UCD School of Chemistry & Chemical Biology

Scoil na Ceimice agus na Ceimbhitheolaíochta UCD

Laboratory Operations Manual

and

Standard Operating Procedures

Version 1: 17th October 2008

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Introduction and Welcome from Head of School

Welcome to this first edition of the SCCB/CSCB Laboratory Operations Manual. The production of this document is timed to coincide with the completion of the A1 Phase of the Science District building works (October 2008). On occupation of the A1 laboratories, for the first time, the majority of preparative chemistry researchers in the School will be working in world-class facilities. This opportunity imposes a duty on us to ensure that our safety and laboratory procedures are also up to the same high standard.

In combination with the School Safety Manual, this document sets out the laboratory practices that require our attention. Some of these procedures have been previously documented and some are already in place. But this is the first time that we have drawn together all of the relevant material. There are also significant new additions such as the operation of the new style fume hoods; the more widespread availability of glove boxes and much more extensive handling procedures for gases.

A number of other initiatives in respect of laboratory operations are also planned for the coming year, mostly in connection with our management of chemicals. These also tie in with the opportunity afforded by the move to the new labs and include: the new (POP) ordering system; a computerized chemical inventory system for the School; and, most significantly, a move towards much more structured chemical risk assessment.

In addition to the above plans, this document has been prepared in advance of occupation of the new Laboratories. It is to be expected therefore that additional material and adjustments will be necessary as we become more familiar with the operation of A1. An SOP committee has been set up to progress this. Therefore we have built in provision for the amendment of this document. Note that some of these amendments may need to be made before the next printed version - so the Web version of this document always takes precedence over the printed version.

Finally, as well as the expected sections on lab practice and safety etc., we have included a section on how research space is assigned. These new facilities have cost a substantial amount of money and new financial arrangements within UCD mean that the School is now charged for its space occupancy. Therefore we have to have a transparent system for the assignment and efficient use of research space.

Who should read this Manual

This document has been prepared for all preparative chemistry research workers in the School and CSCB Buildings including graduate students, postdoctoral researchers, fourth year undergraduate students, Erasmus and other visitors. Researchers in other chemistry disciplines who have the need to do preparative chemistry will also find the material useful. It is expected that an expanded version covering non-preparative chemistry will be necessary when the next phase of the Building Works (A2 Phase) is complete.

Structure of the Manual

The full School Laboratory Manual is in 3 distinct parts:

1. Operations Manual - the first part of this document
2. School Safety Manual - this is unaltered from that already circulated for this academic year. It will be incorporated into subsequent versions of this document.
3. Compendium of Standard Operating Procedures (SOPs) - for convenience the actual SOPs are printed at the end of this document along with some Quick Guides.

It should also be noted that some of the contents herein have already been implemented in the CSCB labs and some have to be implemented in a different manner from the A1 labs (e.g. operation of the fume hoods). Full integration of these will also be undertaken for subsequent versions

Lastly we note that we have not yet included full coverage of many of the various Analytical Services that are already well-covered in the School (e.g. NMR, MS, X-ray, elemental analysis) but again, it is the intention to incorporate these in next version.

Implementation of these Procedures

These protocols have been adopted as policy in the School of Chemistry and Chemical Biology and it is expected that they will be implemented by all research groups in the School. The primary responsibility for implementation rests with Principal Investigators and it is their responsibility to ensure that our laboratories are characterised by culture of safety and best practice. This is a vital part of ensuring the reputation of our School and our Graduates.

The Head of the School is ultimately responsible for implementation of policy and may delegate some of this responsibility as he/she sees fit. The Chief Technical Officer (CTO) will be responsible for day-to-day oversight of laboratory practice. Periodic random inspections will be carried out by the School Safety Officer. Other breaches may be reported to the Chief Technical Officer.

Infringements and how they will be dealt with

It is expected that infringements will be rare occurrences; they will be dealt with as follows:

1. When the CTO identifies an infringement, he/she will bring it to the notice of the person responsible immediately and issue a reminder of the importance of compliance with agreed protocols. The direct supervisor of the person will be informed that an infringement has taken place and the identity of the person responsible for the infringement will be made known to them. It is expected that this will precipitate a discussion in the relevant group about the importance of the protocols.

2. In the event of a second infringement, the CTO will act as above, but a written warning will be issued to the person responsible for the infringement and copied to the Head of School.

3. The Head of School will deal with further infringements and will impose appropriate sanctions up to and including suspension from the laboratory.

4. Where an event is of sufficient seriousness the written report and involvement of HOS will be immediate

Minor infringements would include not wearing safety glasses, lab coats, eating in lab, incorrect/unsafe operation of vacuum lines/glove box/HPLC etc., persistent annoyance to fellow workers.

Major Breaches may be safety related such as ignoring a fire alarm or delaying evacuation, but would also include breaches of scientific ethics and inappropriate behaviour towards fellow workers as outlined in the UCD Dignity and Respect at Work Policy.

Documentation Required before Research commences.

Before starting research work in the School, researchers are required to read this Manual and the School Safety Manual. Before gaining entry to the laboratories to perform research, each researcher is required to sign a "Read & Understood" form relating to both documents (Appendix 2) and return same to the School Office.

Safety

The School Safety Manual is the primary source of safety information for the School and will in future be circulated along with this Manual. However, for this year, since all researchers have already been issued with the Safety Manual, this is not necessary.

If you are one of those workers moving to the new A1 Laboratories, please familiarise yourself with any changes in the primary safety considerations that may result from the move. In particular, note the new fire escape routes and any changes of fire marshals.

In addition there are some other new safety considerations introduced in the move to A1. These are related to the safe operation of fume hoods, glove boxes and Grubbs solvent stills. Also there is a new system of gas detection and alarms, especially for hydrogen. We have included information and procedures on these issues in Appendix 1. Also in Appendix 1, we have noted the standard operating procedures (SOPs) for certain other safety issues that you are likely to encounter, including use of cyanides (nitriles), thiols (mercaptans) and azides.

During the coming year, we will be producing a second (expanded) edition of this Manual. One of the additions is safety related and deserves particular attention, namely Risk Assessments, especially of chemicals, but also of procedures and apparatus. Now that most of the preparative chemists in the School will be working in world-class laboratories, it is the intention of the School to introduce comprehensive new Risk Assessment procedures. This will bring our safety culture fully into line with international practice and, no less important, fulfill our legal requirements.

If working outside of opening hours you must comply with the following:

- Experimental work may be performed only if there is at least one other research worker in the same laboratory or in an adjoining room that has a clear view of the Laboratory.
- Experimental work is restricted to operations with which the individual is familiar. New or hazardous operations should not be undertaken.
- Each research worker is responsible for ensuring that all apparatus is switched off; this implies a thorough check particularly on gas, electricity and water. Windows must be closed and all unnecessary lights switched off after use.

Finally we note that many of the other procedures detailed herein have a safety implication or impact indirectly on safety. Thus a clean, tidy, convenient and social working environment contributes to overall safety.

Comprehensive safety information is available on the UCD Safety Website, www.ucd.ie/safety/

Assignment of Research Space

1. Overview

The School is now charged for its space occupancy. Therefore the allocation of space must be conducted in a consistent, transparent and strategic manner. Space is a central resource and hence the allocation of space is designed to optimize productivity and effectiveness. To this end, there will be no sole ownership of space. Vacant research space may be used to meet temporary research needs.

In the School of Chemistry and Chemical Biology space is allocated with a certain degree of flexibility. Factors influencing space allocation include need, availability and suitability for intended use. Due regard will also be given to compatibility of chemistries being undertaken.

Space is classified as follows:
Primary Research Space - areas where researchers carry out principal tasks (e.g. fume cupboards, lab-benches, write-up areas, instrument rooms, offices)
Secondary Research Space - areas that support researchers e.g. common instrument areas, large communal instrumentation (e.g. NMR, MS, X-ray, workshops, stores, meeting rooms)
Teaching Space - all areas associated with teaching
General Space - e.g. School Office, seminar rooms, reception areas

2. Responsibilities and Scheduling

Space allocation will be the responsibility of the School Management Committee, with the assistance of the Chief Technical Officer. Space allocation will be reviewed twice yearly.

The main space determination takes place in early September, the School Management Committee considers the up-to-date data on Group Size (see sections 7/8) and what changes should be made to space allocations and to group locations. This sets the general space occupancy of labs for the academic year. A subsidiary determination takes place in early March, towards the end of 4th Yr Projects, to identify available gaps that could be used to accommodate additional researchers and to assist projected space requirements.

3. Allocation of Primary Preparative Chemistry Research Space

3.1 Workstations

The School of Chemistry and Chemical Biology operates a policy of one workstation per PhD or Postdoctoral researcher whenever possible (this may be exceeded in some cases, particularly in the early days of occupation of the A1 Laboratories due to building constraints). Workstations are clearly defined and, in most cases, consist of a fume cupboard space with accompanying lab-bench area including storage and a write-up space. Undergraduate researchers such as 4th year Project students and Erasmus students will be assigned up to a maximum of 0.5 Workstation, subject to availability. Other researchers including Visiting Professors, Special Fellows and Emeritus Staff will be treated on a caseby-case basis.

Workstations are assigned to research groups in accordance with the number of researchers in that group. Depending on demand, once a workstation becomes vacant, the workstation may be assigned to a researcher from a different group. The calculation of group size is described in Section 7.

Prior to submission of applications for research awards, the space implications of the award (both personnel and equipment) will be considered by the School Management Committee. Requests from PIs for more space will be directed to the School Management committee who will attempt to find the most appropriate solution.

Where it is appropriate and with the approval of the School Management Committee, specialised equipment that must, for safety reasons, be accommodated in a fume hood will be taken into consideration in space allocation to the Group concerned.

3.2 Common Laboratory Areas

Each Laboratory contains a number of common areas, instrument rooms, shared “dirty” areas, and equipment areas (laboratory areas not designated as workstations) which are all for communal use. In the case where laboratories are shared by a number of research groups, the communal space will be allocated, with a degree of flexibility, in consultation with the Management Committee and the relevant PIs. Research groups should communicate and work together to ensure efficient use of space (i.e. not double up on equipment etc).

Fume cupboards designated for hazardous experiments e.g. hydrogenation, photochemical apparatus (located on some floors in shared “dirty” areas) are for communal use and may be reserved in advance by signing the log book located at each fume cupboard. If such a fume cupboard is required for a significant period of time, please discuss with the Chief Technical Officer/Buildings Officer.

4. Allocation of Primary Non-Preparative Chemistry Research Space

This section will be considered on completion of the A2 Phase of the Science District Building Works

5. Implementation & Sanctions

All research Groups should only occupy space that has been allocated to them. Any misuse of this system should be reported to the Chief Technical Officer/Buildings Officer who will inform the Principal Investigator in question. If the problem is not resolved, the Chief Technical Officer/Buildings Officer will report to the Head of School who will then address the situation directly.

6. Vacating/Occupying Space

Accepted practice is that vacated space should be left in a clean and safe state for the next occupant. This will be enforced in a number of ways. For example, in the case of PhD candidates, the degree will not be awarded without confirmation that the relevant procedures have been carried out. For procedures and forms relating to the vacation of space - refer to Appendix 2. In the case of postdoctoral workers, as a last resort, it may be necessary to withhold final month's salary to secure compliance.

On occupying space, an initial check should be carried out to confirm that the space has been vacated following all guidelines set down in Vacation of Research Space SOP. If it is found that the condition of the space is unacceptable, the new occupier should not carry out any clean-up of the area; instead, they should report directly to the Chief Technical Officer.

7. **Procedure for the Determination of Chemistry Research Group Sizes**

The procedure is operated twice per year and is done by the School Office

Main Space Determination: Late August - Just before Term Starts

Subsidiary Space Determination: Early March - as the end of 4th Yr Projects approaches

Collection of Data This exercise enables the School to determine the precise numbers of PhDs, postdocs, project students and other researchers in the building along with their associated PI, start/finish dates, contact details, grant awarding body, etc. The numbers are then used for a variety of purposes in addition to space allocation: up-to-date FTE data, FTWA requirements and insurance records.

An Excel spreadsheet (SpaceXXX.xls) is circulated to all PIs in the School showing the results of the last exercise with any subsequent additions/deletions known to the School Office. PIs are asked to confirm the accuracy of its contents and to provide updates. In particular they are asked to confirm the names of incoming personnel; details of positions to be filled on grants recently obtained; and whether existing personnel are to be retained by extension contracts (again with grant details).

Estimation of Group Size Based on this collected data, group sizes in no. of workstations are calculated (1 Workstation = Fume Hood + Bench + Write-up):

PhDs: 1 Station, assumed to be full-time laboratory occupation of 4 years. Exceptions due to Special circumstances must be approved by the School Management Committee Postdocs: 1 Station and may not be contracted to leave Fixed Term Worker Act (FTWA) obligations with School (e.g. contracts of greater than 4 years duration).

4th Year Projects and visiting Erasmus-type students: 0.5 station, maximum of 6 months

Others e.g. Visiting Professors, Special Fellows, Emeritus staff treated on a case-by-case basis.

The final updated Excel Spreadsheet is then circulated along with the estimate of Group Size

8. Maximum Occupancies

For the Purposes of Allocation of Preparative Space the following Maximum Occupancies Apply CSCB : 24 Stations per floor A1 : 20 Stations per floor

Building Access and Security Information

The SCCB and CSCB Buildings (Science Centre South) are open from 08.00 to 19.00 on weekdays and are closed at weekends and on those days when the college is officially closed. Postgraduate students and Postdoctoral researchers may work outside the above hours with the agreement of their Supervisor subject to the conditions detailed in the Safety Section of this document. Outside the above hours, access to Science Centre south is available only to authorised personnel.

Access to Science Centre South outside the above hours on Monday to Friday is via either the SCCB electronic access door (located under the pedestrian tunnel on the ground floor) or the CSCB electronic access door (located beside the main revolving door). Authorised researchers may arrange a swipe access card for the relevant door by contacting either the School Office, SCCB, or the CSCB Reception. When using these secure entrances you must wait for the door to close behind you before leaving the area. When working out of hours (before 08.00 and after 19.00 on weekdays), you must sign the relevant Log Book located either beside the SCCB electronic access door or at the CSCB reception.

Access to the A1 Laboratories and also to the CSCB during weekends is via the School of Chemistry and Chemical Biology electronic access door, which is located on the ground floor under the pedestrian tunnel. If you enter/exit the building you must sign the School of Chemistry and Chemical Biology Log Book, which is located near the electronic access door.

All School and CSCB building users must lock all doors to unoccupied rooms and Laboratory areas at all times. All valuables should be kept out of sight, locked in lockers or drawers.

Any unauthorised person observed in the School or CSCB buildings should be reported immediately by calling the First Response Room ext 1200 or the UCD Emergency Services on ext 7999.

The CSCB external doors are fitted with an intruder alarm that will activate if the door is opened/tampered with when armed (CSCB Seminar Room and CSCB rear staircase fire doors are armed at all times). The intruder alarm is audible on the ground floor of the CSCB and can be distinguished from the Fire alarm in tone and also in that no red sensors will be activated during an intruder alarm.

Management of Chemicals

This is an important part of the running of a chemistry laboratory facility. However, this aspect of the Operations Manual is still being developed, except in the case of Waste Disposal. Therefore we provide an indication only of the changes planned over next year.

Ordering of Materials (and Apparatus)

A major transition is underway in UCD in the way that ordering of materials and apparatus is managed. This is the so-called POP (Purchase Order Processing) system. It has been implemented on a pilot basis in some other Schools in the College and now it is proposed to introduce it to Chemistry. This is a large undertaking - for example the whole of the Aldrich Catalog has to be loaded onto the system and therefore its implementation is expected to be complex and be subject to teething difficulties. Please bear with us as we try to carry out these requirements of the University administration.

Risk Assessment Procedures

It is now commonplace internationally that any operation using chemicals and/or specialized apparatus is governed by a strict system of Risk Assessment. The international practice is that all experiments are subject to this requirement. This is something that has been lacking at UCD and will be one of the measures of our attainment of a world-class laboratory operations culture. It will also be introduced in the near future - indeed it is recently enacted legal requirement. A start will be made straightaway by firming up on the use of Material Safety Data Sheets (MSDS). In the meantime, information on Chemical Risk Assessment may be obtained from the UCD Safety Office Web Site: http://www.ucd.ie/safety/riskassess.html

Chemical Stocks Inventory

At the present time, the School has no unified system of Chemical Stock Inventory and Control. This is unsatisfactory from a safety point of view. Also it often happens that excess chemicals are ordered because users are unaware that other Research Groups may have the relevant chemical. Relatively simple computer programs are available for this purpose and one of these will be implemented in the coming year.

Management of Waste Chemical Disposal

In contrast to the above, an efficient and cost effective system is in place for this function.
Details may be obtained from Chemical Stores personnel

Operation of Fume Hoods

Contents

1. Background

Fume cupboards are designed to protect against toxic and dangerous material by dragging airflow away from the user and thus preventing harmful vapours being inhaled or ingested. The aim of this section is to provide a description of the fume cupboard systems in the School of Chemistry and Chemical Biology and the CSCB and to describe how to use them safely and efficiently. Operational details are repeated in the relevant SOP in Appendix 1 along with a Quick Guide.

The design of the fume hoods and the air handling systems in the CSCB differ from those in the A1 laboratories of the School of Chemistry and Chemical Biology. Both are of the built-in ducted type that vent to the outside via outlets on the roof. However those in the CSCB have individual fans whereas those in A1 are arranged in a manifold style with one constant speed extraction fan per laboratory (i.e. all fume cupboards in a lab are served by the same fan). We give here greater details about the A1 style because it is the first time such a system has been implemented in an Irish University. Further details on the CSCB hoods can be obtained in the relevant SOP

2. General Information

2.1 Extraction

This manifold arrangement is energy efficient. However, any malfunction of the fan will affect all fume cupboards in any one laboratory. Each fan has a back-up motor that will automatically take over in the unlikely event of a malfunction. The fume cupboards are equipped with a VAV (Variable Air Volume) controller which maintains a constant velocity in each fume cupboard regardless of the sash position. This controller operates by means of an individual damper for each fume cupboard which adjusts in accordance to the sash position. Extract volume is at a minimum (reduced to 80 % of maximum extract volume) when the sash is in the lowered position.

The extraction fan for each laboratory operates continuously, and so, there is no local on/off control at each fume cupboard. During night-time/weekends/holidays, the fume cupboard system is set to adjust accordingly thus maintaining energy efficiency. Nevertheless, out of hours use is always possible as the system is set to maintain airflow at these times, albeit in a reduced number of fume cupboards.

2.2 Air Handling Units (AHU)

The air extracted by the fume cupboards is replaced by air provided by an Air Handling Unit (AHU) in such a way that a negative pressure is maintained throughout the laboratory. There is a separate AHU for each Laboratory and the amount of air provided is determined by the amount of air extracted by the fume cupboards. Fume cupboards not only extract air but also heat and so the AHU provides heated air in order to maintain a constant temperature. Air and heat extraction from the laboratory is at a minimum when all sashes are in the lowered position. Thus, the laboratory is at maximum energy efficiency when all sashes are in the lowered position. Chemical containment is at maximum efficiency when the sashes are lowered.

2.3 Fume cupboard Alarms

Each fume cupboard is equipped with an alarm that will sound if there is insufficient airflow for a safe working environment (this will sound if the height of the sash exceeds the maximum height for a safe working environment). If this alarm sounds, the user must investigate the cause. If the sash is above the maximum height (indicated by the stop mechanism) for a safe working environment, the user must lower the sash. If the cause is unknown, the user should report to the Chief Technical Officer, Paul Kane, ext 2290 or Dermot Keenan, ext 2353.

2.4 Automatic Sash Closure

The fume cupboards in the School of Chemistry and Chemical Biology are equipped with an "Automatic Sash Closing" device. This device ensures that no fume cupboard sash is inadvertently left open for any significant length of time when the fume cupboard is not in active use. This device is set so that if there is no movement detected at a fume cupboard during a period of 5 mins the sash will automatically close. The advantage to this is that chemical containment will be maintained as much as possible and also the fume cupboard system will run at maximum energy efficiency. A hazard related to this automatic device is that any apparatus left protruding from the fume cupboard will obstruct the sash when it closes. Hence, no apparatus should ever be left obstructing the sash area. For special cases where a piece of equipment (e.g. an immersion cooler) obstructs the sash area the Chief Technical Officer must be contacted and the automatic closure will be disabled temporarily.

Operational Instructions for Automatic Sash Closure

a) The auto sash may be operated manually.

b) The auto sash can be electrically operated by use of the **up / stop / down** switch fitted to the LHS of the fume cupboard.

c) The **up** switch: Sash opens and stops at the maximum working height (500mm above worktop), unless the **stop** switch is pressed.

d) The **down** switch: Sash closes to the low level stop position (just above the front airfoil).

e) Automatic closure: Sash closes automatically to the low level stop position after 5 mins of no activity. The front sash movement stops immediately as soon as a person is detected via the PIR (passive infrared sensor) within the working area of the sash.

f) Detecting obstruction: In addition to the PIR, an additional safety stop is fitted. This low level light barrier will immediately stop the sash moving up or down if the light beam is broken by user operation or by an object protruding from the fume cupboard at this height. Auto closure is deactivated and requires the sash to be manually moved by 30 mm or the **up / down** switch utilised to reinstate auto closure.

2.5 Servicing

The fume cupboards in the School of Chemistry and Chemical Biology will be serviced annually.

3. General Safety Guidelines for Fume cupboard Use

As stated throughout this document, fume cupboard sashes must be lowered as far as possible at all times for maximum efficiency in coping with the removal of vapours. Proper safe work practices for fume cupboard users include:

- All apparatus should be set up at least 15 cm behind the plane of the sash.
- Never put your head inside an operating fume hood to check an experiment.
- Always work with the sash in the lowest position possible.
- Do not clutter your hood with bottles or equipment as this can interfere with the smooth flow of air.
- Keep your fume cupboard clean. Only materials actively in use should be present. Be especially attentive to the presence of acids.
- Do not obstruct the glass with paper of writing - white boards will be provided at the ends of each bank of fume cupboards.
- No apparatus should protrude from the fume cupboard as this will obstruct the automatic closing sash (and also hinder sash closure in the event of an emergency).
- Do not use fume cupboards as storage cabinets for chemicals as these represent potential additional hazards in the event of an accident.
- If airflow fails for any reason, work must stop in all fume cupboards (in any one laboratory) and the sashes must be closed until airflow is restored.
- Clean-up spills immediately.
- When using extremely hazardous chemicals, always prepare a plan of action in case of an emergency, such as a power failure.
- Waste bottles held in the fume cupboard should be kept closed. It is illegal to use fume hoods (especially manifolded ones) for the evaporation of volatile organic compounds.

Guidelines on Fume Cupboard Manifold Issues The UCD Safety Office has supplied the following guidelines on issues related to manifolded fume cupboards:

Where fume cupboards extracts are linked together in a single manifold extract system then some additional precautions need to be taken. Whilst manifolded systems offer an additional degree of protection to the user over single flue units due to the increased dilution in the extract flues and the presence of a backup extract fan, the indiscriminate mixing of exhausts from different fume cupboards could lead to the generation of an unstable / dangerous atmosphere in the common extract flue. To prevent this happening the following materials must not be used in a manifolded extract system: radioactive materials; fuming acids; any concentrated acid including hydrofluoric, perchloric, nitric, etc; any biological material of Class 2 or above. If in doubt do not use a manifolded fume cupboard until advice has been obtained.

4. Operational Notes

The following are useful notes in relation to the general operation of the fume cupboards in the A1 Laboratories of the School of Chemistry and Chemical Biology:

4.1 There are 2 types of nitrogen gas taps (house supply); one being classified as "normal" control and the other classified as "fine" control. The "fine" control taps allow very fine control of the amount of gas and are suitable for use with gas bubblers etc. Fume cupboards with a total of 3 nitrogen taps possess 1x"normal" and 2x"fine" control. Fume cupboards with 4 nitrogen taps possess 2x"normal" and 2x"fine" control.

4.2 All vacuum taps must be fitted with a suitable trap at all times to protect the house vacuum system from solvent ingress.

4.3 All fume cupboards are fitted with a cabinet underneath to house a vacuum pump for reduced pressure distillations and/or Schlenk line connection (if required). This cabinet is connected to the lab venting system, and so, airflow in this cabinet should be sufficient to alleviate any exhaust fumes or heat from the pump. Attached tubing to the pump exhaust to lead the exhaust fumes into the venting system.

4.4 The cabinet should accommodate the most common types of pump used in chemistry laboratories (e.g Edwards RV-5). If your pump does not fit, do not force it in. In such cases, contact the Chief Technical Officer. It is good practice to have oil mist filters on such pumps to reduce contamination of the cabinet.

4.5 Schlenk lines may be connected to the vacuum pump in the cabinet below the fume cupboard by means of the pass through conduit provided. Once the tubing has been passed through this (some lubrication may be required), the hatch at the back of the pump cabinet may be temporarily removed (with a screwdriver) to allow access to the tubing.

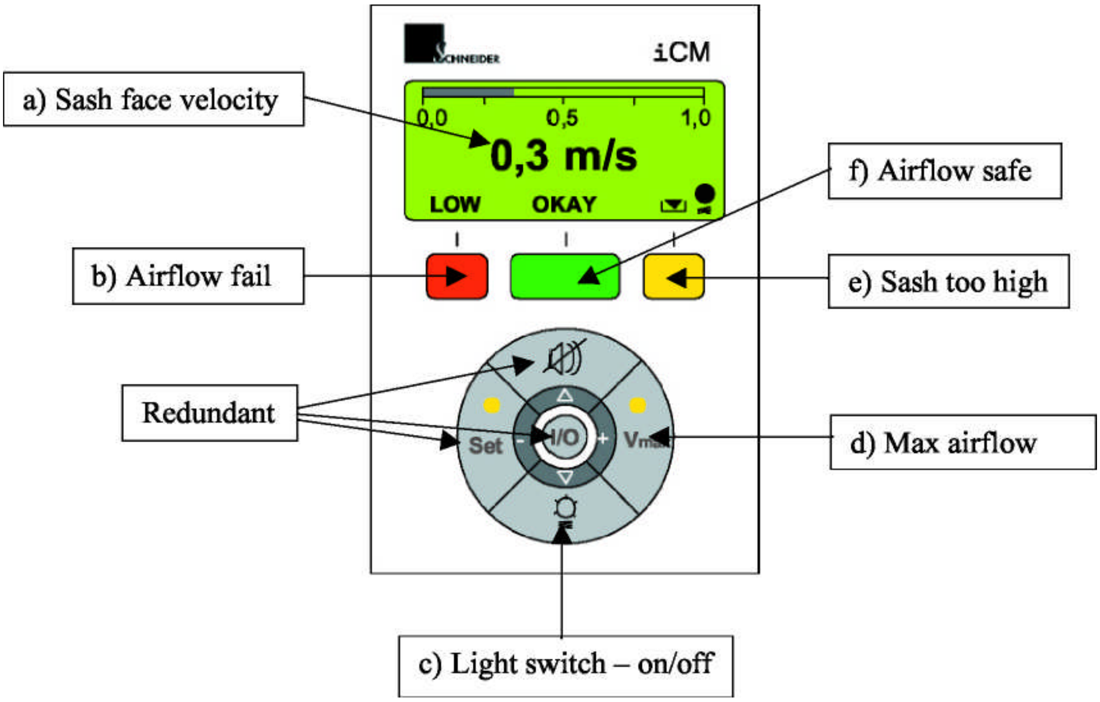
4.6 The airfoil (stainless steel lip at the front of each fume cupboard) is hinged to allow easy access for cleaning.

4.7 All special gas/"X" gas supplies are fitted with "fine" control gas taps.

4.8 There are two power isolation switches on the top left-hand-side of each fume cupboard which allow the user to isolate electrical supply to the power sockets on the fume cupboard and also in the under fume cupboard pump cabinet.

5.

Fume cupboard Control Panel



a)
b) Continuous digital read out of sash face velocity
c) Airflow fail (red indicator) with audible alarm
d) Light switch – on/off

Max airflow option: This option sets airflow to maximum while the sash is in the lowered position (an orange light flashes when this mode is enabled). This mode should only be used in an emergency (e.g. if there is a spill in the fume cupboard) and should be
e) manually disabled (by pressing the button once more) once it is no longer required.
f) Sash too high (flashing yellow indicator)

Airflow safe (green indicator)

6. Emergency Procedures

The activation of the Fire alarm, causes a set of actions to occur automatically: Fume Hood extraction is turned off, and ventilation is shut down following a 5 minute delay. The hydrogen gas supply is also shut-off. In the case of fire, close all sashes and leave lab. In case fan failure for any other reason, again close sashes and report to Chief Technical Officer.

7. Fume cupboard Audits

Fume cupboards in the School of Chemistry and Chemical Biology will be included in the regular

Clean-up/Safety Audits.

Gases and Gas Detection Systems

The method for handling gases is very substantially changed in the A1 Laboratories, to bring it in line with the CSCB labs and there are some additional aspects of gas handling in A1.

Nitrogen. Both buildings have house nitrogen, which now has some extra metering points to distinguish the usage in A1 from that in CSCB. It is intended to install extra metering points so that the nitrogen usage of individual floors can be ascertained. Information on normal nitrogen pressures can be obtained from Dr. Una McCarthy.

Hydrogen and Hydrogen Detection. Both buildings also have hydrogen supplies on each floor, housed in special cupboards that have a vent to the roof. The hydrogen lines go to a limited number of specified fume hoods in each laboratory. Because of the danger of build up of an explosive mixture of hydrogen and air, there are very extensive hydrogen detection systems. These are also connected to the fire alarm system. Further details of the operation of these are described in the relevant SOPs in Appendix 1.

X-gases. This refers to additional gas lines that have been installed. These are somewhat more extensive in the A1 Laboratories and include the possibility on each floor of interchangeably connecting up cylinders of, for example argon, helium, oxygen or air to one of a number of specified fume hoods in each laboratory. In addition there is a possibility to connect carbon monoxide in the CSCB labs, although this facility is suspended at the present time.

Oxygen Depletion Sensors. In the corridors where cylinders of suffocating gases are housed, oxygen depletion sensors have been installed. The operation of these sensors is described in the relevant SOP (Appendix 1).

General Laboratory Practice and Procedures

It is our intention to add very substantially to this section. However this is best achieved by involving all researchers after we have taken occupation of the A1 Laboratories. This will be one of the first tasks of the new SOP committee, which will have to consider issues such as outof-hours working, procedures for Stores runs, storage of chemicals, solvents, glassware, records, laboratory notebooks, etc. We have noted below some areas where an outline can be given.

Facilities in the Laboratry.

As well as hot and cold water, the A1 laboratories are equipped with house nitrogen, compressed air, vacuum and venting. The last of these is distinguishable by its much wider bore black tubing and is connected (by dampers) to the fume cupboard extraction system. It provides a slow steady draw of air to which the exhaust of a pump or rotary evaporator etc, can be attached. In addition a number of the fume cupboards have positions for X-gas - a gas-line to which a number of alternative gas cylinders can be connected. Please familiarise yourself with the operation of these various facilities.

Vacuum Pumps and Rotary Evaporators

Most fume hoods have a cabinet containing a rotary oil vacuum pump. This must be operated in a safe and clean manner (refer to the relevant SOP). Particular issues are (i) the use of cold traps between the pump and the system being pumped is mandatory and if this trap is of liquid nitrogen, special care must be taken to avoid the condensation of liquid oxygen - **an explosion risk;** (ii) the use of oil-mist filters is strongly encouraged (the School will provide funding to fit them); (iii) the pump exhaust must be vented to the hood or connected to the venting system; (iv) routine maintainance requires the oil to be changed and this must be treated as waste chemical disposal. Most benches have rotary evaporators served by membrane pumps, which must be vented using the venting points on the benches. There is house vacuum throughout the laboratories. This also requires a trap in front of the system being pumped so that the vacuum system is not contaminated.

Solvent Usage

Manipulation of solvents outside the fume hoods in the absence of venting is strongly discouraged, including use of acetone for washing up. Individual solvent waste bottles should be maintained in each hood. Solvents and chemicals must be transported using the safety containers provided. Solvents should only be stored in the solvent storage cabinet provided. Every effort must be made to limit the quantity of solvent stored in any laboratory.

Management of Resources

Please familiarise yourself with the various arrangements for the safe, efficient and cost effective use of the following centrally provided resources:

(i) House nitrogen (note that it is planned to have two types of nitrogen taps in the A1 Fume Hoods, one for high flow rates and one for low flow rates)

(ii) Grubbs Stills and Glove Boxes (SOPs in Appendix 1)

(iii) Liquid nitrogen and dry ice (containers provided)

(iv) Solvent, Glass, Chromatography stationary phase and normal waste disposal

Unattended Experiments

Special rules apply to experiments run overnight or otherwise unattended. Every hood is provided a water-safety cut-out switch and it is mandatory to connect this for unattended experiments; all joints have to be securely fastened and correct forms filled in and displayed near the experiment and outside the laboratory (see relevant SOP)

Laboratory Etiquette

1. Introduction

The laboratories in the School of Chemistry and Chemical Biology should be, and can be, areas of productive discovery, development, collaboration and general learning. To this end, all researchers should contribute to create, and maintain, a respectful and supportive environment where research can be carried out in a professional manner.

2. Laboratory Etiquette

All researchers must observe the following guidelines:

- **Respect others' projects/property/laboratory space as you would expect others to respect yours.**
  Never use equipment/apparatus belonging to another researcher without asking permission.
- **Maintain a professional environment, professional techniques, and professional attitude toward others at all times.**
  Provide collegial support for colleagues. Share knowledge and understanding. Keep distracting noise to a minimum.
- **Take responsibility for the facility and your colleagues.**
  The lab is our asset and we must all take the responsibility to maintain it, and the safety of our colleagues. If you observe inappropriate behavior, take responsibility and provide leadership in correcting it. Or, if appropriate, report irresponsible behavior to the Chief Technical Officer.

3. Laboratory Rules

These laboratory rules are aimed at both ensuring the safety of, and as a general courtesy to, all researchers in the School of Chemistry and Chemical Biology.

- Eating and drinking is forbidden in all laboratory areas; this includes write-up areas located within laboratories;
- Personal audio equipment (e.g. MP3/iPod) is forbidden in all laboratory areas. This is for the safety of all researchers. These devices may hinder your ability to react fast and efficiently if a co-worker has an accident and needs your help. It may also hinder your ability to react to various laboratory incidents (alarms etc).
- Mobile phones may not be used in the laboratory areas. They can be used in the write-up areas but please keep ring-tones at low volume.

- Radios are not permitted in laboratory/write-up areas.
- Lab coats must be cleaned regularly. All researchers must possess two laboratory coats each, which are to be cleaned alternately (Chemistry Stores service provided).
- Wearing of lab coats outside of the laboratory areas should be kept to an absolute minimum and should only be done so when absolutely necessary (e.g. when transporting equipment/chemicals etc). Staff members who do not routinely carry out laboratory work but wear lab coats to enter laboratories (e.g. Principal Investigators, Technical Officers) may wear their lab coats as long as they are cleaned regularly.
- It is forbidden to wear laboratory gloves outside of laboratory areas/on door handles/in write-up areas/on computer keyboards/on phone handsets.
- Running is not permitted in the laboratory areas.
- Space is limited. Have only what you need in your work space.
- Coats, bags and other possessions should be placed where they cannot interfere with productivity and safety.
- If entering a laboratory area, please ensure you are wearing appropriate clothing (e.g. no open-toed shoes/shorts etc).
- Only those who have a legitimate reason to be in the laboratory should be there. Guests should always be provided with the necessary PPE (e.g. safety glasses and lab coat) and should be accompanied at all times.

Cleaning and Housekeeping

1. Introduction

As well as leading to efficient work practices, elimination of hazards is aided by attention to detail, environmental hygiene, and tidiness of benches, desks, storage areas, fume hoods and all areas where work is carried out. Cleaning Staff will clean labs / offices on a daily basis. If extra cleaning is required contact Chief Technical Officer (Paul Kane) Room 106 (ext.2290).

2. Correct Disposal Procedures

To ensure safety of staff that do not have a chemical / technical background it is essential the following is observed:

- Floors must not be used as storage areas (no winchesters empty or full, no chemicals).
- Sharps of any type (e.g. Pasteur pipettes, needles etc.) must NOT be placed in plastic bags. This includes yellow (hazardous waste) bags. Such items must be rinsed and disposed of in green bins (waste glass) or sharps boxes.
- Green Bins: to be used only for clear glass and porcelain (triple rinsed prior to disposal)
- Sharps Boxes: dispose of needles ONLY.
- Barrels which have been triple rinsed can be placed in non-hazardous bags.
- Brown Glass: (Winchesters, lab smalls etc.) must be triple rinsed and all labels removed before returning too stores.
- Plastic Containers: must be triple rinsed and labels removed before returning too stores.
- Labels can be removed by heating with a hot gun or by filling container with very hot water. It is advisable to rinse container with cold water prior to heating.

3. Chemical Spillage

Chemical Spillage: in the event of a chemical spillage, clean up IMMEDIATELY. Ensure caution signs are put in place and notify co-workers of any potential hazards.

If the extent of the spillage deems it necessary, two people should deal with the spillage. Where appropriate all others should leave the area. Suitable personal protective equipment (PPE) must be worn by the persons dealing with the spillage. As a minimum, safety glasses, lab. coat and heavy duty gloves (not disposable) must be worn. When dealing with spillages, inhalation of vapours should be avoided. Respiratory protection may be needed.

Spillage Kits are available from the stores or the safety office and are suitable for a wide range of chemicals. Some chemicals will require specialist responses (e.g. elemental mercury, cyanides, strong acids etc.). Before using such chemicals any specialist response equipment required must be readily available.

All wastes and contaminated items generated by a spillage must be disposed of in a suitable manner. Under no circumstances can a chemical spillage be left for cleaning staff to deal with.

4. Waste Solvent Disposal

Solvent waste is disposed of under two headings

1) Halogenated
2) Non-Halogenated

The cost of disposal of halogenated waste is considerably higher than that of non-halogenated. In order to reduce costs, the separation of both types should be a priority. This can be achieved by the correct use of the appropriate waste container. It should be noted waste solvent companies regard waste which contains more than one percent halogenated waste as halogenated and charge accordingly.

Once full 10 litre waste solvent drums must be returned too stores. Do not keep more than one full container in the lab. Rapid return of containers will reduce fire hazard and help rotate the limited supply of same.

The proposed disposal method of chemical waste should form part of the risk assessment prepared PRIOR to commencing work.

Chemicals labelled carcinogen or potential carcinogen (Risk Phrase R45, R40, and R49) must not be entered into waste disposal stream. They should be separated and disposed of in an appropriate manner.

5. Literature

There are many sources of information on waste disposal. Three very useful sources are:

1) Prudent Practices in the Laboratory (Handling and Disposal of Chemicals). Published by National Academy Press, Washington D.C. (Seventh printing 2006) It is recommended each lab should have its own copy.

2) Hazardous Laboratory Chemicals Disposal Guide; M.A.Armour Copy available from School Safety Advisor

3) University College Dublin Chemical Safety Manual. A copy of is issued on an USB key to each participant at the School Safety Induction Day. A hard copy should be available in the lab on the rack used for MSDSs and SOPs.

Analysis Services and Analytical Equipment

In the last five years, there has been very substantial investment in analysis equipment in the School, from large ticket items such as NMR to smaller equipment such as HPLC and IR/UV spectroscopy. Approximately !1M more is being spent to equip the A1 and A2 Developments.

Extensive and well-documented procedures already exist within the School regarding the services provided by the large equipment (NMR, MS, X-ray and elemental analysis). At present this is encountered when the user approaches the equipment and technical staff concerned. It is intended to integrate this material into a subsequent version of this document.

No such documentation exists for the smaller equipment and this will take some time to redress. The problem will become more acute with the delivery of 2 communal HPLCs (with column switchers) in late October - early November 2008. This equipment will be truly communal and will require the development of operating procedures. This will be another of the initial tasks of the SOP committee and we will take the opportunity to develop procedures for other equipment at the same time.

Vacation of Research Space

The laboratory and facilities used should be left clean, tidy and safe on completion of research work within the School. A form confirming that this has been done (in Appendix 2) must be completed by the postgraduate research student and signed by the relevant persons below and a copy submitted to the School Office and the School Safety Advisor.

Review, Modification and Addition of SOPs

This is intended to be a "living" document and will be subject to change in a controlled manner. In the first months of occupation of the A1 Laboratories, it is proposed to convene a number of meetings of the SOP committee to draft the procedures noted as absent above. Thereafter it is envisaged that yearly meetings would suffice for such changes as may be necessary. The SOP Committee will be chaired by a Member of School Academic Staff and will include, among others, the School Safety Officer and representatives of the various laboratories and research groups.

Appendix 1 SOPs and Quick Guides

Operation of Fume Hoods

Operation of Vacuum Pumps

Operation of Grubbs Stills

Operation of Glove Boxes

Hydrogen Gas Detection

Handling of Cyanide

Use of Mercaptans/Thiols and other foul smelling materials

Handling of Azides and other potentially explosive materials

Appendix 2 Forms

Unattended reactions

"Read and Understood" Forms

Vacation of Research Space Forms

Operation of Fume Hoods SOP-SCCB-001
Version 1

Created by: Una McCarthy Date: August 2008

Reviewed by: Chemistry Management Committee Date: 23rd September 2008

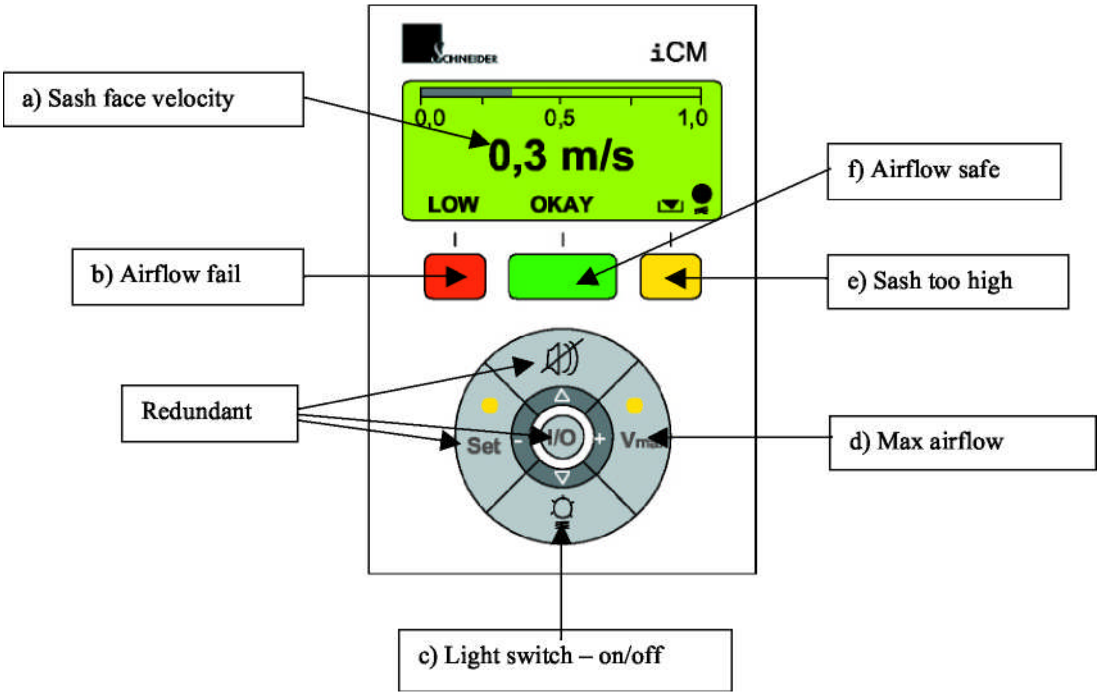
Contents:

Aim

The aim of this document is to provide instructions for the operation of the fume cupboards in Section A1 of the School of Chemistry and Chemical Biology and also those in the CSCB. A Quick Guide is also included providing guidelines on how to use fume cupboards safely and efficiently. For full details of the fume cupboard systems, please refer to the “Operation of Fume Cupboards” section of the Operations Manual.

School of Chemistry & Chemical Biology

A1 Fume Cupboard Control Panel



a) Continuous digital read out of sash face velocity

b) Airflow fail (red indicator) with audible alarm

c) Light switch – on/off

d) Max airflow option: This option sets airflow to maximum while the sash is in the lowered position (an orange light flashes when this mode is enabled). This mode should only be used in an emergency (e.g. if there is a spill in the fume cupboard) and should be manually disabled (by pressing the button again) once it is no longer required.

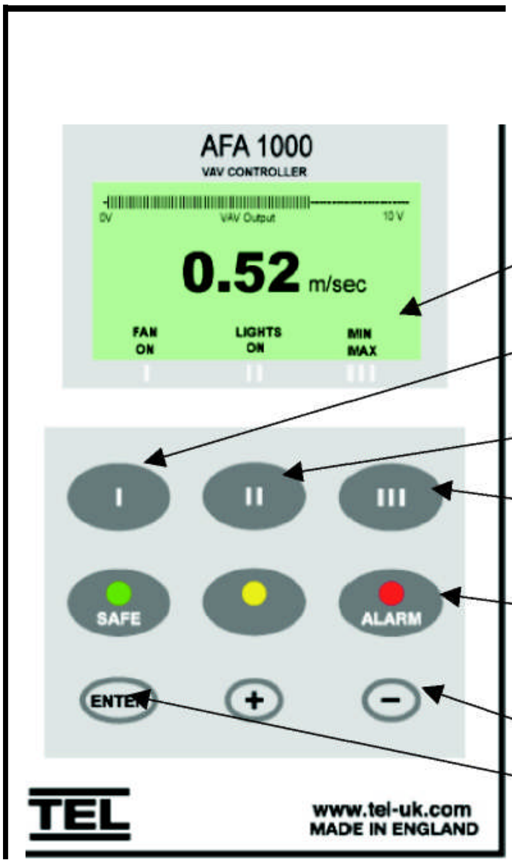
e) Sash too high (flashing yellow indicator)

f) Airflow safe (green indicator)

CSCB Control Panel

AFA 1000/2

The AFA 100
has the foll



- Digital Display with continuous reading of face velocity, alarm condition & pushbutton status
- Programmable Pushbutton I (Fan Stop/Start)
- Programmable Pushbutton II (Light On/Off)
- Programmable Pushbutton III (Spare Function)
- 3 Alarm Indication Lights (Green – safe, Amber – warning, Red – Alarm)
- \+ and – configuration buttons
- Enter Button

OPERATION

1. With the sash in closed position, press **Fan On** (pushbutton 1). Fan will start and after it is allowed to get to running speed, green – safe light will illuminate.
2. Raise sash to safe working height sash stop. Safe light will still be illuminated and digital display should give required face velocity reading.
3. If there is a requirement to raise the sash above the safe working height (Bench mounted fume cupboards only) please **DO NOT try to force the sash past the stop.**
    - If the sash is raised above the normal working height of 500mm, the audible sash high alarm will activate with the amber light flashing and the digital face velocity will toggle with a SASH HIGH display. The alarm can be muted by pushing enter.
    - Upon air failure (caused by sash being high as well as fan / extract system failures) the amber light will flash for a warning it is getting low and then the audible air fail alarm will activate with the red light flashing and the digital face velocity will toggle with a AIR FAIL display. The alarm can be muted by pushing enter.
4. Lower the sash completely and press **Fan Stop** (pushbutton I). The digital face velocity reading will reduce to zero and toggle with AIR FAIL display, all alarms will be muted and the red alarm indicator will flash. Air Fail status will continue until the fan is re-started

Fume Cupboard Quick Guide

School of Chemistry & Chemical Biology

- Never place your head inside an operating fume cupboard to check an experiment.
- Always work with the sash in the lowest position possible.
- Do not clutter your hood with bottles or equipment as this can interfere with the smooth flow of air.
- The under fume cupboard cabinets are vented and are suitable for use as storage areas for chemicals.
- If airflow fails for any reason, stop work in all fume cupboards (in any one laboratory) and lower all sashes until airflow is restored.
- Never ignore a fume cupboard alarm; always investigate the cause.
- Fume cupboards are fitted with an automatic sash closing device. If no movement is detected at the fume cupboard during a period of 5 mins, the sash will automatically close.
- The auto sash can be electrically operated by use of the “up / stop / down” switch fitted to the LHS of the fume cupboard.
- Never obstruct the automatic closing sash area.
- Contact the Chief Technical Officer if deactivation of the automatic closing sash is required (e.g. if using an immersion cooler).
- The auto sash may be operated manually.
- All vacuum taps must be fitted with a suitable trap at all times to protect the house vacuum system from solvent ingress.
- The “Vmax” mode may be useful in the event of a spill (please refer to control panel instructions for more information).
- Fume cupboard electrical sockets may be isolated using the power isolation switch on the top left hand side of the fume cupboard marked “Power Isolator”.
- The electrical socket in the under fume cupboard cabinet (LHS), may be isolated using the power isolation switch on the top left hand side of the fume cupboard marked “Vac Pump Power”.

Operation of Vacuum Pumps and Evacuated Apparatus SOP-SCCB-002 Version 1

Created by: Una McCarthy and Declan Gilheany Date: September 2008

1. Introduction

Vacuum work can result in implosion and the possible hazards of flying glass, splattering chemicals and fire. A risk assessment must be carried out for all vacuum operations. Vacuum systems must always be set up and operated with careful consideration of the potential risks. This SOP contains some basic guidelines on the use/set-up of vacuum systems, laboratory vacuum pumps and the necessary associated traps.

Note-A general risk assessment for the use of laboratory vacuum pumps can be found on the UCD Safety office website at http ://www.ucd.ie/safety/genlabrisk.html.

2. Vacuum Pumps

Vacuum pumps are used in the lab to remove air and other vapors from a vessel or manifold. The most common uses are on rotary evaporators, reduced pressure distillations, drying manifolds, freeze dryers, vacuum ovens and filtration apparatus.

The critical factors in vacuum pump selection are:

Application the pump will be used on Nature
of the sample (air, chemical, moisture) Size of
the sample(s)

When using a vacuum pump on a rotary evaporator, a dry ice alcohol slurry cold trap or a refrigerated trap is recommended. A Cold Trap should be used in line with the pump when high vapor loads from drying samples will occur. Consult manufacturer for specific situations. These recommendations are based on keeping evaporating flask on rotary evaporator at 40 C. Operating at a higher temperature allows the Dry Vacuum System to strip boiling point solvents with acceptable evaporation rates.

Vacuum pumps can pump vapours from air, water to toxic and corrosive materials like TFA and methylene chloride. Oil seal pumps are susceptible to excessive amounts of solvent, corrosive acids and bases and excessive water vapours. Pump oil can be contaminated quite rapidly by solvent vapours and mists. Condensed solvents will thin the oil and diminish its lubricating properties, possibly seizing the pump motor. Corrosives can create sludge by breaking down the oil and cause overheating. Excess water can coagulate the oil and promotes corrosion within the pump. Proper trapping (cold trap, acid trap) and routine oil changes greatly extend the life of an oil seal vacuum. Pump oil should be changed when it begins to turn a dark brown colour and, in any event, at least once per year.

Diaphragm pumps are virtually impervious to attack from laboratory chemical vapours. They are susceptible to physical wearing of the membrane if excessive chemical vapours are allowed to condense and crystallize in the pumping chambers. A five-minute air purge either as part of the procedure or at day’s end will drive off condensed water vapours and further prolong pump life.

Hazardous chemicals can escape from the vacuum pump and pump should be vented to a fume cupboard. Cold traps and acid traps can be helpful, but if allowed to thaw or saturate, they can lose their effectiveness.

3. Operation of Vacuum Pumps

1. No person may operate a pump without first receiving instruction in the safe use of that particular model. It is the responsibility of laboratory supervisors / managers to ensure that all persons under their control using pumps have been trained, and that full records of such training are maintained.
2. Pumps must be visually inspected before each use and damaged units reported to the laboratory manager / supervisor. Damaged units must not be used until they have been examined by a competent person.
3. All moving parts of pumps must be guarded so as to prevent workers coming into contact with moving parts.
4. A trap should be used between system and pump to prevent contaminants reaching the pump oil or being exhausted into the laboratory where possible.
5. Pumps that have the capacity to exhaust chemical contaminants should be vented to the outside, be used within a fume hood or have their own local exhaust ventilation.
6. The exhausts of pumps must be free from obstruction.
7. Where possible mercury diffusion pumps should be replaced by oil versions. Mercury pumps must have secondary containment and their use must be subjected to a risk assessment.
8. As far as possible, pump oil should be drained with the pump in a fume hood.
9. Refer to *UCDE19 Electrical Safety In The Lab Risk Assessment.*

4. Vacuum Apparatus

Vacuum work can result in an implosion and the possible hazards of flying glass, splattering chemicals and fire. All vacuum operations must be set up and operated with careful consideration of the potential risks. Equipment at reduced pressure is especially prone to rapid pressure. Such conditions can force liquids through an apparatus, sometimes with undesirable consequences.

Personal protective equipment, (PPE) such as safety glasses or chemical goggles, face shields, and/or an explosion shield should be used to protect against the hazards of vacuum procedures, and the procedure should be carried out inside a hood;

Do not allow water, solvents and corrosive gases to be drawn into vacuum systems. Protect pumps with cold traps and vent their exhaust into an exhaust hood;

Assemble vacuum apparatus in a manner avoiding strain, particularly to the flask neck;

Avoid putting pressure on a vacuum line to prevent stopcocks from popping out or glass apparatus from exploding;

Place vacuum apparatus in such a way that the possibility of being accidentally hit is minimized. If necessary, place transparent plastic around it to prevent injury from flying glass in case of an explosion;

When possible, avoid using mechanical vacuum pumps for distillation or concentration operations using large quantities of volatile materials. A water aspirator or steam aspirator is preferred. This is particularly important for large quantities of volatile materials;

5. Vacuum Trapping

When using a vacuum source, it is important to place a trap between the experimental apparatus and the vacuum source. The vacuum trap:

protects the pump and the piping from the potentially damaging effects of the material
protects people who must work on the vacuum lines or system
prevents vapors and related odors from being emitted back into the laboratory or system exhaust.

Proper Trapping Techniques

For **particulates,** use filtration capable of efficiently trapping the particles in the size range being generated

For most **aqueous or non-volatile liquids,** a filter flask at room temperature is adequate to prevent liquids from getting to the vacuum source.

For **solvents** and other volatile liquids, use a cold trap of sufficient size and cold enough to condense vapors generated, followed by a filter flask capable of collecting fluid that could be aspirated out of the cold trap.

For **highly reactive, corrosive or toxic gases,** use a sorbent canister or scrubbing device capable of trapping the gas.

Cold Traps

For most volatile liquids, a cold trap using a slush of dry ice and either isopropanol or ethanol is sufficient (to -78 deg. C). although acetone may be used, ethanol and isopropanol may be cheaper and less likely to foam.

Liquid nitrogen may only be used with sealed or evacuated equipment, and then only with extreme caution. If the system is opened while the cooling bath is still in contact with the trap, **oxygen may condense from the atmosphere** and react vigorously with any organic material present, leading to the **potential for an explosion.** It is the responsibility of each Research Group to consider what cold traps are most appropriate to their needs and take appropriate precautions.

6. Amendments Record

Amended/created on	Amended/Created by	Reason for amending	Version
01-Sep-08	Una McCarthy	Original	1

Operation of Grubbs-Type Dry Solvent Stills
SOP-SCCB-003

Created by: Una McCarthy Date: September 2008

Contents:

1 Aim

This SOP describes the operation of the PureSolv 400-3-MD Solvent Purification System. It includes the general procedures for:

- Dispensing dry solvent
- Refilling solvent kegs

2 Safety/additional information

2.1 When dispensing solvent, never fill the flask more than 2/3 full

2.2 Gauges should be set as follows at all times:

- Nitrogen supply gauge (building supply): ~ 50 psi
- Gas manifold gauges: ~ 3 psi
- Keg regulator gauges: ~ 15 psi

2.3 The gas manifold gauges are equipped with pressure relief valves. These safety valves are set to release any pressure above 7 psi. If pressure exceeds 7 psi a "hissing" sound may be observed. If this happens check that all the gauges are set as described above.

2.4 Pump exhaust should be vented.

2.5 Over time or through frequent use, the so-called “5-way valve” (evacuate/dispense/refill valve) may become loose and require tightening. If any of the following are observed please contact the Chief Technical Officer:

- If valve feels loose when turning between evacuate/dispense/refill
- If solvent is observed in vacuum line

2.6 To protect the 5-way valve, the green solvent tap should be kept closed when the system is not in use.

2.7 To ensure optimum quality, solvent should be dispensed into a Straus flask or a drop-funnel type collection vessel which is kept in place at all times (to protect dispensing line from atmospheric moisture)

2.8 Solvent used in the system must not contain any inhibitors/stabilisers.

2.9 The quantity of solvent added to each keg during the refill processes must be recorded in order to allow estimation of when cylinder reactivation is due.

2.10 Each cylinder should be reactivated once approximately 400 L of solvent has passed through that particular system. Contact the Chief Technical Officer when this is due.

2.11 Never leave “evacuate/dispense/refill” valve on “refill” if there is solvent in the dispensing vessel (this creates route for solvent to ingress and damage gas manifold gauges).

3 Procedures

3.1 Dispensing of Solvent

Before commencing, refer to Section 2.

3.1.1 Turn on house nitrogen supply for dry solvent system.

3.1.2 Switch on pump.

3.1.3 Open the green solvent tap by turning half a turn anticlockwise.

3.1.4 Check that collection vessel is empty and tap is closed.

3.1.5 Turn to “Evacuate” until level on vacuum gauge reaches approx. 25 psi.

3.1.6 When vessel is evacuated turn valve to “Refill” (*via* “Closed” rather than “Dispense”) until level on nitrogen gauge reaches approx. 3 psi.

3.1.7 Repeat steps 4 & 5 a total of three times.

3.1.8 Evacuate once more and then turn valve to “Dispense”.

3.1.9 When dispensing complete, turn valve to “Refill” momentarily in order to back fill collection flask with inert gas, also clearing dispensing line of solvent preventing dripping of solvent. (After dispensing, do not turn to closed *via* “Evacuate”, this will cause solvent to enter system causing solvent degradation of parts, if this accidentally occurs leave the pump on for *ca*. 1 hour to clear solvent in system.)

3.1.10 Turn valve to “Closed” position.

3.1.11 Switch off pump.

3.1.12 Close green solvent tap by turning half a turn clockwise.

3.1.13 Turn off house nitrogen supply at wall.

3.2 Refilling Solvent Kegs (Sparging/Purging)

Figure 3.2.1 Keg set-up during purge

3.2.1 Before commencing refer to notes 2.6 to 2.8 in Section 2.

3.2.2 Close solvent line (at Port #4) and disconnect it.

3.2.3 Disconnect the solvent keg from solvent purification system at nitrogen regulator (Port #1).

3.2.4 Connect a ! inch line (supplied in safety cupboard) to the two-way valve at Port #2 and run line to fumehood or venting point

3.2.5 Carefully pull keg out of safety cupboard and depressurize it slowly by turning the two-way valve at Port #2 to open position.

3.2.6 Once depressurized, close two-way valve at Port #2 and disconnect nitrogen line.

3.2.7 Move keg to fumehood.

3.2.8 Carefully open the two-way valve at Port #2 to ensure depressurization is complete.

3.2.9 Remove lid and fill " full with solvent (NB-note quantity of solvent added). Replace lid (to “hand tightness”).

3.2.10 Close two-way valve at Port #2. Return keg to safety cupboard and reconnect line to two-way valve at Port #2 and open it.

3.2.11 Connect nitrogen regulator to the three-way valve at Port #4.

3.2.12 Turn handle on three-way valve very slowly toward the regulator until a gentle nitrogen flow is achieved (feel side of keg for indication).

3.2.13 Purge solvent for 45 mins (this is the time required for a 15 L/4 gallon keg).

3.2.14 When purging is complete, turn inlet (Port #4) and outlet (Port #2) valves on keg to closed positions.

3.2.15 Disconnect nitrogen regulator from Port #4 and reconnect to Port #1 (head pressure inlet).

3.2.16 Re-secure keg in the solvent purification safety cupboard and open solvent line (Port #4) to system.

Sections 4 Quickguide

Quick Guide

Grubbs-Type Dry Solvent

Stils PureSolv 400-3-MD

1. Turn on house nitrogen supply slowly and ensure the pressure is *ca* 50 psi.
2. Switch on pump.
3. Open the green solvent tap by turning half a turn anticlockwise.
4. Ensure solvent collection vessels are empty and taps are closed.
5. Turn valve to “Evacuate” until level on vacuum gauge reaches approx. 25 psi.
6. Turn valve to “Refill” (*via* “Closed” rather than “Dispense”) until level on nitrogen gauge reaches approx. 3 psi.
7. Repeat steps 4 & 5 a total of three times.
8. Evacuate once more and then turn valve to “Dispense”.
9. When dispensing complete, turn valve to “Refill” momentarily and then turn valve to closed position.
10. Switch off pump.
11. Close green solvent tap by turning half a turn clockwise.
12. Turn off house nitrogen supply.
13. Dispense collected dry solvent with the aid of a nitrogen balloon.

Note:

- After dispensing do not turn to closed *via* “Evacuate”.
- Never leave on “refill” if solvent in is the dispensing flask

Section 5 Amendments Record

Amended/created on	Amended/Created by	Reason for amending	Version
13-Sep-08	Una McCarthy	Original	1

Operation of Glove Boxes SOP-SCCB-004

Version 1

Written by Andrew Philips / Dominique Schreiber, October 2008

1. Aim

The following document provides guidelines on how to adequately and safely operating a standard laboratory glove box and its associated devices.

2. General Information

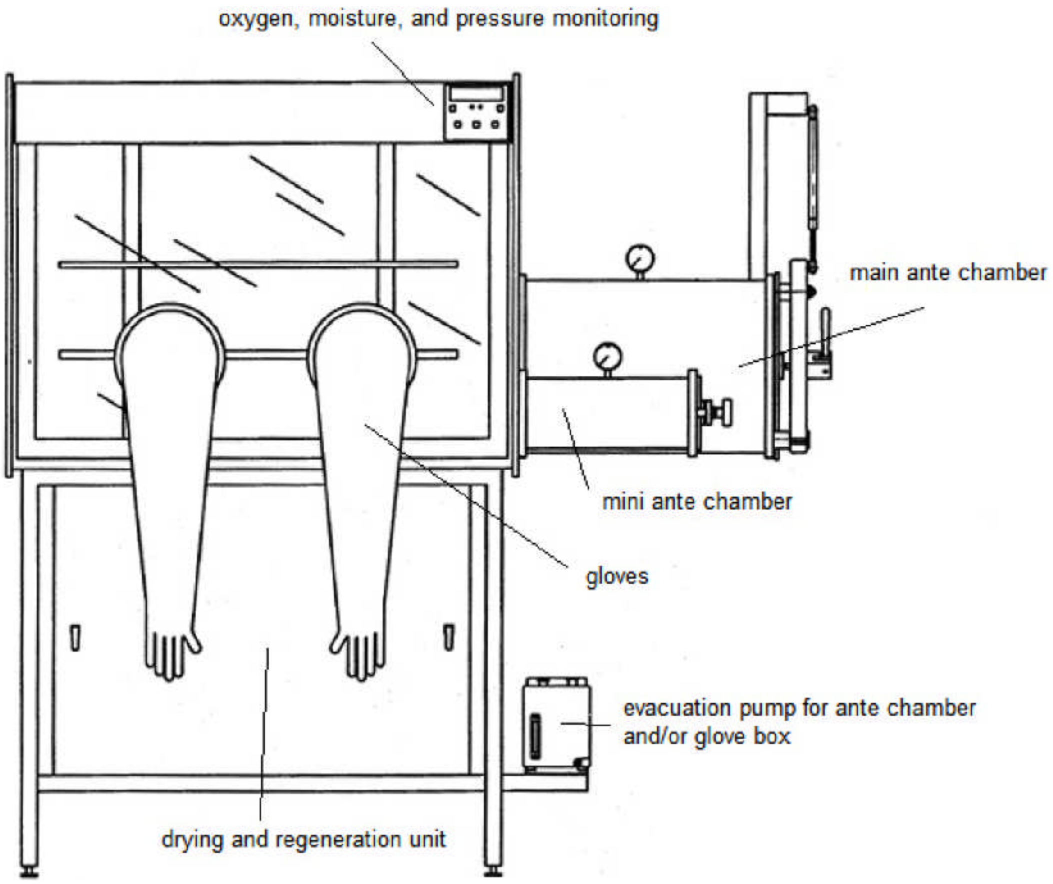
A glove box, also called a dry box, is a confined gas tight space allowing the manipulation of chemicals under an inert atmosphere. To some extent a glove box can be regarded as a substitute for conventional inert-gas Schlenk techniques employed on the laboratory bench. However, the best performance is obtained when combining glove box and Schlenk techniques. For this reason, every user should be familiar/comfortable with standard Schlenk techniques prior to use of a glove box.

As described, a glove box is a confined volume containing an inert gas, different to air, allowing the handling of air and moisture sensitive compounds. In most cases, these boxes contain nitrogen or argon as an inert gas together with a purification unit to prevent the accumulation of moisture and oxygen within the box. The bigger the glove box, the more difficult it is to maintain a dry and air-free atmosphere. Even if the glove box is initially air-free and well setup, every user needs to take as much care as possible not to contaminate the glove box with air, moisture, and corrosive volatile chemicals. Therefore, every user needs to plan very carefully all manipulations to be performed in advance and to make sure that the desired compounds are absolutely air and moisture free before bringing them into the box. When working within such a glove box, great care must be taken in order to avoid any spillages and accumulation of waste – especially volatile and corrosive chemicals should in general not to be handled in a glove box as they damage the associated purification and generation unit.

In many cases, the glove box will appear to be the method of choice compared to conventional Schlenk techniques because less glassware and simpler reaction setups are involved, however, clean and safe glove box manipulation will turn out to be as time-consuming as the corresponding bench techniques.

3. General Overview

The following figure shows the components of a standard glove box, similar to the ones installed in the A1 Laboratories at UCD.



3.1 Oxygen, moisture, and pressure monitoring

Every glove box is equipped with a water content measuring gauge and a pressure gauge. An operational glove box should contain less than 1 ppm of water. Some glove boxes also allow the continuous monitoring of the oxygen content, which should not exceed 1 ppm as well to guarantee an inert atmosphere. The pressure in a glove box is usually between 1 and 1.5 atm. Significant over pressure should be avoided by all means.

A glove box is designed to provide a gas-tight environment around standard pressure. To pressurize a glove box above 3 atm will cause the glove box to explode. Usually, the most pressure sensitive part are the gloves which easily burst above atmospheric pressures and may causes severe harm to the operator.

Likewise, excess vacuum should be avoided. Therefore, the glove box should never be put under continuous vacuum. Most modern glove box devices do have an automated pressure control that balances the pressure increase caused by manipulation.

3.2 Evacuation pump

The glove box and the ante chambers will be connected to a rotary vacuum pump. A single stage rotary pump can achieve pressures of about 1 torr, whereas a double stage pump can go down to about 10^{-4} torr. One of the most common reasons for bad vacuum with these types of pumps is a too low **oil level –** so the oil level needs to be checked regularly. In addition, the pump exhaust valve needs to be covered with oil in order to obtain a good seal. As a rough rule, any deviation in sound of the operating rotary pump indicates technical problems of which it should be taken care of immediately. For further specifications about rotary pumps, refer to the corresponding SOP.

3.3 Drying and regeneration unit

The glove box is equipped with a recirculation system that exchanges the glove box atmosphere over an oxygen scavenger and a drying unit. These oxygen scavengers are based on copper (RIDOX and BTS catalysts) or manganese oxide, whereas the drying unit consists of molecular sieves (4A or 5A). In order to maintain an inert atmosphere in the box, its volume is exchanged about 50 times per hour, which should allow for oxygen and moisture levels lower than 1 ppm respectively. When the level of O2 and/or H2O go beyond 1 ppm for an extended period of time, it is necessary to regenerate, see 5.2.

4. Operating the Glove Box

4.1 General use of a glove box

The following guidelines are a prerequisite to start working in a glove box.

- If the glove box is used by more than one person on a routine bases, a log book should be filled in advance to book the box for the desired time
- If reactions are run over a longer time in the box, every user should keep in mind that the next person using the box will need enough space to perform its manipulations. Therefore, the working area in front of the gloves should be cleared and cleaned after every manipulation
- Every item in the glove box should be placed and stored within a reachable distance. If necessary, tweezers can be employed to reach for distant objects

- Every user should have properly labelled plastic containers inside the box where personal samples can be stored
- After finishing work in the glove box, the gloves are wrapped inside-out and cleaned with a slightly watered tissue
- After having performed work in the glove box, one has to make sure that the ante chambers are left under vacuum

Remember that some chemicals when stored in a glove box are inert, but when these are taken out of the box, they can react violently with the moisture and oxygen containing laboratory atmosphere (e.g. alkyl metals). Therefore, these items should not be directly exposed to the laboratory atmosphere. It has to be made sure that these highly reactive substances are not mixed with other chemicals and items within the glove box, and thus taken out accidentally.

4.2 Purging and filling the ante chamber

If one considers the ante-chamber a gas-tight system, the number of cycles of alternating vacuum application and inert-gas filling which are necessary to obtain a fraction of less than 1 ppm of air is given by the following equation:

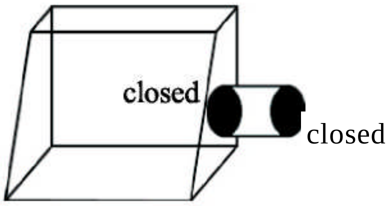
$$R_{air} = P_n$$

where R_{air} is the fraction of air remaining in the ante chamber after n cycles using a vacuum pump able to achieve a pressure P. Therefore, for a typical ante-chamber of an approximate volume of 40 dm^3, 3 cycles of applying moderate vacuum (1 torr) will result in a remaining air fraction of only 2 ppb.

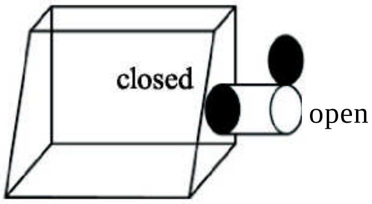
- Check if the inner port door is closed, otherwise shut the inner door prior to evacuate the ante chamber (By default, the inner door has to be shut and the ante chambers set under vacuum)
- Shut off the vacuum
- Start filling the ante chamber with nitrogen until the ante chamber pressure gauge indicates approximately 1 bar, then open the outer door of the ante chamber
- Insert the required items in the ante chamber under a continuous flush of nitrogen. EVERY ITEM HAS TO WITHSTAND THE APPLIED VACUUM - **SOLIDS** are dried and brought into the ante chamber in a closed vial. **AIR SENSITIVE SOLIDS** need to be put in a strong vacuum withstanding Schlenk flask. **NON-VOLATILE** and **VOLATILE LIQUIDS** need to be in a strong vacuum resistant flask (equipped with a teflon stopcock) containing 1 bar of inert gas.
- The outer door of the ante chamber is closed and the nitrogen is shut off.

- Evacuate the ante chamber for 15 or 5 minutes for the big or small ante chamber respectively. Flushing with nitrogen until 1 bar is reached

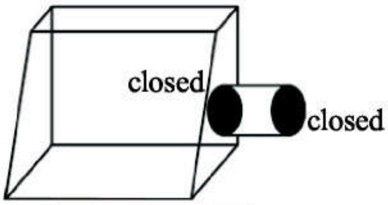
  THIS STEP IS REPEATED 3 TIMES
- After the above described cycles are performed, flush again with nitrogen until 1 bar is reached.



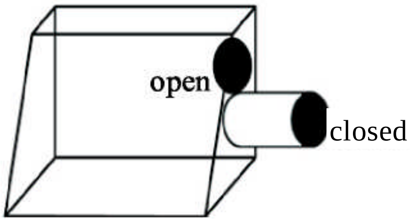
Ante chamber is under vacuum and then filed with nitrogen



If 1 atm of nitrogen is reached, the outer door is opened



Vacuum is applied, followed by flushing it with nitrogen – this step is repeated 3 times!



The inner door is opened and the items brought into the box – then the inner door is closed

4.3 Perform manipulations in the glove box

- Before you start working in the glove box, make sure that either all the steps listed under 4.2 are completed or if no items need to be taken into the box, make sure that the vacuum in the ante chambers is shut off and that they are refilled with approximately 1 bar of nitrogen
- Remove jewellery from your hands, as they can damage the gloves
- Immerse your hands and arms into the gloves very slowly to avoid a sudden increase of pressure which will cause the glove box to automatically shut down. When you are able to handle the vacuum system manually (for example with an external foot pedal), apply a gentle vacuum in order to balance the pressure increase associated with the immerging the gloves into the box
- Avoid manipulating sharp objects with the gloves, as the gloves may be damaged

- After finishing working in the box, make sure that the ante chamber doors are closed – the ante chamber is then put under vacuum. Switch the circulation system to standby (allows for lower nitrogen renewal frequency)
-

4.4 Taking objects out of the glove box

- Check if all the steps described in 4.2 are completed, otherwise check if the ante chamber external doors are closed (if not, close them and perform 3 vacuum –nitrogen cycles as described in 4.2)
- If the ante chamber contains approximately 1 bar of nitrogen – make sure that the nitrogen supply is shut off
- Once inside the glove box, open the inner door of the ante chamber and place in there the items to be taken out from the box – make sure that they are properly sealed
- Close the inner ante chamber door – once, outside the box, apply a gentle flow of nitrogen when opening the outer ante chamber door
- If all the items are removed, close the outer door of the ante chamber and put the ante chamber under vacuum
-

5. Regeneration and Servicing

5.1 Cleaning and Labeling

The glove box needs to be cleaned after every manipulation. To clean the box inside, a box of Kleenex will do nicely. However, to avoid accumulation of moisture within the tissues, they should be dried in a vacuum desicator prior to bring them into the box. In addition, the glass window should be cleaned at the outside on a regular basis, using a slightly watered tissue. As already mentioned in 4.1, each flask and vial inside the box needs to be labeled properly. It is also suggested that every user has a plastic tray in the box where personal samples/items can be stored.

5.2 Regeneration

To maintain an inert and dry atmosphere containing in maximum 1 ppm of oxygen and moisture respectively, the circulation unit needs to be generated at a level of about 0.95 ppm. Regeneration of the oxygen scavengers, namely BTS or RIDOX, is achieved by heating to about 200 – 300 °C under a stream of nitrogen, followed by flushing the scavenger with hydrogen (5 % hydrogen in 95 % nitrogen) to obtain again the reduced form. Molecular sieves can be regenerated by heating under vacuum. One has to be aware that the use of hydrocarbons and polar solvents presents a contamination problem of the regeneration unit. In particular, **volatile sulfides and halocarbons** should not be handled within the glove box if the regeneration unit isn't preceded by Linde 13X molecular sieves, which are able to trap these compounds.

In order to use hydrogen gas as a regenerating agent, the user must be familiar with hydrogen gas SOP and the corresponding emergency procedures.

5.2 Diffusion of air and leaking

The major part of air and impurity diffusion into the box is due to the gloves. It is estimated that about 59 ppm/h of atmospheric impurities diffuse through the gloves under stand-by conditions. When the glove box is in use, the diffusion of the impurities is increased by a factor of 10. Therefore, it is of crucial importance to choose the right type of gloves. In general, butyl rubber gloves are preferred over neoprene gloves as they show reduced diffusion of moisture and oxygen. To prevent impurity diffusion in stand-by modus, the glove ports should be sealed using glove port shuts.

If the values of oxygen and moisture within the glove box keep rising even after the drying unit and the oxygen scavenger had been regenerated, the box should be tested for leaks. Small leaks are detected by slightly pressurizing the box and cover the potential leak areas with soap solution – if bubbling is observed, the leak is detected.

5.3 Safety guidelines

- Gas cylinders for regeneration of the oxygen scavenger need to be fixed to the wall and freely accessible
- The good functioning of the moisture and pressure gauges needs to be verified at least once a year
- Be familiar with the MSDS of every substance you intend to work and/or to store in the glove box
- When leaks are suspected to be present, increase pressure slightly to avoid contamination of the glove box – locate and fix the leaks immediately

IF A SUDDEN PRESSURE BUILD UP IS OBSERVED, THE RECIRCULATION UNIT NEEDS TO BE SHUT DOWN

6. Literature

1) D. F. Shriver and M. A. Drezdzon: The Manipulation of Air-Sensitive Compounds, 2nd 1986, Wiley Interscience, New York.
2) J.R. Errington: Advanced Practical Inorganic and Metalorganic Chemistry, 1997, CRC Press, London.

Hydrogen Gas Detection System SOP-SCCB-005

Version 1

Created by: Una McCarthy Reviewed by: Chemistry Management Committee
Date: August 2008 Date: 23rd September 2008

This SOP applies to the CSCB Buiding only

It will be updated when the A1 system has been fully installed.

SOP Read & Understood Form required

Contents

1. Aim

This SOP provides important information for CSCB Hydrogen gas users in relation to the Hydrogen Gas Detection Systems that are installed in the CSCB Building. Labusers intending to use Hydrogen gas for the first time must notify the CSCB Technical Officer.

All CSCB Hydrogen Users must sign an "SOP Read & Understood" form for this SOP.

2. Safety Information (See MSDS for full details)

All Labusers intending to use Hydrogen gas must consult the MSDS and the UCD Chemical Safety Manual located at http://www.ucd.ie/safety/index.html.

Hydrogen is a colourless, odourless, extremely flammable gas.

Hazards Identification:

- Burns with an invisible flame
- High pressure gas
- Can cause rapid suffocation

- Extremely flammable
- May form explosive mixtures in air
- Immediate fire and explosion hazard exists when mixed with air at concentrations exceeding the lower flammability limit (LFL)
- High concentrations that can cause rapid suffocation are within flammable range and should not be entered
- Avoid breathing gas
- Self contained breathing apparatus (SCBA) may be required

3. General Information

Figure 3.1 Hydrogen Detection Control Panel

Figure 3.2 Hydrogen Detection Amber Strobe

- There are 3 separate Hydrogen Detection Systems in the CSCB, one on each floor 1, 2 and 3
- The control panels are located outside the door to Labs: L1.12-1.17, L2.12-2.17 and L3.12-3.17 (see Figure 3.1).
- Each system is designed to detect the presence of hydrogen gas.
- The hydrogen alarm is connected to **AMBER coloured strobes/sirens** in each laboratory (see Figure 3.2) and instrument room and the siren is not as loud as a Fire alarm siren.
- The hydrogen alarm registers on the fire alarm panel but does not activate the audible fire alarm unless a very serious leak is detected.
- The systems on each floor are completely separate. Therefore, if an alarm is activated on one floor it is audible/visible only on that floor.
- The hydrogen alarm sounds if a level of above 10% LEL is detected by any one sensor.
- The locations of all sensors are clearly marked on each control panel.
- The system monitors the areas served by the hydrogen cylinder in the gas cylinder storage cupboard located on the corridor of each floor (1, 2 and 3).

- **If a high level (20% LEL) of hydrogen gas is detected an automatic safety shut off valve located in each gas cylinder cupboard is triggered and the gas supply is shut off.**
- There is a "Panic Button" located in each instrument room on each floor. When activated, this triggers the safety shut off valve, cutting off hydrogen supply from this point in the gas cylinder cupboard to the areas served by the hydrogen gas pipeline.
- The hydrogen gas pressure should never be increased above 5 bar.
- In the case of a leak occurring directly from a gas cylinder, the safety shut off valve cannot resolve this situation. In this case, the sensor located inside the gas cylinder cupboard (Sensor No. 4) will activate the full audible fire alarm and the building will be evacuated.

4. Procedure if you intend to use Hydrogen gas

If it is the first time you use Hydrogen gas please notify the CSCB Technical Officer.

1. Turn on Hydrogen cylinder. You are now responsible for the Hydrogen gas supply on your floor. **Never increase the hydrogen gas pressure above 5 bar.**
2. Fill in relevant details in logbook at control panel.*
3. When finished using the Hydrogen gas, turn off the supply.
4. Remove your name from the board provided.

* If you are working out of hours or if you leave the lab while H_2 is turned on you must fill in your phone number in the logbook.

5. Procedure in the event of alarm activation

When a hydrogen alarm sounds, the amber strobe in each laboratory and instrument room on the effected floor will flash and the siren will sound.

1. **Turn off the Hydrogen cylinder immediately.**
2. Open the windows on the corridor and open the doors to the lab.
3. Contact:

During Office hours contact	Out of Hours contact
(a) Úna McCarthy* ext 2963	(a) Services ext 2600
(b) Dermot Keenan ext 2353	(b) First Response Room ext 1200
(c) Jimmy Muldoon* ext 2958	

4. Leave the area.
5. It is not necessary to evacuate the building unless the Fire alarm sounds.

*Mobile phone numbers are held by Science Services and First Response Room

Important

If the fire alarm sounds evacuate immediately and DO NOT open the gas cylinder cupboard.

6. Additional information

An alarm sounds if a level of 10% lel is detected by any one sensor. This is a warning that there is a hydrogen leak in the gasline. You can tell which sensor activated the alarm by looking at the Control Panel. There are 4 sensor lights (C1-C4) (see Figure 6.1). The system cycles through each sensor and gives a reading. This should be approximately zero% lel. The Sensor which activated the alarm will be denoted by a red light. If the level should rise to over 20% lel the safety shut off valve will be activated which shuts off supply to areas served by Sensors 1, 2 and 3. Therefore the situation is now safe if the alarm was caused by Sensor 1, 2 or 3. However, if the alarm was caused by Sensor 4 which is located in the gas cylinder cupboard the leak could possibly be directly from the cylinder. In this case, the system has been set to activate the full fire alarm if a level of 25% lel is detected. Therefore, if you hear the fire alarm at any point, do not open the gas cylinder cupboard doors, evacuate immediately. For your information, the gas cylinder cupboard is vented.

IMPORTANT: NEVER RESET THE ALARM

("Reset" button in some cases simply mutes the alarm)

Sensor lights C1-

Figure 6.1

Cyanide Handling Procedures SOP-SCCB-006

Created by John Coffey

Prepare a written Hazard Identification and Risk Assessment, which must be countersigned by Academic Supervisor (see Risk Assessment for cyanide compounds in UCD Chemical Safety Manual page 55) url: ucd.ie/safety/chemagentrisk.html.

Deposit a copy with supervising First Aider* and John Coffey.

Before commencing procedure, ensure oxygen resuscitation devices and designated First Aider are on hand in the event of an incident.

When using these materials at least one other person must be present in the lab at all times. This person must be aware of the procedures to be followed in the event of an emergency.

Procedures using these materials must never be carried out, outside of normal hours or over the lunch period when first aiders may not be available.

The following are trained in the use of oxygen resuscitation devices:

Mick Dunne - Ext.2289 (Lab 309)
Andy Rous – Ext.2099 / 2301 (Room 105 / Lab 210)
John Coffey – Ext.2410 (Lab 132 / 109)

Procedures for Handling Foul Smelling Compounds Esp Thiols SOP-SCCB-007

Created by John Coffey

Most thiols have an obnoxious smell and volatile ones must be handled with great care in order to prevent the release of foul smelling vapours. In addition to the discomfort caused, the smell is often confused with that of gas and this can give rise reports of gas leaks. **The escape of foul smelling thiols may result in this or adjacent buildings being evacuated.** The following procedure must be used when handling volatile sulfur compounds.

1. Consult the safety data books to find out how volatile the thiol is and to check for hazards other than the stench.

2. Tell John Coffey (Ext.2410) when and where the experiment is to be carried out, so that he can inform the Maintenance Department(Ext.1111). Out of hours ring Services (Ext.1200).

3. Consult your supervisor regarding techniques for minimising the release of the compound, during the reaction, the workup and the purification.

4. Do the experiment in a fume cupboard in which no other experiments are being carried out.

5.Treat the thiol as though it was an air sensitive material, i.e. do not expose it to the atmosphere. Thus, the container should be sealed with a septum, and the thiol should only be transferred using syringe techniques, ensuring minimal exposure to the air. Wear a lab. coat and disposable gloves and take care to avoid contact with the thiol. Do not take the lab coat or gloves out of the hood if they are contaminated. The vapours from the reaction should be passed through bleach solution.

6. Do not workup the reaction or isolate your product until you have worked out a procedure, with your supervisor, for dealing with any possibility of escape of residual thiol. **Note that the aqueous washes from the reaction will also require bleach treatment.** **If you do not workup the reaction the same day, you must inform John Coffey when you do carry out the workup.**

7.All glassware which comes in contact with the thiol should be allowed to soak in bleach solution for several hours, before cleaning in the normal way. Use bleach solution on any spillages.

NO RESEARCHER WILL BE ALLOWED TO GRADUATE WITHOUT FIRST COMPLETING THIS FORM

Procedures for Handling Azides and other potentially Explosive materials SOP-SCCB-008

Created by Declan Gilheany / Una McCarthy

Organic azides are potentially explosive substances that can, and will, decompose with the slightest input of energy from an external source (e.g. heat, light, pressure). Additionally, small molecules containing the azido functionality tend to decompose violently, which may result in injury if proper safety precautions are not utilized. Generally, azides of high molecular weight are considered a low risk, whilst azides of low molecular weight are more problematic. High nitrogen content is another warning sign. If you intend synthesising, purifying or handling azides you must consult your Supervisor and the CSCB Technical Officer. The following safety precautions should be adhered to:

- Always use appropriate gloves when handling azides (the azide ion has a similar toxicity to the cyanide ion) and also use a plastic spatula for weighing.
- Sodium azide reacts violently with some common laboratory organics. When planning your experiment, always research the reactivity of sodium azide to ALL reaction components.
- Never use chlorinated solvents as reaction media (dichloromethane and chloroform will result in the formation of explosively unstable di- and tri-azidomethane respectively).
- All organic azides decompose with introduction of external energy and therefore must be stored below room temperature and in darkness.
- Never use distillation or sublimation as purification techniques. Purification should be limited to extraction and precipitation.
- Azide waste should be placed in a separate, labelled container and kept away from acid.

The following general precautions apply to any compounds that are potentially explosive (an extensive list of such compounds is given in the UCD Chemical Safety Manual.

- As with any chemical agent, its MSDS must be consulted to establish if it is explosive
- Known explosive materials must be stored in a suitable manner away from incompatibles
- No more than one day's supply of explosive material should be stored at the bench
- All containers holding explosive materials must be clearly labelled as such
- Potentially explosive materials must be only used in a fume hood behind a safety screen
- Chemicals known to become explosive when dry (e.g. picric acid) should be regularly inspected and where necessary wetted
- Chemicals known to become explosive after a period of time should be dated as to when they were opened and disposed of / stabilised before they become a risk

- Keep all sources of ignition away form potentially explosive materials

8. **If you notice an obnoxious smell at any stage, you must inform John Coffey at once.**

School of Chemistry & Chemical Biology
Unattended Experiment Form

Lab:	Date:	Fume Cupbd No:

Name (of person responsible):

24 hour Contact Phone No:

Signature:

List solvents and all hazardous chemicals giving approx. volumes:
(Do not write formulae)

Possible Hazards (circle):

Fire *Explosion* *Toxic Fumes* *Corrosion*

Avoid contact with skin *other (specify)*

Apparatus:

Services required (circle):

Water electricity heat inert gas

vacuum other (specify)

Emergency action (e.g. failure of services):

Supervisor (or Alternative Supervisor):

Signature:

- 2 copies required. Please place one on fume cupboard and the other in the box provided outside the lab-door
- NB: Remove this form from the box when experiment is complete
- Leave light on in fume cupboard containing this experiment
- Please refer to SCCB SOP for Unattended Experiments

SCCB/CSCB Read & Understood Form

This is to confirm that the undersigned have read, understood and agree to follow any relevant instructions detailed in:

SCCB/CSCB Operations Manual   Version: 1 (17th October 2008)

Laboratory: ______________________________

Principal Investigator: ______________________________

Researcher (Please print)	Signature	Date

Research worker finishing up form-- CS RES 1

This form can be obtained from the school office/school safety officer and should be completed by the postgraduate research student and signed by the relevant people below and a copy submitted to the school office and the school safety advisor. The laboratory and facilities used should be left clean, tidy and safe on completion of research work within the school.

PLEASE TICK RELEVANT BOX BELOW:

RESEARCHER CHECKLIST	**Y**	**N**
Is the bench space clean, cleared and left in a safe manner?		
Have the underbench units been cleaned and left in a safe manner?		
Have the drawers been cleared and cleaned and left in a safe manner?		
Donate any remaining chemicals/synthesized compounds to your colleagues/supervisor?		
Have you removed and disposed of your chemical waste in the correct manner?		
Have you tidied your fumehood workspace in a safe and correct manner?		
Have you cleared data stored on instruments/computers etc.?		
Have you cleaned equipment used in these areas?		
Have you returned keys for the building, laboratory, lockers etc.?		

Name:

Room no.:

Laboratory room number/s used:

I understand that the forms relating to the examination of my thesis will not be presented to the appropriate Faculty Board for Research degrees, until such time as this form is signed by ALL persons indicated below, in the order given.

Student Name: **Signature:**

Supervisor: **Signature:**

School Safety Advisor: **Signature:**

N.B. Signatures signify that all procedures required have been fully complied with.

A copy of this completed form should be given to the school safety advisor and to the School Office.