Loeb Research Group (LRG): Standard Operating Procedures

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1. Introduction

It is essential for all researchers in the Loeb Research Group (LRG) to undertake work in the laboratory with due consideration and care not only for their own health and safety but also for their colleagues and others who may be working nearby. This document outlines the duties and expectations of safe conduct for all members of the LRG.

1.1 General Comments

This document is applicable to ALL research activities undertaken in the LRG laboratories and offices as well as the day-to-day management of the LRG in order to provide practical advice on Health and Safety issues. In addition, this document is applicable to safe working practices in other areas of the Department of Chemistry and Biochemistry related to experiments and instrumentation including, but not restricted to, operation of X-ray diffraction, NMR spectroscopy, TGA and DSC instruments.

2. Working in the Laboratory

Personal Protective Equipment *(PPE) – Safety glasses and a lab coat, as a minimum – must be worn in the laboratory at ALL times regardless of the actions been undertaken; this includes when in transit between laboratories. When not in use, lab coats should be hung upon pegs provided in the laboratory. They should not, under any circumstances, be brought into non-laboratory or office space.*

2.1 Before Commencing Work in the Laboratory

New LRG Researchers must:

- Obtain a valid *UWin ID* in order to complete on-line and *WHMIS* training (below).
- Complete the Department of Chemistry and Biochemistry *Laboratory Safety Orientation Checklist (see UWChemBiochem website)*, undertake all appropriate training indicated, sign, obtain supervisor approval and submit to the LRG Administrative Assistant (G. Loeb).
- Complete **mandatory** WHMIS training and familiarize yourself with the Chemical Control Centre (CCC) Chemical Waste Handling guidelines. WHMIS training can be found on-line at: **http://www.uwindsor.ca/safety** → Health & Safety Training → WHMIS
- Make sure that you are aware of the locations of the nearest:
    - safety shower
    - fire extinguisher
    - eye wash
    - First-aid kit
- Make where you know where to locate the pertinent Materials Safety Data Sheet (MSDS) for each chemical substance you will use: **www.uwindsor.ca/msds**
- Make sure you are aware of whom to contact in case of an emergency. A current list emergency contact numbers is available on the Safety Notice boards in each LRG laboratory.

2.2 Before Commencing Any Experiment

- Ensure you are aware of the hazards associated with EVERY chemical you are using including solvents (MSDS). This is a VERY important, initial assessment stage.
- Ensure you have taken suitable precautions, in light of your assessment, particularly with regard to your own protection and that of your colleagues. A reaction should be only be undertaken in a working fumehood. Suitable PPE must be worn; this should comprise not only safety glasses and a lab coat, but also gloves (appropriate for the chemistry undertaken) and any specialized protective gear that might be appropriate. If you are not sure about suitable PPE, this should be discussed with your immediate supervisor prior to using opening any chemicals containers.
- Whenever undertaking a new reaction or experiment for which you have no prior experience, the FULL procedures must be checked by your immediate supervisor before commencing. If any of the reagents are classified as toxic, carcinogenic or pyrophoric you MUST obtain the signature of your immediate supervisor before commencing. For example: Pd/C hydrogenations may ignite if proper precautions are not taken. A detailed SOP is available at *www.cchem.berkeley.edu/rsgrp/SOPs/Hydrogenation.doc*.
- Ensure that a record of your experimental protocol is available in your laboratory notebook. All chemicals, reagents and solvents, including amounts MUST be recorded, including MSDS numbers for any chemical that you are using for the first time. A clear and concise coding system should be established for reactions, products and their respective characterization data. This coding system MUST be obvious and clear to anyone else who needs to follow your protocol. The date for each experiment, procedure and data set (NMR, MS, X-ray etc.) must be recorded.

2.3 Before Using Any Instrument/Equipment

- Familiarize yourself with the individual SOP available for the instrument. Discuss how you will operate the instrument with your immediate supervisor OR another member of the LRG who is listed as a trained operator of this equipment. For some major pieces of shared departmental instrumentation (*e.g.* NMR, MS, scXRD, pXRD, TGA, DCS etc.) VERY specific training may be required before being granted access to the instrument. You MUST consult with the technical personnel in-charge of managing the facility (*e.g.* NMR – Matt Revington; MS – Janine Auld) or the faculty member assigned responsibility for managing access to the instrument.
- If there is ANY doubt, ask Professor Loeb.
- Some instrumentation requires special safety protocols prior to training (*e.g. X-ray radiation dosimeter*). These protocols and any extra safety training should be undertaken OR renewed if necessary before accessing the instrument.
- Radiation dosimeters are required when using any X-ray equipment.

2.4 During Experiments

- Check intermittently on any apparatus to make sure it is operating properly and as designed for your experiment.
- Always ensure heated reactions or reactions which may release vapours which build up pressure have an exit needle or appropriate venting apparatus attached.
- Always ensure water continues to flow in condensers and that the condenser hoses are securely attached with clips or wires (floods are almost as bad as fires!). Always keep fumehood sashes DOWN to their maximum when the fumehood is not being used.
- If you are running an experiment overnight, a note detailing the chemicals used in the reaction should be left on the fumehood in case of an emergency. Leave the fumehood light on and check that the reaction is secure prior to leaving the Department. In particular, check that water connections are firmly secured as they will be susceptible to fluctuations in water pressure overnight (floods that last for hours are disastrous and cause a great deal of water damage!!).

2.5 After an Experiment is Complete

- Dispose of solvents and residues in accord with proper protocols;
- Solvents should be disposed of in chlorinated or non-chlorinated solvent containers as appropriate; replace the lid on the solvent container so as to minimize evaporation. However, **do not tighten the lid completely** as this may lead to pressure build-up inside the solvent waste drums.
- Solid waste may be disposed of in the solid waste bin.
- Kim wipe tissues and paper towels may be disposed of in the regular waste if they are not contaminated.
- Disposable syringe needles should be placed in the sharps disposal bin.
- If residual solutions are corrosive and/or noxious, they should be decomposed prior to disposal. Seek advice from your immediate supervisor about appropriate ways to decompose your waste if necessary.
- Clean up glassware, apparatus and any other equipment and replace it in the appropriate cupboard for future use. Any broken glassware should be disposed of in the glass disposal bin if it cannot be repaired. Ground glass joints which are not broken should be retained as they are expensive and can be recycled. A reasonable effort should be made to clean broken glass of residual chemicals before disposal or submitting for repair, though extreme caution should be taken to avoid personal injury from broken glass.
- Broken glassware which can be repaired should be collected in the *Glassware for Repair* container located in EH 372-7.

2.6 Working Hours

- The health and safety of the research group is paramount. Normal working hours for commencing experiments can be considered to be between 8:00 AM and 8:00 PM, Monday through Friday. On-going experiments, *e.g.* overnight reactions, should be set-up as per section 2.4.
- **Under NO circumstances** should out-of-hours laboratory work be undertaken without someone else present.
- **Researchers should only undertake experimental work outside of normal hours if there is someone else in the laboratory** (or nearby AND aware of the work being undertaken) in case of an accident. In the event of a serious accident, it is **essential** that someone be close enough to respond to any calls for assistance and summon help. It is NOT advisable to undertake a new reaction for the first time, outside of normal working hours.
- Data collection and interpretation (outside of the chemical laboratory) can be undertaken outside of normal hours if this presents minimal risk to researchers.
- When conducting experiments outside of normal working hours, it is the responsibility of the researcher to ensure someone else is available nearby, ideally within the LRG laboratories or offices. Alternative arrangements with researchers in other groups are acceptable provided they are nearby and aware that lab work is being undertaken.

2.7 Music/Noise

- Be considerate with music and noise volumes. Music/noise can be a hazard and can drown out important sounds which might otherwise indicate a malfunction or some other problem, *e.g.* the sound of air being sucked into a cold trap on a vacuum line (condensation of liquid O_2 with volatile organics can be an **explosive** hazard). Turn music/noise down or off when requested to do so.

3. General Lab Maintenance

3.1 Your Work Area

- Your assigned fumehood and laboratory workbench is your responsibility. All permanent glassware, such as a Schlenk line should be kept clean. All hoses should be in good shape. All electrical, mechanical and plumbing should be in good working order. If any item is in need of repair, please contact the LRG administrative assistant (G. Loeb), who can help you with the appropriate paperwork required for a repair.
- You are responsible for your pump maintenance. Periodically check the level and quality of the oil – change the oil if necessary. Pumps are VERY expensive to repair and replace
- There should be NO chemicals in the fumehood other than your reaction(s) or work-up(s) – *i.e.* NO chemical containers, NO waste containers; storage of any type is not permitted.
- It is OK to write a current reaction scheme on the glass fumehood sash as a way to inform every one of the fumehood contents in case of an emergency, BUT it is NOT appropriate

to doodle, write messages or apply stickers etc. that are not essential to the chemistry being conducted or the safety of the fumehood user.

- You are responsible for the content and organization of the cupboards below your fumehood EVEN if you do not use the contents. Storage in the LRG laboratories is such that this may be a designated storage area for a particular class of chemical. Familiarize yourself with the contents of the cupboard and make sure you are aware of the safety protocols related to these items

3.2 Communal Items and Space

- All glassware should be cleaned thoroughly and stored in a timely fashion after usage.
- All bench tops used should be cleaned immediately after usage.
- Reaction vessels, especially round-bottom flasks and test-tube racks, are NOT for storage. This is both inefficient and dangerous.
- Dispose of cracked or broken glassware that cannot be repaired.
- Make every attempt to reduce clutter/garbage on all bench tops.
- Clean the balance after every use.
- Clean the rotary evaporator after use, disposing of any unwanted solvents.
- When you notice that any bottled or still solvent and other common reagent is running low, order replacements. This is best done ***before*** they are used up! Remember some chemicals may take several days/weeks to arrive depending on supplier.
- "Dry" solvents are sourced from the solvent still in EH 374. This still is maintained by the LRG. It is vital that you sign their log book and record as accurately as possible the amount of solvent taken.

3.3 Use of Syringes

- Syringes are a hazard. **Should you have a need to walk along the corridor or around the lab with a unsheathed syringe, place a rubber septum over the needle point to avoid injury to yourself and others!**
- Use disposable syringes and needles for corrosive reagents. Clean syringes/needles after use – BUT do not reuse – dispose of syringe in the garbage can and the needle in the sharps bin after decontamination.

3.4 Compressed Gases

- Most fumehood, Schlenk lines use the departmental nitrogen supply. However, some instruments or reactions may use other compressed gases such as hydrogen chloride (HCl), ammonia (NH_3) or hydrogen/deuterium (H_2/D_2) as laboratory reagents. The appropriate regulator specific for the gas type MUST be used to control gas pressure. Before using cylinders of compressed gases, researchers should familiarise themselves

with the specific gas handing protocol. Training is available from the CCC *via*: ***www.uwindsor.ca/labsafety***

- When using any compressed gas for the first time, consult with an experienced user such as your immediate supervisor for advice and instruction.

3.5 Chemical Inventory and Chemical Storage

- All chemicals and reagents in the LRG laboratories should have been catalogued into an inventory Excel Sheet database available on ALL LRG Office PCs.
- For reasons of safety and inventory (WHMIS), all organic compounds are to be stored in one of the large blue cabinets in EH 374-A and all inorganic compounds in the gray cabinet in EH 374-A. It is NOT appropriate to store chemicals in regular bench drawers no matter how convenient. It is NOT appropriate for chemical source containers to be on the bench or in the fumehood unless the compound is being dispensed.
- Acids, bases and strong oxidizers are stored in separated and designated areas. It is important that these items are returned to their designated storage area.

4. Day-to-Day Laboratory Operations

4.1 End-of-Day Check-List

- Markers, pens, spatula etc. should be removed from the fumehood, cleaned and placed in a drawer.
- Solvent wash bottles should be placed in the fumehood or other identified storage area.
- Used syringes and needles should be cleaned and disposed of.
- Any chemical source bottles, reagent bottles or solvent bottles (particularly flammable solvents) should be returned to their cupboards or cabinets.
- The base of the fumehood is wiped clean.
- All apparatus, including vacuum pump and Schlenk line in your fumehood, are switched "off" **OR** overnight reactions are secured as outlined previously.
- Do not forget to remove the cold trap when switching off your vacuum line: ***Condensation of liquid O_2 into a trap containing organic solvents is a potential explosive hazard!***
- Fumehood sashes should be pulled down – with no obstructions present.
- Your lab coat should be hung on a peg, not left on a fumehood knob, bench or stool. **Lab coats and gloves are not permitted to be left in the office where food and drink may be consumed.**
- If there are no overnight reactions then your fumehood light should be switched off. This makes it easier to identify who is still working in the laboratory or where overnight reactions are taking place.
- If you **are** leaving a reaction on overnight, leave the light in your fumehood on and a note (outlining the procedure and a phone contact number) in case of emergency.

4.2 If you are the Last to Leave:

- Make sure that the radio is switched off.
- Make sure that the office light is switched off.
- Make sure that the office door is locked, the laboratory lights are switched off and the laboratory door is locked.

4.3 Spot-Checks

- Dr. Loeb is ultimately responsible for the safe operation of the laboratory, including implementation of improved safety procedures or further necessary actions. He will undertake intermittent spot-checks of the laboratory and identify issues to individual group members which need attention. If you witness any action or item which feel is unsafe pleas report this to Dr. Loeb so corrective action can be taken.

- **In cases of persistent infringement by any individual, of University, Departmental or LRG Safe Operating Procedures, as outlined in this document or any other document made available to the researcher on health and safety, policies: 1) a report describing of the non-compliance issues will be forwarded to the Department of Chemistry and Biochemistry Safety Committee, which will result in an official reprimand and filing of the incidents, 2) removal of laboratory privileges for a set period of time or 3) in extreme cases, a permanent ban from laboratory related activities. NOTE: depending on the work status of the researcher, this could result in termination of employment or expulsion from a degree program.**

5. Working in the Office

5.1 General Maintenance

- The office space is a shared communal area where researchers may be drinking and/or eating. Therefore ***No lab coats or chemicals are allowed in the office under any circumstances.***
- It is appropriate to maintain a clean and tidy environment in which to work. Graduate research group members typically have their own desk area and it is their responsibility to keep their own area clean and tidy.
- For undergraduates, general seating is available but it might not be possible to assign individual desks. It is, imperative they tidy up at the end of each day.
- All LRG members have access to the refrigerator and microwave etc. inside EH 372-3. Please clean up after using the microwave and do not store/forget food items for lengthy periods in the refrigerator. All LRG members are equally responsible for cleaning up any mugs/cups/plates/crockery/cutlery which they have used.

5.2 At the End of the Day

- Replace catalogues, books and theses on the shelf when you have finished with them.
- Put any rubbish in the bin.
- Tidy your desk space.
- Wash up used mugs/plates.

5.3 Noise/Music

- Music/noise levels in the office should be maintained at a low level so as not to disturb those who may be working or discussing research. Be considerate of others working in the office and adjacent offices. In particular, if your office is adjacent to Dr. Loeb's office you should be considerate as he may be in meetings with academic visitors, faculty, other students or video-conferencing.

5.4 LRG Computers

- An HP computer (PC) running Windows-8 is supplied in each of the LRG offices. The primary use of these computers for LRG research related work. It is acceptable to access the internet and sites unrelated to LRG research, when no one is in need of the computer for valid LRG research reasons. HOWEVER, this access for personal use is a privilege and should NOT be taken for granted or abused.
- NO software should be installed or settings changed on any of the LRG office computers without Dr. Loeb's permission. Installing pirated software is strictly forbidden under any circumstances. If you feel there is a piece of software or a program that would be of benefit to the LRG efforts then please bring it to Dr. Loeb's attention.

6. Signatures of Agreement

This version updated: September 3, 2014
Next tentative review date: January 5, 2014

I, _____ certify that I have read the Loeb Research Group (LRG) document on Standard Operating Procedures specific to the research group. I am familiar with all applicable topics and fully understand them.

Supervisor: Name Dr. Stephen J. Loeb

Signature _____

Date _____

Researcher: Name _____

Signature _____

Date _____