Laboratory Guidelines

The tenet "Work Hard and Be Nice" will propel a scientist far in their career. In the laboratory, however, there are many new procedures to learn and rules of order to ensure safe, proper, and efficient function. These protocols, if followed, will improve your happiness and chances for success. We expect that you will follow these guidelines to the best of your abilities and with good intentions. Any suggestions to this working document are welcome.

Ethics:

Our laboratory has a zero tolerance policy for plagiarism, falsified data, or cheating. (A commonly overlooked example of unethical behavior in science is not attributing an idea to its rightful source. Use "we" as often as possible when speaking about work. Avoid minimizing other's contributions.)

Expectations:

Work Hours: Set work hours are not enforced for Graduate Students. Undergraduates will be encouraged to join our laboratory if they demonstrate a willingness to work diligently, safely, and responsibly. Undergraduates must also carry a 3.0 or higher in their most recent semester to continue working in the laboratory. Students registered for CHEM4380 should plan to work 15-25 hours per week. All lab members will be evaluated based on research productivity. If the quantity and quality of research you are producing is sufficient, no one will question the hours you are working. It has been my experience that counting hours leads to poor performance. Obviously, more time spent in lab will lead to a greater likelihood of success. The average week for a graduate student involves 6 days of work, but the beauty of our work is that it can be performed at any time of day and there are many opportunities for increasing productivity and getting the most out of your time.

Remember, you are learning. Only time, dedication, and motivation will get you to the point of being productive in an 8-10 hour day.

Group Meetings: Researchers will present their most recent data, an analysis of that data, and the next planned experiments in weekly group meetings. This should be a formal presentation to the group members, but may be presented on the board. PowerPoint is not required.

Formal Presentations: Each member of the laboratory is expected to give one formal group meeting presentation per semester, whether through joint meetings with the organic division, or through our weekly group meetings. In this case, a hard copy of your presentation should be filed with the laboratory. Voluntary presentations outside of the group/department are encouraged, but may not always replace your formal presentation. The idea of the formal talk is to get experience performing a presentation

and fielding question in a situation resembling second year proposal, or a scientific meeting.

Project Management:

A successful research experience (Ph.D., or other) is measured by your progress toward becoming an independent scientist. To do this, one must practice more than just proper laboratory techniques. After discussing your work with me, you should be taking the following steps to practice management of your project.

Every Month:

Set/evaluate goals for the next **one**, **three,** and **six** months.

Plan and prioritize experiments necessary for the paper(s) you are working towards. What control reactions will you run? What is the best method of analysis? What characterization will be required for publication?

Sketch-out/Update a table of desired data needed to complete your proposed work.

Every week.

Spend a few hours planning your goals/experiments for the week.

Force yourself to learn something new (a mechanism, background, a reaction).

Complete on average 7 complete synthetic steps.

Each day.

Read the literature related to your project/experiments.

Read a paper **not** related to your project.

Obtain on average two useful pieces of data.

Do not put off until tomorrow something that you could do after dinner.

If you have some time at the end of the day, but not enough to set up a crucial experiment, set the experiment up in your notebook and find all of the reagents.

Evaluate needed characterization and control experiments.

Check to be sure you have the equipment/chemicals needed for upcoming steps.

End your day by compiling a “to do” list for the following day.

Laboratory Safety:

Safety is a must.

Wear your goggles and gloves when in the lab. Wear a coat if your clothing is not sufficient to keep you and your work separate . . . or if you are performing any work that may explode, splash, or spill due to some procedure or particular reactivity.

Do not wear your gloves or lab coat in the student office or department hallways. This is not tolerated in industrial settings and is not safe for your colleagues.

Read all containers as you handle them – become familiar with common statements about chemicals and the correct procedure for handling classes of chemicals. A quick glance at any chemical container will help you prepare for what may happen. MSDS sheets are available through our inventory and in the department.

Rehearse the actual experiment in your head. Train yourself to ask questions about when things may go wrong for the reaction. Developing an intuitive sense for foreseeing events during the experiment creates a safe environment and *leads to more successful reaction outcomes*.

Have an exit strategy for dangerous experiments.

Displaying improper safety habits will receive a warning that may be accompanied by limitations for your individual work. Dangerous habits may be cause for dismissal from the laboratory without notice.

Laboratory Notebooks:

Proper notebooks can be obtained from Dr. Foss as needed. Do not use the notebooks provided from the stockroom, as they do not provide a blank slate to write on. The notebooks are the collective property of the University and are not to leave the Laboratory without permission. Do not remove any pages from your notebook.

Do not place laboratory notebooks on contaminated surfaces in the laboratory.

Write in your notebook with non-erasable ink. If you make an error, cross through the mistake with a single line, write the correction in the closest blank portion of the page, and initial beside the line. Be sure to write neatly and describe all experimental methods. Dates should be included with any new entry, or when there has been a break in work within the same experiment.

Comment on the outcome of spectroscopic data obtained and include TLC examples helpful to rerunning the experiment in your notebook. NMR, MS, IR, Elemental Analysis and other spectroscopic data should be kept in a second location (as well as electronically. For NMR data, always save the raw FID file). Your printed data are best kept in three ring binders. Large binders are impressive, but one-inch binders are easier to handle and store. Be sure to copy your electronic data to a CD or DVD when finishing a notebook. This disc should be affixed to the inside cover of your completed notebook if possible or kept at your desk until graduation.

All reactions should be labeled and referred to by the researcher's initials, notebook #, then page number. ie, XYZ1014, would refer to XYZ's first notebook, page 14. When rerunning a reaction, refer to the previous page number.

Examples of proper notebook layout can be obtained from Dr. Foss or your colleagues.

Be critical of each other's notebooks and keep each other accountable.

Glassware and General Equipment:

There is enough glassware for all to keep a working set of flasks and daily equipment. However, laboratory glassware and equipment belongs to everyone. Please allow other's to borrow your favorite glassware, on condition that they return the glassware when finished, in good order.

Occasionally, items have a specific use (CH_2N_2 distillation apparatus, non-metal stir bars, etc.) that must be respected for safety reasons or for reaction reproducibility.

Breaking will occur, but excessive breaking shows a lack of care and respect for your lab mates. Excessive breakage will limit your ability to work in the laboratory until better skills are learned.

Keep your hands and area dry and clear. Most broken glassware arises from cluttered workspaces and wet gloves (nitrile gloves are particularly suspect).

Clean your glassware on a daily basis. During your favorite radio show, after lunch, or during rotovap usage is a great time to wash your dishes.

General Cleaning:

1) Use alconox baths with scrub brushes;
2) Rinse with warm water,
3) Rinse with deionized water, then
4) Rinse with a minimal volume of acetone.
5) Place items on the racks or in a location for clean dishes (a clean bin).

Base Bath:

The base bath is not for common use, but for difficult cleanups.
Do not place volumetric glassware or fritted glass in base baths.
Do not expect the weight of a piece of glass to remain the same if placed in the bath.

Acid:

Strong acids or aqua regia can be used when necessary, this is especially useful for fritted funnels with metal impurities.

If you need a dishwashing tutorial, this can be provided, but wet and spotty dishes will not be tolerated. Expect to be criticized by your lab mates for shirking these duties. A manual for cleaning is on our website.

Common glassware, especially NMR tubes, separatory funnels, columns, and large RBFs, should be cleaned promptly and stored properly.

Avoid cleaning your columns and graduated cylinders with alconox and water, unless needed.

Instrument Training:

Before using any instrumentation in the laboratory, you must be properly trained by the lab member responsible for that piece of equipment. Consult the lab job list to determine who is responsible for a given instrument. If you have any questions, ask before "experimenting" with dangerous or expensive equipment. Remember that acetone and other solvents can damage equipment housing and computers. Instruments should be kept clean and clear of clutter. We do not work around instruments with caustic materials. We do not operate computers with gloves on!

Resources:

Our laboratory and my office contain many books on *laboratory techniques regarding purification, reaction operations, air sensitive methods, and characterization*. There are also numerous texts regarding *synthetic strategies* and *synthetic mechanisms*. You should always refresh your memory if you have any questions, by thumbing through an appropriate text or asking a colleague. *SciFinder* and online search engines such as *PubMed* and *Web of Science* are extremely helpful for finding information regarding your project background and individual steps of your experimental design.

Storing Samples:

Chemicals should be made at an appropriate scale for the task; however, it is often a good idea to make enough so that you do not have to repeat the same reaction over and over again. When you can plan appropriately, calculating expected product yield from your known reaction yields is a prudent practice. For storage of materials, you should always attempt to store your material in a sealed vial that is two to five times the volume of your material. Vial adaptors are available for attaching vials to the rotary evaporators and high vacuum pumps. You must remove the solvent gently to avoid bumping, but your lab mates will be very happy that another RBF is freed up for working up current reactions. Everyone frowns at the flask that lives for years in the freezer or refrigerator. Your samples should be appropriately dried and purged with argon before affixing a screw cap/septum and sealing with parafilm, or better yet, parafilm and electrical tape. The container should be stored upright, in a box containing your compounds or similar compounds for a single project, in the freezer.

Chemical Inventory:

It is our policy to share inventories with other labs and only borrow/lend chemicals when needed (the caveat being that large amounts or expensive reagents are replaced). SOLVENTS should not be given to other labs unless the stockroom is OUT of solvent or there is an urgent need. If a non-synthetic person asks for help though, you could be nice and let them use a little bit of our good stuff, but you should help them use it correctly.

Bottles (oldest first) should be checked out and returned as soon as possible. Using a secondary container, often leads to the transfer of additional reagent to another lab. That reagent, then often sits hidden on a shelf. Lend out bottles, but ask how long they'll be out of the lab. If someone needs a chemical for more than three days, they should be able to buy it themselves. If they happen to know the amount they need, then they are welcome to measure it into the proper container using our scales or volumetrics.

If a chemical is checked out for over a week (this requires noting the "date borrowed"), we should follow up with the borrower and see if we can get it back or have it replaced with a new container.

You should write our laboratory name (FWF, Foss, etc) on bottles as they arrive and include the date on bottles as they are opened (just another reason you need a sharpie at hand).

Reagents are entered into our inventory system by the stock room electronically. When a bottle is empty it should be placed in the "empty bottle" container and removed from the inventory before discarding the bottle. A lab cleanup day will occur every six months. At this time, the inventory should be updated and laboratory jobs should be

switched so that everyone learns a new technique. If pump oil has not been changed recently, people should be encouraged to do so at this time.

Sustainable lab practices:

Purchasing: It is always tempting to buy kilogram scale materials at 10% more cost than gram-scale materials. However, this practice often leads to unused materials (that may be slowly deteriorating) taking up space in the laboratory storage areas. Please be reasonable about the amount of material you need. Most materials can be purchased and delivered within three days, so planning ahead and ordering in a "just in time" manner will limit the amount of waste our laboratory produces and keep our work area clutter free.

Waste: Proper waste disposal is a must. Please quench harsh reactants before adding to mixtures of waste. Correctly separating *Aqueous, Non-Halogenated Organic, Halogenated Organic, Solid, and Heavy Metal wastes* into different containers and faithfully describing their contents is the best way to cut costs on waste and ensure that we are not creating dangerous situations for Environmental Health & Safety workers. Be aware that improperly labeling waste can lead to a serious impact on the environment and your health. Any old materials that are suspicious should be removed from the lab, not left at the back of the shelf.

Lab Job Description:

Argon/Nitrogen: Inert gasses must be on hand to replace tanks that become empty, without the disruption of ongoing experiments. To accomplish this, it is the job or one or two people to monitor the use of these gases, in the back room AND at the solvent still. We should have one back up tank for each working tank. When a tank needs to be changed, switch the empty tank for a new tank and replace the empty tank immediately with a full tank from the stockroom. **DO NOT LEAVE EMPTY TANKS IN THE BACK ROOM.** If no tanks are available, the person responsible for this job should inquire immediately with Jim or Jill as to the arrival of new tanks.

Consumables: Each week you should check the status of consumable products (gloves, paper products, needles, syringes) and place an appropriate large-scale order to replenish diminishing supplies.

HPLC-MS: *This system is not to be used for characterization of unknown materials.* The LC-MS will be used to aid in methodology development and biological assays that have been thoroughly investigated, with proper calibration and standards already tested. No member of our laboratory or another laboratory will be able to use this instrument without approval and training.

Inventory: The inventory should be updated on a weekly basis. All of our spent reagents should be properly removed from the electronic system before the bottles are discarded. The inventory should be checked by area (acids, bases, oxidizers, reducing agents, organics, inorganics) for proper contents on a biannual basis.

Stills/Solvents: Solvents (20L) should be inventoried weekly and ordered from the state contract or from another special bulk order rate. The stills should be checked multiple times a week for proper dryness, temperature, cooling, and Argon use.

TLC Station/Silica Gel: Stains should be kept fresh and the area should be free of spills and old TLC plates. Silica Gel orders should be coordinated with other organic laboratories to secure bulk order rates.

Waste: Waste should be properly separated, labeled, and discarded. Sharps containers and glass waste containers should be replaced before they fill to full capacity. EH&S will be contacted to remove full waste cartons on the day they are filled and in the case that a dangerous chemical container is found.