School SOP for Operation of Vacuum Pumps and Evacuated Apparatus:

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.

Vacuum Pumps:

Vacuum pumps are used in the lab to remove air and other vapours 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 of which 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 vapour loads from drying samples will occur. Consult manufacturer for specific situations. These recommendations are based on keeping an 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 should be vented to a fume hood. Cold traps and acid traps can be helpful, but if allowed to thaw or saturate, they can lose their effectiveness.

School of Chemistry SOP For Operation Of Vacuum Pumps And Evacuated Apparatus

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.*

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.

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 vapours and related odours 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 vapours 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.