

DINCĂ GROUP
web.mit.edu/dincalab

LAB MANUAL

Frequently unanswered questions:

Q: What is graduate school? What does getting a PhD mean?

A: Graduate school is not a 9-to-5 job. You are here to become a scientific leader, not a technician. To get a PhD means that YOU contribute something original to the scientific community. When getting a PhD you should know more about your project than anyone else.

Q: How do I make sure I have a successful PhD?

A: This requires a multi-tiered answer, all of the following bearing equal importance:

1. Take ownership of your project. You will learn to love it and hate it, but you will have a special connection with it.
 - a. Know the background information better than anyone else.
 - b. Know the caveats of your research better than anyone else.
 - c. Don't become an automaton. Don't simply follow SOPs without understanding how an instrument works. Don't take instruments and software programs as black boxes. Educate yourself about each and every technique/theory that you are using to collect and analyze data. Learn from others, but understand what you're learning.
 - d. Get the help you need. Take full advantage of group meetings, seminars, other members in lab, and especially the *MIT faculty* and your colleagues. You are surrounded by some of the best experts in the world in almost every science and engineering discipline.
 - e. **VERY IMPORTANT:** don't get too focused. It is easy to lose yourself in your project. Keep up with literature, go to seminars **OUTSIDE** your area (e.g. Materials Science, Excitonics, Bioengineering - use RSS Feeds from MIT Events to learn about many of these), and take classes that you are interested in. An MIT graduate should be broadly educated.
 - f. Learn how to train people. If you want to do research only with your own two hands, you should not be getting a PhD. People with PhDs lead research teams (either in industry or academia). Train UROPs and TA classes. Take the time to show others in the group how to do something.

Q: How much am I expected to work?

A: As much as you need to move your project along. Ambiguous? Yes. Some projects are harder than others, so don't be frustrated that you need to work late on weekends, when your neighbor doesn't (although your neighbor probably should, too). As a general rule, however, you will be expected to work **~60 hrs/week (including weekends)**, whether it is experimental work at your hood, bench, or glovebox, writing manuscripts, preparing presentations, etc.

Q: Am I expected to write papers?

A: Yes. Whatever you do with your life after grad school, it is important to be able to communicate effectively. You should learn how to write papers and you will write the first draft of all the papers you are a first author on. You will learn how to make appropriate figures and how to give effective presentations. As a first author on a manuscript, you should double check

every reference and every tick mark or symbol on a graph. You should know your paper inside and out, you are the expert.

Q: Is there a specific way I need to keep my notebook?

A: Yes. People will come after you and will need to repeat experiments that you've done. It is of paramount importance to keep a good record. See details in the following pages.

Q: How do I plan for the future?

A: Regardless of what you do after grad school, you will need letters. Recommendation letters are more important to your future than anything else. Do the best job you can on oral presentations and oral exams. Your letter writers won't have the time to read your papers, so you need to impress through your presentations and your thesis. When you meet with your advisor/chair, bring a sheet of paper with some discussion points, don't show up with a blank notepad. Finally, treat faculty with respect and be a good citizen of the department. Research is about teamwork. Show that you can get along with others and help out when the situation calls for it.

LAB SAFETY

General Requirements

All graduate students, UROP students, postdocs, visiting scientists, and other researchers working with chemicals and other hazardous materials must complete the Training Needs Assessment which is accessible through the EH&S Office's training web site <http://web.mit.edu/environment/training/>. All new students/postdocs must complete the following steps **prior to** beginning work in lab. Even if you do not work in areas where hazardous chemicals are in use you still need to complete step 5, listed below.

- (1) New personnel must attend the **Chemical Hygiene and Safety Lecture** presented in January or view a video recording of the lecture. The video is accessible through the training web site listed above.
- (2) Read and understand the Chemistry Department **Chemical Hygiene Plan and Safety Manual**. Copies of the book can be obtained in Chemistry Headquarters or from Jim Doughty (4-469).
- (3) Receive **Initial Lab Specific Chemical Hygiene and Safety Training** from the group's EH&S Representative. This is an orientation to the location of safety equipment in the laboratory and to special hazards associated with the research in the group.
- (4) Complete the training course, **Managing Hazardous Waste**. This is offered as a web based course and is accessible through the EH&S Office's training website listed above. This is an annual requirement.
- (5) Obtain the **EH&S Clearance Form** from Chemistry Headquarters or from Jim Doughty (4-469). New members must sign the EHS Clearance Form and obtain Mircea's signature and submit it to Jim Doughty (4-469).

No individual is permitted to work in areas in which chemicals are in use until all of the above steps have been completed and a signed EHS Clearance Form is submitted.

Note: Additional training is required for individuals working with other hazardous materials including (but not limited to) **lasers, radioactive materials**, and certain **biohazardous materials**. Completing the Training Needs Assessment Form will dictate your training needs. It is the responsibility of the researcher to ensure that all of these training requirements are met.

Contact Jim Doughty EH&S Coordinator (324-6132, jdoughty@mit.edu) with any questions.

Additional Dincă Lab requirements

- 1) Make sure you are trained by a senior group member or by Mircea before handling toxic, caustic, flammable, and explosive compounds and sign the training log in the group room to keep a record of your training. Examples of compounds to be particularly careful with:
 - **perchlorates (NEVER** use a perchlorate without prior approval by MD)
 - **cyanides and azides (NEVER** use acid wash or work outside the hood - HCN and HN₃ are extremely potent poisonous gases and the latter is also explosive)
 - **diazoalkanes** – they explode
 - **azides** -in addition to the above, NEVER use azides in chlorinated solvents – they can form diazoalkanes, which are explosive.
 - **^tBuLi** - Avoid it if you can. If you cannot, let Mircea know before you use it. In principle, you can only use it in the glove box. Tert-BuLi burns violently when encountering air. **Do not use ^tBuLi in THF and ether. It burns.**
 - Metallic **Li/Al/Cs** 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.
 - **Pd over Carbon catalysts** are known to be flammable. So it is normal if one sees fire when working reaction up. This is probably why some people use Pd/C that contains water. Water is compatible with this catalyst. So when you do hydrogenation reaction using Pd/C, be cautious. Read appropriate literature for handling. **Pd/C with methanol is a dangerous combination**, so should be avoided. With EtOH it might work. In any case, make sure you are aware of the danger and be ready for any small fire. In particular, work in the hood where there are no other flammable materials nearby (such as solvents, reagents). Have quick access to water to put out a small fire.
 - **Toxicity Hazards:** 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). Dispose of gloves (in solid waste container beneath the hood) whenever you may have come in contact with these reagents. Dispose of all contaminated waste in a separate Ziploc bag before removing it from the box, and purge the box after the use of these compounds (and before opening the antechamber).
 - Thallium salts (e.g., TlOEt).
 - Alkyl mercury salts (e.g., HgMe₂). **Warning: never use HgMe₂.**
 - Tin reagents (especially tetra-alkyl or tri-alkyl aryl Sn compounds).
 - Alkylating agents (e.g., MeI): potent carcinogenics
 - Hydrazines (esp. MeNH-NH₂ and anhydrous H₂N-NH₂): potent carcinogens
 - Be careful with sulfur containing molecules as they can be quite smelly!

2) It is forbidden to leave the following types of experiments unattended:

- ° use of toxic gas: CO, phosgene, phosphines, chlorine
- ° very exothermic reaction as diazotization, Grignard, hydrogenation, nitration, etc.
- ° manipulation of alkali metals.
- ° prepare a reaction in autoclave

3) Notify the group's EHS representative and Mircea immediately if you have been injured or if a major spill occurred (toxic, caustic, or flammable compound).

EMERGENCIES – Dial **100** from any campus phone for assistance in **any emergency**.

NOTEBOOKS

General

ALL members of the group will use the group sanctioned laboratory notebook:

Rediform Computation Notebook

4x4 Quad, 11-3/4"x9-1/4"

Upon joining, each group member will obtain **15 GB** of storage on the group server, where they should back up ALL electronic raw data (**do not store large pictures**). Each group member will also set-up a free TSM backup account with MIT, which will allow them to store **15 GB** of data free of charge.

Keeping a proper notebook

- Leave 6 blank pages in the beginning for the Table of Contents. Be sure to update the TOC frequently, both for your own good, and especially for people coming after you that will try to replicate/study your work.
- Each experiment will bear a label containing the notebook owner's initials followed by an underscore and notebook page number. For instance, the label MD3_45 corresponds to an experiment in Mircea's third notebook on page 45.
- Characterizations do not qualify as new experiments. For instance, the IR, UV, mass spec, or TLC of a product or a new MOF should not receive new numbers. It is **required** that you paste reduced size copies of IR and UV spectra that you take directly into your notebook using magic tape. NMR and other data need not be pasted as images in the notebook, but this is recommended too.
- Full size copies of ALL spectra must be kept in dedicated spectra folders to accompany each (or multiple) notebook, as necessary.
- If a major new experiment in UV analysis or gas sorption is undertaken, for instance, they should be considered new experiments and should receive proper labeling in the Table of Contents.
- Combinatorial-type small scale exploratory reactions will be recorded in a table and each table will be counted as a *single* experiment.
- If you use ChemDraw to set-up your reactions, always check that the formula and MW given by ChemDraw's recognition software agrees with your own calculation for your starting materials. Getting the stoichiometry right is the first important step in any chemical experiment.
- Do not erase anything in the notebook. Simply use ~~strikethrough~~ and continue your text. You learn from mistakes, so don't delete them.
- Date your notes every time you write something in your notebook. If an experiment takes multiple days, you will have multiple dates on the same page.
- Make detailed observations. The more detail you provide, the easier it will be for yourself and those who will come after you to follow your work.
- **BACKING up data:** all electronic data (spectra, raw adsorption data, e-chem data, etc) **MUST** be backed up **WEEKLY** on the group's Network-Attached Storage Drive. If you do not have an account on the network drive, contact the computer person/Mircea to get one.

RUNNING REACTIONS

General

For a great reference on synthesis and useful lab techniques, see the Not Voodoo page from Alison Frontier's website at the University of Rochester (linked from our group's webpage under "Links"). This website has information on many basic techniques such as: TLC staining, temperature of various baths, flash chromatography, etc. For additional information on flash chromatography see "*Rapid Chromatographic Technique for Preparative Separations with Moderate Resolution.*" *J. Org. Chem.* **1978**, *43*, 2923-2925.

If you are not sure how to set up a reaction or an experiment, ASK a senior member of the group or Mircea for help.

Details

- Start with pure reagents and chemicals. See "*Purification of Laboratory Chemicals*" by Armarego and Chai for information on purifying common reagents and solvents.
- For information on how to handle air-sensitive compounds and perform air-sensitive manipulations outside of the glovebox see "*Manipulation of air-sensitive compounds*" by Shriver and Drezdson.
- Wipe off balance surface and pan with a balance brush after each use.
- Run new reactions on a small scale (~100-200 mg). After successful identification of reaction conditions and purification procedure, you may scale up the reaction. This is especially true for reactions involving expensive 2nd and 3rd row transition metals or similarly priced organic chemicals.
- Monitor organic reactions by TLC. Crude NMR and IR can be used, but only in addition to TLC.
- ALWAYS work up reactions immediately upon completion. Leaving a reaction sitting overnight because it is too late is not OK. Plan your reactions accordingly.
- If you observe major by-products take the time to identify them. This may help in devising more efficient/higher yielding routes towards your product.

Glove Box Usage

Glove boxes can be very useful if you know how to use them. They need careful maintenance. **Always follow the rules. If you are not sure, ask!**

The glove box is meant to provide the convenience for working under inert atmosphere, i.e. without O₂ and H₂O. But before it can do it, you need to make sure you do not release even traces of O₂ and H₂O into the box. The general rule is that everything is bad for the glovebox. With a few exceptions, nothing goes into the glove boxes as it is.

Solvent scales:

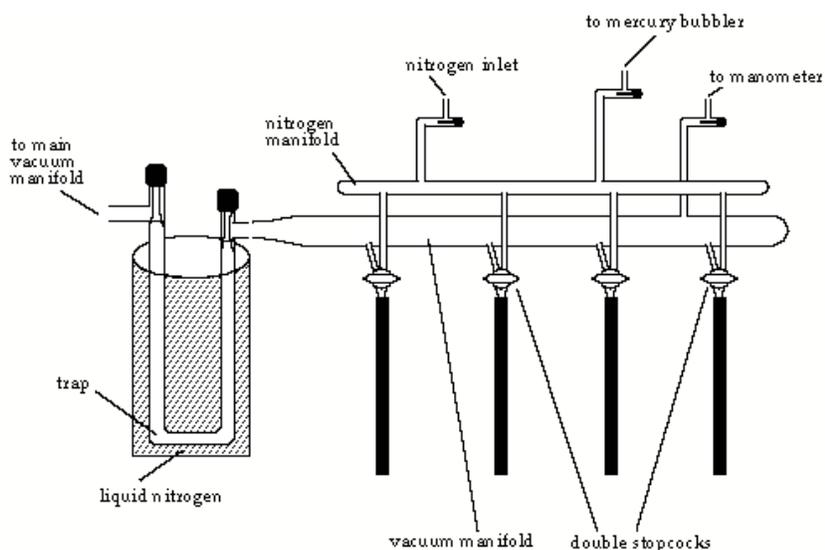
- I. Solvents **compatible** with the purification catalysts: Pentane, Hexane, Benzene, Toluene, Heptane, and other hydrocarbons.
- II. Solvents **bad** to the catalysts: THF, Ether, DME, etc.
- III. Solvents **very bad** to the catalysts: Methylene Chloride, Acetonitrile.
- IV. Solvents **extremely bad** to the catalysts: Pyridine, Methanol, Chloroform, DMSO, DMF, Pyridine, volatile Sulfur-containing compounds.
- V. Never use as solvents: acids, water etc.

1. The box can work under three modes: circulation (blower on), purging, or neither. Under circulation, the box atmosphere goes through the catalyst for purification. The box should be under the circulation mode when not in use and/or when only compatible solvents are being used. **Switch the circulation off when you are working with solvents/vapors that are toxic to the catalysts, e.g., solvent II to IV.** After that, purge the box to regenerate the atmosphere. Normally a 30-min purge is required. If you just used small amounts of THF and ether, then a 20 min purge is enough. Do not turn back to circulation without purging.
2. Every morning, before using the box, you should purge it for 15 min.
3. Regenerate the box every 3 months (depending on the performance) to reactivate the catalyst. Regenerate the activated carbon.
4. Do not leave anything open in the box for too long. Solution reactions running in vials should be capped.
5. If the box atmosphere contains other vapors, purge the box for 15 min before running a routine NMR, and 30 min before making samples for a very clean NMR.
6. Be careful of solvent contamination. If you are using ether, do not open a bottle of other solvents because you don't want those solvents to contain ether as well. Purge generously if you are concerned.
7. For big antechambers, evacuate/refill for 3 times, every time 15 min. For small antechambers, evacuate/refill for 3 times, every time 5 min. Exception can be made under special circumstances (e.g. transferring crystals), but use good judgement.

- **ALWAYS use the log book to see the status of the antechambers**
- **Clean up after yourselves – especially the balance and the hot plates**

Schlenk Procedures — Using the Vacuum Line

Make sure that you never pump air in a cold trap. Oxygen will condense and explode! The Schlenk techniques are named for Schlenk, who investigated the complex structures and equilibria for common Grignard reagents in solution. These techniques have been modified and refined to deal with transition metal compounds that are sensitive to the components of normal air (dioxygen and water are typically the most problematic) and are thus also referred to as “Inert Atmosphere Techniques”. Moderate to quite air-sensitive compounds are routinely manipulated by these procedures in modern laboratories. Extremely air sensitive or very volatile compounds, however, are not amenable to Schlenk techniques and are handled using “Vacuum Line Techniques”.



I. Schlenk Techniques

The basic piece of equipment used in this project is the double manifold, or Schlenk line: The designs of Schlenk lines vary in individual labs. At MIT, we are not allowed to use mercury bubblers, so we use silicon oil bubblers. A digital vacuum manometer can be used to monitor the vacuum. Inert gas (argon or nitrogen) is provided through the top manifold. The inert gas enters from a tank via the indicated stopcock and any over pressure exits through a oil bubbler (not shown). The vacuum manifolds in Mead are all connected to one main manifold, which is isolated from the oil pump by a liquid nitrogen trap (**solvents degrade pump oil**).

There are certain hazards associated with this apparatus. First of all, any time there is pressure or vacuum in use there is a possibility of **glassware failing due to fatigue**. Using the oil bubbler as an outlet for the over pressure of nitrogen greatly reduces the chance of explosion, but the risk of implosion is not as readily controllable. Even glassware that is in apparently good condition can fail under vacuum as small as that provided by a water aspirator – the probabilities increase somewhat on a vacuum manifold, especially when the apparatus is subjected to thermal shocks. Liquid nitrogen in an open dewar presents no hazards beyond frost bite, however, **liquid N₂ condenses O₂ at reduced pressure**. Should a vacuum trap cooled with liquid N₂ be left exposed to the air, O₂ will condense. **Liquid O₂ is a deep blue color** -- if you ever see a deep blue color in a trap, get Mircea immediately and follow his instructions. If

Mircea is not available, a general advice is to **keep the vacuum on the system to pump the trap, and slowly warm up the trap (e.g., leave the trap on but not add more liquid N₂).**

LIQUID OXYGEN IN THE PRESENCE OF ORGANIC SOLVENTS PRESENTS AN EXTREME EXPLOSION HAZARD. You should always assume there are trace organic solvents in the trap.

II. PROCEDURE for working on the Schlenk Line:

1.1 Set-up

- 1.1.1 Always wear safety glasses whenever working in the hood area!
- 1.1.2 Examine vacuum manifold to insure that it ready to be evacuated
 - 1.1.2.1 Turn stopcocks to the horizontal position.
 - 1.1.2.2 The liquid trap {looks like a giant glass finger} is empty and securely clamped in place.
- 1.1.3 Turn on vacuum pump with the switch located near the motor of the pump
 - 1.1.3.1 Pump should become quiet within minutes indicating that there are no significant leaks
- 1.1.4 Connect the vacuum to the Schlenk line. This can be done by opening the corresponding stopcocks.
- 1.1.5 (optional) Check that the vacuum line is functioning correctly.
 - 1.1.5.1 Test vacuum by placing thumb over one of the hoses descending from the manifold
 - 1.1.5.2 Rotate the corresponding stopcock 90° CW such that the vacuum line is connected to your manifold.
 - 1.1.5.3 Return stopcock to the starting position by rotating 90° CCW
- 1.1.6 Fill the Dewar with a small amount of Liquid N₂ (20%). Place the Dewar under vacuum trap. Adjust lab jack to appropriate height. Fill the trap with liquid N₂.
 - 1.1.6.1 The Dewar is made of glass and is under vacuum. It will implode violently if the glass is shattered. Handle with care
 - 1.1.6.2 Liquid N₂ is a cryogenic coolant and will cause burns to the skin if handled with bare hands
- 1.1.7 The vacuum line is now ready to be used.**

1.2 Shut Down

- 1.2.1 Always wear safety glasses whenever working in the hood area!
- 1.2.2 Disconnect the pump from the manifold by turning off a connection (if your vacuum line has one) or by turning off the vacuum pump. **This is very important because we do not want to have the vacuum on while opening the Schlenk line to the air, or else we will condense liquid O₂!**
- 1.2.3 Open one of the stopcocks to relieve the vacuum in the line by rotating 90° CW and connect the Schlenk line to air. Valve can be left in the open position
- 1.2.4 Carefully lower the lab jack in order to remove the Dewar.
- 1.2.5 Remove the trap. **DO NOT** add the contents of the Trap to a waste container unless it is > 0°C
- 1.2.6 Vacuum trap can then be left in the hood to dry.

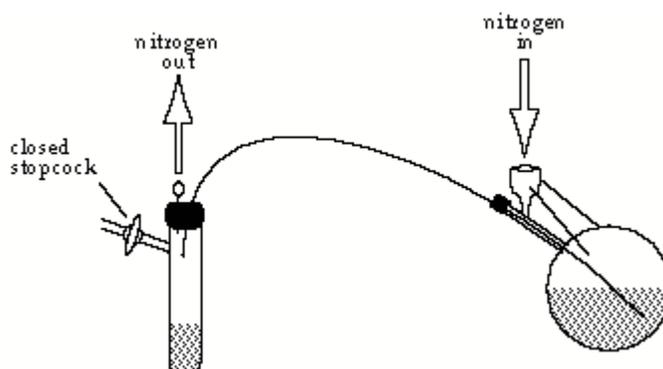
Vacuum Glassware (Schlenkware) is made with a side arm for evacuation of the apparatus and for the entering inert gas used to flush the apparatus. **Vacuum Grease** should be used when assembling an apparatus for use on the double manifold. Grease should be removed using pentane, hexane, or petroleum ether, kimwipes, pipe cleaners and a pair of forceps.

A **septum** (plural "septa") is a stopper with a thin section in the middle to allow transfer of liquids in and out of the vessel with needles. Septa should always be folded down and wired when in use. **Never pump down on a septum capped flask-- always use ground glass stoppers.** Septa do not hold vacuum very well, even when they have not been pierced, and are best used only with a positive pressure of N_2 .

Exchanging a ground glass stopper for a septum or vice versa, requires a moderately strong flow of N_2 . **Pump/fill cycles** are used to establish an inert atmosphere in a vessel. The vessel is sealed, but attached to the line via pressure tubing. The vessel is evacuated by opening the double stopcock so that the vacuum manifold and the hose are connected, then filled with argon by moving the stopcock until the argon manifold and the hose are connected. The argon flow should be monitored via the bubbler during this procedure. For best results, pump/fill cycles should be repeated three times.

A **cannula** is a long double ended needle. It is used to transfer liquids from one vessel to another. Cannulas should be kept in the oven, and purged with N_2 while still warm. The other end of the cannula is inserted into the receiving flask, and the stopcock closed so that the only flow of dinitrogen is through the cannula. A needle is placed in the receiving septum to vent, and the cannula is pushed into the liquid to be transferred.

Do not transfer liquids through a cannula with vacuum -- the interface of the septum and needle will leak air.



A **cannula filter** is a long needle with a piece of filter paper tightly wired onto a lipped glass attachment at one end. These can also be made from plain cannulas.

Syringes are used to transfer liquids without exposing them to air, but unlike a cannula, a flow of N_2 is not required. An inert atmosphere should be established in a syringe by repeatedly drawing N_2 into the syringe and expelling it. *Do not pull hard on a syringe to create a vacuum -- the syringe will leak.* Allow the positive pressure of the N_2 flow to push the barrel out. By the same token, **beware that the barrel is not forced all the way out of the syringe and broken.** If the compounds in question are water sensitive, the syringe should be dried in the oven and cooled in a desiccator.

For more info on air-sensitive techniques: Shriver, D. F. *The Manipulation of Air-Sensitive Compounds*

SPECIFIC BATH TEMPERATURES

- common baths in red

Bath temperature / °C	Bath Composition
13	p-Xylene/CO ₂ (s)
12	Dioxane/CO ₂ (s)
6	Cyclohexane/CO ₂ (s)
5	Benzene/CO ₂ (s)
2	Formamide/CO ₂ (s)
0	Crushed Ice
-5 → -20	Ice/Salt
-10.5	Ethylene Glycol/CO ₂ (s)
-12	Cycloheptane/CO ₂ (s)
-15	Benzyl alcohol/CO ₂ (s)
-22	Tetrachloroethylene/CO ₂ (s)
-22.8	Carbon Tetrachloride/CO ₂ (s)
-25	1,3-Dichlorobenzene/CO ₂ (s)
-29	o-Xylene/CO ₂ (s)
-32	m-Toluidine/CO ₂ (s)
-41	Acetonitrile/CO₂(s)
-42	Pyridine/CO ₂ (s)
-47	m-Xylene/CO ₂ (s)
-56	n-Octane/CO ₂ (s)
-60	Isopropyl Ether/CO ₂ (s)
-77	Acetone/CO₂(s)
-77	Butyl Acetate/CO ₂ (s)
-83	Propyl Amine/CO ₂ (s)
-83.6	Ethyl Acetate/Liq N ₂
-89	n-Butanol/Liq N ₂
-94	Hexane/Liq N ₂
-94.6	Acetone/Liq N₂
-95.1	Toluene/Liq N ₂
-98	Methanol/Liq N ₂
-100	Ethyl Ether/CO ₂ (s)
-104	Cyclohexane/Liq N ₂
-116	Ethanol/Liq N ₂
-116	Ethyl Ether/Liq N ₂
-131	n-Pentane/Liq N ₂
-160	Isopentane/Liq N ₂
-196	Liq N₂

NMR SPECTROSCOPY

- Always check solubility in regular solvents before deciding on a Deuterated solvent.
- Do not process data on the spectrometer computers. Use dedicated computers for data processing to avoid unnecessary charges for spectrometer time.
- Print your spectrum (to be filed in your hard-copy folder) and save the electronic file for future use.
- Your spectrum title should ALWAYS start with the experiment number, followed by a description of your choice (e.g. *MD1_125 Ligand1 – column fraction 2*)
- Printing: be consistent with your choice of ppm range. A good range is usually 0-10 ppm. Use the same page for zoomed-in ranges and print them as insets to the full range spectrum.
- mystery peaks in your NMR spectrum are often solvents – consult “*NMR Chemical Shifts of Common Laboratory Solvents as Trace Impurities*” *J. Org. Chem.* **1997**, *62*, 7512-7515.
- Always take NMR's of your starting compounds and use the same solvent to compare peaks/identify potential starting material as impurity.

FINISHING AN EXPERIMENT

General

- Put all chemicals back into their designated locations in the chemical storage area
- If you used up a chemical inform the person in charge of the chemical inventory to update it.
- Important: if you find that a chemical is bad, do not put it back – inform the person in charge of the inventory and discard it.
- Put all single-use needles in a sharps container.
- clean and DRY all reusable needles and syringes immediately after usage or once the experiment is done. Otherwise, they will corrode and may need to be discarded. Never dry glass syringes with the plunger in the syringe. This is generally true for any ground-glass joints. They should always be dried disassembled.
- Dispose of all waste in the appropriate Satellite Accumulation Areas

Storing home-made chemicals

- For air-stable products, store them in capped scintillation vials. For air- or moisture-sensitive compounds, store them in the glove box or in vacuum-sealed Pyrex ampules (contact a senior member or Mircea if you have never sealed an ampule).
- Label vials and/or ampules with a white paper label reinforced with clear tape. NO SHARPIES. Each label should have Experiment number and molecular structure. For more complicated compounds, molecular formula will suffice.
- Each screw cap should have the experiment number and, if possible, the molecular formula/structure for the compound.

Glassware

Clean all glassware pertaining to that experiment, including filtration flasks, etc. If you have crap and you know it is crap, discard it. Laboratory glassware will not be used for storing chemicals. If glassware is broken, make sure it cannot be salvaged before discarding it. If it can be salvaged, put it in a box and when more accumulates, contact the glassblower (Ed – 781-829-0967) to submit a repair order. Make sure it is worth repairing the glassware (i.e. it is not one of the hundreds of r.b.'s or erlenmeyer's we have in lab).

NMR tubes

If you know you will not take the same spectrum again (almost always the case), clean your NMR tubes as soon as you have used them, or at the very least at the end of the week. Do not let used NMR tubes go dry because of solvent evaporation, they will be difficult to clean.

Rotavap use

Empty the collection flask and clean the bump trap after each use. The person coming after you will not know what solvent you used and will not know how to discard it safely. If the glass tube connecting the trap to the collection bulb is dirty, disconnect it and clean it.

WEEKLY CLEANUP DUTIES

- Clean your bench and hood space area of any clutter, dirty glassware, needles, filter paper, mysterious powders and stains, dust, etc.
- If you have taken notes on pieces of paper (which you should NEVER do), transcribe them into your notebooks with the appropriate time stamp.
- Dispose of all cardboard boxes and other packing material that clutters the floor.
- Clean your hotplates thoroughly of any oil or chemical spills. If necessary, change the Al foil covering the ceramic top.
- Return chemicals to the chemical storage area (see above).
- You MUST change your vacuum pump oil every 3 months, or every month if you use your Schlenk line heavily. Use only TKO-19 Ultra Oil from Kurt J. Lesker Company.

ORDERING CHEMICALS

- 1) Determine necessary purity. Do you need the fifth 9 digit? Otherwise, 99.5% is MUCH cheaper than 99.999%.
- 2) Check our own chemical storage room and electronic group inventory to see if we have it (get a ChemTracker account for this).
- 3) Check other groups' inventories through ChemTracker, especially under Surplus. If other groups have the chemical, see if we can reasonably borrow some.
- 4) If 2 and 3 fail, get prices using SciFinder from at least 2 vendors (do not go directly to Aldrich E-cat, this is often the most expensive option). Lancaster(Acros?), CityChemical (Wako) , and TCI America are often good options.
- 5) If you are buying multiple chemicals, see if a single vendor sells them all and if the price makes sense (cheaper shipping).
- 6) Once you have determined the best source, add it to the items to be ordered that day (white board) and the person in charge of ordering will take care of ordering at the end of the day. Make sure to include:
 - a. Vendor
 - b. CAS Number and Chemical name (person ordering will use CAS to verify)
 - c. Vendor Catalogue Number
 - d. Quantity
 - e. Price/item
- 7) Once you receive a chemical, it is your responsibility to update the group inventory in ChemTracker.

GROUP FUNCTIONS

Group Meetings

Group meetings will be weekly formal presentations (Powerpoint or equivalent) and will take place on Thursdays at 5 PM unless otherwise noted, according to the schedule on the group website.

Literature Group Meetings

Students may choose either: 1) a significant recent paper of interest – group critique; 2) a topic to be discussed by the group (if this is chosen, have the topic approved by Mircea) – the topic cannot be too general; 3) a new research proposal that deviates from a student's designated project, and hence merits discussion in a group setting.

Vacation Time

Group members should inform Mircea about planned vacations **at least two weeks in advance** and should provide contact information while away.

Keeping up with Literature

Everyone should set-up an RSS feed (or have an equivalent system) that will allow them to keep up with literature daily, or at least weekly. Several hours per week could be dedicated to this activity. You should read both general science and chemistry journals, to gain breadth, and project specific journals, to gain depth. Suggestions:

Science – weekly

Proc. Nat. Acad. Sci. USA - monthly

Nature Publishing Group

Nature – weekly

Nature Chemistry – monthly

Nature Materials – monthly

ACS Publications

J. Am. Chem. Soc. – weekly

Inorg. Chem. – weekly

Chem. Mater. - weekly

Chemical Reviews – monthly

Accounts of Chemical Research – monthly

Wiley Publications

Angew. Chem. Int. Ed. – weekly

Chemistry – A European Journal

Advanced Materials – weekly

Advanced Functional Materials – weekly

European Journal of Inorganic Chemistry

Royal Society of Chemistry Publications

Chemical Science – weekly

Chemical Communications – weekly

Chemical Society Reviews – monthly

Dalton Transactions

Energy and Environmental Science – monthly

INDIVIDUAL INSTRUMENT USES (see Schlenk and Glove Box Usage as well)

Never use an instrument before being trained by person in charge or a senior member!
Consult instrument manuals or home-written SOP's even after you've been trained.

Balances

- Clean balances after each use using the balance brush. This is **very important** because chemicals will corrode the weighing pan and can also contaminate other users' reactions.

Agilent UV-Vis Diode Array

- Wait for lamp to warm up for collecting stable spectra
- Turn lamp off when you are done to lengthen the lifetime of the lamp!

Ultrasonic bath

- Only use distilled water to refill the bath and never use the bath unless water is at required level. Never use tap water for the sonicator!
- If you drop any solution in the bath, empty it, clean it, and refill the bath to required level

Rotavap

- Clean traps when you are done and only use clean traps. Garbage in trap = garbage in your reaction!
- Only use distilled water to fill the bath. Never use tap water!

Vacuum Oven

- Do not dry compounds that sublime. If you do so by mistake, unplug the oven, wait for it to cool down and thoroughly clean the inside surface with water and acetone.

Tools

- Return all tools to the tool drawer once you are done using them.

Potentiostat(s)

- always polish your electrodes before doing any e-chem.
- do not mix the Al₂O₃ polishing powders - cross-contamination will make them unusable. Do not use the same polishing surface for polishing powders of different mesh.
- make sure there are no air bubbles in your reference electrode.

Sorption Analyzer

- make sure that the solvent traps are full of liquid nitrogen before starting an analysis/degas sequence.

- thoroughly inspect the O-rings of the Transeal and for the sample tubes for cracks each time you perform an analysis/degas.
- make sure that you are not ruining someone else's sample by putting yours on the same vacuum line as theirs.

Bruker X-ray Diffractometer

- under no circumstances should you touch the instrument if you have not taken the X-ray safety class.
- Carl must observe you collecting a set of data before getting checked out.
- if anything does not seem to be right with the diffractometer, **DO NOT** attempt to solve the problem yourself. Contact Carl about it.

Vacuum Pumps

- **NEVER** turn a vacuum pump on without knowing that there is oil or not.
- change your vacuum pump oil once a month if you are using your Schlenk line often, or as needed if use is more infrequent. Use **only TKO-19 Ultra** vacuum pump oil.

GROUP JOBS

The following guidelines are for regular maintenance only. If you make a mess, you clean up yourself, you do not wait for the designated member in charge of the instrument to clean up.

Glove boxes

- Regenerate catalyst every 4 months or more frequently if necessary – consult manual for doing this properly
- Regenerate solvent trapping system every 4 months or more frequently if necessary – consult manual on how to do this properly
- Order nitrogen for the box as needed
- Change pump oil (both box pump and filtration pump) once a month.
- Keep an eye for clutter and unlabeled samples. Warn people of this once, then discard them.
- The person in charge of the box is not in charge of supplies! Whoever used up the last item (pipettes, vials, solvents, kimwipes, etc) should refill them. See above.

Potentiostats and other Electrochemical Equipment

- Make sure potentiostat/s and other electrochemistry instruments is/are operational and that reference electrodes are in good condition.
- Keep polishing supplies in order and train people on using the polishing kit properly.

UV-Vis

- Train new users on the UV-Vis
- Keep an eye on the lamp and make sure it is off when not in use
- Make sure files are being stored in user folder, not on desktop. Warn once, then delete files from unauthorized storing folders.

Rotavap

- Train new users
- Refill water if needed
- Clean dry ice finger once a month (dirt accumulates from dry ice)

Chemical Inventory

- Train new users on ChemTracker
- Make sure inventory is up to date (especially with used-up chemicals)

Balances

- Check that balances are clean every week. If weighing pan needs to be cleaned, turn balance off, then take out weighing pan and clean it thoroughly. **Never** clean the pan while balance is on or directly on the balance.
- Replenish weighing paper as needed.

Solvents and Bulk Supplies

- Make sure there is a steady supply of bulk solvents, paper towels, gloves, lab notebooks, vials, Pasteur pipettes, and office products (pens, etc). This group job includes providing paper and toner cartridges for the group printer.

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Powder X-ray Diffractometer

- Make sure every user has taken the MIT X-ray safety class.
- Train new users and watch them do several data collection cycles before allowing self-use.
- Keep sample prep table organized and clean
- **Stress the importance that users do not attempt to fix problems by themselves.**