

1. General Lab Conduct

All members of the Chemistry Department share responsibility for the safety of their coworkers as well as themselves.

Excessive noise and horseplay are forbidden.

Eating is not permitted in the laboratories proper (i.e. on/near bench tops, fumehoods, sinks), due to the risk of chemical contamination. Please use the desk areas or a common area when you wish to eat/snack/drink beverages.

2. General Laboratory Rules

Unauthorized experimentation and work in the laboratory is forbidden.

Unauthorized personnel are not permitted in the laboratory research area.

Only use the equipment you have been trained on and you are allowed to use. Use it only for what it was intended to be used for.

It is required that each group member know the location and proper use of fire extinguishers, safety showers, eye wash stations, and first aid kits within the laboratory.

Flooding - do not leave water running unattended. Secure all water hoses with copper wire. Open faucets just enough to obtain the desired flow rate. Avoid opening faucets all the way.

No hazardous laboratory work may be carried out alone. If you're working alone, make sure someone in another lab knows and will check up on you at regular intervals. Never quench chemicals alone.

Don't work in the lab if you're ill, intoxicated, or otherwise not able to be 100% focused on what you're doing. Pay attention to what you're doing. Inattention causes accidents!

When the lab is to be left empty for an extended period of time (i.e. coffee breaks, lunch, and closing), all bottles (> 1 L) of highly flammable organic solvent should be placed in cabinets designated for that use.

3. Accidents

Use your common sense and always THINK before doing something in the lab.

Ask yourself, "What is the worst that could happen from this activity?" and, prepare for the worst case scenario.

Any personal injuries that occur in the laboratory must be reported to the research supervisor and the ChemDept Safety Office.

4. Chemical Exposure during Pregnancy

Female scientists who become pregnant must immediately inform your supervisor and suspend all experimental research activities.

5. Personal Protection Equipment

At a minimum, personnel working in a synthetic laboratory must wear department standard PPE: safety glasses, lab coat, long pants, and closed toe shoes. More elaborate PPE should be worn (face shield, polymer gloves etc.) when more potentially dangerous experiments are planned.

Wear appropriate gloves while working. For a guide to what gloves are compatible with which chemicals, see: http://www.science.uottawa.ca/fac/spe-ehss/glove_main.htm

Suggestion: Keep a change of clothes in the lab.

6. Fumehoods

All experimental procedures must be carried out inside the fumehood. Any large scale experiments which will not fit inside the fumehood must be reviewed with the supervisor prior to proceeding.

Extra care must be taken to ensure that the fumehood sash can be closed rapidly in an emergency, without breaking glassware, bottles, or other items that may accidentally be in its way. It is essential to keep the fumehood in a tidy condition and free from clutter at all times.

Conduct all operations that may generate airborne contaminants inside a fumehood. Do not raise the sash higher than the labeled height. Leave the sash lowered when the fumehood is unattended. Keep the bypass grill clear. Keep apparatus at least 15 cm from the face of the fumehood. Do not place electrical receptacles or other ignition sources inside the fumehood when flammable liquids or gases are present. No permanent electrical receptacles are permitted in the fumehood. Avoid blocking the rear ventilation slot. Material stored at the back of the fumehood should be stored on an elevated shelf so that the slot airflow is not impeded.

Keep your face outside the plane of the fumehood opening. Avoid cross drafts at the face of the fumehood. Minimize foot traffic past the fumehood and position windows and supply air diffusers to direct airflow away from the fumehood. Do not store chemicals inside the fumehood. Hazardous chemicals should be stored in an approved safety cabinet.

Experiments to be left overnight should be left with an information sheet attached to the fumehood (experiment type, contact person and information).

If the fumehood is used for semi-permanent experiments, post the name of the person in charge, experiment title, and possible hazards.

7. Clutter

Good housekeeping is essential.

- Aisles must be kept clear of debris (i.e. empty solvent bottles).
- Chemicals and active reactions must be labeled.
- The bench tops must be kept free from unnecessary chemicals/glassware/ equipment. Clean your bench top regularly.
- Clean up chemical spills (including water) immediately. Do not leave spilled chemicals on the bench top or floor.

8. Chemicals-General

All compounds that are produced in a synthetic lab are potentially deadly. Nothing is known about the bioactivity of most of the compounds that are prepared in a synthetic laboratory. Please handle all substances with utmost care and respect: wear gloves, avoid skin contact, and avoid breathing vapors.

If contact with a chemical compound occurs, please wash the contaminated area of the body with plenty of soap and water. **DO NOT USE ORGANIC SOLVENTS** to clean your skin, as these may facilitate the absorption of chemicals through the skin. Be aware of the location of eye wash stations, safety showers, and fire equipment.

A vocal warning must be given to other laboratory personnel when performing a potentially dangerous procedure that may result in a fire, explosion, or release of fumes (toxic or otherwise). Before performing a procedure that has serious potential hazards (e.g. fire, explosion or release of fumes), individuals should inform other lab personnel in the immediate area.

Reactions that are chemically or mechanically hazardous (i.e. external overhead stirring; distillations) must not be left unattended.

Procedures for reactions to be performed on large scale (> or =100 mmol) should be double-checked by a more experienced colleague (postdoc or supervisor).

When setting up an experiment in your notebook, there should be a column entry labeled “ms-ds”. The first time that you use a particular chemical, you should read the designated ms-ds. This procedure should take only a couple of minutes using the Internet.

9. Fire Safety

Know what to do in case of fire (review fire safety training)

Never use water to extinguish small lab fires!! Always use the fire extinguishers.

If a major, uncontrollable fire breaks out, pull the fire alarm and evacuate the building, leaving lab doors closed but not locked.

Never use a free flame to heat organic mixtures

No flammable solvents must ever be placed around the Bunsen burner/torch.

Use the appropriate fire extinguisher. Use the D extinguisher for fires involving combustible metals, and the ABC extinguisher for everything else

Attempt to fight fires only in the instance that they are small enough for you to handle. Otherwise, evacuate the lab, pull the fire alarm and call 911

10. Spills

DO NOT WORK ALONE when cleaning up spills.

For chemical spills greater than 1 L, contact the following for backup:

- your supervisor
- the Chemistry Department Safety Office (Room A237, phone **7-5216**)
- UBC - HSE Office (phone **2-2029**)
- the Fire Department at **911**

Only clean up spills small enough to handle yourself. Otherwise, clear the lab and call the research supervisor and the safety office.

Alert everyone in the lab when a spill has occurred and block the spill so that people don't walk through it.

Spill carts are located outside Rooms A352, B170, D413 and E415. **Hydrofluoric Acid Kit** is located in D113 (Glassblower's Shop).

Wear appropriate personal protective equipment when cleaning up spills

11. Waste chemicals

Under no circumstances is any material that has been involved in a chemical reaction to go into the normal garbage bin! Specialized protocols for solid waste etc. should be generated.

Waste chemicals are to be stored in the designated red solvent containers provided by the UBC Health, Safety & Environment (HSE) using the following general categories: halogenated, organic, and aqueous. Halogenated materials must be stored separately from non-halogenated materials. Labels provided by the HSE are to be properly filled out for each solvent container.

Waste corrosive chemicals including inorganic acid chlorides, active metals, metal hydrides, and organometallics must be deactivated in the laboratory.

Empty glass reagent bottles must be cleaned and placed in a designated area with the lids left off. A designated group member will collect all empty glass bottles when appropriate for disposal

Toxic metals, such as tin and chromium, must be separated into separately labeled waste containers

Waste generators should take care of their own waste, not leave it for “future generations” of the group or later occupants to deal with. At the very least, waste bottles should be clearly labeled with the waste type, date, the individual who generated the waste and (*insert the research supervisor's name*). Suggestion: *Subsequent to your leaving the department, if material that was formally your responsibility to clean up is found—and you have left it for others to deal with, then the supervisor will include this information on future reference letters!*

12. Specific Chemical Hazard Guidelines

When opening a new bottle, write the date it was opened on the bottle on the label and remove the plastic ring.

When receiving ordered chemicals, alert the inventory manager so that they can be entered into your group's inventory

After use, place all chemicals under an atmosphere of argon/nitrogen before closing. Remove all old parafilm and parafilm the bottle after closing.

If the chemical is in an Aldrich "Sure Seal" bottle:

- insert a balloon of argon/nitrogen before removing any reagent
- place a folded swatch of Teflon tape (*not* Parafilm!) over the opening and replace the swatch with a new one on every use
- parafilm the cap of the bottle after use

Put chemicals back where they belong after use

13. *Known problem chemicals:*

- HCN (and metal cyanide salts)
- HF
- HN₃ (and metal azide salts)
- picric acid

Certain chemicals and reactions have over time become recognized as being responsible for a significant number of laboratory accidents.

- Organic azides**
Explosion hazards, especially with ground glass joints
- Perchlorate salts of organic, organometallic, and inorganic complexes**
Explosion hazards
- Diethyl ether**
Fires (has an extremely low flash point)
- Lithium aluminum hydride**
Fires on quenching

Notes:

- use a Teflon wrapped or Teflon coated spatula with weighing paper (the use of a metal spatula and a plastic weigh boat will result in a fire)
- add LAH to a cooled solvent (adding hot or room temperature solvent from a still to the solid reagent will result in a fire). Place a foil shield over the cold bath before the LAH is added to the solvent.

To workup a reaction containing x g lithium aluminum hydride:

- dilute with ether and cool to 0°C
- slowly add x mL water
- add x mL 15 % aqueous sodium hydroxide
- add 3x mL water
- warm to RT and stir 15 min
- add some anhydrous magnesium sulfate
- stir 15 min and filter to remove salts

To workup a reaction containing x mmol of an agent such as diisobutylaluminum hydride (Dibal-H):

- dilute with ether and cool to 0°C
- slowly add 0.04x mL water
- add 0.04x mL 15 % aqueous sodium hydroxide
- add 0.1x mL water

- warm to RT and stir 15 min
- add some anhydrous magnesium sulfate
- stir 15 min and filter to remove salts

e) **Sodium, potassium**

Fires on quenching

f) **Potassium hydride, sodium hydride (90%)**

Fires

g) **Sodium-benzophenone ketyl still pots**

Fires on quenching

h) **Palladium on carbon**

Fires on removal from the inert atmosphere, especially if wet with organic solvent or when contacting combustible materials such as filter paper

Notes:

- do not add more Pd/C to an ongoing hydrogenation, this will result in a fire

i) **Heat**

Exothermic reactions causing violent spills on scale-up due to inadequate provision for heat removal

j) **Ethers with alpha-hydrogen atoms**

Dangerous peroxide concentration during distillation; explosion hazards, especially with ground glass joints

k) **Carbon monoxide**

Toxicity

l) **Organic peroxides**

Sensitivity to shock, sparks, and other forms of accidental detonation, sensitivity to heat, friction, impact and light, as well as to strong oxidizing and reducing agents

m) **Pyrophoric organometallic reagents (t-BuLi, BuLi, alkylaluminums, alkylzincs)**

Can ignite in air, fires on reaction setup and quenching

Sample Protocol for quenching pyrophoric alkyl-metal reagents (BuLi, MeLi, AlMe₃, ZnMe₂)

IF YOU HAVE NEVER QUENCHED A STILL BEFORE, DO NOT ATTEMPT THIS PROCEDURE. QUESTIONS?-CONTACT THE SAFETY COMMITTEE FOR A DEMONSTRATION.

- a) NEVER QUENCH CHEMICALS ALONE.
- b) Clear out flammable materials from your fumehood. Place a fire extinguisher at the ready. Be prepared for the quench to catch fire-it should not surprise you.
- c) Take a large beaker (> 1 L). Find a watch glass or lid that can cover (smother) the top of the beaker. Aluminum foil can also be used to cover the top to avoid problems with spattering.
- d) Make a methanol-Dry Ice (~1:1) slurry in the bottom of the beaker. Methanol is recommended as ethanol or isopropanol do not react instantaneously—there may be a dangerous induction period.
- e) Add your pyrophoric reagent slowly. If it flames, smother. Reagents like diethyl zinc can be syringed directly into the solution (wide-bore needle needed-do not trickle-add directly under the carbon dioxide vapor layer). The slurry should be (manually) stirred—with the quenching of powders this is especially important.
- f) Do not let the liquid volume in the beaker exceed more than 1/3 of the beaker capacity.
- g) Rinse out “empty” bottles with hexanes prior to popping off the lid and adding water. Make sure the lid is off before water is added.
- h) Allow these quenching materials a day or two to rest in the back of the fumehood to ensure of no nasty surprises. Add liberal amounts of water (carefully).
- i) Paper towels and gloves should be placed in the back of the fumehood.

Sample Protocol for quenching sodium stills

IF YOU HAVE NEVER QUENCHED A STILL BEFORE, DO NOT ATTEMPT THIS PROCEDURE. QUESTIONS?-CONTACT THE SAFETY COMMITTEE FOR A DEMONSTRATION.

- a) NEVER QUENCH CHEMICALS ALONE.
- b) Have a class D fire extinguisher handy. Clear out flammable materials from your fumehood. Place an *empty* plastic tub in the fumehood. Put a cork ring in the bottom of the tub and clamp the still pot so the weight is distributed to the clamp and the cork ring. Place a blast shield in front of the still pot.
- c) Make sure there is enough solvent to cover the sodium. Add more solvent until the sodium is completely submersed.
- d) SLOWLY, add 1-5 mL of pure ethanol.
- e) If the solvent in the flask is bubbling vigorously, do not add more ethanol.
- f) Wait at least 15 minutes before proceeding.
- g) Once the bubbling subsides (or if there is not substantial bubbling) repeat step 3.
- h) If there is no bubbling after the addition of 5 mL of ethanol, add 20 mL.
- i) Wait at least 1 hour before proceeding.
- j) Continue to add ethanol until no sodium remains.
- k) Slowly add 20 mL methanol and let the still sit overnight.

14. Protocol for quenching calcium hydride

- a) Take a large beaker (> 1 L). Find a watch glass or lid that can cover (smother) the top of the beaker. Have a fire extinguisher handy. Clear out flammable materials from your fumehood.
- b) Fill 1/2 of the beaker with ice water.
- c) 3 SLOWLY add calcium hydride (with stirring)

15. Recent Safety Incidents in the UBC Department of Chemistry

- d) ***Runaway reaction:*** A researcher who needed to prepare a benzyl ether of a phenolic compound injected benzyl bromide into a solution of the sodium salt of phenol that had been previously by reaction of the substrate with NaH. The flask containing this solution of sodium phenoxide had been immersed in an ice bath to control the temperature of the reaction, and the experiment was carried out inside a fumehood. Evidently, the rate of addition of benzyl bromide was so rapid as to trigger a runaway exotherm. Having noticed that the reaction was about to become uncontrollable, the researcher inexplicably took the reaction flask out of the ice bath and the fumehood, apparently with the intention of cooling it under running cold water in the sink. This action caused an immediate increase of the temperature inside the flask and a consequent pressure buildup. The septum that was sealing the flask blew off while the researcher was transferring the flask from the hood to the sink. The now very hot reaction mixture was forcefully projected out of the flask into the air, onto the floor and all over the researcher, who fortunately suffered no major injuries, but still had to be taken to the hospital for treatment. The powerful lachrymatory properties of benzyl bromide immediately caused severe distress to all lab occupants.

What went right? The individual was wearing safety glasses, lab coat, long pants and closed shoes.

What went wrong? The researcher ignored the fact that alkylating agents react exothermically and that they must be added to reactive mixtures slowly, carefully, dropwise and with good stirring. This large scale reaction was not monitored with a thermometer in the reaction solution. Taking the reaction mixture out of the ice bath would cause an increase in internal temperature, increasing the potential for a runaway reaction. If a reaction becomes uncontrollable, one can only close the fumehood and get out of the way.

- e) ***Major Fire Caused By Incorrect Disposal of Palladium-on-Carbon:*** A researcher carried out a catalytic hydrogenation on gram scale. When the reaction was deemed to be completed, the palladium-on-carbon was vacuum filtered away from the reaction mixture

using a Celite-filter paper pad. The active palladium-on-carbon catalyst was then placed in a plastic garbage can lined with a plastic bag. The researcher and other laboratory personnel left the lab for lunch. Speculation: A fire ignited in the plastic trash can, melting the trash can down until the fire came into contact with solvent bottles stored on the floor. A major fire ensued. Unfortunately, these items (garbage can and solvent bottles) were also placed in a spot in the room where the automatic overhead sprinkler system could not douse the flames. The fire department was called. Fire and water damage to the laboratory resulted in a total loss of infrastructure. The room required extensive renovation, taking six months to complete.

What went right? No individuals were hurt.

What went wrong? Palladium-on-carbon (activated after reaction) was incorrectly handled and disposed. (*Interesting note: two days after this major incident, a similar mistake was repeated in another UBC Chemistry laboratory, resulting in a small fire*). Solvents stored on the floor enabled the fire to spread. The plastic garbage can provided fuel for the fire, melting down.

- f) **Minor Fire Caused By Quenching Procedure:** As part of a general laboratory cleanup, a number of vials containing compounds believed to be air-sensitive were removed from a glove box, opened, and placed in the back of a fumehood. After a prolonged period of time (overnight), a researcher began a quenching procedure by adding solvent to a vial. Material within the vial ignited. The researcher was surprised, and in a panic, brought the material outside the fumehood onto the laboratory floor. The fire was then put out using fire extinguishers by other researchers in the laboratory.

What went right? No individuals were hurt.

What went wrong? The researcher performing the quench was not wearing a lab coat. The researcher was taken aback that the material to be quenched would ignite, and did not have immediate access to a fire extinguisher.

**University of British Columbia
Department of Chemistry**

Practical Laboratory Safety Guidelines (updated June 2010)

_____ research group

This document contains safety rules and procedures that everyone in the _____ research group must follow. Failure to do so may result in monetary fines or expulsion from the group.

The specifics presented within augment the overall safety training experience in the UBC Department of Chemistry. Enrollment and completion of appropriate UBC HS&E safety courses are required as well as specific informal safety training for techniques and methods used within the _____ research group.

I, _____ (print name), have read and understand this document and agree to comply with all UBC and Chemistry Department policies as outlined in this document.

_____ (signature)

_____ (location, date)

_____ (witness)