Chem., Int. Ed.
Science
Nature

9.4. General tips for reading the chemical literature:

- 9.4.1. You cannot expect to read everything (that is why we have literature group meeting).
- 9.4.2. Try to read papers that are (i) the most interesting to you and (ii) the most relevant to your and the group's research projects.
- 9.4.3. No one has time to read the entire text of every article. Read the abstract and introduction and then try to discern the major point of the paper from the Figures and Schemes. If you find something especially interesting or unclear consult the text for further details. Keep in mind when writing your own papers that these are the sections that are usually the most read.
- 9.4.4. Whenever possible, discuss with others what you have read (that is what lit meetings are for)! This will solidify your general knowledge as well as improve your understanding of what you have read.
- 9.4.5. Keep an eye out for molecules that could be assembled using the methodology that you are developing. This will be helpful for those of you who are interested in applying methodology in total synthesis, as well as for writing proposals.

9.5. Other journals to keep an eye on are:

J. Polym. Science
Adv. Synth. Catal.
Chem. Comm.
Chem. Reviews
Acc. Chem. Res.
JOC
etc.
