CMMP Laboratory Guidance Notes

Researchers must follow the <u>UCL</u> and <u>Departmental</u> Health & Safety Policies and Procedures. The following are guidelines for the use of some of the equipment and substances encountered in F7, F8, F10C, F16 and F19. Please note that all activities must be covered by a specific active <u>riskNET</u> risk assessment.

Before undertaking any work in the Laboratories, you <u>must</u> hold a CMMP Work Permit, and you <u>must</u> familiarise yourself with and follow the <u>UCL</u> and <u>Departmental</u> Safety Guidelines, Local Rules, and Standard Operating Procedures. For example, you <u>must</u> abide by the Detailed Arrangements specified in the Departmental Health & Safety Handbook: 4.4 & 6.3 After Hours and Lone Working; 6.7 Chemical Safety; 6.9 Compressed Gases and Gas Cylinders; 6.11 Cryogenic Substances.

The following mandatory courses must be completed by all researchers before using laboratories:

- Mandatory Course: <u>UCL Safety Induction</u>
- Mandatory Course: Fire Safety
- Mandatory Course: <u>Principles of Laboratory Safety</u>
- Mandatory Course: Principles of Risk Assessment

Other training courses are listed <u>here</u>. These include Gas Cylinder Safety, and Gas Safety in Laboratories.

Before commencing any new experimental work within the laboratory the special risks that apply to that particular work <u>must</u> be assessed and recorded in a Risk Assessment. The experimental method <u>must</u> be documented in your risk assessment *along with all the associated risks* from the chemicals required and any proposed reaction to be performed, along with measures to dispose of any waste generated. Risk Assessments should be uploaded and approved from your <u>riskNET</u> account:

- Departmental Health and Safety Handbook
- H&S links and resources: <u>http://www.ucl.ac.uk/~ucapnsz/safety.html</u>
- Equipment booking: <u>https://teamup.com/ks2a7cf70f258a3aa9</u>

If you are in any doubt about the risks associated with any part of the experiment, you should consult with your supervisor or if still in doubt with the Department Safety Officer, Lee Bebbington, before proceeding.

Oxygen depletion monitors have been installed in rooms F7, F8, F10c – see RA for Compressed Gases. The alarms are located outside the rooms by the entrances to F8 and F10c. Do not enter the rooms if the alarm is sounding. If you are in the room when the alarm sounds, isolate any gas supply you are using and then leave the room. Do not re-enter the room until the oxygen level is safe.

Equipment marked * is restricted access, and you must receive specific instruction and be placed on the supervisors list of registered users before undertaking this work.

Contacts:

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Tube & Muffle Furnace Annealing*

The tube and muffle furnace annealing experimental apparatus in Rooms F7 and F10E may be used safely as long as the following points are appreciated and followed at all times:

- 1. <u>Risk of burns</u>: At no time while a furnace is being used should the protective grill be removed, and the hands or any part of the body be brought into contact with the white 'work-tube' which rests inside the cylindrical bore of the tube furnace.
- 2. <u>Risk of fire</u>: At no time should any inflammable materials such as paper, cardboard or fabric of any kind be left on the bench top on which the furnace rests or on/in/near any of the furnaces. You must not use an external temperature sensor to control the furnace. The furnaces must be switched off when not in use.
- 3. <u>Gas bottles</u>: Pressurised gas bottles may sometimes used to pass inert gas through the work-tube. You must be trained to use pressurised gases, and at all times the gas bottle containing the pressurised inert gas must be kept upright, and must be firmly attached (chained) to the wall bracket provided.
- 4. <u>Insertion/Removal of Samples</u>: whenever samples are being placed inside or removed from the furnace work-tube, the leather gauntlets (gloves) and perspex face-guard provided should be worn, and the long-handled tools provided should be used. The muffle furnace door must be closed before the furnace is heated. Hot samples should be placed on the heat-resistant ceramic tile to cool, and the area labelled accordingly.
- 5. <u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof. Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Pavlo Zubko (<u>p.zubko@ucl.ac.uk</u>)

Ultrasonic Probe*

The 750W Ultrasonic Probe located in F10c may be used only in the fume cupboard within the ultrasound enclosure and while wearing ear protection. A warning sign must be placed on the lab door. Do not run the probe at the same time as ammonia condensation or other procedures are underway in the fume hood.

- 1. <u>Risk of Bone Damage</u>: At no time should the probe tip be touched.
- 2. <u>Risk of fire</u>: Surround solvents in an ice bath/liquid coolant. Maximum Power operation at 60%.
- 3. <u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>)

Cryogenic Systems and Compressed Gases (Cylinders, Regulators)*

Cryogens and compressed gases should only be used in accordance with Departmental Guidelines.

- 1. <u>Disturbing the Storage Dewar</u>: When liquid helium or liquid nitrogen is being used in any cryogenic system, care should be taken not to violently disturb the storage dewar. The dewar must be kept upright at all times.
- 2. <u>Pressure Build Up</u>: When operating the system, you must ensure that the appropriate valves are opened and closed according to the instructions provided by the manufacturer.
- 3. <u>Gas Vessels, Cylinders and Regulators:</u> Gases should only be contained in tested vessels and pipework, manufactured from appropriate materials, and should be leak tested before every experiment.

Only qualified staff (*ie* those who have attended the UCL Courses on cylinders and regulators, links below) should operate and/or change regulators. Mandatory courses:

- <u>https://www.ucl.ac.uk/safety-services/policies/2021/aug/connecting-regulators-and-safe-cylinder-set-training</u>
- <u>https://www.ucl.ac.uk/safety-services/policies/2020/dec/using-gas-cylinders-safely-within-universities-training</u>
- 4. <u>Risk of Burns and Skin Damage</u>: The leather gauntlets and goggles provided must be worn whenever you are working with either liquid helium or liquid nitrogen, to avoid skin damage. Be careful not to touch the leg of the cryostat transfer tube without wearing the leather gauntlets.
- 5. <u>Risk of Asphyxiation</u>: caused by gradual or sudden replacement of air with nitrogen or helium. Low oxygen levels are not readily discernible. Considered "low" if oxygen content drops to 19% or less (21% normal). Below 16%, uptake of oxygen in the body is impaired and initial symptoms are irrational and disorderly behaviour (UMIST Safety Manual). Concentration of oxygen will vary with position in the room. Effects of oxygen depletion are rapid and potentially fatal fainting occurs (<11%): death is within a few breaths (<8%). The degree of hazard depends on the amount of gas released and on the volume of the room, local ventilation and airflow within the room. Ventilation cannot be relied upon, out of hours.</p>

Room ventilation, where it is present, can normally be expected to supply make-up air at the rate of about 0.1 to 0.2 cubic metres per second. Exact figures are given in Appendix 2.

Estimate: a broken nitrogen gas line at a nominal pressure of 50psi could release gas at a rate of about 50-100 litres a minute (0.1 cubic metres per <u>minute</u>, or rather less than the makeup air). There would be considerable noise associated with this flow rate, which is likely to attract attention. The risk of asphyxiation is considered low

because there is enough time to get out and seek help. There is a small possibility of lower oxygen content in air during a power cut, or when ventilation is no longer operational (many valves are manual). Nitrogen isolation valves should be indicated on the risk assessment for each room.

Of particular concern is the scenario where someone falls unconscious in such a room. Supervisors should be alerted if a potential room user has a known medical condition where this is more likely, for instance, diabetes or epilepsy, and the case should be discussed with Occupational Health.

- 5. <u>Oxygen Depletion Monitors</u>: Rooms considered at risk are F7, F8, F10 (lobby) and F10c. These rooms contain gas cylinders (argon and nitrogen) and have oxygen depletion monitors fitted, and alarms are placed outside F8, F10 and F10c.
- 6. <u>Emergency Procedures</u>: Do not enter a room if the oxygen depletion alarm is sounding or if low oxygen level is suspected (for example if you suspect a faulty cylinder or manifold). If you are in the room when the alarm sounds, if possible isolate any gas supply you are using and then leave the room.

Isolation values for the F10c N_2 supply are in the F10 lobby, under the spiral stairs. Isolation values for the F6 N_2 supply are in F7. Isolation for the remaining Ar supplies are located by the apparatus itself (for example, the Ar gloveboxes in F10c).

Do not re-enter the room until the oxygen level is safe, and until at least 2 complete air changes have taken place (see Appendix 2). Do not loiter in the F10 area corridor. In the case of an alarm sounding in F7, F8 must also be vacated. If the alarm sounds in F10c, F10 and F10a must be vacated.

Air change rates and room volumes are given in Appendix 2. A typical K-size cylinder contains 7.2m³ of gas at 175 bar.

An incident/accident report form must be completed whenever an O₂ alarm is set off.

7. <u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof. Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>) Prof Pavlo Zubko (<u>p.zubko@ucl.ac.uk</u>)

Chemical Preparation of Samples

General experimental practice, Rooms F10c, F7, F8 F16 Chemistry Laboratories

Users working in the samples preparation laboratory, rooms F7, F8 and F10c are required to follow the Local Rules, Departmental and <u>UCL</u> safety procedures. These include:

- 1. <u>Experiments in progress</u>: To complete and display an 'experiment in progress' form (as below) when working in F10c, F7 or F8. These forms are stored in F10c, F7 and F8.
- 2. <u>Staffing</u>: To adhere to a maximum staffing level at any given time of at most; 3 people in either room, and a total of at most 5 people in both rooms F7 & F8, and 5 people in room F10c.
- 3. <u>Faults</u>: To immediately report any faults with equipment to Professor Chris Howard (c.howard@ucl.ac.uk).
- 4. <u>Protective clothing</u>: To wear suitable protective clothing as appropriate, such as a lab coat, eye protection and gloves. This is essential due to the corrosive nature of many of the chemicals stored in the laboratory.
- 5. <u>Marking containers</u>: To keep all bottles and sample containers clearly marked including information on Hazards so as to avoid confusion for others.
- 6. <u>Tidiness</u>: To keep the work space as free of clutter and unnecessary items as practical.
- 7. <u>Fire</u>: To be aware of the location of basic fire-fighting equipment; fire extinguisher, fire blanket and sand bucket.
- 8. <u>First aid</u>: To be aware of the location of the first aid kit and emergency eye wash in case of accident.
- 9. <u>Disposal of chemicals</u>: To follow correct procedure when disposing of chemicals:
 - **Small** quantities of water soluble chemicals may be disposed of in the fume cupboard sink, along with copious amounts of water.
 - **Organic solvents** should be disposed of in bottles, marked for **halogenated** and **non-halogenated** solvents.
 - Waste must be stored and collected regularly in the manner approved under the UCL policies at:
 - <u>https://www.ucl.ac.uk/estates/our-services/waste-and-recycling/laboratory-waste</u>
- 10. <u>Sharps</u>: To dispose of sharps in the correct manner i.e. in the supplied Sharps bin.
- 11. <u>Storage</u>: To store chemicals in a sensible manner. Cabinets are provided for solvents and for acids.
- 12. <u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof. Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>) Prof Pavlo Zubko (p.zubko@ucl.ac.uk)

X-ray Diffraction*

Only trained approved users can conduct experiments using the D2 Phaser diffractometer and Rigaku Smartlab in room F19 (Registered Radiation Area). <u>UCL</u>, <u>Departmental</u> and F19 local rules guidance should be followed at all times, and you must complete the following to become an authorised user and before starting work:

- <u>Become a radiation worker</u>
- Principles of Radiation Safety
- Basic X-ray Physics
- Safe Use of X-rays

All persons must have completed the training, operator induction, and their name and signature must be added to the 'Authorised Persons List' **prior** to commencing work with the equipment.

Before loading sample and running a programme:

- The X-ray warning light on the equipment exterior is illuminated
- Check the red warning light inside the equipment is not illuminate. If it is, this indicates that the X-ray shutter is removed and no attempt should be made to open the door. Where necessary equipment fault procedures outlined in section 8 must be followed.
- There is no obvious damage to the equipment.
- If there is any malfunction of the equipment of visible damage, the equipment must not be used and must be immediately reported to the Principal Investigator in charge of the equipment or the RPS using the contact details in this document.

Once the programme has finished:

- Persons must check the shutter has returned to the closed position prior to attempting to retrieve their sample. This is indicated by the red illuminated button inside.
- 1. <u>Contacts</u>: Prof. Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Pavlo Zubko (<u>p.zubko@ucl.ac.uk</u>)

Handling Toxic Gases including Ammonia & Methylamine (Gas Rigs)*

Toxic gases should be handled in the fume-cupboard in F8 or F10c, according to the practices described in the appropriate Materials Safety Data Sheet (MSDS). In particular, users should ensure that clear warning signs are used. In all cases, the minimum quantities of gases should be used.

- 1. <u>Emergency Procedures</u>: In all cases, the user should follow the guidelines set out in the appropriate Materials Safety Data Sheet (MSDS), and be aware of the Emergency Procedures. In the case of suspected ammonia or methylamine leak, if possible isolate the source, and then evacuate the room. All ammonia and methylamine condensation work must be conducted in a fume-hood.
- 2. <u>Labels and Warnings:</u> The area around the gas should be labelled clearly, with a warning sign and summary of emergency procedures. An experiment in progress form must be completed, and refer to the appropriate RiskNet risk assessment.
- 3. <u>Gas Vessels, Cylinders, Gas Rigs:</u> Gases should only be contained in tested vessels and pipework, manufactured from appropriate materials, and should be leak tested before every experiment. Only qualified staff (*ie* those who have attended the UCL Course on Laboratory gases or an equivalent) should operate regulators. For ammonia or methylamine based condensations, the main supply bottle must be isolated by at least two valves, including the main bottle valve.

Gas must not be placed back into the original supply bottle.

Gas must be decanted into a separate steel Swagelok supply bottle, and dispensed to a buffer volume. There must always be at least one empty buffer volume available at the start of each condensation.

Supply bottles must be labelled with contents.

All cylinders must be securely clamped to the gas rig or a suitable stand.

Only qualified staff (*ie* those who have attended the UCL Courses on cylinders and regulators, links below, and Swagelok joints course or equivalent) should operate and/or change regulators and gas rig valves and fittings. Mandatory e-learning courses:

- <u>https://www.ucl.ac.uk/safety-services/policies/2021/aug/connecting-regulators-and-safe-cylinder-set-training</u>
- <u>https://www.ucl.ac.uk/safety-services/policies/2020/dec/using-gas-cylinders-</u> <u>safely-within-universities-training</u>

Gas rigs must be tested, stage-by-stage, under vacuum to $< 10^{-5}$ mbar before use, and must be fitted with a pressure release valve that vents into a fume-hood.

Pumping stations must be vented into a fumehood or safe suction line.

- 4. <u>Cryopumping</u>: use of liquid nitrogen for cryopumping must only be conducted on stainless steel Swagelok bottles. Care must be taken to keep the top valve and at least the tapered top section of the supply bottle ice-free.
- 5. <u>Risk of Exposure to Ammonia:</u> use of the ammonia gas rig presents the risk of exposure to this toxic and flammable gas. The STEL limit is 25ppm this will be achieved in F8 or F10c if 5 bar are released from a 300cc buffer bottle (1.6 bar litre). This limit must not be exceeded in the buffer or decanting cylinder. In addition, the main ammonia supply bottle must be isolated by at least two valves.

The vapour pressure of liquid ammonia is around 8.5 bar at 20°C.

- Ammonia: Classification according to Regulation (EC) No 1272/2008
 Flammable gases (Category 2), H221
 Gases under pressure (Compressed gas), H280
 Acute toxicity, Inhalation (Category 3), H331
 Skin corrosion (Category 1B), H314
 Acute aquatic toxicity (Category 1), H400
 Chronic aquatic toxicity (Category 1), H410
- 6. <u>Risk of Exposure to Methylamine:</u> use of the methylamine gas rig presents the risk of exposure to this toxic and flammable gas. The STEL limit is 15ppm this will be achieved in F8 or F10c if 3 bar are released from a 300cc buffer bottle (1.0 bar litre). This limit must not be exceeded in the buffer or decanting cylinder. In addition, the main methylamine supply bottle must be isolated by at least two valves.

The vapour pressure of liquid methylamine is around 3.0 bar at 20°C.

- Methylamine: Classification according to Regulation (EC) No 1272/2008 Flammable gases (Category 1), H220 Gases under pressure (Compressed gas), H280 Acute toxicity, Inhalation (Category 4), H332 Skin irritation (Category 2), H315 Serious eye damage (Category 1), H318 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335
- 7. <u>Supply and Storage of Gases</u>: gases must be purchased in the smallest available volume (usually a lecture bottle), and must be stored securely in properly labelled vented gas cabinet.
- 8. <u>Disposal of Gases</u>: Should only be conducted in the recommended manner. See: <u>https://www.ucl.ac.uk/estates/estates-services/waste-and-recycling</u>

- 9. <u>After Hours Experiments:</u> At least two people should be present in the laboratory during after hours experiments. Ammonia condensation should never be conducted after hours.
- 10.<u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>)

Highly Flammable Gases including Hydrogen *

Highly Flammable gas cylinders must only be used in F10 or the fume hoods and according to the practices described in the appropriate Materials Safety Data Sheet (MSDS) and within the ATEX <u>http://www.hse.gov.uk/fireandexplosion/atex.htm</u> and the Dangerous Substances and Explosive Atmospheres Regulations 2002 (DSEAR) <u>http://www.hse.gov.uk/fireandexplosion/zoning.pdf</u>.

Hazardous places are classified in terms of zones on the basis of the frequency and duration of the occurrence of an explosive atmosphere.

For gases, vapours and mists the zone classifications are:

- Zone 0; A place in which an explosive atmosphere consisting of a mixture with air of dangerous substances in the form of gas, vapour or mist is present continuously or for long periods or frequently.
- Zone 1; A place in which an explosive atmosphere consisting of a mixture with air of dangerous substances in the form of gas, vapour or mist is likely to occur in normal operation occasionally.
- Zone 2; A place in which an explosive atmosphere consisting of a mixture with air of dangerous substances in the form of gas, vapour or mist is not likely to occur in normal operation but, if it does occur, will persist for a short period only.Users should ensure that clear warning signs are used. In all cases, the minimum quantities of gases should be used.

Within our laboratories, researchers must ensure that procedures are in place to ensure that an explosive atmosphere cannot occur, and that the laboratories are therefore not classified as hazardous areas in this respect. Researchers must ensure that release of gases is extremely unlikely to occur and/or the quantities released are small enough to ensure that they disperse so that an explosive atmosphere cannot arise.

For example, if a dangerous substance is being carried through a seamless pipe, and that pipe has been properly installed and maintained, it is extremely unlikely that the substance will be released. An explosive atmosphere would not be expected to occur from this source and the area surrounding the pipe would be non-hazardous.

A spillage from a small bottle of solvent would release so little flammable material that no special precautions are needed other than the general control of ignition sources (for

example, no smoking) and cleaning and disposing of the spillage. It would not be classified as a hazardous area. When considering whether hazardous area classification is necessary for "small" quantities of dangerous substances the actual circumstances of use and any specific industry guidance should also be taken into account.

We give examples of calculations in the Appendices, but have also given explicit procedures in the case of Hydrogen in point 5 below.

- <u>Emergency Procedures</u>: In all cases, the user should follow the guidelines set out in the appropriate Materials Safety Data Sheet (MSDS), and be aware of the Emergency Procedures. If hydrogen leak is suspected, the source should be isolated and the area evacuated. Do not return until the air has been replaced (please see appendices for air-change rates in each room).
- 2. <u>Labels and Warnings:</u> The area around the gas should be labelled clearly, with a warning sign and summary of emergency procedures.
- 3. <u>Gas Vessels and Cylinders:</u> Gases should only be contained in tested vessels and pipework, manufactured from appropriate materials, and should be leak tested before every experiment. Only qualified staff (ie those who have attended the UCL Course on Laboratory gases or an equivalent) should operate regulators. For hydrogen based preparations, the main supply bottle must be isolated by at least two valves, including the main bottle valve. Hydrogen gas cylinders will be leak tested on installation by BOC.
- 4. <u>Risk of Explosion (hydrogen)</u>: hydrogen may be used as the working gas on the IGA in F10, and presents a risk of explosion. The Lower Explosion Limit (LEL) for H₂ is 4%, and the Upper Explosion Limit (UEL) is 74.5%. Autoignition occurs at 570°C.

The volume of F10 is approximately 365m³ and the a/c units change the atmosphere approximately 10 times per hour. A Nederman sparless hood is fitted over the IGA system, and should be placed over the gas supply rig/vent.

Only one B-size hydrogen cylinder should be used in F10, and this must be connected via a 1/8th or less Swagelok fitted pipe. The total volume of hydrogen in this cylinder is 1.2m³ at STP. The cylinder must be fitted by BOC with a BOC approved anti-flashback regulator, and the pipe work leak tested by BOC at the time of installation.

Please note that cylinder sizes B and K filled to 175bar contain 1.5 and 7.2 m^3 respectively.

The maximum volume of hydrogen used in any experiment must be >100L at STP, and preferably > 20L at STP.

The maximum pressure delivered from the regulator must be 20bar.

5. <u>Disposal of Gases</u>: Should only be conducted in the recommended manner. See:

http://www.ucl.ac.uk/efd/cleaningandwaste/waste/

- 6. <u>After Hours Experiments:</u> At least two people should be present in the laboratory during after hours experiments.
- 7. <u>Contact</u>: Prof. Neal Skipper (<u>n.skipper@ucl.ac.uk</u>)

Alkali and Reactive Metals Requiring Glovebox Handling*

When possible Alkali and Reactive Metals should be handled in F7/F8, according to the practices described in the appropriate Materials Safety Data Sheet (MSDS). In particular, users should ensure that clear warning signs are used. In all cases, the minimum quantity of chemical should be used. When it is essential to use such materials outside F7/F8, for example for experiments to be conducted on the X-ray equipment, the following procedures should be followed.

- 1. <u>Emergency Procedures</u>: In all cases, the user should follow the guidelines set out in the appropriate Materials Safety Data Sheet (MSDS), and be aware of the Emergency Procedures.
- 2. <u>Labels and Warnings:</u> The area around the gas should be labelled clearly, with a warning sign and summary of emergency procedures.
- 3. <u>Containers:</u> Metals and their products should only be contained in tested containers, manufactured from appropriate materials. Suspected leaks should be reported immediately.
- 4. <u>Disposal of Metals and Products</u>: Should only be conducted in the recommended manner. See: <u>http://www.ucl.ac.uk/efd/cleaningandwaste/waste/</u>
- 5. <u>After Hours Experiments:</u> At least two people should be present in the laboratory during after hours experiments.
- 6. <u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>)

Toxic and Flammable Solvents

When possible Toxic and Flammable Solvents should be handled in F7/F8, according to the practices described in the appropriate Materials Safety Data Sheet (MSDS). In particular, users should ensure that clear warning signs are used. When it is essential to use such materials outside F7/F8, for example for experiments to be conducted on the X-ray equipment, the following procedures should be followed.

- 1. <u>Emergency Procedures</u>: In all cases, the user should follow the guidelines set out in the appropriate Materials Safety Data Sheet (MSDS), and be aware of the Emergency Procedures.
- 2. <u>Labels and Warnings:</u> The area around the solvent should be labelled clearly, with a warning sign and summary of emergency procedures.
- 3. <u>Containers:</u> Solvents and their products should only be contained in tested containers, manufactured from appropriate materials. Suspected leaks should be reported immediately.
- 4. <u>Disposal of Toxic and Flammable</u>: Should only be conducted in the recommended manner. See: <u>http://www.ucl.ac.uk/efd/cleaningandwaste/waste/</u>
- 5. <u>After Hours Experiments:</u> At least two people should be present in the laboratory during after hours experiments.
- 6. <u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (f.cacialli@ucl.ac.uk)

Handling Toxic Chemicals

When possible Toxic Chemicals should be handled in F7/F8, according to the practices described in the appropriate Materials Safety Data Sheet (MSDS). In particular, users should ensure that clear warning signs are used. When it is essential to use such materials outside F7/F8, for example for experiments to be conducted on the X-ray equipment, the following procedures should be followed.

- 1. <u>Emergency Procedures</u>: In all cases, the user should follow the guidelines set out in the appropriate Materials Safety Data Sheet (MSDS), and be aware of the Emergency Procedures.
- 2. <u>Labels and Warnings:</u> The area around the gas should be labelled clearly, with a warning sign and summary of emergency procedures.
- 3. <u>Containers:</u> Metals and their products should only be contained in tested containers, manufactured from appropriate materials. Suspected leaks should be reported immediately.
- 4. <u>Disposal of Metals and Products</u>: Should only be conducted in the recommended manner. See: <u>http://www.ucl.ac.uk/efd/cleaningandwaste/waste/</u>

- 5. <u>After Hours Experiments:</u> At least two people should be present in the laboratory during after hours experiments.
- 6. <u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>) Prof Pavlo Zubko (<u>p.zubko@ucl.ac.uk</u>)

Oxy-Propane Micro-Blowtorch*

The oxy-propane blow torch should only be used to seal small glass ampoules by qualified staff according to the operating safety guidelines supplied by the manufacturer, in the presence of at least two people. Sealing must be conducted in the fume hood.

- <u>Emergency Procedures</u>: In all cases, the user should follow the guidelines set out in the appropriate Materials Safety Data Sheet (MSDS), and be aware of the Emergency Procedures.
- 2. <u>Risk of Explosion</u>: Sealing must be conducted in the fume cupboard. Sealed samples must be contained in the fume hood when not in actual use.
- 3. <u>Disposal of Samples and Products</u>: Sealed samples should be disposed of as soon as possible after use, in the recommended manner.
- 4. <u>Labels and Warnings:</u> The area around the blowtorch should be labelled clearly, with a warning sign and summary of emergency procedures. A label must be placed on the door to F8 while sealing is in progress, and no other work may be carried out in F7 or F8 while sealing is in progress.
- 5. <u>Contact</u>: Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>)

Oxygen Plasma Treatment ("Ashing")*

The Tegal Oxygen plasma asher, oxygen bottle, argon bottle and vacuum pump are located in F8. The guidelines for the handling of Flammable gases and Laboratory gases must therefore be followed at all times. Users must be familiar with the procedures set out in the Operating Manual.

- 1. <u>Emergency Procedures</u>: In all cases, the user should follow the guidelines set out in the Operating Manual and appropriate Materials Safety Data Sheet (MSDS), and be aware of the Emergency Procedures.
- 2. <u>Gas Vessels and Cylinders:</u> Gases should only be contained in tested vessels and pipework, manufactured from appropriate materials, and should be leak tested before

every experiment. Only qualified staff (ie those who have attended the UCL Course on Laboratory gases or an equivalent) should operate regulators. Turn off gas bottles after use.

- 3. <u>Risk of Explosion:</u> Pure oxygen is used, and is a potential hazard if a fire breaks out in storage or in use. Danger of explosion if pump oil is replaced with mineral oil during pure oxygen pumping operations. Take especial care during maintenance to replace with appropriate pump oil (not mineral oil).
- 4. <u>Contact:</u> Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

High Concentration HCL

<u>Room:</u> F6, F10c

Equipment Used: Beakers, pipettes, HCI and deionised water.

<u>Nature of hazards + frequency of encounter:</u> Possible acid burns, risk of inhalation of Cl₂ gas, risk of strong exothermic reactions. Causes burns and is irritating to the respiratory system.

<u>Precautions:</u> Use small quantities only. Use in fume hood. Make FULL use of personal safety equipment available. Never add water to acid, only acid to water.

<u>Emergency action</u>: Flush any acid burn with plenty of cool water for 15 min, irrigate the eye thoroughly. Contact a physician.

<u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>) Prof Pavlo Zubko (<u>p.zubko@ucl.ac.uk</u>)

<u>Supporting documents:</u> Material safety data sheet held in Chemcials cupboard in F8, also the COSHH form in F6.

Electroabsorption Rig*

<u>Room:</u> F34

<u>Equipment Used:</u> Xe –lamp, power units, rotary oil pump, optical alignment equipment, computer

Nature of hazards + frequency of encounter:

- 1) Electrical Risks
 - High voltage (2-3 kV) for experiments on planar devices with 100 micron or so electrodes spacing.
 - High voltage on the Xe-lamp.

2) Non-Electrical risks

- Possibility of explosion of the Xe-lamp upon replacing
- Handling of liquid nitrogen for sample cooling (NB both burns and asphyxiation)
- Handling of Helium cryogenerator, risk for cold burns and asphyxiation
- Exposure to minor levels of ozone (produced by the Xe-lamp), and to UV radiation from the lamp during alignment and operation.
- Implosion of the samples holder under vacuum.

<u>Precautions:</u> Do not use without training. (Contact Prof. F. Cacialli) Follow instructions. Never attempt to change the bulb alone. Wear heavy gloves, coat and face mask when changing the bulb. Be aware of the UV output of the lamp. Wear UV- goggles and protective coating when working with UV radiation from the lamp. Oxygen deficiency sensors are mounted in the lab. Check the integrity of the cabling and vacuum system regurlarly. Do not enter if the alarm sounds.

<u>Emergency action</u>: Switch off electrical supply (if safe to do so) and seek help from others. If first aid is needed contact first aider in the department.

<u>Contacts</u>: Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

Measurement of IVL Characteristics of Light Emitting Devices

<u>Room:</u> F34

Equipment Used: Rotary Pump, PC+VDU, EL Rig, Voltage supply, Keithley multimeter

<u>Nature of hazards + frequency of encounter</u>: Electrical Hazard – rare occurrence. Rotary Pump breakdown – rare occurrence. Risk of implosion of the samples holder under vacuum - rare occurrence. <u>Precautions:</u> Check cabling and the integrity of the vacuum system regularly. Maintain rotary pump (please see Appendix 5) and switch off the pump when not in use. Also return the rig to air after measurements.

Emergency action: Switch all off electrical supplies.

<u>Emergency contact(s):</u> Prof. Franco. Cacialli (f.cacialli@ucl.ac.uk)

Measurement of Photovoltaics IV under Solar Simulator

Room: B106 (LCN Building)

<u>Equipment Used:</u> Solar Simulator Abet Technologies "Class AAA", Keithley multimeter, RV12 rotary oil pump.

<u>Nature of hazards + frequency of encounter</u>: Electrical Hazard (rare occurrence). Rotary Pump breakdown (rare occurrence). Skin (rare occurrence) - Excessive short-term exposure causes sunburn and can result in an increased risk of skin cancer. Eyes (rare occurrence) - Exposure can cause acute damage to the cornea and conjunctiva causing pain, light sensitivity and tearing. These effects can be felt between 30 minutes and 24hrs after exposure. Prolonged exposure can cause permanent retinal damage. Risk of implosion of the samples holder under vacuum (rare occurrence).

<u>Precautions:</u> Hands must be protected by wearing gloves with low UV transmission (nitrile/latex gloves are suitable). Arms will be protected by wearing long sleeve lab coat but care must be taken to ensure there is no gap between cuff and glove (avoid prolonged exposure, >1 minute, of the skin to the light). Use UV goggles when using the equipment. Elimination of reflected UVR by avoiding shiny surfaces and using nonreflective UVR material. Good ventilation is needed, as the lamp produce some ozone. Check cabling and the integrity of the vacuum system regularly. Maintain rotary pump (please see Appendix 5) and switch off the pump when not in use. Also return the rig to air after measurements. Do not turn on lamp if hot. Make sure all instruments are turned off after use.

Emergency action: Switch all off electrical supplies.

<u>Contacts</u>: Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

Measurement of Photovoltaics IV and photocurrent spectra

Room: B106 (LCN Building)

<u>Equipment Used:</u> Rotary Pump, PC+VDU, PV Rig, two power supplies for the preamplifier, Keithley multimeter, electrometer, monochromator and Xenon arc lamp

<u>Nature of hazards + frequency of encounter</u>: Electrical Hazard – rare occurrence. Rotary Pump breakdown – rare occurrence

Arc lamp: Do not use without training, high voltages from supply. (Contact Prof. F. Cacialli) Follow instructions. Never attempt to change the bulb alone. Wear heavy gloves, coat and face mask when changing the bulb. Be aware of the UV output of the lamp. Wear UV-goggles and protective coating when working with UV radiation from the lamp. Good ventilation is needed, as the lamp produce some ozone.

Risk of implosion of the samples holder under vacuum - rare occurrence.

<u>Precautions:</u> Check cabling and the integrity of the vacuum system regularly. Maintain rotary pump (please see Appendix 5) and switch off the pump when not in use. Also return the rig to air after measurements. Do not turn on lamp if hot. Make sure all instruments are turned off after use.

Emergency action: Switch all off electrical supplies.

<u>Contacts</u>: Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

<u>Thermal Evaporation of Metals/Organics & Annealing of Substrates in</u> <u>Gloveboxes*</u>

Rooms: F6 and F10c

<u>Equipment Used:</u> K. J. Lesker Evaporation chamber, Hotplate, Glovebox, rotary and turbomolecular pumps. Various metals and organics. Glass substrates.

<u>Nature of hazards + frequency of encounter:</u> Water leak – rare occurrence. Electrical Hazard – rare occurrence. Trapped hand in chamber – rare occurrence. Oxidising agents - low occurrence.

<u>Precautions:</u> Switch off water at stopcock when not using chamber. Ensure cabling is in good condition. Operate chamber hoist only when no one is working in the glovebox. Keep all oxidising agents in nitrogen atmosphere

<u>Emergency action</u>: Switch all off electrical supplies. If there is a nitrogen leak, vacate the room

<u>Contacts</u>: Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>) Supporting documents: Evaporator and pump manuals kept in F6 and F10c

Use of Gloveboxes*

Room: F6, F8, F10c, F17

Equipment Used: Glovebox, Nitrogen, Argon

<u>Nature of hazards + frequency of encounter:</u> Asphyxiation, caused by gradual or sudden replacement of air with nitrogen. Incorrect use of pass tube and air lock chamber. Tear in gloves.

<u>Precautions:</u> Complete training on glovebox before operation, see Dr Howard, Prof Skipper or Prof Cacialli for this. Nitrogen/argon isolation valves should be indicated on the risk assessment for each room. Use Oxygen monitor before entering the room to verify that the oxygen levels are within acceptable limits. Be familiar with the asphyxiation Risk Assessment. Be gentle with the gloves and remove all jewellery before using them.

<u>Emergency action</u>: Vacate the room quickly if lack of oxygen is suspected. Fixed oxygen meters are available in Rooms F6, F7, F8, F10 and F17, Contact the lab emergency number. Turn off the nitrogen supply (if safe to do so) in F7/8.

<u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Prof Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>) Prof Pavlo Zubko (<u>p.zubko@ucl.ac.uk</u>)

Use of Spin Coater

<u>Room:</u> F6

<u>Equipment Used:</u> Vacuum Pump, Spin coater, Substrates (usually glass or mica), Solutions

<u>Nature of hazards + frequency of encounter</u>: Substrate not adhering to spin coater chuck well and hence spinning off – very infrequent. Solution spins off and hits user in the face.

<u>Precautions:</u> Check that vacuum has been established before spinning, close lid of spin coater and wear goggles. Drop "enough" solution, again wear goggles and gloves, close spin coater lid.

Emergency action: Seek First Aid if injury occurs.

<u>Contacts</u>: Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

Scanning Near-field Optical Microscopy/Lithography

<u>Room:</u> F17

<u>Equipment Used:</u> Computer, Electrical equipment, Lasers (HeCd, Argon ion and GaN diode lasers) – see separate laser risk assessment, Optical components such as lenses, mirrors, a shutter and optical fibres, Substrates (Glass)

<u>Nature of hazards + frequency of encounter:</u> Risks to eyes and skin from exposure to laser light - see separate laser risk assessment (frequent), Risk of eye damage from optical fibre (occasional), Risk of electrocution from high voltage power supply (rare), Risk of tripping when main lights are switched off for an experiments (occasional)

<u>Precautions:</u> Follow laser risk assessment guidelines. In particular, use goggles when there is a risk of scattered laser light. Also alert other users of the lab when lasers are being aligned

Emergency action: Seek First Aid if injury occurs.

<u>Contacts</u>: Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

Supporting documents: Laser Risk Assessments for F17 – held in F17

Optical Absorption Measurements of Films and Solutions

Room: F34 (soon to be moved to F19)

<u>Equipment Used:</u> Agilent 8453 UV-Vis Spectrophotometer with a Peltier Temperature Controller (Agilent 89090A), PC+VDU, glass substrates, various solutions

<u>Nature of hazards + frequency of encounter:</u> Broken Glass (rare), Fumes from heated solutions (occasional), Exposure to UV light (frequent), Electrical hazard (rare), Spillages of solution (occasional)

<u>Precautions:</u> Be cautious when mounting glass substrates. Do not heat liquids near their boiling points, ensure good ventilation in lab at all times. UV Light is directional and only

on during a scan so do not place hands/body in scan area during scan. Replace bulb whilst kit is switched off and unplugged and replace all protective coverings. Ensure all electrical cables are in good order and that when not in use the mains supply is switched off. Do not place cuvettes on equipment, keep the lid on and carry in the cuvette carrier.

<u>Emergency action:</u> Switch all off electrical supplies. In case of spillage, clean up accordingly to the COSHH. Put broken glass in the sharps bin.

<u>Contacts</u>: Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

Supporting documents: Aglient Spectrophotometer Manuals – kept in drawer in F34

Use of Soldering Station

Room: F19

Equipment Used: Soldering station, fume extractor and iron wire

Nature of hazards + level of risk:

- Risk of burns MEDIUM RISK
- Risk of exposure to lead LOW RISK

Precautions:

- Never touch the element or tip of the soldering iron. They are very hot (up to 400°C).
- Take great care to avoid touching the mains flex with the tip of the iron.
- Always return the soldering iron to its stand when not in use.
- Never put it down on the workbench, even for a moment!
- Work in a well-ventilated area. The fumes formed as you melt solder can be irritating.
- Avoid breathing it by keeping your head to the side of, not above, your work.
- Use lead-free solder as far as possible.
- Wash your hands after using solder. Solder contains lead which is a poisonous metal.
- Never leave the soldering iron unattended when switched on
- Never used without the soldering fume extractor.

Emergency action:

Switch off electrical supplies. Stop soldering immediately if you develop a headache.

Contact(s): Prof. F. Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

Use of Kelvin-probe

Room: F19 – soon to be moved to F17

Equipment Used: Kelvin probe equipment

Nature of hazards + level of risk:

- danger of electric shock LOW RISK
- exposure to nanoparticles or other molecules by touching substrates LOW RISK

Precautions:

- Wear gloves and handle substrates/samples with tweezers.
- check MSDS for materials used and wear appropriate protective clothing

Emergency action: Switch off electrical supply (if safe to do so).

Emergency contact(s): Prof. F. Cacialli (f.cacialli@ucl.ac.uk)

Optical inspection of samples

Room: F19

Equipment Used: Optical Microscope, computer

<u>Nature of hazards + frequency of encounter:</u> Electrical Hazard from electronics components.

<u>Precautions:</u> Ask for a brief introduction from a group member. Handle samples with nitrile gloves. Remember to turn off the lamp at the end of experiment.

<u>Emergency action:</u> Switch off electrical supply (if safe to do so) and seek help from others. If first aid is needed contact first aider in the department

Contact(s): Prof F. Cacialli (34467)

Supporting documents: Procedural risk assessment for room F19

Use of centrifuge

<u>Room:</u> F19

Equipment Used: Centrifuge 5414 C (Eppendorf)

<u>Nature of hazards + frequency of encounter:</u> Fast moving parts can cause dangers when equipment is used improperly.

<u>Precautions:</u> Operate centrifuge in accordance with the operating instructions given in the manual. Make sure the centrifuge is installed on a good, stable base. Before using,

check the rotor for firm placement. During operation, no persons, dangerous substances or objects may be within a safety margin of 0.3 m. Use only samples listed in the appendix of the manual. Don't use the centrifuge with inflammable or explosive materials. Balance loads around the circumference of the rotating drum.

<u>Emergency action</u>: In case of fault or emergency, switch off all electrical supplies. Never touch the rotor before it has stopped running.

<u>Contact(s)</u>: Prof. F. Cacialli (f.cacialli@ucl.ac.uk)

<u>Supporting documents:</u> Centrifuge manual in bookshelf in F6

Use of Benchtop Muffle Furnaces

Room: F19

<u>Equipment Used:</u> Benchtop Muffle Furnaces (FB1310M-33-UK, ThermoFischer Scientific).

<u>Nature of hazards + frequency of encounter:</u> Risk of burns from the internal heating table.

<u>Precautions:</u> Do not touch the heating table when the heat is on and the front lid is open. Close the front lid at every experiment. Report with a 'experiment in progress' form. Do not leave next to the furnace thermal sensitive materials. Wear oven protective equipment (coat, gloves, goggles) when operating the equipment.

Emergency action: Switch off the furnace. Seek First Aid if injury occurs.

<u>Contact(s):</u> Prof. F. Cacialli (f.cacialli@ucl.ac.uk)

Use of Blade Coater

<u>Room:</u> F19

Equipment Used: Blade coater (TQC AB3400), Substrates (usually glass or mica), Solutions

Nature of hazards + level of risk: Be cautious when mounting glass substrates

<u>Precautions:</u> Perform deposition only under the fume hood. Avoid using it in too high or too low temperature environments. Avoid humidity. Always make sure the instrument is connected to an earthed electric socket. Always make sure that the power of the

instrument is turned off while adjusting any electric component. Drop "enough" solution, again wearing gloves and any other appropriate PPE. Never put your head into the fume hood.

<u>Emergency action:</u> Switch off electrical supply (if safe to do so). Seek First Aid if injury occurs.

<u>Contact(s):</u> Prof. F. Cacialli (f.cacialli@ucl.ac.uk)

Preparation of Polymer & Organic Solutions

<u>Room:</u> F6, 7, 8

Equipment Used: Dry Polymers, Pipettes, Organic Solvents, Water, Balance

<u>Nature of hazards + frequency of encounter:</u> Spillage of solvent – if handled carefully is rare. Breakage of glass pipette. Dry Polymer are toxic, declared in the COSHH.

<u>Precautions:</u> Wear goggles, gloves and lab coats. Use small quantities of solvent. Know the correct procedure for dealing with solvents and how to deal with waste solvents. Deal with glass breakages by placing in the correct waste bin.

<u>Emergency action:</u> Seek First Aid if injury occurs. For spillages read and follow the appropriate COSSH and materials data sheet guidelines.

Contacts:Prof Neal Skipper (n.skipper@ucl.ac.uk)Prof Chris Howard (c.howard@ucl.ac.uk)Prof Franco Cacialli (f.cacialli@ucl.ac.uk)Prof Pavlo Zubko (p.zubko@ucl.ac.uk)

<u>Supporting documents:</u> "Use of Solvents" kept in Safety folder in F6. "Disposal of waste solvents" kept in Safety folder in F6.

Use of Nitrogen Lines (Asphyxiation risk)

<u>Room:</u> F6, F8, F17

<u>Equipment Used:</u> Nitrogen lines used for purge, glovebox supply, Cryogenic liquids: diffusion pumps, cooled experimental rigs

Nature of hazards + frequency of encounter:

Asphyxiation: caused by gradual or sudden replacement of air with nitrogen or helium. Low oxygen levels are not readily discernible. Considered "low" if oxygen content drops to 19% or less (21% normal). Below 16%, uptake of oxygen in the body is impaired and initial symptoms are irrational and disorderly behaviour (UMIST Safety Manual). Concentration of oxygen will vary with position in the room. Effects of oxygen depletion are rapid and potentially fatal - fainting occurs (<11%): death is within a few breaths (<8%).

The degree of hazard depends on the amount of gas released and on the volume of the room, local ventilation and airflow within the room. Ventilation cannot be relied upon, out of hours

Room ventilation, where it is present, can normally be expected to supply make-up air at the rate of about 0.1 to 0.2 cubic metres per second.

Estimate: a broken nitrogen gas line at a nominal pressure of 50psi could release gas at a rate of about 50-100 litres a minute (0.1 cubic metres per minute, or rather less than the makeup air). There would be considerable noise associated with this flow rate, which is likely to attract attention. The risk of asphyxiation is considered low because there is enough time to get out and seek help. There is a small possibility of lower oxygen content in air during a power cut, or when ventilation is no longer operational (many valves are manual).

Precautions

Nitrogen isolation valves should be indicated on the risk assessment for each room.

Of particular concern is the scenario where someone falls unconscious in such a room. Supervisors should be alerted if a potential room user has a known medical condition where this is more likely, for instance, diabetes or epilepsy, and the case should be discussed with Occupational Health.

<u>Precautions:</u> Rooms considered to be at particular risk should have a fixed or portable alarm system fitted and maintained. Rooms that do not have a fixed sensor should always be connected to an external oxygen monitor.

<u>Emergency action:</u> Vacate the room quickly if lack of oxygen is suspected. A portable oxygen meter is available in Rooms F6 and F17. Contact the lab emergency number

<u>Contacts</u>: Prof Neal Skipper (<u>n.skipper@ucl.ac.uk</u>) Dr. Chris Howard (<u>c.howard@ucl.ac.uk</u>) Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>) Dr Pavlo Zubko (<u>p.zubko@ucl.ac.uk</u>)

Photoluminescence Rig

<u>Room:</u> F17

Equipment Used: Andor-Newton spectrometer, Ocean Optics spectrometer, EPL-375, -405 -450, -520, -633, -670 and -780 laser diode, Minilite Nd-YAG Thorlab CW Diode Laser 405 nm and 532 nm, Spectra-Physics Argon-ion laser, Kimmon IK3301R-G HeCd, NEC GLC 5002 He-Ne, High-power LEDs, optical components, computer

Nature of hazards + frequency of encounter:

Medium risk to eyes and low risk for skin in the case of direct exposure to the class 3B laser. High risk to eyes and skin in case of exposure to direct or scattered light from a class 4 laser. Risk of electrocution from the 1000V power supply.

Precautions:

Complete training on Andor-Newton spectrometer before operation, ask Prof F. Cacialli for this. Complete the "laser safety training course" provided by UCL safety services. Before operating the laser follow this procedure:

- All jewellery must be removed, protective goggles must be worn.
- Turn on laser interlocks (F17).
- Set mirrors and optical components so that the laser beam will be on a horizontal plane.
- Clamp all optical components.
- Partially enclose the laser beam path using the aluminium shields provided in room F17.
- Advise all group members of the running experiment, in particular if the laser beam is not completely enclosed.

When the laser is on:

- Locate and terminate all stray laser beams (beware that mirrors can transmit part of the laser beam).
- Don't bend down below beam height.

Samples:

- Wear nitrile gloves while handling solid samples (films) or water solutions.
- Wear latex gloves while handling solutions in organic solvents.
- All solutions should be used in sealed cuvettes, in case of spillage of the solution, wipe it off immediately with a paper towel.
- Avoid reflection/refraction (from the sample) of the excitation source outside the horizontal plane of the laser beam
- Dispose of solutions or solid samples in F6

<u>Emergency action:</u> In case of fire use fire alarm and evacuate the room. If first aid is needed contact first aider in the department.

<u>Contacts</u>: Prof Franco Cacialli (<u>f.cacialli@ucl.ac.uk</u>)

Supporting documents: Laser risk assessments, Local rules for lab F17

Use of Vacuum Ovens

Room: F7 and F8

<u>Equipment Used:</u> Vacuum pump and pressure gauge. Sample chamber including heating table. Thermocouple and temperature display.

<u>Nature of hazards + frequency of encounter:</u> Danger of implosion of vacuum chamber. Risk of burns from the heating table.

<u>Precautions:</u> Do not touch the heating table when it is on. Fill in an 'experiment in progress' form (available on F8 door) and leave next to the rig when the rig is left unattended. Wear oven protective equipment (coat, gloves, goggles) when operating the equipment.

Emergency action:

Contacts:Prof Neal Skipper (n.skipper@ucl.ac.uk)Prof Chris Howard (c.howard@ucl.ac.uk)Prof Franco Cacialli (f.cacialli@ucl.ac.uk)Prof Pavlo Zubko (p.zubko@ucl.ac.uk)Supporting documents:Local rules for lab F8 and F7

Use of Off-Axis Sputtering Equipment

<u>Room: F7</u>

<u>Equipment Used:</u> Vacuum pumps and pressure gauges. Sample chamber with optical viewports and removable heater. Thermocouple. High voltage radiofrequency generator. Bottled, compressed gases (N₂, O₂, Ar). DC power supply. Cooling water pump. Gas flow controllers. Magnetron sputter sources. Plasma equipment operated using a PC.

<u>Nature of hazards + frequency of encounter:</u> Danger of implosion of optical viewports (rare). Risk of burns from the heater (frequent). Risk of eye damage from UV radiation from plasma (frequent). Risk of electrocution from the RF generator and DC power supply (rare). Risk of inhalation of toxic vacuum exhaust fumes (frequent). Strong magnetic field near the sputter sources may affect pacemaker operation or wipe magnetic memory devices (frequent).

<u>Precautions:</u> Handle the heating block with oven gloves (located drawer under furnace in F10e) and only when cold. Wear protective goggles at all time in the room. Do not look into the viewing ports when the plasma is on. Do not touch or disconnect any electrical cables without first disconnecting the equipment from the mains. Never remove the protective casing of the RF generator. Do not remove sputter source unsupervised. If wearing a pacemaker, consult lab manager before operating equipment. Ensure the fume extractor is positioned over the outlet of the scroll pump when sputter source is in operation. Fill in an 'experiment in progress' form (available on F8 door) and leave next to the system when in use.

Emergency action: In case of identified or suspected electrical fault, disconnect equipment from mains and contact lab manager.

Contacts: Prof Pavlo Zubko (p.zubko@ucl.ac.uk)

Supporting documents : "Off-axis magnetron sputtering" risk assessment (document RA02316/1 on RiskNet)

Appendix 1: Examples of Solvents and Gases Used

Maximum Exposure Limits (MEL), Occupational Exposure Standards (OES) and Short Term Exposure Limits (STEL) are taken from Royal Society of Chemistry data books and the current edition of the HSE's "EH40/2000 Occupational Exposure Limits 2000".

Solvent	OES/MEL	STEL	Risk	Comments †
Acetone (dimethyl ketone, 2-propanone)	1780 mg m ⁻³	3620 mg m ⁻³	Highly flammable	
Isopropanol (IPA, propan-2-ol)	980 mg m ⁻³	1250 mg m ⁻³	Highly flammable	
Ammonia	25ppm	35ppm (IDLH 500ppm)	Toxic, flammable	Burns to skin, eyes, respiratory tract
Methanol	260 mg m ⁻³	333 mg m ⁻³	Flammable, toxic	affects eyesight, cumulative
Xylene (mixed)	435 mg m ⁻³	662 mg m ⁻³	Flammable, harmful, irritant	
Dichloromethane (DCM)	350 mg m ⁻³	1060 mg m ⁻³	Poss. carcinogen	
Dimethylformamide (DMF)	30 mg m ⁻³	61 mg m ⁻³	Harmful, may be harmful to unborn child	
n-butyl acetate	710 mg m ⁻³	966 mg m ⁻³	Flammable, irritant	
Chloroform	9.8 mg m ⁻³	-	Harmful, poss. carcinogen	
Toluene	188 mg m ⁻³	574 mg m ⁻³	Highly flammable, harmful	
1,1,2-trichloroethane	45 mg m ⁻³	90 mg m ⁻³	poss. carcinogenic, mutagenic, toxic, irritant	attacks liver readily absorbed through skin
Nitrobenzene	5.1 mg m ⁻³	10 mg m ⁻³	very toxic	hits central nervous system
Tetrahydrofuran (THF)	300 mg m ⁻³	600 mg m ⁻³	irritant, flammable	drying THF could be hazardous
Ethanol	1920 mg m ⁻³	-	flammable	toxicity enhanced by denaturing

† for COSHH forms, information should not be taken from this table but from the current or most recent edition of the publications named above, or from suppliers.

Appendix 2: Laboratory Rooms in CMMP

This appendix indicates the approximate volume of certain rooms within CMMP and is useful for determining the likelihood of vapour concentrations exceeding threshold limits.

Room No.	Use	Floor area/m²	Height m	Volume m ³	Comments
F7	Glove boxes Furnaces Gas bottles to F6	15	4	60 8.5 air changes per hour	N ₂ bottles, Ar bottles. 4 Furnaces. O ₂ monitor present.
F8	Simple chemical preparation, use of fume hood.	20	3	60 6.7 air changes per hour	Ammonia condensation Sample sealing Oxygen plasma + gas O ₂ monitor present.
F10*	X-ray diffraction, magnetometry, cryogenics, IGA including hydrogen	84	2.4 or 5	365 9.5 air changes per hour	Cryogenics and IGA, including Hydrogen, XRD.
F10c*	Sample preparation, glove boxes	20	2.4	48 9.5 air changes per hour	Gloveboxes, ammonia condensation, fumehood. N ₂ bottles, Ar bottles. O ₂ monitor present.

In normal operation, there is extraction in Rooms F7, F8 and F10.

*Currently undergoing refurbishment – September-November 2008.

Appendix 3: Examples of Calculations

Query: what is the quantity of solvent for which there is no possibility of reaching the Maximum Exposure Limit (MEL) in a given room?

<u>Scenario</u>

- A given quantity of spilt liquid or gas from a bottle completely vaporises
- There is no ventilation
- The vapour is uniformly mixed throughout the air in a room

From Appendix 1 and data sheets (8 hour values):

Acetone - 1780 mg m⁻³ - in **Room F8** - volume 60 m³ : Weight of liquid that would give this MEL = 1780(mg m⁻³) x $60(m^3) = 107g$ Density of acetone = 0.79 g cm⁻³ This corresponds to a volume of 174 cm³, or about a washbottle full.

Chloroform - 9.8 mg m⁻³ - in **Room F8** - volume 60 m³: Weight of liquid that would give this MEL = $9.8(mg m^{-3}) \times 60(m^3) = 0.59g$ Density of chloroform = 1.48 g cm^{-3} This corresponds to a volume of 0.5 cm^3 , or a small squirt from a pipette.

Isopropyl alcohol - 980 mg m⁻³ - in **Room F8** - volume $60m^3$: Weight of liquid that would give this MEL = $980(mg m^{-3}) \times 60(m^3) = 59g$ Density of IPA = 0.79 g cm⁻³ This corresponds to a volume of 97 cm³, or about half a washbottle.

Ammonia – from gas bottle. **Room F8** – volume $60m^3$. Volume of gas that would give MEL = $60 \times 25 \times 10^{-6} = 0.0015m^3 = 1,500cm^3$. This corresponds to 5 bar ammonia in a standard 300cc buffer bottle.

Hydrogen – from gas bottle. **Room F10** – volume $365m^3$. Volume of gas that would give LEL = $0.04 \times 365 = 14m^3$. The volume in a K-type cylinder is $7.2m^3$, and B-type is $1.2m^3$.

Conclusions

Small quantities of certain solvents outside fume-cupboards need not give cause for concern. These might be encountered in silver dag, aerosols etc. From the quantities associated with the STEL above, even a full washbottle of acetone or IPA, is unlikely to enter the air rapidly, so occasional use might be considered acceptable. Filling such a washbottle would probably <u>not</u> be acceptable without extraction.

For ammonia gas (used in condensation) the maximum limit to be condensed should be 5 bar in a 300cc buffer. The main supply bottle must be isolated by at least two valves. The acetone/dry ice bath must only be used in a fume-hood.

For hydrogen, the K-type cylinder in F10 will not reach LEL, but MSDS precautions must be followed at all times. Cylinder must only be changed by BOC, and must be leaked tested on installation. The volume of hydrogen used must be kept to a minimum, and where at all possible kept to below 100L at STP.

In general, the use of solvents outside an extracted workbench should <u>not</u> be encouraged because of the temptation to treat potentially more dangerous materials in the same way. Chloroform, particularly, presents a greater risk than other solvents and is highly volatile. Some solvents have suspected carcinogenic properties. Authorities differ about the extent of carcinogenic risk. This may depend on how the material is encountered. It is safest to assume the worst case in the literature[†] and note that many materials are reviewed as evidence becomes available.

[†] This is what the HSE approach would be, whatever they list in their publications.

Appendix 4: Waste Disposal

Waste disposal is carried out in collaboration with the Departmental Safety Advisor, currently Mr John Fordham, available on 33433. Contacts to remove waste are on the waste management site and the relevant documents are also in this file: <u>http://www.ucl.ac.uk/efd/cleaningandwaste/waste/</u>

- 1) If you are not sure if an item cannot easily be moved or dealt with, contact Derek Attree (dja@hep.ucl.ac.uk) for advice or waste management http://www.ucl.ac.uk/efd/cleaningandwaste/ on 37001 or dhws@ucl.ac.uk.
- 2) Contact John Murphy to arrange disposal (usually taken to Physics Yard area).

John Murphy Disposal of Hazardous Waste Manager Tel: 020 7679 2585 (UCL internal 32585) Fax: 020 7679 7008 Email: john.murphy@ucl.ac.uk

- 3) Write down the quantity and nature of the waste, including hazards.
- 4) Photocopy completed form for your records. Photocopy further copies to attach to each container (I like to reduce to A5 or less to fit on the bottle).
- 5) Highlight the contents of each bottle on the photocopied form (*) and attach firmly to each container. I find short lengths of parcel tape preferable to sellotape, which has a habit of falling off in the adverse environment of the waste store.
 - (*) a pink highlight pen is preferred to yellow, being visible in yellow room lights
- 6) Call waste management to arrange access to the Waste Chemical Store to return waste and collect new waste containers.
- 7) Paperwork: Copy to NS or FC for records. Further copies attached to container.

Solid wastes - paper, plastic, contaminated with small quantities of polymers

Do not place in ordinary bin. Place in designated yellow bin. Designated yellow bins should not be emptied by cleaning staff. Glass (sharps waste) and no solvents should be placed in the chemical waste bins.

Solvent wastes, contaminated with small quantities of polymers

Solvent waste should be stored separately as chlorinated and non-chlorinated solvents, preferably in containers similar to those in which they originally arrived. Solvent waste

should be stored in ventilated fume cupboards, or, sealed, either in metal solvent cupboards or the Departmental Waste Store.

Pressure build-up in waste bottles

Bottles have been known to explode. The problem originates not from any chemical reaction, but simply from filling waste solvent bottles too full. If there is no air space, then the liquid cannot expand easily if the temperature increases. This causes a large increase in pressure - sufficient to rupture the vessel.

Procedure before (as stated in Health and Safety Handbook):

- do not overfill bottles fill to "shoulder" or lower
- Transfer part to another bottle if waste bottle overflows.
- notify Waste Management once a bottle is 90%.
- Place appropriate label on empty bottles to indicate contents or write on with pen.

Appendix 5: Replace the oil in a rotary pump

Replacement of the oil is part of the rotary pump maintenance. This operation must be carried out every 3000 hours (~ 4 months) of operation. The replacement of the oil is low risk procedure (risk: oil spill on the floor; hazard: slip) during the procedure.

- 1. Operate the pump for approximately ten minutes to warm the oil, then switch off the pump (this lowers the viscosity of the oil and enables it to be drained from the pump more easily).
- 2. Isolate the pump from the electrical supply and disconnect it from the vacuum system.
- 3. Remove one of the oil filler-plug.
- 4. Place a suitable block under the pump-motor to tilt the pump and place a suitable container under the drain-plug. Remove the drain-plug and allow the oil to drain into the container.
- 5. If the oil drained from the pump is contaminated, pour clean oil into the filler-hole and allow it to drain out of the pump. Repeat this step until the oil reservoir in the pump has been thoroughly cleaned.
- 6. Refit the drain-plug, remove the block and reconnect the pump to the vacuum system.
- 7. Fill a suitable container with clean oil and pour the oil into the filler hole until the oillevel reaches the MAX level mark on the bezel of the sight-glass.
- 8. Allow a few minutes for the oil to drain into the pump. If necessary, add more oil. Refit the filler-plug.

For the disposal, please refer to the appendix 4 "Waste disposal".