

CMK & FMK RESEARCH GROUPS, Lab 5006

Protocol for Good Laboratory Practice

1. Availability and use of safety equipment

The following equipment should be readily available:

- (i) **Safety glasses** (to be worn at **all** times for experimental work)
- (ii) **Full face masks** (for the handling of cylinders of compressed gas; for any reaction which involves the vigorous evolution of gas or performed at a higher pressure than atmospheric; for the handling of any azide compounds)
- (iii) **Breathing masks** (for use with hazardous dust)
- (iv) **Disposable gloves** (to be worn when carrying out any chemical reaction, remembering that compounds may permeate through latex gloves after about five minutes, whereas acetone rapidly dissolves many types of disposable gloves. N.B. Wet glassware is very difficult to handle with disposable gloves, care must be taken when cleaning glassware. Also, do not touch door handles, keyboards etc. with gloves, you will transfer and contaminate these surfaces with chemicals)
- (v) **Thick rubber gloves** (to be worn when handling toxic and/or corrosive solutions or compounds, especially concentrated acid and base solutions. N.B. Wet glassware can be difficult to handle with rubber gloves, care must be taken when cleaning glassware)
- (vi) **Oven gloves** (to be worn when handling hot or cold apparatus, remembering that thick gloves may compromise your grip of a piece of apparatus)
- (vii) **Lab coats** (must be worn when handling toxic or corrosive materials)
- (viii) **Fire extinguishers** (fire blankets, CO₂, dry powder fire extinguisher for sodium fires. All lab members should be proficient in handling a fire extinguisher and know where the nearest fire safety equipment and fire alarm are)
- (ix) **First aid kit** (including eyewash)
- (x) **Emergency number is 4100 or 9-911**

2. Records

- (i) **Lab notebooks** (these must contain an accurate record of what you have done and what you plan to do. Include comments on hazards where appropriate. Undergraduate students and starting PhD students must not carry out new experiments without authorisation from their supervisor)
- (ii) **Inventory** (must be kept up-to-date and chemicals must not be used before they have been entered in the inventory. The inventory is electronic and available on the PC in lab 5006)
- (iii) **Orders** (store hard copy of purchase orders in binder and note arrival)
- (iv) **Servicing** (record faults and servicing in the instruments book, instrument books will be by the equipment or with a technician or supervisor)
- (v) **Loans book** (all equipment loans to other research groups must be recorded in the loans book and be updated upon return, *N.B. Chemicals are not loaned. Researchers from other groups should bring a sample vial and take what they need.* If unsure whether something should be loaned ask CMK or FMK)

3. Chemicals

- (i) **New chemicals** (add to inventory, with notes on hazards, make sure MSDS sheet is available)
- (ii) **Storage** (store chemicals appropriately (in freezer, fridge, fire-proof solvent cupboard etc.), solvents must not be kept in fume hoods without good reason)
- (iii) **Labelling** (this should be done clearly and indelibly: all materials kept in “communal” areas (e.g. fume cupboards, freezer, fridge) **must** be labelled)
- (iv) **Fume cupboards** (must be used according to safety guidelines. When handling known toxic materials (e.g. metal carbonyls, organomercury compounds) always use fume cupboards. Otherwise ensure that all dust is collected and cleared away. Treat compounds with unknown properties similarly. Take particular care with **volatile materials** (wear lab. coat and disposable gloves). When fumehoods are left unattended the sash must be closed leaving a small gap to improve efficiency and encourage the ‘chimney effect’ in case of a power failure)
- (v) **Corrosive materials**, acids, bases, etc. (must be handled with the appropriate safety equipment wear appropriate protection [gloves, face mask, lab. coat] according to hazard).
- (vi) **Flammable materials** (must be handled away from all sources of ignition, this includes sparks caused by electrical equipment such as electric motors, light switches, thermostats and vacuum pump switches. Diethyl ether must be handled with special care; fumes from open reaction vessels must be ‘piped’ away to below bench level or preferably to a fume hood)
- (vii) **Toxic materials** (must be handled according to safety guidelines)
- (viii) **Mutagens, teratogens, carcinogens, intercalators, sternutators, sensitisers** (must be handled with very special care using all appropriate protective garments and after having considered all possible alternatives. The meanings of these words must be understood)
- (ix) **Disposal** (Solvents in waste bottles. Metal residues (Ru, Rh, Hg, Ag, Pd, Pt, Ir, Mo, Sn, Ln) in appropriate bottle for recycling where appropriate. Residues containing alkali metals or CaH₂ to be treated with propan-2-ol/toluene (50/50) in fume cupboard)
- (x) **Liquid nitrogen** (must be handled with the appropriate insulating gloves and eye protection. Air or oxygen must not be pumped through a trap cooled by liquid nitrogen)
- (xi) **Dry ice** (solid carbon dioxide, must be handled with insulating gloves)
- (xii) **Organic azide compounds** (must not be used without prior permission of supervisor. All precautions must be taken, including the use of small quantities, blast shields and appropriate glassware)
- (xiii) **Dimethyl sulfoxide** (solutions are capable of transporting solutes across skin, Rubber gloves must be used when handling these solutions)
- (xiv) **Drying agents** (must be disposed of according to established procedures. Nearly all drying agents react vigorously with water; care must be taken to avoid a violent reaction with water. Residues containing alkali metals or CaH₂ to be treated with propan-2-ol/toluene (50/50))
- (xv) **Ethereal solvents** (must be dated upon opening. Empty and near empty ethereal solvent bottles represent an explosive hazard due to the possible presence of organic peroxides; care must be taken to avoid these situations)

- (xvi) **Reactions using greater than 2L of solvent(s)** (must be authorised by your supervisor before use)
- (xvii) **Methylating agents** (e.g. methyl iodide, must be used with extreme caution. They must be stored in a well ventilated area and precautions must be taken to avoid spillage)
- (xviii) **Strong bases / Reducing agents** (such as butyl-lithium, lithium aluminium hydride, lithium di-isopropyl amide, sodium, potassium and sodium borohydride are pyrophoric or react explosively with water. Special care must be exercised when using these compounds especially alkyl-lithiums. These chemicals must be handled only in the presence of at least one other person who is fully aware of its use)
- (xix) **Formaldehyde and paraformaldehyde** (must be handled only in fumehood. Its aqueous solution must not be mixed directly with neat liquid amines due to a potential explosive hazard)

4. Housekeeping

- (i) Keep lab. clean and tidy at all times.
- (ii) Avoid build-up of dirty glassware. Sinks must not become cluttered with glassware
- (iii) Dispose of sharps in appropriate bin: do not use this bin for disposal of other material
- (iv) Floor must be kept clear of trip hazards
- (v) Food must not be consumed in the laboratory. This will be viewed as a serious breach of laboratory practice.
- (vi) Broken glassware must be disposed of immediately in the proper container. Ground glass joints should be recycled where appropriate.
- (vii) All samples in the fridge freezer must be correctly labelled otherwise **they will be removed without warning**
- (viii) All samples in the glove box must be correctly labelled otherwise **they will be removed without warning**
- (ix) Check the atmosphere in the glove box once a day before use.
- (x) All chemical substances must be correctly and fully labelled

5. Use of heating devices

- (i) Take special care with stills: do not leave residues to build up. Avoid leaving unattended if possible.
- (ii) Label oil baths using a pen to indicate whether they are paraffin or silicone
- (iii) If a reaction involving external heating must be left running outside hours:
 - a) Where possible, use graphite or silicone oil in preference to paraffin or a heating mantle.
 - b) Where possible, fit with a safety thermometer.
 - c) Inform supervisor and label with overnight permit
- (iv) Sand baths must not be used unless absolutely necessary and only after prior consultation with the lab technician or your supervisor

6. Use of flames

- (i) Use butane torch if possible

- (ii) Remove all inflammable solvents (e.g. in wash bottles) from the surrounding area beforehand.

7. Use of cooling water

- (i) Any water cooling system which is left running overnight must be fitted with hoseclips, install a flow indicator if possible
- (ii) Cooling water outlets must not be allowed to discharge anywhere than a sink

8. Vacuum lines and condensation of gases

- (i) **Schlenk lines and equipment** (must not be used without training. 'Using Schlenk lines' software provides a useful electronic reference manual to be used in conjunction with training)
- (ii) **Beware of explosion hazard from condensed gases:** liquid N₂ can condense Ar, air, C₂H₄, etc.
- (iii) Remove liquid nitrogen from traps when switching off vac. lines. Let in air. Clean out trap.
- (iv) Use face shield for more hazardous materials (keep quantities small!).
- (v) Vent pump exhausts and mercury bubblers to fume cupboards or outside.
- (vi) Check that mercury bubblers are not blocked
- (vii) **Mercury bubblers** must be contained in a beaker
- (viii) **Vacuum pumps** must be protected by liquid nitrogen traps
- (ix) **Cannula wires** must be handled carefully so that they cannot cause a puncture wound. They must not be bent to an extent that they represent a hazard if they are accidentally released.
- (x) **Electronic pressure gauges** must only be turned on when the vacuum system is operating
- (xi) All grease must be assiduously removed before a vacuum line is sent for repair

9. Glove boxes

- (i) All operators must be trained prior to use to prevent contamination of the inert atmosphere
- (ii) Operators should be aware of how to purge the box and isolate the purification columns in case of air contaminating the atmosphere
- (iii) Regeneration of the columns by junior researchers should only be undertaken alongside your supervisor or a senior lab member

10. Pressurised gases

- (i) **Large gas cylinder** (must be fitted with an appropriate regulator. They must not be free-standing. They must not be fully emptied. They are heavy and cumbersome and must be moved carefully)
- (ii) **Reactions involving pressures greater than 1.25 atm** (must be carried out using specialised equipment after training from your supervisor. Never perform reactions at higher than atmospheric pressure in conventional glassware or before appropriate training has been given)

11. Solvent drying stills

- (i) **Drying stills** must be kept clean and tidy at all times
- (ii) Where available '**Grubbs**' columns should be used
- (iii) **Stills** must **not** be left running unattended
- (iv) **Stills** must **not** be used without a water flow meter
- (v) **Stills** must **not** be used by unauthorised personnel
- (vi) Solvents for drying must not be prepared by pouring solvent directly from the storage bottle onto the drying agent
- (vii) **Tetrahydrofuran** drying can be hazardous, consult your supervisor

12. Overnight running

- (i) Leave overnight card with details.
- (ii) Consult your supervisor about reactions involving heating (see 5 above).

13. Undergraduate or visiting students

- (i) Inform of protocol
- (ii) Ensure training in new techniques and on new instruments
- (iii) No new operations to be attempted alone
- (iv) Work in laboratory only in the company of a full-time research group member

14. Use of equipment by non-members of the research group

Permission for use by anyone outside the group only from supervisor or technician

15. Visitors

- (i) Insist on safety glasses
- (ii) Non-members of the department must have permission to visit from FMK or CMK

16. Out of hours working

- (i) Out of hours working should be restricted to office work where possible
- (ii) Any out of hours laboratory work should be authorised in advance by your supervisor
- (iii) No out of hours work can be carried out without at least one other person being present in either the adjacent office or lab