¹Guidelines for Working With High Pressure or Vacuum Apparatus

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**Introduction**

Working with hazardous chemicals at high or low pressures requires planning and special precautions. Procedures should be implemented to protect against explosion or implosion through appropriate equipment selection and the use of safety shields. Care should be taken to select glass apparatus that can safely withstand designated pressure extremes.

### **High Pressure Vessels**

### High-pressure operations should be performed only in pressure vessels appropriately selected for the operation, properly labeled and installed, and protected by pressure-relief and necessary control devices.

* Vessels must be strong enough to withstand the stresses encountered at the intended operating temperatures and pressures and must not corrode or otherwise react when in contact with the materials they contain.
* Systems designed for use at elevated temperatures should be equipped with a positive temperature controller. Manual temperature control using a simple variable autotransformer, such as a Variac, should be avoided. The use of a back-up temperature controller capable of shutting the system down is strongly recommended. All pressure equipment should be inspected and tested at intervals determined by the severity of the equipment's usage. Visual inspections should be accomplished before each use.
* Hydrostatic testing should be accomplished before equipment is placed in initial service. Hydrostatic testing should be re-accomplished every ten years thereafter, after significant repair or modification, or if the vessel experiences overpressure or over temperature.

### **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](http://web.princeton.edu/sites/ehs/labsafetymanual/sec6c.htm), 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 fume 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 a fume hood or into the exhaust hood of other ventilated pieces of equipment.
* Assemble vacuum apparatus in a manner that avoids strain, particularly to the neck of the flask.
* 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 when large quantities of volatile materials are involved.

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

* To prevent contamination, all lines leading from experimental apparatus to the vacuum source should be equipped with filtration or other trapping as appropriate.
* 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. Avoid using acetone.
* 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.

*Use with Hazardous Chemicals*

* Vacuum pumps which are used to evacuate systems containing toxic, corrosive or volatile substances must be vented into the building’s exhaust system.  Failure to properly vent the pump can result contamination of the lab with hazardous chemical vapors

### **Rotary Vacuum Pumps**

Rotary vacuum pumps are used in a wide variety of experimental set ups.  If vacuum pumps are not properly installed, trapped and exhausted they may expose you to hazardous chemicals and vapors.

 *Operation Checklist*

* Belt guards must be in place.
* Service cords and plugs must be free of defects.
* Plug the pump directly into an outlet.  Do not connect to an extension cord or power strip.  See the Electrical Safety Guideline for more information.
* Locate in a vacuum pump cabinet (if one is available) or other ventilated cabinet.
* Place the pump on a tray so that spilled oil is contained.
* Shield any glassware under vacuum.
* Pump oil may be contaminated and should be disposed as chemical waste.
* Pump oil must be compatible (i.e. do not use hydrocarbon pump oil with oxidizing gases or vapors) with the vapors that will pass through the pump.
* Use a cold trap to prevent the degradation and contamination of pump oil.

### **Glass Vessels**

Although glass vessels are frequently used in pressure and vacuum systems, they can explode or implode violently, either spontaneously from stress failure or from an accidental blow. Conduct pressure and vacuum operations in glass vessels behind adequate shielding.

* Ensure the glass vessel is designed for the intended operation.
* Carefully check glass vessels for star cracks, scratches or etching marks before each use. Cracks can increase the likelihood of breakage or may allow chemicals to leak into the vessel.
* Seal glass centrifuge tubes with rubber stoppers clamped in place. Wrap the vessel with friction tape and shield with a metal screen. Alternatively, wrap with friction tape and surround the vessel with multiple layers of loose cloth, then clamp behind a safety shield.
* Glass tubes with high-pressure sealers should be no more than 3/4 full.

* Sealed bottles and tubes of flammable materials should be wrapped in cloth, placed behind a safety shield, then cooled slowly, first with an ice bath, then with dry ice.
* Never rely on corks, rubber stoppers or plastic tubing as pressure-relief devices.
* Glass vacuum desiccators should be made of Pyrex or similar glass and wrapped partially with friction tape to guard against flying glass. Plastic desiccators are a good alternative to glass, but still require shielding.
* Never carry or move an evacuated desiccator.