UNIVERSITY OF UTAH

Department of Physics and Astronomy

Safety Manual

Revised: August 2019

Part 3

General, cryogenic and laser safety

I. Protective Equipment

What protective equipment must be used for a particular operation/ laboratory procedures is determined by Supervisors.

A. Eye Protection

To minimize the risk of eye injury, all personnel and visitors MUST wear eye protection while in a departmental lab where some chemistry work is done. Eye protection in laser labs are described below,

Eye protection is required by law (Code of Federal Regulations, Title 29, Section 1910.133) for all personnel working in labs that work with chemicals. This protection is required whether or not one is performing an experiment. Groups that work with chemicals must maintain a supply of safety glasses for visitors near lab entrances, and group members are responsible for enforcing the policy of wearing eye protection whilst in the lab.

Safety glasses can be purchased in the stockroom or from VWR or Fischer Scientific. Groups that work with lasers or other light sources should also have a supply of glasses with the appropriate filters.

Ordinary prescription glasses do not provide adequate protection against injury, as they lack side shields and may break upon impact. If one wears prescription glasses, one can either wear safety goggles on top of the glasses or obtain a pair of prescription safety glasses.

Contact lenses provide no protection against eye injury and are no substitute for safety glasses. Further, if chemicals get in the eye, the lens can trap the chemical and interfere with first aid and eye-flushing procedures (note, the lens may not come out with eye flushing). It is imperative that each lab have an up-to-date list of members who wear contact lenses so that appropriate measures can be taken in an emergency (ie, removal of the lenses).

Goggles offer more protection than safety glasses, and should be worn when carrying out operations that have a high risk of splashing chemicals, flying particles, etc.

Face shields must be worn when there is a risk of explosion. This is also encouraged when working with particularly hazardous chemicals that will cause severe burns upon exposure to skin.

B. Lab coats

All personnel working in the lab must have at least one lab coat that is appropriate for their needs. Lab coats must be worn in the chemistry lab at all times.

In most instances, a standard 100% cotton lab coat will be sufficient. These are available for purchase in the chemistry stockroom. Those working with pyrophoric chemicals should also have access to a Nomex flame-retardant lab coat to minimize the risk of catching on fire. These can be purchased from VWR and Fischer.

Lab coats should remain in the lab, and under no circumstances should be worn in the bathrooms.

Lab coats can be laundered on campus (2-day turnaround). Bring a completed campus order form to the basement of the University Hospital, room AA120 between 7AM-3PM (M-F). Lab coats must have pockets emptied and be labeled with the group name. You are responsible for dropping off/picking up laundered coats.

C. ***Physical Hazards***

These hazards include compressed gases, nonflammable cryogenics, high-pressure systems, vacuum work, UV-vis-near IR radiation, electrical hazards, magnetic fields, sharp edges, and ergonomic hazards.

Compressed Gases

Compressed gases expose laboratory personnel to both chemical and physical hazards. If the gas is flammable, flash points lower than room temperature compounded by rapid diffusion throughout the laboratory present the danger of fire or explosion. Additional hazards arise from the reactivity and toxicity of the gas. Asphyxiation can be caused by high concentrations of even inert gases such as nitrogen. An additional risk of simple asphyxiants is head injury from falls due to rapid loss of oxygen to the brain. Death can also occur if oxygen levels remain too low to sustain life. Finally, the large amount of potential energy resulting from the compression of the gas makes a highly compressed gas cylinder a potential rocket or fragmentation bomb.

To minimize the risks of gas mixing/release, all gas cylinders and accessories are labeled with a CGA number (Figure 5-7), which is specific to each gas (See Appendix G). To minimize accidental equipment mismatch/mixing, flammable gases are reverse-thread.

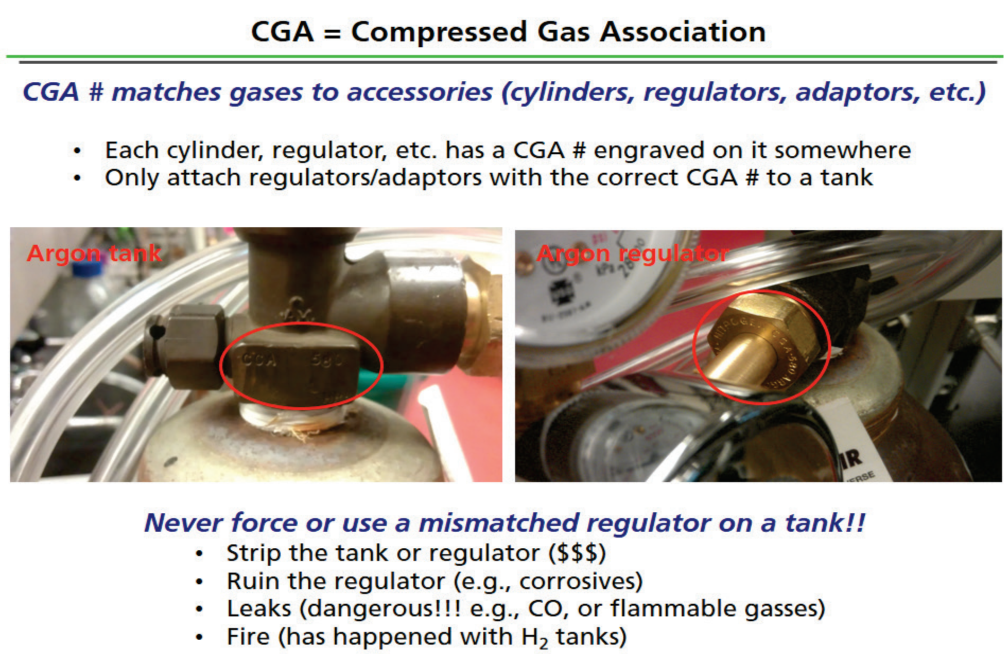


Figure X-4. Information on CGA numbers. Consult Harold Simpson on classification of adapters and regulators.

All gas cylinders must be double-chained to the wall, and when not in use, stored with a cap and no regulator. Proper training is required for use of compressed gases.

Nonflammable Cryogens

Cryogenic liquids are fluids with boiling points less than -73°C (-100°F). They are also characterized by a high volume-expansion ratio in the liquid to gas phase. Some physical properties of three reference cryogenic liquids are outlined in Table 11-2.

Identity	Boiling Point	Vapor Pressure (at -196°C)	Volume-Expansion Ratio (at 1 atm, 20°C)
Nitrogen (*l*)	-195.8°C	730 mm Hg	1:696
Oxygen (*l*)	-182.9°C	150 mm Hg	1:861
Carbon Dioxide (*l*)	-78.5°C	1.33×10^{-8} mm Hg	1:553

Table 5-8: Common Characteristics of Cryogenic Liquids.

Hazards to consider when working with cryogenic liquids include cold burns/frost bite from liquids in contact with skin or eyes. Appropriate PPE includes safety goggles, lab coats, closed toe shoes, and clothing that covers arms and legs. Insulated gloves which are easily removed can be worn, but are not recommended due to the high risk of cold burns (with liquid nitrogen). Likewise latex or nitrile gloves can capture splashes of cryogens and hold them close to the skin causing burns as the liquid expands as it warms. Additionally, inadequate ventilation poses asphyxiation hazards as oxygen-containing air is displaced. Do not use cryogenic fluids in small, closed rooms or rooms with inadequate ventilation. Embrittlement of structural materials is also a hazard of cryogenics, as is pressure buildup.

Explosion hazards should also be considered, as many cryogenic fluids can condense oxygen from the air to generate liquid oxygen. Liquid oxygen is flammable and explosive; it is characterized by a light, clear blue color. When in combination with organic compounds, the explosive nature of liquid oxygen is exacerbated.

Work and transportation of cryogenics requires additional training.

High-Pressure Reactions

Experiments that generate high pressures, are carried out at elevated pressures, or are run in supercritical fluids, can lead to explosion from equipment failure. Additional safety training is required to carry out such work.

Vacuum Work

Precaution must be taken when working at sub-ambient pressures. The main danger is injury from glass breakage, and release of chemicals, which may catch fire, explode, or be toxic. Additionally, most vacuum lines require cold traps, which pose additional hazards. Additional training is required for vacuum line work that makes use of liquid nitrogen cold traps.

UV, visible, and near-IR Radiation

UV, visible, and near-IR radiation from lamps and lasers can produce many hazards. UV lamps used in biosafety cabinets and light boxes can cause serious skin and corneal burns. Powerful arc lamps can cause eye damage and blindness within seconds. Some compounds are explosively photosensitive.

Incorrect use of lasers can pose hazard to the eyes, and is also a potential fire hazard. Depending on the type of laser, additional hazards can include: mutagenic, carcinogenic, or otherwise toxic laser dyes and solvents; flammable solvents; UV or visible radiation from the pump lamps; and electric shock from the lamp power supplies.

Additional training is required for use of UV, visible, and near-IR radiation.

Electrical Hazards

Electrocution hazards of electrically powered instruments, tools, and other equipment are essentially eliminated by safety features built into the systems. However, the possibility of serious injury or death by electrocution is very real if attention is not paid to engineering, maintenance, and personal work practices. It is advised that researchers check the integrity of their electrical equipment. Chemicals in the lab can lead to eroded insulation on wires. These should be repaired immediately, particularly if they are located in near proximity to water (or cold environments). Equipment malfunctions can also lead to fires. If equipment results in tripping of a circuit, lab personnel should not reset the circuit breaker but instead have the electric shop check the integrity of the electronic equipment. *Repair and modification of electrical equipment should be done by the electronics shop*.

All lab personnel should know the location of the lab power shut off. This is usually located by a door, and is a large red switch. Personnel should also be aware of hazards that may arise after power outages when power returns to the lab. Each lab should have a plan of action for such a scenario.

Magnetic Fields

Many instruments, such as NMR spectrometers, have magnetic fields on the order of 14,000 to 235,000 G. The magnitude of these static magnetic fields drop off rapidly with distance. Also, many instruments have internal shielding, which reduces the strength of the field outside the instrument. Strong attraction occurs when the magnetic field is greater than 50 to 100 G and increases as the separation is reduced. Those with metal implants should be cautious of the distance they maintain with these instruments. These instruments should be marked with the “safe” distance. If in doubt, contact the manufacturer of your medical implant.

Sharp Edges

A common lab injury has to do with cuts and punctures from broken glasses, needles, and other sharp objects. To minimize such injuries, personnel should take the following precautions:

- Use correct procedures (for instance, do not over-pressurize a glass vessel)
- Check glassware for chips and cracks. These render the glassware prone to breakage, but can readily be fixed by the glassblower.
- Dispose of broken glass in glass disposal, not the regular trash.
- When using razors, ensure that the edge is sharp, and keep hands out of the line of the cut. Also stand off-line from the direction of the cut.

Ergonomic Hazards

Personnel should not ignore ergonomic hazards that come with lab work. Repetitive motion and awkward posture due to instrument positioning can lead to strains and over-use injuries. More information on ergonomics can be found here: http://www.cdc.gov/niosh/topics/ergonomics/. A checklist that helps evaluate ergonomic hazards in the lab can be found here:

http://www.ehs.ucr.edu/ehsacademy/presentations/ergonomicslaboratorychecklist.pdf.

II. Emergency Preparedness

A. ***Fire Extinguishers, Fire Blankets, Safety Showers, and Eyewash Facilities***

Fire Extinguishers

The University of Utah does not require that personnel extinguish fires that occur in their area, though under certain circumstances, researchers who have been trained to use extinguishers can put out fires. Personnel are not allowed to use extinguishers unless they have been properly trained to do so. Contact the University Fire Marshal at 801-581-6590 for information on the next extinguisher training session.

Fire extinguishers are located in the hallways (A/B/C). All extinguishers are electronically monitored. Any time an extinguisher is used, contact Facilities Maintenance Fire Prevention Shop at 801-581-7221 so that it can be inspected and recharged.

There are various types of fires; those pertinent to the chemistry department are:

A (for ash): fires that are comprised of solid combustibles, including paper/trash can fires

B (for barrel): flammable liquids/gases, including solvents

C (for current): electrical fires

D (for dynamite): metal fires (note, requires a different extinguisher!)

There are several types of extinguishers that can be used to put out different types of fires.

Carbon Dioxide extinguishers are effective against Class B and C fires (burning liquids and electrical fires). They are less effective for type A fires (burning paper), and should not be used for type D fires (burning metals and pyrophoric organometallic/main group reagents). These extinguishers are clean, leaving no chemical residue behind.

Dry Powder extinguishers are effective against Class A, B and C and should not be used for type D fires. They contain sodium bicarbonate and leave a powder behind.

Met-L-X extinguishers are effective for class D fires. These are available upon request from OEHS.

Sand can be used to smother any type of fire, and is particularly useful for putting out small type D fires. Note, smothering type D fires does not quench the pyrophoric material, which must still be properly quenched (see Appendix F).

To put out a fire, using an extinguisher, remember PASS: Pull, Aim, Squeeze, Sweep. A/B/C extinguishers should be aimed in front of the flames, whilst D extinguishers should be aimed on top of the flames.

Fire Blankets

Fire blankets are located in certain labs, but are not recommended as a first aid measure; smothering a person on fire can result in more severe burns. If a person is on fire, it is recommended that they be moved to the emergency shower immediately and/or for the burning clothes removed.

Emergency Showers and Eyewash Stations

Emergency showers are located within all labs, and should be checked periodically by the group safety officer to ensure proper function. Issues should be reported to Facility Operations at 801-581-7221. Modesty should never be an issue in an emergency, and all researchers should devise a plan that they have discussed with their lab peers/PI on what they will do if they must strip and use the safety shower (clothes on fire, major chemical exposure to skin, etc.). For instance, it may be that all those of the opposite gender leave and get help, while those of the same gender use the fire blanket as a makeshift curtain. Personnel are encouraged to keep a spare change of clothes in the office for these instances.

Eyewash stations are located near all safety showers, and should be checked weekly by the group safety officer to ensure proper function and to flush out any bacteria that grow in stagnant water. Issues should be reported to OEHS. Note, eyewash stations will not necessarily flush out contact lenses, so it is up to each researcher to notify the rest of the research group that he/she wears contact lenses. This information can readily be stored as part of the emergency contact information.

B. General Emergency Preparation

- Every group should have a safety officer/fire warden, whose responsibility is to familiarize lab members with emergency and evacuation procedures, carry out monthly visual inspections of the safety equipment, and conduct “sweeps” of the working area during evacuations. They also keep track of emergency contact info.

- *All lab members MUST have cell phone #s of every group member programmed in their cell phones*. Note: University policy is that all labs must have functioning land line telephone in, or close to, the lab.

- Every group member should be trained on using the safety equipment in the lab as part of the initial safety training to work in the group.

- For work with dangerous chemicals or procedures, researchers must be trained by appropriate personnel and checked out by the safety officer/PI. This must be documented within the group.

- Every lab should post a map of where safety equipment is on lab doors. This includes spill kits, fire extinguishers, first aid kits, safety showers, eye wash stations, and fire blankets.

- Every lab should have Campus Emergency Flip Chart from OEHS. Updated flipcharts can be requested from OEHS (our tentative contact is Matt Lundquist matt.lundquist@oehs.utah.edu.)

- Under no circumstances should exits be blocked (in the lab or hallways).

- Each lab should have an up to date hazard warning sign posted, with numbers of who to contact in case of an emergency. To request new signage go to http://oehs.utah.edu/resource-center/forms/hazard-warning-signage-questionnaire

- Additionally, each lab should have up-to-date emergency contact information. It is highly recommended that this information be shared with neighboring research groups. These lists should be kept in an envelope taped to the door so that they are easily accessible when evacuating.

- Always be prepared with what you are doing in the lab- know the hazards, minimize your exposure to chemicals, do not under-estimate the risks, and be prepared for accidents.

- Hood sashes should list what is going on in the hood (reactions), with hazards and what to do in case of an emergency, and/or notebook name/number/page. Hoods should also have a phone number for who to call in case of an emergency. Alternatively, an open notebook page detailing this information near the hood is also appropriate.

- Do not work alone- if you must, have someone check in on you or call you periodically and NEVER use chemicals that have increased hazards, such as pyrophorics, etc.

C. ***Emergency Procedures***

- To summon emergency police, fire, or ambulance, call campus police at 911 or 9-911 from a campus phone. Report the location of the emergency (building and room), and be as specific as possible about nature of emergency and what is needed. If you are unsure, you may get a “full force” response.

- You can call OEHS for advice and counsel. OEHS personnel are available 24/7 – during normal business hours by calling 801-581-6590 and after hours by calling UUPD dispatch at 801-585-2677 and asking them to page OEHS Occ Hygiene on-call.

- Notify other workers in the area of the nature of the emergency, if necessary, activate the fire alarm to order evacuation of the building.

- If a coworker has ingested a toxic substance, follow the first aid information on the SDS and seek medical attention.

- If a coworker is bleeding profusely, elevate the wound above the level of the heart and apply pressure with a clean cloth or your hand. Seek immediate medical assistance. Note - if there is glass shrapnel in wound, do not remove.

  Do not touch a person in contact with a live electrical circuit- disconnect the power first.

D. ***Evacuation***

Departmental Evacuation Plan

Follow the departmental guideline for evacuation and meeting points. (to be added)

Assessing the lab prior to evacuation

During an evacuation, personnel should assume that they will not return for several hours. Therefore, it is the responsibility of lab personnel to ensure that the lab is safe. This may require instruments be turned off, reactions placed under inert atmospheres, chemicals be put away, and reactions/procedures stopped (for example, distillations) as long as it is safe to do so.

E. ***Power Outages***

In the case of power outages, it is advised that personnel secure the lab and evacuate. Researchers should ensure that chemicals are properly stored, reactions/equipment in a stable state, etc. It may be advised to unplug equipment if power surges are a concern.

F. ***Treatment of Injured or Contaminated Persons***

- Ingestion: Call emergency personnel (911). Do not encourage vomiting except under the advice of a physician. Call the Poison Control Center **(800-222-1222)** immediately and consult the SDS for the appropriate action. Save all chemical containers and a small amount of vomitus, if possible, for analysis. Stay with the victim until emergency medical assistance arrives. Send a copy of the MSDS with the victim.

- If a chemical gets in the eye of a victim, flush for a minimum of 15 minutes with eyewash station. Note that soft contact lenses will not necessarily be flushed out, so the victim (or you!) may have to remove contact lenses (hard or soft). Hold the individual's eyelids away from the eyeball, and instruct him or her to move the eye up and down and sideways to wash thoroughly behind the eyelids. Follow first aid by prompt treatment by medical personnel or an ophthalmologist who is acquainted with chemical injuries. Send a copy of the MSDS with the victim. Note: these eye wash stations should be flushed weekly to ensure proper function.

- For spills covering small amounts of skin: immediately flush with water for at least 15 minutes. If there is no visible burn, wash with water and soap, and remove any jewelry/clothing in the area. Check the SDS to see if any delayed effects should be expected (example). It is advised to seek medical attention for even minor chemical burns.

- For spills on clothes: The emergency responder should wear appropriate PPE during emergency treatment to avoid exposure. Do not attempt to wipe the clothes. To avoid contamination of the victim's eyes, do not remove the victim's eye protection before emergency treatment. Quickly remove all contaminated clothing, shoes, and jewelry while using the safety shower. Seconds count; do not waste time or limit the showered body areas because of modesty. Take care not to spread the chemical on the skin or, especially, in the eyes. Cut off garments such as pullover shirts or sweaters to prevent spreading the contamination, especially to the eyes. Immediately flood the affected body area with water for at least 15 minutes. Resume if pain returns. Get medical attention as soon as possible. The affected person should be escorted and should not travel alone. Send a copy of the SDS with the victim. If the institution's SDS is digital, hardcopies of the relevant information should be provided to responders. If the SDS is not immediately available, it is vitally important that the person in charge convey the name of the chemical to the medical personnel.

- *In the event that use of a shower or eyewash is warranted we recommend that someone call 911 immediately – that way medical personnel should be on scene by the time the 15 minute flush is done.*

Additional information on handling emergency situations is outlined in Prudent Practices page 117.

G. __Leaking Gas Cylinders__

- Occasionally a gas cylinder or one of its components will develop a leak (usually at the valve threads, safety device, valve stem, and valve outlet). If you suspect a leak, do not use a flame for detection; use soapy water. If the leak cannot be remedied by tightening, contact OEHS.
- Note, when using gas cylinders, it is critical that you use regulators with the correct CGA number (no exceptions!) to avoid unnecessary leaks, injury, damage to tank, and $$. See Appendix G for a table of CGA Connection numbers.
- When attaching a regulator, do not use Teflon tape.
- Note, gases that pose toxic hazards (such as CO) should have detectors attached to the tank ($20 at Home Depot for CO detector).
- For minor leaks, do the following:

- Flammable, inert, or oxidizing gases: Move the cylinder to an isolated area (away from combustible material if the gas is flammable of an oxidant) and post signed describing the hazards/state warnings. If possible, move the cylinder into a fume hood. Call OEHS for assistance if needed (they have a cylinder leak response kit that can handle most leaks).
- Corrosive gases: May increase the size of the leak as they are released and some are also oxidants or flammable. Move the cylinder into an isolated, well-ventilated area and use suitable means to direct the gas into an appropriate chemical neutralizer. Post signs that state the hazards and warnings. Note: for these gases, always use a regulator with a cross-purge assembly (for an inert gas) to minimize the risks of developing leaks in the regulator. Also, promptly flush out/remove regulator (esp. if without a cross-purge assembly) from tank, to avoid permanently having regulator on tank (example). Corrosives regulators are very $$$$!
- Toxic gases: Follow the same procedure as for corrosive gases.

- If the nature of the leaking gas or the size of the leak constitutes a more serious hazard, evacuate personal and call campus police from a distance to obtain emergency assistance (pull the fire alarm if a MAJOR leak).

H. <u>Fires</u>

Researchers should be familiar with the types of fires: A, B, C, D (see section 8). Only researchers who have completed fire extinguisher training should attempt to use a fire extinguisher. If a researcher is not trained or is not comfortable putting out the fire, the fire alarm should be pulled. Fires that typically can be put out by researchers include:

- Fires from quenching small amounts of pyrophoric reagents
- Trash can fire (move to an empty hood before extinguishing)
- Fires in hoods (if large or there are many flammables present, lower sash and call for help).

Contact the Fire Marshall (801-585-9122) to set up a fire extinguisher training.

I. <u>Working with Cryogenics</u>

Using cryogenic fluids improperly may produce physical and personal hazards that are not always obvious. The primary hazard to people is skin or eye contact with splashing liquid as it warms and expands. Injuries similar to a burn will result. Safety goggles or a face shield should be worn. Clean, insulated gloves that can *easily* be removed is suggested.

All cryogenic fluids are capable of causing asphyxiation without warning by displacing oxygen-containing air. Areas where they are used or stored should be adequately ventilated. These fluids should not be used in closed rooms or other enclosed spaces. Also, cryogenic fluids are capable of condensing oxygen from the air, causing oxygen enrichment or oxygen entrapment in confined spaces, which may result in increased flammability and subsequent explosion hazard.

Liquified gases are generally stored at atmospheric pressure in an insulated container, which keeps them near their boiling point, with some gas present. The large expansion in volume that takes place when the liquid becomes a gas means that pressure can build up in an unvented or unrelieved container and in transfer lines and piping. System design and maintenance must take this expansion ratio into account. Only containers designed for cryogenic fluids should be used. The selection of materials to be used with cryogenics is important because of the changes in physical properties of materials at very low temperatures. Some materials become extremely brittle. Chemical interactions between the cryogenic liquid and its container or equipment must also be evaluated.

The Dewar flask is the most common container used for storage and transfer of cryogenic fluids. When using the Dewar, follow these procedures.

- Cover the Dewar with a cap that allows escape of built-up pressure and keeps air and moisture out.
- Transfer cryogenic liquids from large Dewar vessels with special transfer tubes designed for the particular application.
- Tipping or tilting to pour the liquid may damage large Dewars.
- Do not use heat guns or similar equipment to warm transfer tubing quickly for disconnection.
- Handle containers carefully to protect the vacuum insulation system of Dewars.
- Place large Dewars on dollies that move freely so there is no possibility of personal injury or damage to the supported Dewars.

If there is a cryogenic spill, immediately leave the area. If you believe the cryogen has caused significant oxygen depletion, do not re-enter the area unless the oxygen content of the atmosphere is at least 19.5% and there is no flammable or toxic mixture present.

Cold Traps

- When using a cryogenic fluid in a Dewar for use on a vacuum line, it is imperative that the system be under vacuum when the Dewar is set in place.
- Only place dewars around traps after the system has been leak checked, and is under vacuum.
- Cold traps should be checked frequently/re-filled and not left unattended for extended periods of time.
- To remove dewars, isolate the traps from your line and vacuum, drop the dewars and vent the system. Place dewars in the back of a hood and allow trapped solvent to thaw.
- Important things to remember: never open a cold trap being cooled with a cryogen to the atmosphere as you will condense liquid oxygen.

Vacuum Transfers

Vacuum transfers and distillations often use cryogen cooled receiving flasks to increase the efficacy and speed of transfer. In order to prevent the condensation of liquid oxygen, take the following precautions:

- Verify that there are no leaks in the line (check the vacuum gauge / monometer). Never use a cryogen cooled receiving flask on a system with a leak.
- Monitor the pressure of the system under static vacuum – leaks not only allow air to enter the system, but also slow the transfer of solvents.

- Never leave your vac transfer unattended for extended periods of time.

Emergency Procedures

In the event of a large cryogen spill, immediately leave the area until oxygen in the atmosphere is at least 19.5% in order to avoid asphyxiation hazards.

If you suspect your cold trap has condensed liquid oxygen, you (and everyone in your vicinity) are in immediate danger. Make sure that the hood sash is down, or that a blast shield is in front of the flask if the operation is not being performed in a hood.

1. Notify all lab occupants and your supervisor, and place a blast shield in front of the traps (even if in the hood).
2. Inspect the system, taking as many precautions as possible.
3. Try to ensure that the liquid nitrogen dewar in which the flask resides has ample liquid nitrogen in order to maintain a temperature of ~ -196 ^{o}C. This is to ensure that the Dewars are filled with plenty of cryogen to ensure the liquid oxygen is kept at an adequately low temperature. Warming of the liquid oxygen could result in pressure buildup and/or a violent oxidation reaction (if organics are present).
4. At this stage, you have several options:
    a. Further venting the system to the open atmosphere is not advised, as this provides an ample supply of oxygen to the system, which may exacerbate the problem. If there are organics present, an explosion may result.
    b. After ensuring that the dewar is filled with liquid nitrogen, try to locate the leak. If it can be fixed (as in a valve was left open, etc.), quickly seal the leak and put the system under dynamic vacuum (if not already so). If you cannot seal the leak effectively (such as if a valve broke), clear the area immediately and notify your supervisor. Do not re-enter the hood until the liquid oxygen has been pumped out of the cold trap (this may take several hours).
5. Your vacuum gauge/ monometer will indicate if you have fixed the leak and if liquid oxygen has been pumped off (changes by about 100 mm Hg). Evacuate the area. The entire system should be regarded as an explosive danger until no liquid oxygen remains (e.g., the vacuum is returned to its best reading).
6. Once the liquid oxygen is removed, get help from your advisor or another qualified researcher. Remove the dewar, then detach the flask from the line and allow it to warm slowly behind a blast shield.

If you suspect your cold trap has condensed liquid oxygen in the presence of an organic compound, the risk and extent of explosion is amplified. Follow the procedure outlined above, and notify the campus emergency service, at 4911. Absolutely ensure all lab occupants are evacuated, and that all entrances to the lab are closed off.

Date/initials of student and PI next to each task once complete. Space is provided to give details of the training.

III. Working with Lasers (from Morse Group, Department of Chemistry)

Introduction

The purpose of this document is to inform personnel who will be using Class 3b or 4 lasers within this laboratory of the hazards and necessary safety procedures surrounding the use of such equipment. It is extremely important to be safe when operating or present near lasers, especially those classified as Class 3b or 4, as they can produce injuries (including burns, blindness, and electrocution) if improperly used.

What is a laser?

LASER is an acronym that stands for Light Amplification by Stimulated Emission of Radiation. The radiation emitted by the laser is in or near the optical portion of the electromagnetic spectrum, and is emitted in a collimated beam. Energy is amplified to extremely high intensity by a process called stimulated emission. The term "radiation" is often misinterpreted because this term is also used to describe the ionizing radiation that is emitted by radioactive materials. The electromagnetic radiation emitted by lasers is quite different from that emitted by radioactive materials, and is generally not considered to be ionizing radiation. As a result, the hazards associated with laser radiation are completely different than those associated with radioactive materials. As used in the context of a laser, radiation refers to the transfer of energy in the electromagnetic field. Energy moves from one location to another by conduction, convection, and radiation. In a laser, the energy transfer only occurs through radiation. The color of laser light is normally expressed in terms of the laser's wavelength. The most common unit used in expressing a laser's wavelength is the nanometer (nm). There are one billion nanometers in one meter (1 nm = 1×10^{-9} m). Laser light is nonionizing and includes ultra-violet (100-400 nm), visible (400-700 nm), and infrared (700 nm-1 mm).

Electromagnetic Spectrum

The electromagnetic spectrum is vast, ranging from the short wavelength gamma rays to the long wavelength radio waves. Most importantly, the visible portion of this spectrum is very small. The vast majority of the electromagnetic spectrum is invisible to the human eye, which can lead to safety concerns, depending on the type of radiation.

Classification of Lasers

Lasers are divided into a number of classes depending upon the power or energy of the beam and the wavelength of the emitted radiation. Laser classification is based on the laser's potential for causing immediate injury to the eye or skin and/or potential for causing fires from direct exposure to the beam or from reflections from diffuse reflective surfaces. Since August 1, 1976, commercially produced lasers have been classified and identified by labels affixed to the laser. In cases where the laser has been fabricated in house or is otherwise not labeled, Radiation Safety should be consulted on the appropriate laser classification and labeling. Lasers are classified using physical parameters of the laser, power, wavelength, and exposure duration.

- **Class 1 Lasers**
    - Class 1 lasers are considered to be incapable of producing damaging radiation levels, and are therefore exempt from most control measures or other forms of surveillance.

- Example: Laser printers and CD players.

- **Class 2 Lasers**
  - Class 2 lasers emit radiation in the visible portion of the spectrum, and protection is normally afforded by normal human aversion response (blink reflex) to bright radiant sources. In general, the human eye will blink within 0.25 seconds when exposed to Class 2 laser light. This blink reflex provides adequate protection. However Class 2 lasers emit laser light in the visible range and are capable of creating eye damage through chronic exposure.
  - Examples: Laser pointers, surveying lasers.

  - Class 2a lasers are special-purpose lasers not intended for viewing. Their power output is less than 1 mW. This class of lasers causes injury only when viewed directly for more than 1,000 seconds. The 1,000 seconds is spread over an 8-hour day, not continuous exposure.
  - Example: Many bar-code readers fall into this category.
- **Class 3 Lasers**
  - Class 3a lasers are those that normally would not produce injury if viewed only momentarily with the unaided eye. They may present a hazard if viewed using collecting optics, e.g., telescopes, microscopes, or binoculars.
  - Example: HeNe laser above 1 mW but not exceeding 5 mW radiant power, or some pocket laser pointers.

  - Class 3b laser light will cause injury upon direct viewing of the beam and specular reflections.
  - Example: Visible HeNe laser above 5 mW but not exceeding 500 mW radiant power.

- **Class 4 Lasers**
  - Class 4 lasers include all lasers with power levels greater than 500 mW radiant power. They pose eye hazards, skin hazards, and fire hazards. Viewing of the beam and of specular reflections or exposure to diffuse reflections can cause eye and skin injuries. All of the control measures explained in this training must be implemented.
  - Example: Most Nd:YAG Lasers, Excimer Lasers, pulsed dye lasers.

<u>Laser Beam Hazards</u>

The laser produces an intense, highly directional beam of light. If directed, reflected, or focused upon an object, laser light will be partially absorbed, raising the temperature of the surface and/or the interior of the object, potentially causing an alteration or deformation of the material. These properties, which have been applied to laser surgery and materials processing, can also cause tissue damage.

In addition to these obvious thermal effects upon tissue, there can also be photochemical effects when the wavelength of the laser radiation is sufficiently short, i.e., in the ultraviolet or blue region of the spectrum. Today, most high-power lasers are designed to minimize access to laser radiation during normal operation. Lower-power lasers may emit levels of laser light that are not a hazard.

The human body is vulnerable to the output of certain lasers, and under certain circumstances, exposure can result in damage to the eye and skin. Research relating to injury thresholds of the eye and skin has been performed in order to understand the biological hazards of laser radiation. It is now universally accepted that the human eye is more vulnerable to injury than human skin. It is useful to understand the damage that can occur to the human eye as a result of laser exposure, which depends significantly on the wavelength.

190-315 nm:

The cornea (the clear, outer front surface of the eye's optics), unlike the skin, does not have an external layer of dead cells to protect it from the environment. In the deep ultraviolet regions of the optical spectrum (190-315 nm), the cornea absorbs the laser energy and may be damaged. Indeed, radiation of these wavelengths is used for laser radial keratotomy, which is the reshaping of the cornea to improve vision by correcting the refraction of the eye, so that images are focused on the retina. At much lower exposures than is used in radial keratotomy, the cornea can become inflamed (keratitis), leading a delayed sensation of sand in the eyes 6-12 hours after exposure. This condition is typically treated by rest and usually heals after 24-48 hours. Exposure of the skin to wavelengths of 190-315 nm leads to a "sunburn", which is not typically any more serious than a sunburn caused by exposure to sunlight.

315-400 nm (UV A radiation):

In the 315-400 nm range, the lens of the eye may be vulnerable to injury. Exposure to these wavelengths may increase the risk of cataract formation in the lens of the eye.

400-1400 nm:

Of greatest concern is laser exposure in the retinal hazard region of the optical spectrum, approximately 400 nm (violet light) to 1400 nm (near-infrared) and including the entire visible portion of the optical spectrum. Within this spectral region collimated laser rays are brought to focus on a tiny spot on the retina. In order for the worst case exposure to occur, an individual's eye must be focused at a distance and a direct beam or specular (mirror-like) reflection must enter the eye. The light entering the eye from a collimated beam in the retinal hazard region is concentrated by a factor of 100,000 times when it strikes the retina.

Therefore, a visible, 10 mW/cm^2 laser beam would result in a 1000 W/cm^2 exposure to the retina, which is more than enough power density (irradiance) to cause damage. If the eye is not focused at a distance or if the beam is reflected from a diffuse surface (not mirror-like), much higher levels of laser radiation would be necessary to cause injury. Since this ocular focusing effect does not apply to the skin, the skin is far less vulnerable to injury from these wavelengths.

Within this wavelength range, pulsed lasers present serious hazards to the eye. In the Morse group, we work with lasers having a 5 ns pulse duration, which is far shorter than the blink reflex (about 0.25 s). Thus, the entire pulse is focused onto the retina before the blink reflex kicks in. Further, the focusing effect magnifies the danger, so that even weak partial reflections from a flat surface (such as a prism or clear window) become quite hazardous. In addition to burns to the retina, shock and acoustic waves caused by the focused radiation can also cause damage, including retinal detachment. For this reason, it is essential that all reflections or partial reflections should be blocked in the lab, and that laser beam blocks should always be left in place. Reflected beams having energies as low as 25 μJ have been known to cause permanent eye damage, so extreme care is required in blocking all specular reflections from surfaces such as prisms, optical filters, lenses, and windows.

Non-Beam Hazards

In addition to the direct hazards to the eye and skin from the laser beam itself, it is also important to address other hazards associated with the use of lasers. These non-beam hazards, in some cases, can be life-threatening, e.g. electrocution, fire, and asphyxiation. The only fatalities from lasers have been caused by non-beam hazards.

- **Chemical Hazards:**
    - Compressed Gases – careful handling of compressed gas tanks is important. Reinforce all tanks by attaching them to sturdy objects, e.g. walls or other fixed objects. Poisonous gases must be housed within a well-ventilated cabinet whose exhaust is piped into the fume hood exhaust.
    - Laser Dyes and Solvents – Many dyes, and some solvents, used in this lab are toxic, carcinogenic, or mutagenic. Even if it is not known whether a dye or solvent falls within one of these categories, it should always be assumed that it does. Take care when handling these substances, wearing gloves and safety glasses to prevent splashes to the eyes. Take extra caution when using dimethylsulfoxide (DMSO) as a solvent. This solvent is known to carry dissolved substances (such as laser dyes) through the skin and into the bloodstream. Make sure to dispose of any used dyes and solvents appropriately by collecting them in labeled containers and arranging for EHS (Environmental Health & Safety) to pick them up.
- **Electrical Hazards**
    - Shock and electrocution are the primary lethal hazards associated with lasers and laser systems, especially their high voltage power supplies. In order to avoid electrical hazards, care should be taken during servicing, testing, modification, maintenance, or any other activity that requires contact with energized components of the laser system. Electrical protection parameters, connection to the utilization system, and safety training should comply with OSHA, the National Electric Code (NEC), National Fire Protection Association (NFPA), and any other applicable state and local laws and regulations.
    - Class 3b and 4 lasers should have a separate circuit and local disconnect switch for the circuit.
    - It is good practice to have at least two persons in an area while working on high-energy power systems.
    - Keep cooling water connections away from main power and high voltage outlets and contacts. Use double hose clamps on cooling water hoses. Inspect cooling water hoses and connections, and power cables and connectors periodically as part of a regular equipment inspection.
    - In labs where laser power supplies are opened or serviced by lab personnel, staff should be trained in cardiopulmonary resuscitation.
- **Fire Hazards**
    - Fires can result from contact of a laser beam with flammable materials, unprotected wire insulation, and plastic tubing. While class 4 laser beams present the highest hazard potential for fire (irradiance exceeding 10 W/cm^{-2} or beam power above 0.5 W) class 3b lasers may pose a hazard under some conditions. Each of the laser laboratories in the Morse group is equipped with an emergency power shutoff switch and a fire extinguisher. Personnel working in the labs should know the location of these items and how to use them in the event of an emergency.

Eye Protection

Laser protective eyewear is specific to the types of laser radiation in the lab. Each laser laboratory must provide laser-specific appropriate eye protection for persons working with the laser. Windows where Class 2, 3, or 4 beams could be transmitted causing hazards in uncontrolled areas shall be covered or otherwise protected during laser operation. The following guidelines are suggested for maximum eye protection.

- Whenever possible confine (enclose) the beam and provide non-reflective, nonflammable beam stops, to minimize the risk of accidental exposure or fire. Use fluorescent screens or secondary viewers to align the beam; avoid direct beam exposure to the eyes.

- Use the lowest power possible for beam alignment procedures. Use lower class lasers for preliminary alignment procedures, whenever possible. Keep optical benches free of unnecessary reflective items.
- Confine the beam to the optical bench unless necessary for an experiment, e.g., use barriers at side of benches or other enclosures. Do not use room walls to align Class 3b or 4 laser beams.
- Use non-reflective tools. Remember that some tools seem to be non–reflective for visible light, but may be reflective in the invisible part of the spectrum.
- Do not wear reflective jewelry when working with lasers. Metallic jewelry also increases electrocution hazards.

Wear protective glasses whenever working with Class 4 lasers with open beams or when unblocked reflections can occur. This is especially important when working with infrared beams (such as the Nd:YAG fundamental), since the unblocked reflections cannot be seen, and are therefore difficult to locate and block.

Generally, protective eyewear may be selected to be adequate to protect against stray reflections. Wearing such glasses allows some visibility of the beam, preventing skin burns, making it more likely that persons will wear the eye protection. Also, the increased visibility afforded by this level of protection decreases potential for other accidents in the lab, i.e., tripping, etc.

Factors to consider in selection of Laser Protective eyewear include the following:

- Wavelength(s) or spectral region(s) of laser radiation
- Optical density at the particular wavelength(s)
- Maximum irradiance (W/cm^2) or beam power (W)
- Type of laser system
- Power mode, single pulse, multiple pulse, or cw
- Possibilities of reflections, specular and diffuse
- Field of view provided by the design
- Availability of prescription lenses or sufficient size of goggle frames to permit wearing of prescription glasses inside of goggles.
- Comfort
- Ventilation ports to prevent fogging
- Effect upon color vision
- Impact resistance
- Ability to perform required tasks while wearing eyewear

Since laser protective eyewear is subject to damage and deterioration, the lab safety program should include periodic inspection of these protective items.

Engineering Controls for Laser Systems

In the Morse group, lasers shall not be modified to defeat the engineering safeguards without review and approval of the PI, or other authorized personnel, to ensure that appropriate controls are instituted.

Appropriate design standards for laser system are as follows:

- Laser should be equipped with a protective housing, an aperture that is clearly identified, and a clearly marked switch to deactivate the laser or reduce its output to less than maximum permissible exposure (MPE). If this is not possible, Radiation Safety should be consulted to assess the hazards and to ensure that appropriate controls are in place. Such controls may include, but are not limited to the following:
    - Access restriction
    - Eye protection
    - Barriers, shrouds, beam stops, etc.

    - Administrative and/or procedural controls
    - Education and training
- Protective housings should be interlocked for Class 3a, 3b and 4 lasers.
- A keyed master switch or password protected operating computer should be provided for Class 3b and 4 lasers. Lasers should be disabled by removing the key when the laser is not in use for prolonged periods.
- Viewing ports and collecting optics shall provide adequate protection to reduce exposure at viewing position to below the MPE level. (Classes 2, 3a, 3b, or 4).
- If the beam path is not enclosed, then the Nominal Hazard Zone (NHZ), the areas where the exposure level exceeds maximum permissible exposure level, need to be assessed and a controlled area established.
- Commercially manufactured Class 3b and Class 4 lasers must come equipped with a connection for external interlocks.
- Laser beams should be terminated in a suitable “beam stop.” Most laser heads come equipped with a permanently attached stop or attenuator, which will lower the beam power to less than the MPE at the aperture from the housing. Additional beam stops may be needed in the beam path to keep the useful beam confined to the experimental area.

Control of Laser Areas

Laser labs are to be locked when lasers are operating, with warning signs on the doors. Only personnel who have received laser training in the group will be issued keys to these laboratories. Laser beams will be restricted so that they never are accessible within a minimum distance of 6 feet from the door. Windows into laser labs will be blocked with opaque materials.

For Class 4 lasers that have open beams, the ANSI Standards call for interlocked doors or devices that turn-off or attenuate the laser beam in the event of an unexpected entry into an area. An alternative method of protection is to provide a suitable barrier (screen or curtain) just inside the door or wherever most appropriate to intercept a beam or scatter so that a person entering the room cannot be exposed above the MPE limits. In the Morse group, a barrier will be present to prevent laser beams (and reflected beams) from reaching the doorway area.

Other conditions related to control of laser areas include the following:

- Never direct the laser beam toward the entry.
- Use shields and barriers around the laser work area so that the beam, reflections and scatter are contained on the optical table. Try to keep the unenclosed beam path out of the normal eye-level zone. (The normal eye-range is from 4 to 6 feet from the floor.)
- Ensure that only diffuse reflection materials are in or near the beam path to minimize the chance of specular reflections. When this is not possible, locate and block all specular reflections with suitable beam blocks.
- Ensure that locks or interlocks do not prevent rapid egress from the area in the event of an emergency situation.

Unauthorized persons are to be prevented from entering the laboratories by keeping the doors closed and locked when the lasers are in use. The locks on the doors will be such that they do not impede egress from the laboratory.

Posting and Warning Systems for Laser Controlled Areas

Entrances to laser areas are to be posted in accordance with ANSI Z136.1-2000. In particular, areas where Class 3b or 4 lasers are used must be secured against persons accidentally being exposed to beams, and be provided with a proper warning indication. All windows, doorways, and portals should be covered or restricted to reduce transmitted laser levels below the MPE.

Personnel who do not read the English language, and who may need to enter areas where lasers are used are to be given appropriate instruction as to the meaning of warning signs and labels.

All visitors to the operating laser labs will be escorted by trained personnel while in the lab, in order to keep them safe at all times.

Training

Only qualified and trained employees may operate Class 3b and 4 lasers. To be qualified, a laser operator must meet the training requirements outlined below. The Principal Investigators are responsible for ensuring that all persons who work in areas where Class 3b or 4 lasers are used are provided with appropriate training and written safety instructions (work rules), so that the workers can properly utilize equipment and know and follow safety procedures.

For personnel who work with Class 3b and 4 lasers, the training will included the following topics:

- The biological effects of laser radiation
- The physical principles of lasers
- Classification of lasers
- Basic safety rules
- Use of protective equipment
- Control of related hazards including electrical safety, fire safety, and chemical safety
- Emergency response procedures

All alignment, operation, and maintenance procedures are to be trained by a qualified employee or learned by studying the respective laser manuals.

Because of the hazard of electrocution, it is a recommendation that the lab personnel take a course in cardiopulmonary resuscitation (CPR) and proper rescue techniques to follow in the event of electrocution.

An employee already qualified and trained with the laser system(s) must train any non-University personnel that may need to work on/with Class 3b or 4 lasers. A qualified employee will also closely supervise these individuals until they are comfortable with their level of expertise.

IV. Working with Gas Cylinders (Saouma Group Department of Chemistry)

Standard Operating Procedure

Task: Gas Cylinders
Created by: Saouma Group
Date: 05/08/2017
Revision Date (Author): Moumita Bhattacharya 05/09/2017

Training Requirements:

- ✓ Department of Chemistry Safety Training
- ✓ Saouma Group Safety Training
- ✓ Fully read and understand this SOP
- ✓ Checked out by a senior lab member that is trained in using gas cylinders

Potential Hazards:

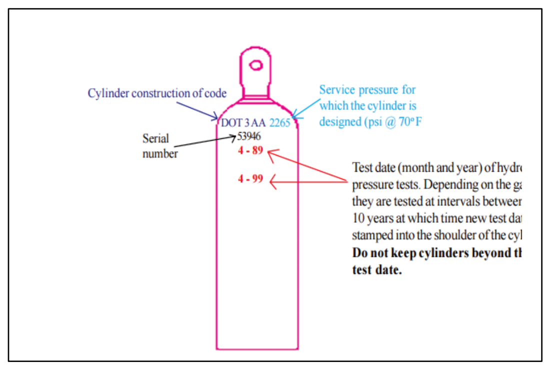
- Falling gas cylinders
- High pressure gases
- Potential toxic/flammable gases
- Fire (flammable gases)
- Stripping (tank or regulator)

Special PPE Requirements:

- Labcoat, safety glasses, nitrile gloves

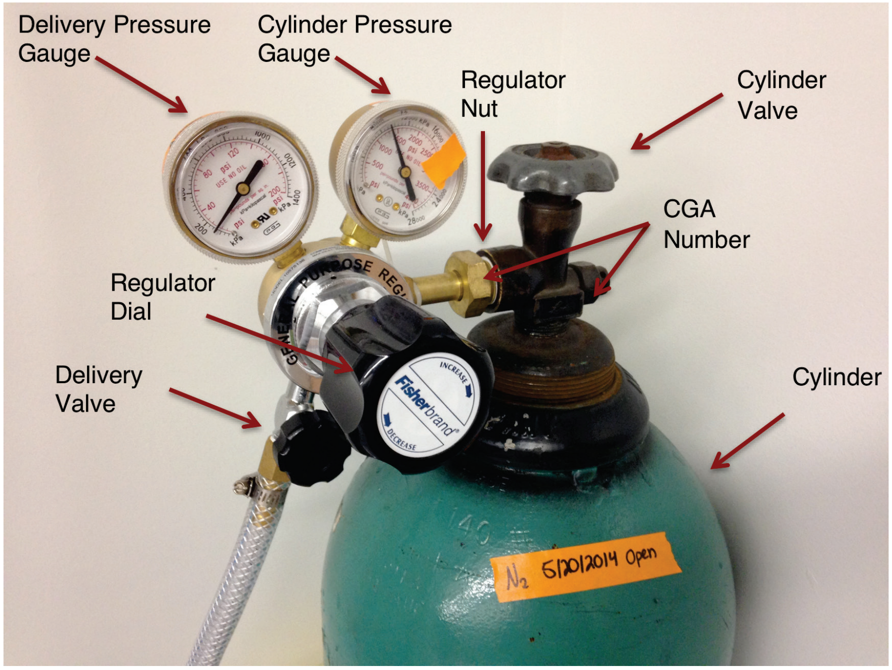
Materials Needed:

- Wrench
- Gas cylinder cart
- Straps
- Teflon tape
- Regulator
- Hoses, adapters, etc.
- Alligator clips for grounding (flammable gases)



Background:

- Users should understand basic regulator operation before manipulating gases at high pressure.
- The user should be familiar with the markings on the gas cylinder. The top mark is either a DOT or an International Code Council (ICC) marking indicating pertinent regulations for that cylinder. The second mark is the serial number. Under the serial number is the symbol of the manufacturer, user or purchaser. Of the remaining marks the numbers represent the date of manufacture, and retest date (month and year). A (+) sign indicates the cylinder may be 10% overcharged, and a star indicates a ten-year test interval.



Procedure:

Regulator Information

- Check the CGA numbers on *both* the cylinder and the regulator to make sure that they match. This number is usually printed on the flat portion of the cylinder directly below the valve. On most regulators, the number can be found printed on the regulator nut or underneath the dials. **Note**, *NEVER use a regulator that is incompatible with the gas you are using.*
- The cylinders and regulators for flammable gasses are reverse threaded to ensure that they are only used together. Usually, the nuts on these regulators are notched to indicate this.
- Common CGA numbers are in the table below. If a gas or gas mixture is not listed, refer to supplier to confirm the CGA number.
- For lecture bottles: CGA 170 (non-corrosive) or CGA 180 (corrosives).

PURE GASES CGA SELECTION CHART FOR FITTINGS

CGA Fittings Required	Pure Gases
510/300	Acetylene
590/346/347/702	Air
240/660/705	Ammonia
580/680/677	Argon
350	Arsine*
320	Carbon Dioxide
350	Carbon Monoxide
660	Chlorine
510	Cyclopropane
350	Deuterium
350	Ethane
350	Ethylene
510	Ethylene Oxide
580/680/677	Helium
350/695/703	Hydrogen
330	Hydrogen Chloride
330	Hydrogen Sulfide
580	Krypton
350/695/703	Methane
510	Methyl Chloride
580/680/677	Neon
580/680/677	Nitrogen
326	Nitrous Oxide
540/577/701	Oxygen*
350	Phosphine
510	Propane
350	Silane*
668/660	Sulfur Dioxide
590	Sulfur Hexaflouride
580/680/677	Xenon

MIXED GASES CGA SELECTION CHART FOR FITTINGS

CGA Fittings Required	Mixed Gases	
	Minor Component in	Major Component
240/660/705	Ammonia	Nitrogen
350	Butane	Nitrogen
296	Carbon Dioxide	Oxygen
580	Carbon Dioxide	Helium or Nitrogen
580	Carbon Dioxide and/or Nitrogen	Helium
590	Carbon Monoxide	Air
330	Chlorine	Nitrogen
350	Diborane	Argon, Helium, Hydrogen, Nitrogen
580	Freon-12	Nitrogen
296	Helium	Oxygen
350	Hexane	Nitrogen
350	Isobutane	Nitrogen
580	Krypton	Argon
590	Methane	Air
580	Moisture	Argon, Helium or Nitrogen
660	Nitric Oxide	Nitrogen
660	Nitrogen Dioxide	Air or Nitrogen
590	Nitrous Oxide	Nitrogen
590	Oxygen	Nitrogen or Helium
350	Propane	Nitrogen or Helium
590	Propane	Air
660	Sulfur Dioxide	Air or Nitrogen
590	Sulfur Hexaflouride	Argon, Helium or Nitrogen
350	Sulfur Hexaflouride	Hydrogen

Attaching a Regulator

- Make sure that the threads of both the regulator and the cylinder are free of Teflon tape and debris.
- Unless specifically noted, apply one complete wrap of new Teflon tape to the exposed threads in the direction of the thread.
- Tighten the nut while supporting the weight of the regulator. With your fingers.It should travel smoothly as long as the threads are matching correctly. Remember, righty tighty, lefty loosy. (reverse threads are opposite).
- Use a wrench to fully tighten the nut (usually another ¼ to ½ turn).
- If the regulator has not been used recently, or you are unsure of the pressure setting, turn the regulator dial all the way to the left so that it will be set to its lowest pressure delivery setting.

Leak Testing

- Once the regulator is attached, it should be leak tested. One simple method is to use an electronic leak detector. This is located in room 4163.
- Make sure that all tank and regulator valves are closed before you begin.
- If working with a flammable or toxic gas, ensure that the cylinder is in a well ventilated area and if applicable, that the person is wearing a detector.
- Turn on the leak detector and wait for it to equilibrate (about 10 seconds, light will shine a steady green).
- Open the tank valve on top of the cylinder. The cylinder pressure gauge should increase to ~ 2000 psi for most full cylinders. The delivery pressure gauge should read 0 psi. Make a note of the pressure - a piece of tape is a good method.
- Inspect the joint between the tank and regulator using the black and yellow probe. The detector will show red dots if a leak is detected; the number of dots corresponds to the severity of the leak.
- Open the regulator dial. Using the detector, inspect the connection and all local joints. Repeat for the delivery valve.
- If a leak is detected, try re-assembling the regulator using Teflon tape, if applicable.
    - **Note**, it an uncontrolled leak occurs that cannot be contained, contact OEH&S immediately!


Removing a Regulator

- Only remove regulators when the tank is not in use, and the *cylinder valve* is shut.
- Be sure to purge any residual gas into a hood (esp. if toxic, like CO): once the *cylinder valve* is closed, vent the residual gas through a bubbler or hose leading into a hood. The pressure gauge should read 0 psig for both cylinder and delivery pressure.
- Close the *delivery valve*.
- Using a wrench, loosen the regulator nut that connects the regulator to the tank. If there is any residual pressure in the regulator you will hear a hiss as the gas is released.
- Once loosened, untighten the regulator from the cylinder by hand. Take care to support the weight of the regulator with your hands, to minimnize the risk of stripping.
- Cap the cylinder, and put the regulator away.

Moving Cylinders

- Cylinders can only be moved when properly capped! If a regulator must remain attached, then a special regulator protector safety cap must be installed.
- Bring the gas cylinder cart next to the cylinder you want to move.
- Unstrap the cylinder and carefully roll it onto the gas cylinder cart.
- Secure the cylinder with the chain, and move the cylinder to its storage area.
- Carefully tip the cart, unstrap the cylinder, and roll the cylinder to its storage location.
- Double strap the cylinder.

  If the cylinder is empty, promptly take it to the stockroom. If after hours, mark the cylinder with the letters “MT” or word “empty” and take it down the following day.

Storage

- When not in use, the cylinder must be stored with a cap and no regulator. It should be double strapped to a cylinder rack.
- If you have to use a cylinder and there is no nearby rack, you can either keep it strapped into the cart, or setup the portable cylinder racks. The cylinders must only be stored in this manner during active use.
- Cylinders should be stored in well ventilated areas, away from sources of heat and electricity.
- Oxygen should be stored away from flammable gases.

V. Working with Cryogenics (Saouma Group)

Standard Operating Procedure
Cryogenic safety
Created by: Saouma Group (Department of Chemistry)
Date: 05/11/2017

Example of SOP

Training Requirements:

- ✓ Department of Chemistry Safety Training
- ✓ Saouma Group Safety Training
- ✓ Fully read and understand this SOP

Potential Hazards:

- *Cold burns/frostbite*. Appropriate PPE includes safety googles, lab coats, closed toe shoes, and clothing that covers arms and legs. Insulated gloves which are easily removed can be worn, but are not recommended due to the high risk of cold burns (with liquid nitrogen). Likewise latex or nitrile gloves can capture splashes of cryogens and hold them close to the skin causing burns as the liquid expands as it warms.
- *Asphyxiation*. Inadequate ventilation poses asphyxiation hazards as oxygen containing air is displaced. Do not use cryogenic fluids in small, closed rooms or rooms with inadequate ventilation.
- *Explosion*. Many cryogenic fluids can condense oxygen from the air to generate liquid oxygen. Liquid oxygen is flammable and explosive; it is characterized by a light, clear blue color. When in combination with organic compounds, the explosive nature of liquid oxygen is exacerbated.
- *Pressure build-up*. AS fluids warm, they expand, which can result in explosions.
- *Embrittlement*. Materials become more brittle and are prone to breakade when cold.
- *Material contraction*. Different materials expand at different rates with temperature, so care must be taken to not break equipment upon cooling (or heating). In particular, Schlenk tubes with Teflon plugs should never be fully submerged in cryogenics, as this will result in a leak at the Teflon/glass interface, allowing for air (and oxygen) to enter and condense.

Background:

Cryogenic liquids are fluids with boiling points less than -73°C (-100°F). They are also characterized by a high volume-expansion ratio in the liquid to gas phase. Some physical properties of three reference cryogenic liquids are outlined in the table below.

Identity	**Boiling Point**	**Vapor Pressure (at -196°C)**	**Volume-Expansion Ratio (at 1 atm, 20°C)**
Nitrogen (*l*)	-195.8°C	730 mm Hg	1:696
Oxygen (*l*)	-182.9°C	150 mm Hg	1:861
Carbon Dioxide (*l*)	-78.5°C	1.33×10^{-8} mm Hg	1:553

Special PPE and Engineering Requirements:

- Labcoat, safety glasses, nitrile gloves
- Face shield (recommended)
- Dish towels (to open/close the lN_2 dewar)

Materials Needed:

- Wrench (for removing hose from IN_2 dewar)
- Blast shield (if suspect liquid oxygen)

Procedure:

IN_2 Dewar: Filling and Transport

1. When the large dewar is empty, take a wrench and remove the hose.
2. Take the dewar to the elevator, and be sure that the “Do not ride” sign is pointing out.
3. Send the elevator down to the stockroom. DO NOT RIDE THE ELEVATOR!
4. Go down the stairs with the elevator, ensuring no one gets on. This is easily done with multiple lab members, with someone on each floor. If you are not sure, you can send the elevator down one floor at a time, so you can keep resending it down.
5. Take the dewar off the elevator and to the stockroom. Ask them to refill it, and be sure that they give you the BULK price.
6. Once it is filled, the stockroom will put it in the hall. **Note**, at this time the dewar is very prone to venting.
7. Carefully place the dewar in the elevator, again with the “Do not ride” sticking out. If the dewar starts to vent upon moving, allow it to vent before closing the elevator door.
8. Send the dewar up to the fourth floor. As before, you must ensure that no one gets on the elevator.
9. Take the elevator to the lab, and tighten the hose with your fingers (do not wrench tighten as the dewar is cold!)

IN_2 Dewar: Taking IN_2

- Never touch any of the valves on the dewar other than that which dispenses. The other valves are for filling and venting, and are safety precautions!
- It is recommended that you use a small towel to handle valves, hoses, etc. that are cold- this minimizes the chance to trap cold close to your skin and cause burns.
- If the dewar begins to hiss, check the gauge- this usually indicates that the dewar is almost empty. If this is the case, then take it down to get refilled.

1. Place the small 4 L dewar at the end of the hose, and slowly open the dispensing valve on the dewar. At this time, the hose will “jump” as it is displacing warm air in the dewar (more so if the dewar has not been used in a while).

2. Once the dispensed nitrogen is staying as a liquid, you can slowly increase the flow rate.

3. When you have enough IN_2, close the valve and remove the smaller dewar.

Cold Baths

- When reactions must be run at reduced temperatures, use the appropriate cold bath. You should try to avoid toxic chemicals, and avoid the use of IN_2 if you can.
- See the table below. Note, for temperatures below -77 ^{o}C, you must use IN_2 rather than CO_2. Only use high quality IN_2, and ensure that the system remains open.

Temperature	Composition	Temperature	Composition
0°	Crushed ice		
-5° to -20°	Ice-salt mixtures	-77°	Solid CO_2 with chloroform or acetone
Up to -20°	Ice-MeOH mixtures	-78°	Solid CO_2 (powdered; CO_2 snow)
-33°	Liquid ammonia		
-40° to -50°	Ice (3.5-4 parts) - $CaCl_2 \cdot 6H_2O$ (5 parts)	-100°	Solid CO_2 with diethyl ether
-72°	Solid CO_2 with ethanol	-196°	liquid nitrogen (see footnote*)

Alternatively, the following liquids can be used, partially frozen, as cryostats, by adding solid CO_2 from time to time to the material in a Dewar-type container and stirring to make a slush:

13°	*p*-Xylene	-55°	Diacetone
12°	Dioxane	-56°	*n*-Octane
6°	Cyclohexane	-60°	Di-isopropyl ether
5°	Benzene	-73°	Trichloroethylene or isopropyl acetate
2°	Formamide	-74°	*o*-Cymene or *p*-cymene
-8.6°	Methyl salicylate	-77°	Butyl acetate
-9°	Hexane-2,5-dione	-79°	Isoamyl acetate
-10.5°	Ethylene glycol	-83°	Propylamine
-11.9°	*tert*-Amyl alcohol	-83.6°	Ethyl acetate
-12°	Cycloheptane or methyl benzoate	-86°	Methyl ethyl ketone
-15°	Benzyl alcohol	-89°	*n*-Butanol
-16.3°	*n*-Octanol	-90°	Nitroethane
-18°	1,2-Dichlorobenzene	-91°	Heptane
-22°	Tetrachloroethylene	-92°	*n*-Propyl acetate
-22.4°	Butyl benzoate	-93°	2-Nitropropane or cyclopentane
-22.8°	Carbon tetrachloride	-94°	Ethyl benzene or hexane
-24.5°	Diethyl sulfate	-94.6°	Acetone
-25°	1,3-Dichlorobenzene	-95.1°	Toluene
-29°	*o*-Xylene or pentachloroethane	-97°	Cumene
-30°	Bromobenzene	-98°	Methanol or methyl acetate
-32°	*m*-Toluidine	-99°	Isobutyl acetate
-32.6°	Dipropyl ketone	-104°	Cyclohexene
-38°	Thiophene	-107°	Isooctane
-41°	Acetonitrile	-108°	1-Nitropropane
-42°	Pyridine or diethyl ketone	-116°	Ethanol or diethyl ether
-44°	Cyclohexyl chloride	-117°	Isoamyl alcohol
-45°	Chlorobenzene	-126°	Methylcyclohexane
-47°	*m*-Xylene	-131°	*n*-Pentane
-50°	Ethyl malonate or *n*-butylamine	-160°	Isopentane
-52°	Benzyl acetate or diethylcarbitol		

For other organic materials used in low temperature slush-baths with liquid nitrogen see R.E.Rondeau [*J Chem Eng Data* **11** 124 *1966*]. ***NOTE**: Use high quality pure nitrogen; do not use liquid air or liquid nitrogen that has been in contact with air for a long period (due to the dissolution of oxygen in it) as this could EXPLODE in contact with organic matter.

Cold Traps

Appropriate order of operations when using cryogens in cold traps is critical to avoid generating hazards such as condensation of liquid oxygen – an explosion hazard. Cold traps should be checked frequently/re-filled and not left unattended for extended periods of time. When using a cryogenic fluid in a Dewar for use on a vacuum line, it is imperative that the system be under vacuum when the Dewar is set in place. A labelled scheme of a Schlenk line is shown below (Figure 1).

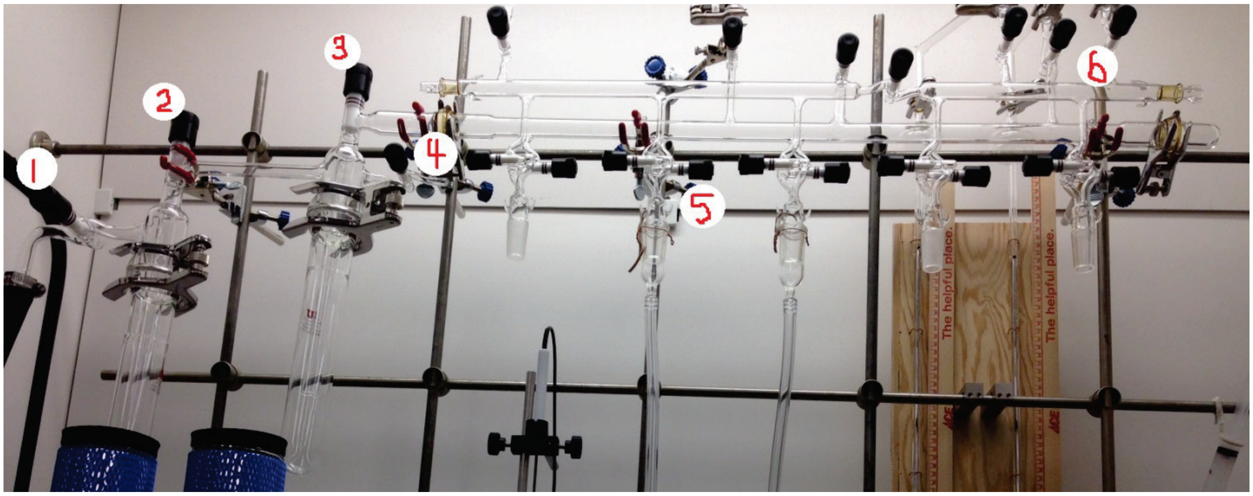


Figure 1: Labelled Scheme of a Schlenk Line.

To put a cryogen filled Dewar on to a schlenk line to create a cold trap, the individual valves (5) and the vent, 4, should be closed before starting. Valve 6 leading to the vacuum gauge and nitrogen bubbler should always be open, unless testing for leaks. Thus, the vacuum manifold should always be under static vacuum. To put up the traps, place a trap lower body/o-ring on the trap upper bottom closest to the vacuum (valve 1). Making sure that valve 2 is closed, valve 1 is then opened. Once you feel that the trap is securely in place and you no longer hear the pump grumbling, clamp the trap (finger tight). This trap is now under dynamic vacuum. With valves 3 and 4 closed, repeat the procedure for the second trap, now opening valve 2 to introduce dynamic vacuum. Finally, open valve 3 to expose you vacuum line/monometer to dynamic vacuum. Note if the mercury level changes0 it shouldn't if your line is holding vacuum. If it doesn't hold vacuum, leak test your line by closing valve 1 and noting any changes in the monometer level (over at least 10 minutes). Once you have established a good dynamic vacuum, place the dewars around the traps, then fill the Dewar with your cryogenic fluid. Check your vacuum gauge and ensure the line is holding vacuum. For most all applications, use liquid nitrogen. The exception is a dry ice/isopropanol mixture for drying celite, sieves, etc.

To remove your Dewars, start by closing the individual valve you were using. Systematically work back toward the pump in the following way: close valve 3 from the main line (creates static vacuum), close valve 2, quickly remove the cryogen filled Dewar, open valve 4 to vent, and carefully remove the cold trap (no longer under vacuum – don't drop it). Then proceed to the next trap: valves 2 and 3 are both closed and valve 4 is still open from the previous step, close valve 1 leading to the pump, remove the Dewar quickly, open valve 2 to the vent, and carefully remove the cold trap. Valve 1 will remain closed and under vacuum leading to the pump.

Important things to remember: never open a cold trap being cooled with a cryogen to the atmosphere as you will condense liquid oxygen (always double check your valves, especially vent valve 4 and individual vales, 5). Always work systematically away from or toward the pump.

A similar flow is used when putting traps on and off of the gloveboxes.

Any extra lN_2 can be put back in the 4L dewar.

Transport

Cryogenic fluids are best kept stored in insulated containers at atmospheric pressure to help keep the liquids near their boiling point. Containers not designed for charge with liquid cryogens should never be used, due to potential pressure building in unvented containers, transfer lines, and pipes from expansion during the liquid to gas transition. The container must also be able to withstand low temperatures without becoming brittle and breaking. Commonly, the Dewar flask is used to store and transfer cryogenic fluids. Considerations when using a Dewar flask include: caps which allow pressure to escape while keeping air and moisture out, charging the flask with transfer tubes designed to withstand cryogenic temperatures, keeping the flask upright, never using a heat gun to warm transfer tubing, protecting the flask's insulating abilities, and placing Dewars on dollies for transport. ***You should NEVER ride on an elevator with cryogenics.***

Liquid Oxygen

Liquid oxygen poses a serious danger. While large wuantities of it have a characteristic blue hue to it, do not rely on this hard-to-see color. Most chemicals are solids at liquid nitrogen temperature, so ***the presence of any liquid in your traps is cause for alarm***.

If you suspect your cold trap has condensed liquid oxygen, you (and everyone in your vicinity) are in immediate danger. Make sure that the hood sash is down, and that a blast shield is in front of the flask if the operation is not being performed in a hood.

1. Notify all lab occupants and your supervisor, and place a blast shield in front of the traps (even if in the hood).
2. Inspect the system, taking as many precautions as possible.
3. Try to ensure that the liquid nitrogen dewar in which the flask resides has ample liquid nitrogen in order to maintain a temperature of ~ -196 ^{o}C. This is to ensure that the Dewars are filled with plenty of cryogen to ensure the liquid oxygen is kept at an adequately low temperature. Warming of the liquid oxygen could result in pressure buildup and/or a violent oxidation reaction (if organics are present).
4. At this stage, you have several options:
    a. Further venting the system to the open atmosphere is not advised, as this provides an ample supply of oxygen to the system, which may exacerbate the problem. If there are organics present, an explosion may result.
    b. After ensuring that the dewar is filled with liquid nitrogen, try to locate the leak. If it can be fixed (as in a valve was left open, etc.), quickly seal the leak and put the system under dynamic vacuum (if not already so). If you cannot seal the leak effectively (such as if a valve broke), clear the area immediately and notify your supervisor. Do not re-enter the hood area until the liquid oxygen has been pumped out of the cold trap (this may take several hours).
5. Your vacuum gauge/ monometer will indicate if you have fixed the leak and if liquid oxygen has been pumped off (changes by about 100 mm Hg). Evacuate the area. The entire system should be regarded as an explosive danger until no liquid oxygen remains (e.g., the vacuum is returned to its best reading).
6. Once the liquid oxygen is removed, get help from your advisor or another qualified researcher. Remove the dewar, then detach the flask from the line and allow it to warm slowly behind a blast shield.

If you suspect your cold trap has condensed liquid oxygen in the presence of an organic compound, the risk and extent of explosion is amplified. Follow the procedure outlined above, and notify the campus emergency service, at 4911. Absolutely ensure all lab occupants are evacuated, and that all entrances to the lab are closed off.

Minimize the risk of liquid oxygen by:

- Never leave a system under static vacuum in a liquid nitrogen bath. A small leak could lead to condensation of liquid oxygen. An active vacuum is required when cooling with liquid nitrogen so that any oxygen that leaks into the system is efficiently removed.
- Use dry ice/acetone when possible, such as when you are drying sieves.

- Regularly check your line for leaks. Do not assume new glasswear is void of leaks, as pinholes may be present. Therefore it is important to always test your glassware for leaks.
- Have a contingency plan for power outages. Loss of power to the vacuum pump while the pumps are being coolds with liquid nitrogen is a common source of liquid oxygen. Don’t leave traps unattended unnecessarily and drop all traps promptly if a power outage occurs.