Northrop Lab

Guidelines & Helpful Information

WESLEYAN
UNIVERSITY

(updated 1/4/2010)

Laboratory Safety

All students doing research in the lab must have completed laboratory safety training, even if you are doing computational and/or analytical research without doing synthetic chemistry (aka "wet" chemistry). Safety training and knowledge of safe laboratory techniques can be obtained by taking upper level laboratory courses such as CHEM258 or Integrated Lab (CHEM375 and 376). During the summer Hughes program there is a safety training course taught by Bill Nelligan, Wesleyan's Director of Environmental Health, Safety, and Sustainability. Any student doing research in the lab over the summer should attend this training course. All incoming graduate students are required to take a safety training course from Bill Nelligan.

Some laboratory safety is common sense (don't eat or drink any chemicals) while some is more subtle (do you add acid to water or water to acid?). For your and everyone elses safety it is imperative that everyone becomes familiar with safe lab techniques and practices laboratory safety at all times. If you have any questions about safe techniques please ask me or, if I'm not around, Bill Nelligan (x2771, wnelligan@wesleyan.edu).

Basic guidelines for laboratory safety include:

(i) You must wear safety glasses/goggles in the lab at all times; the only exception is when you are in the student desk area (there is a piece of tape marking the "goggle/no goggle" line).

(ii) Any work with hazardous compounds (volatile solvents, flammable compounds, highly reactive compounds, etc.) must be done in a fume hood.

(iii) Chemicals that are especially reactive/hazardous are marked with blue tape. Before using any of these chemicals for the first time you must either talk to someone in the lab who has used them or come talk to me about the proper procedure for working with such chemicals.

(iv) All chemicals must be disposed of properly. The satellite accumulation site is located in the small hood along the back wall; it's for waste only and no chemistry. There are containers for organic, aqueous, solid, and silica gel waste.

(v) Be aware of the locations of the eyewashes, fire extinguisher, and first aid kit in the laboratory, and the nearest safety shower (in the hall just outside HA80).

(vi) Do not wear gloves when using the computers, instruments, or opening/closing doors to the lab. Anything on your gloves will contaminate those surfaces.

(vii) The only place where you can have food or drinks in the lab is at the student desks. No food or drinks can ever be stored in the lab freezer.

(viii) Working at night: I would prefer that people do not do experiments in the lab at night when there isn't at least one other person in the lab. If you absolutely must set up and run an experiment when nobody else is around then please exercise extra caution and do not work with any particularly hazardous materials.

Ethics

The lab has a zero-tolerance policy on cheating, plagiarism, and/or falsification of data.

Laboratory Notebooks

Incoming students joining the lab will receive a lab notebook. Notebooks should stay in the lab at all times. If you need to briefly take your notebook outside the lab please come ask me first; I'll likely let you, I just need to know where the notebooks are.

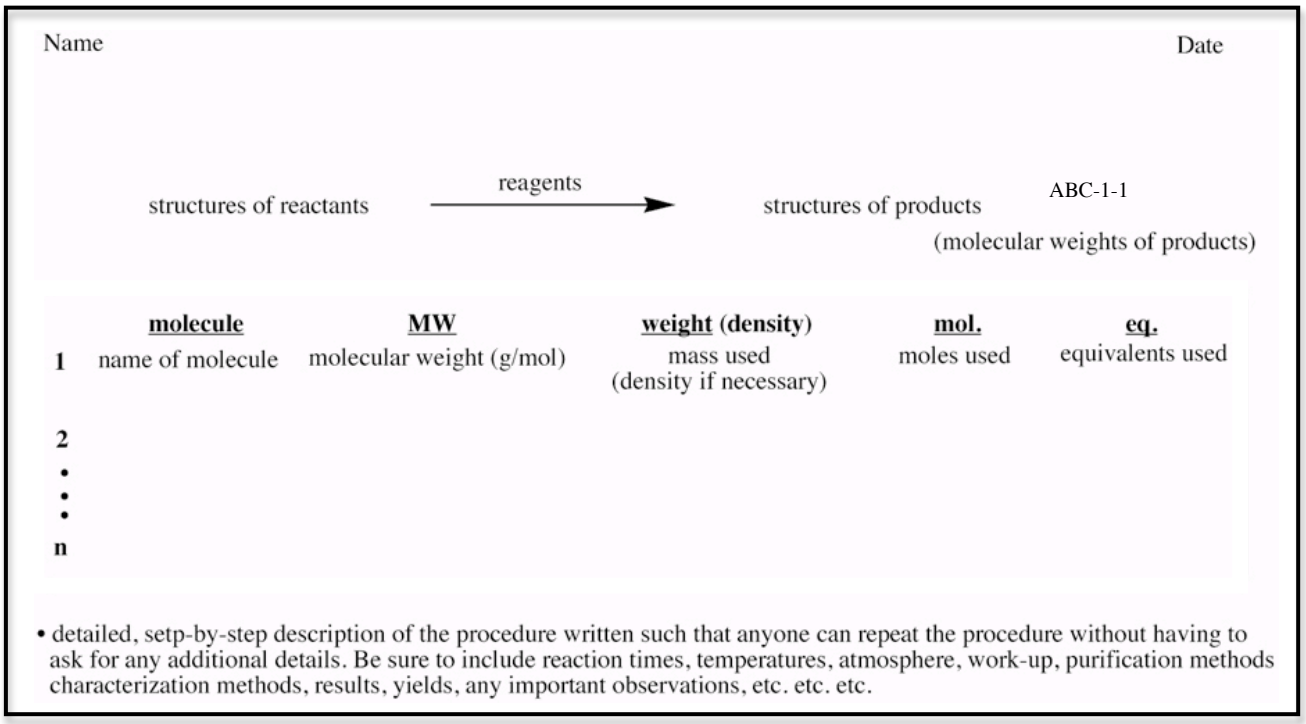
Notebooks are meant to contain detailed records of the experiments you do in the lab. They should be written in such a way that someone unfamiliar with the procedure/experiment could reproduce it without needing to consult any additional source.

The first few pages of your notebook should contain a Table of Contents indicating what page each experiment is on.

A new page should be started for each experiment.

Compounds should be labeled as: initials–notebook number–notebook page number. For example, the label "BHN-1-45" would refer to the product on page 45 of my notebook number 1. If multiple compounds are made during an experiment then you should add the descriptors a, b, c, …etc. (e.g. BHN-1-45b).

Here is an example of the proper recording of an experimental procedure in your notebook:



Name Date

structures of reactants —reagents→ structures of products ABC-1-1

(molecular weights of products)

	molecule	**MW**	**weight (density)**	**mol.**	**eq.**
1	name of molecule	molecular weight (g/mol)	mass used (density if necessary)	moles used	equivalents used
2					
•					
•					
•					
n					

• detailed, setp-by-step description of the procedure written such that anyone can repeat the procedure without having to ask for any additional details. Be sure to include reaction times, temperatures, atmosphere, work-up, purification methods characterization methods, results, yields, any important observations, etc. etc. etc.

You can use ChemDraw to determine accurate molecular weights. You are welcome to abbreviate the names of molecules (or simply use their ABC-1-1 style label) so long as it's clear which molecule you're referring to.

Hard copies of important/relevant spectra should be labeled with their chemical structure as well as their "ABC-1-1" label and saved in a folder. Electronic copies of important spectra should be saved in the "spectra" file on WesFiles (don't know how to scan and upload your results to WesFiles? Just ask).

Storing/Labeling Your Compounds

Upon isolation and purification of a product it should be properly labeled and stored. Compounds may be temporarily stored in chemical glassware such as round bottom flasks, however long term storage of compounds should be in glass vials with screw-top caps and labeled with one of the white labeling stickers. Write the compound label on the sticker (e.g. “ABC-1-1”) and, if there’s enough space, draw the structure of the compound. After sticking the label on the vial, cover it with a piece of masking tape so it’s less likely to fall off or get damaged.

Light sensitive compounds should be stored either in a dark amber glass bottle or in a clear glass bottle wrapped in aluminum foil. Store light sensitive compounds inside a cabinet or drawer where they can be kept in the dark.

If a compound is air sensitive then flush the vial with nitrogen gas before capping it. Wrap the top of any air sensitive compound with parafilm or Teflon tape.

Samples that are heat sensitive should be stored in the freezer.

If you have any questions about the proper storage of a compound just ask.

Chemical Waste Disposal

All chemical waste must be disposed of properly. Waste containers for organic solvent waste, aqueous solvent waste, solid waste, and silica gel waste are all located in the satellite accumulation site, which is inside the small 4’ hood along the back (west) wall of the lab. Chemical waste must be emptied into the correct container. Solutions that are strongly acidic or basic should be neutralized prior to being added to the proper container (neutralize basic solutions with acetic acid, neutralize acidic solutions with triethylamine).

When a waste container is about 90% full place a dated, yellow “Full Container” sticker on it and contact Bill Nelligan (x2771) or Don Albert (x2729) so that it can be removed. Full containers must be removed within 3 days.

Follow all instructions on the Hazardous Waste tag on each waste container.

After removal of a full waste container, replace it with a new empty waste container and be sure to start a new Hazardous Waste tag for the type of waste to be stored inside the container. Extra Hazardous Waste tags are available in the stockroom and empty waste containers can be found in the large round bin just outside the stockroom.

In addition to the satellite accumulation site, there is also one 4 L plastic jug next to each sink that can be used for routine rinsing of glassware, typically with acetone (no acetone or any other organic solvent is allowed to go down the sink). These 4 L jugs are not to be used for general organic waste disposal. When a 4 L jug is greater than half full with wash solvent, empty it into the organic solvent waste container in the satellite accumulation site.

Cleaning Glassware

Easiest way to increase your reaction yield: clean your glassware.

Glassware should be cleaned as soon as is possible following its use. As a first step, any chemical waste should be properly disposed of in the satellite accumulation site as outlined above. Rinse the emptied glassware with a compatible solvent at least three times (aka "triple rinse") to remove trace amounts of solvents and reaction mixtures, emptying each rinse into the appropriate container. You may also need to use a spatula, a brush, and/or elbow grease to remove stubborn waste that's "stuck" to the glassware.

Once the waste is disposed of and your glassware has been rinsed, triple rinse the glassware with DI water and finish with one final rinse with acetone and place the glassware on the drying rack (the last acetone rinse helps the clean glassware dry quickly).

For really stubborn stains place the glassware in the base bath located under the sink closest to the satellite accumulation site. Don't let the base bath come into contact with your skin! Use the blue PVC gloves that are kept with the base bath. Allow the dirty glassware to sit in the base bath for several minutes and be sure that the solution contacts all sides of the glassware. When the glassware is clean, remove it while wearing the PVC gloves (it will be slippery), place it in the sink so you don't drop it (it's slippery remember) and thoroughly rinse the glassware with DI water. Do one final rinse with acetone and place the clean glassware on the drying rack. DO NOT put the following glassware into a base bath: fritted glass funnels, volumetric glassware, cuvettes, any glassware contaminated with an oxidizing agent, or any glassware contaminated with a metal-containing compound.

Fritted glass funnels can be cleaned by inverting them, adding a compatible solvent to the stem, and allowing the solvent to flow through the frit slowly by gravity.

Cuvettes should only be cleaned by washing with a compatible solvent and wiping with a KimWipe. Do not scrub cuvettes with a brush.

General Lab Cleanliness

The lab in Hall Atwater 40-42 went through a complete renovation during the summer of 2009. As a result, essentially everything in the lab is new (as of fall 2009) and the lab in general looks great. If you ever find yourself in another lab and thinking to yourself "wow, I'm glad our lab is much cleaner than this one" then please remember that the only way our lab can be kept in great working condition is if everyone in the lab does their part to keep it clean and organized.

- Put chemicals, glassware, supplies, etc. back in their appropriate location once you're done using them.
- Dispose of trash in the correct place: recycling, broken glass, sharps, or general trash.
- Periodically take the clean/dry glassware off the drying rack and put it away in its proper place.
- The bench and fume hood areas will often be shared by more than one person, so be sure to label anything you're using and dispose of or put away anything you're not.

Chemical/Equipment/Supply Orders

Chemicals, equipment, and supplies for routine lab work (e.g. common solvents, pipettes, dry ice, gloves, etc.) can be purchased downstairs in the stockroom. If you're using the purchasing system for the first time and don't know how to charge items to the lab please come ask me, another student in the lab who has bought something from the stockroom, or Doug Allen. If you need anything that isn't available in the stockroom then we have to purchase it using the WFS (Wesleyan Financial System). Grad students should have accounts with the WFS that will allow them to make purchases, but undergrads will have to let me know what we need to buy and I'll place the order.

Journal Reading

Reading articles in the current literature is a great way to learn about the most recent and exciting research that's being done around the world. Graduate students should periodically search through different journals (see the list of recommended journals below) for titles or abstracts they find interesting, and read through those more relevant/interesting articles. Undergraduates are encouraged to do the same. Research articles can be very dense and even the most accomplished researcher can't be expected to understand everything in every article. If you have any questions about something you read in an article you're always welcome to come and ask me about it, and I will make every effort to help out.

For graduate students it's important to keep up with the current chemistry literature, especially as it relates to your own research. The fact that journals have online tables of contents makes it especially easy to skim through the titles and/or abstracts of current research articles to find relevant material. The following are journals that often publish research articles relating to organic, materials, and/or nanochemistry:

ACS journals:

Journal of the American Chemical Society	*Organic Letters*
Journal of Organic Chemistry	*Nano Letters*
Macromolecules	*ACS Nano*
Accounts of Chemical Research	*Chemistry Reviews*

Wiley journals:

Angewandte Chemie

Royal Society of Chemistry Journals:

Journal of Materials Chemistry *Chemical Communications*

High impact general science journals:

Nature *Science* *Proceedings of the National Academy of Sciences*

Reading journal articles allows you to (i) keep up with recent research, (ii) learn about new techniques, methods, and procedures, and (iii) become familiar with the way current research is carried out and presented, which will help you learn how to organize, conduct, write about, and publish your own research. There are far too many journals and articles for anyone to read everything, but skimming through titles/abstracts that pique your curiosity and reading full articles you find interesting is a good way to keep up with what's going on.

Searching the literature: Aside from skimming through the online tables of contents on publishers' websites, knowing how to effectively and efficiently search through the literature is a very valuable skill to develop. The two search engines that I have found most helpful are:

- SciFinder (scifinder.cas.org)
- Reaxys (reaxys.com, just click on "login")
- Web of Knowledge (isiknowledge.com)

You can access Reaxys and Web of Knowledge when on campus and you can sign up for a SciFinder account at https://scifinder.cas.org/registration/newUserRegistration.html.

For me Reaxys and SciFinder are the most helpful when I want to search for a particular chemical structure or its derivatives, find out if a compound has been prepared before (and if so, how), search for particular reaction conditions, and to determine if compounds are commercially available.

Web of Knowledge is especially helpful when searching for literature on a particular topic, searching by author name, and for finding out what articles have had the most "impact" (as judged by times cited) in a particular area.

Research Expectations

One of the great advantages of Wesleyan is its size and the close interactions between faculty and students. Wesleyan has incredible resources and great research facilities and yet a relatively small number of students, making it a wonderful place to do research. Getting a chance to do undergraduate research is not only a great experience for students interested in going on to graduate school, industry, medical/veterinary/dental/pharmacy/etc school but also for students interested in law, business, or practically any profession that requires problem solving, critical thinking, and good communication skills. Because lab research isn't structures in the same way a course is, here are some general guidelines:

Undergraduate students: Students doing undergraduate research, i.e. those enrolled in CHEM 409/410 or 421/422, should expect to spend around 8-10 hours per week in the lab if registered for 1 credit, or around 4-5 hours per week in the lab if registered for 0.5 credits. I will not keep track of your hours and you can divide them up throughout the week as needed. I will, of course, keep track of your progress throughout the semester. If things are going great then I won't question your hours. If you're absent from the lab for long periods of time and you're not getting any research done then we will have to discuss what changes need to be made.

Graduate students: One of the keys to success in graduate research is time management. During your first few semesters you will be taking courses, progress exams, TA-ing, and starting your research. It is important to find a way to manage your time so that you can be productive in the lab as well as in your courses and teaching.

I will not keep track of your hours. As a graduate student performing independent research your time is quite flexible. That can be a benefit or a detriment depending on your work ethic and how you manage your time. While I won't enforce or keep track of your hours I will keep track of your research productivity. If I am satisfied with the quality and quantity of your research then I won't question your hours. If the quality and/or quantity of your research drops or if it appears you are not taking your research seriously then we will discuss what changes need to be made.

Phone Numbers to Know

Northrop Lab Phone	3356
Northrop Office Phone	3987
Brian's Cell Phone	801-680-5776
Public Safety	3333
Wesleyan Department of Environmental Health & Safety	2771
Wesleyan Davidson Health Center	2470
Middletown Fire Department	
Routine	347-8621
Emergency	911
Middletown Police Department	
Routine	347-6941
Emergency	911
Middlesex Hospital Emergency Services	
Routine	344-6686
Emergency	911
American Association of Poison Control Centers	1-800-222-1222