Chapter 11: Cryogenic Vacuum Pumps

In the preceding units we have described vacuum pumps that operate by isolating volumes of gas, compressing the gas and exhausting to atmosphere (mechanical pumps) and vacuum pumps that move gas through the interaction of high velocity particles or surfaces (diffusion pumps and molecular pumps). Gases may be removed from a vacuum vessel by a third pumping mechanism: gas capture. In this scheme, gas molecules are removed from the gas phase by one of several techniques. Gas capture may be accomplished by solidifying the gas on extremely cold surfaces. This form of gas capture is called cryo-condensation. Some gases, such as helium, neon and hydrogen have such low boiling points that they are not readily condensed. Pumping of these gases may be accomplished by adsorption. If, through a series of collisions with cooled surfaces, a helium atom loses kinetic energy, it may become "adsorbed" onto a surface. In this state, the helium gas molecule is weakly attached to the cold surface and is, for all purposes, removed from the gas phase. Other mechanisms for gas capture will be detailed in the following unit on sputter-ion pumps.

One other difference between gas capture pumps and positive displacement or momentum transfer pumps is that gas capture pumps have a finite capacity; once they are full, pumping action will cease until the pumping media can be renewed or regenerated. For this reason gas capture pumps are seldom used on vacuum systems which are designed for continuous high gas throughput. This fact has an associated safety issue: gas capture pumps collect and concentrate all the gas species they have pumped during their service time. If the pumping media is to be regenerated for further pumping, considerable care should be taken to carefully exhaust the gases which will be released from the pump during regeneration.

The Effect of Temperature on the Vapor Pressure of Gases

Gas molecules, upon collision with cooled surfaces, lose a significant amount of their thermal energy to the cooled surface. In general it may be said that the thermal energy of a gas molecule is determined almost entirely by the temperature of the last surface the gas molecule desorbed from. If a surface is intentionally cooled to the temperature of liquid nitrogen (-196°C or 77K), all gas molecules which have a boiling point higher than -196 °C can be cryo-condensed on this surface. These gas molecules will literally freeze, transforming from a gas to a solid. As solid material, these condensed gases are captured and eliminated from the gas load inside the vacuum vessel.

![Diagram showing cryo-condensation](image-url)
**Figure 11.1** Molecules in the gas phase, upon contact with a cryo-cooled surface, condense on that surface. The residence time for molecules is dependent upon the specie of gas, the temperature of the cryo-surface and the heat of adsorption (see equation 4.8).

**Sample Problems:**

11.1 The boiling point of liquid nitrogen is -196 °C. Which of the following gases can be condensed on a surface cooled by liquid nitrogen?

11.2 For gas molecules which are not cryo-condensed onto a surface cooled by liquid nitrogen, what would the temperature of the desorbed gas molecules be after they leave the cold surface? How would the velocity of these gas molecules be affected?

11.3 Calculate the residence time for common atmospheric gases which have been condensed onto a surface cooled to liquid nitrogen temperature.

**Cryo-sorption Pumps**

Cryo-sorption pumps provide a safe, quiet, clean and reasonably inexpensive method for evacuation of a vacuum vessel to a pressure of $10^{-3}$ Torr. Most commercial cryo-sorption pumps resemble the diagram in figure 11.2. Liquid nitrogen is used to cool the exterior of the welded aluminum pump body to -196 °C. The interior of the pump body has radially arrayed heat transfer fins that aid in extracting the heat from the sorbent, which is usually activated carbon or alumina. A metal screen, often made of stainless steel creates an open channel that runs vertically through the pump body. This channel aids in exposing the pumping media, or sorbent to gases entering the pump inlet. Linde 5A is a popular sorbent material, which has micro pores of approximately 5Å diameter, which are optimal for trapping most atmospheric gases. Other advantages of this sorbent are that it is chemically inert and will not thermally decompose during a bake-out at 250 °C.
Figure 11.2 Cutaway drawing of a cryo-sorption vacuum pump.

Figure 11.3 Schematic of a vacuum vessel rough pumped by a bank of three cryo-sorption pumps.

Several cryo-sorption pumps may be arranged in a bank as shown in figure 11.3. This configuration has several advantages over the use of a single cryo-sorption pump. A multiple pump system provides the capability of regenerating one pump while using others, increasing the capability of the vacuum system for repetitive pump down cycles. Other advantages include the flexibility of pumping with cryo-sorption pumps in parallel connection (all roughing valves opened at once) or in series (opening one valve, utilizing
the first pump until it is saturated, then closing its valve and opening the valve of the second pump). In general, if one wishes to achieve maximum pumping speed at the expense of base pressure, the parallel mode of operation is used. Conversely, if a low base pressure is of utmost importance, the cryo-sorption pumps are operated in series (sequentially).

Sample Problem:

Parallel and series operation of cryo-sorption roughing pumps will produce very different pump down curves. For the vacuum system pictured in figure 11.3 draw the pump down curves (pressure versus time) that you would expect for series and parallel arrangement of the cryo-sorption pumps.

**Liquefied Gas Cryogenic Pumps**

Figure 11.4  Liquefied gas cooled cryo-panels installed in a diffusion pumped vacuum vessel.

Cryo-panels, placed inside a vacuum vessel, and cooled with liquefied gas (most commonly liquid nitrogen) are often employed to reduce the base pressure that may be
attained using a high vacuum pump such as a diffusion pump or a turbomolecular pump.

Cryo-panels should be designed to allow easy filling and purging of the liquefied gas, and also must allow for boil-off of the gas during operation. Typically, cryo-panels are filled after a pressure of less than $10^{-5}$ Torr has been attained. This reduces the loading of the cryo-panels with atmospheric water vapor, which the diffusion or turbo pump can normally handle. Prior to venting the vessel to atmosphere, the cryo-panels should be warmed to room temperature. Cold cryo-panels exposed to air would ice up, and the ice, upon melting would drip water into the vacuum vessel.

**Theory of Operation of Compressed Helium Cryogenic Pumps**

Everyone who has pumped up a bicycle tire using a hand pump has experienced the effect of gas heating upon compression. As the piston in the air pump is forced down, air is compressed and forced through the inner tube valve stem. At this point the compression of gas is high and the heat generated is conducted through the valve stem to the fingers. In just the opposite way, gases may be allowed to expand rapidly to pull heat from their surroundings. This is why the tip of aerosol cans become cold when the compressed gas is released. This effect is particularly noticeable for the cans of compressed freon (microdusters) used to blow dust off of microelectronics parts. Compressed helium refrigerators take advantage of the cooling effect of expanding gases to produce extremely cold surface onto which gas molecules may be captured. It should be noted here that at no point in the operation of the helium compressor is the helium condensed to a liquid. All helium refrigerators used to produce cold surfaces for cryo pumping have three basic components: the helium gas compressor, the connecting lines and the cold head. These components are carefully matched to work together properly. With very few exceptions, components from different manufacturers cannot be intermixed and be made to operate properly.

![Figure 11.5 Components of a compressed helium refrigerator.](image-url)
Figure 11.6 Block diagram of the functional components in a compressed helium refrigeration circuit.

The unit referred to as the "Compressor" actually serves several functions in addition to compressing the helium gas. Following compression, the gas is forced through a heat exchanger which is cooled using flowing water. The cooled helium may contain some residual oil vapor from the compressor. This oil vapor would condense in the cryo-pump regenerator and severely hamper its ability to produce the cold temperatures required for cryo-pumping. To remove oil vapor, an oil separator and an oil adsorber are used in series as shown in figure 11.5. The oil adsorber has a finite service life, and must be replaced with a new unit periodically. Typically, adsorbers are renewed every 6 months.

The lines which transport the high purity compressed helium between the compressor and the cryo-pump are specially designed to contain the high pressure helium gas. These lines have special fittings on each end which allow connection and disconnection without losing the helium in the lines. Maximum line length varies from manufacturer to manufacturer, but most models allow the cryo-pump to be at least 20 feet from the compressor. This permits one to place the compressor outside a clean room to reduce contamination, or to isolate the vacuum vessel from heat or vibration generated by the compressor.

The operation of a compressed helium refrigerator is based upon the cooling cycle as described by Gifford and McMahon in several articles published in 1960. The following series of diagrams and footnotes are presented to demonstrate the principles of operation of this type of refrigerator.
Figure 11.7 Cross-section of a compressed helium refrigerator. In the first part of the refrigeration cycle, the displacer, which is made of a thermally insulating material (usually micarta) is at the lower end of its stroke. The compressed helium supply valve is opened, and high pressure (300 PSI) helium gas at room temperature is flowed into the cylinder in which the displacer oscillates.

Figure 11.8 As the high pressure gas is admitted into the cylinder, the displacer moves upwards, forcing the gas to pass into and through the regenerator. The regenerator is made up of tightly packed material of high thermal inertia or heat capacity. (Heat
capacity may be defined as the amount of thermal energy required to raise a specified amount of material from one temperature to another temperature. A material having a high heat capacity required more thermal energy input to change its temperature than a material of low heat capacity. The materials most often used in the regenerator are lead or copper spheres. Even though the regenerator is tightly packed with these spheres, gas flow is not seriously impeded.

Figure 11.9 The supply valve admitting compressed gaseous helium into the cylinder is closed as the displacer moves upwards nearing the top of its stroke. At this point the helium gas has traveled through the regenerator, and assuming several cycles have already occurred, the helium gas will lose some of its thermal energy to the cooler regenerator.
In the next stage of the cycle, the return valve is opened. The gas in the cylinder is at 300 PSI while the pressure in the return line is at approximately 80 PSI. The gas responds to the pressure differential by expanding into the return line. This expansion is what causes cooling in this type of heat pump. Heat flows from the external heat load (dark rectangle at the bottom of the drawing through the cylinder walls to the cold interior of the cylinder. As the helium passes through the regenerator it also cools the metal spheres.
**Figure 11.11** In the final stage of the cycle, the displacer is forced downwards to push any remaining helium gas through the regenerator and into the return line to the helium compressor. The return valve is closed and the helium is again compressed in the compressor for the next cycle.
Figure 11.12 Cross-section of a two-stage compressed helium refrigerator. The motor serves to rotate the valve disc which is ported to control flow of high pressure gas into the regenerator and flow of low pressure gas back to the helium compressor.

Figure 11.13 Simplified cross-sectional drawing of a compressed helium cryogenic pump body.

The majority of commercially available cryogenic pumps are similar to that represented in figure 11.13. At the pump inlet is the 80 K array, which is thermally connected to the first stage of the refrigerator by the radiation shield. Indium foil is used at the mechanical junctions to improve thermal conductivity. Water vapor is the primary gas that is condensed on the inlet array. Without the optically opaque inlet array, water vapor would condense on the 15 K array severely limiting its ability to pump oxygen, nitrogen and the non-condensable gases, helium, hydrogen and neon. The diagonally positioned plates of the 15 K array serve two functions: the top surfaces are used to pump oxygen, nitrogen and argon, while the sorbent attached to the underside of each array is used to cryo-adsorb the three non-condensable gases.
As with all high vacuum pumps, the compressed helium cryogenic pump is unable to evacuate vessels which are at atmospheric pressure. Unlike diffusion and turbo pumps, the appropriate conditions for crossover for a cryo pump are a function of the amount of gas in the vessel rather than simply the pressure in the vessel. This is best illustrated by example. If a manufacturer's specification for the cross-over of a cryo-pump is 150 Torr-liters, and the vessel to be pumped has a volume of 100 liters, then the cross-over pressure is given by:

\[
\text{Cross-over [Torr-liters]} = \frac{\text{Vessel volume [liters]}}{} \times \text{maximum pressure at cross-over [Torr]}
\]

\[
\frac{150 \text{ Torr-liters}}{100 \text{ liters}} = 1.5 \text{ Torr}
\]
It has been mentioned that cryogenic pumps, being of the gas capture type, have a finite capacity. Once these pumps have reached their capacity (become saturated) pumping action will cease. At this point the pump needs to be warmed up in a controlled manner to allow the release of the condensed gases in the pump's cryo arrays. This process is referred to as regeneration. During regeneration, all of the gases which have been captured by the pump will be released in concentrations much greater than normal in the atmosphere. It is possible, during regeneration, to release explosive or toxic gases in dangerous concentrations. For this reason the process of regeneration must be performed safely, following the pump manufacturer's directions. In general, one regenerates a compressed helium cryogenic pump following this procedure:

**Regeneration Procedure for a Compressed Helium Cryo-pump:**

1. Close the head gate valve between the pump inlet and the vacuum vessel. Turn off any pressure gauges that are exposed to the cryo-pump body.
2. Stop the cold head motor.
3. Check the poppet valve on the cryo-pump body to insure that it is in good condition and is not physically obstructed. Dangerous over pressurization of the pump body will occur if the pressure release valve fails to operate properly.
4. Begin purging the pump body with dry, inert gas such as nitrogen or argon. (In some cases it is possible to speed regeneration if the purge gas is heated by flowing it through an electrically heated tube on its way to the cryo pump body).
5. If the cryo-pump is equipped with a blanket heater, turn this heater on.
6. Allow the pump to be purged with gas for a sufficient amount of time to allow removal of all trapped gases inside the cryo-pump. This time is a function of pump size and design; check manufacturer's specifications for the time duration for this operation.

Following proper regeneration of the cryo-pump, the pump will be ready to resume service.

**Operation of a Cryo-pumped Vacuum System:**

Assuming all of the compressed helium lines are connected and properly purged, the sequence of operations is as follows:

1. Check the compressor to verify cooling water flow to the heat exchanger.
2. Start the compressor, allow it to operate for 30 minutes (the auto bypass circuit in the compressor will cycle compressed helium from the high pressure side of the compressor to the low pressure side).
3. Close all valves on the vacuum system.
4. Start the roughing pump; allow time for the roughing pump to warm up.
5. Open the roughing valve to the cryo-pump; pump the cryo-pump body down to a pressure of less than 50 milliTorr.
6. Close the roughing valve.
7. Record the rate of pressure rise inside the cryo-pump body. If the rate of rise is less than 10 millitorr per minute, begin operation of the cold head. If the rate of rise is greater than 10 milliTorr per minute, the gas load in the pump is unacceptably high, and the cause must be identified and eliminated.
8. Observe the temperature of the cooled surfaces inside the pump using the pump's temperature monitor. Assuming the temp. probe is reading the second stage, when the temperature is below 10K, the pump is in a stable operational mode.
9. Refer to the manufacturer's literature for the crossover pressure specification for the pump model you are using. Calculate the crossover pressure.
10. Evacuate the vacuum vessel to a pressure below that calculated in step 10.
11. Close the vessel roughing valve and open the head gate valve.
12. Turn on vessel ionization gauge.

Sample Problems:
11.5 Why is the regenerator filled with lead spheres?
11.6 Is the helium in the refrigeration cycle of a compressed helium cryogenic pump ever in the form of a liquid?

For Further Reading:


Laboratory Exercise 11.1:

Inspection of a cryo-sorption pump.

Equipment required: a cryo-sorption pump, Linde 5A sorbent, a heating jacket or "callrod" heating assembly, a manually operated vacuum valve to fit the inlet of the cryo-sorption pump.

Procedure: Locate the manufacturer's literature for the model cryo-sorption pump to be used in this activity. Answer the following questions:
1. What is the capacity of the pump for atmospheric gases?

2. What type of sorbent is recommended? What amount of sorbent is recommended?

3. What are the time and temperatures recommended for regeneration of the cryo-sorption pump?

4. Identify the pressure relief valve on the cryo-sorption pump. How does it work? Is the pressure relief valve in good operating condition? What are the potential dangers associated with regeneration of this type of vacuum pump?

5. Can this pump be mounted on a vacuum vessel in any orientation?

**Laboratory Exercise 11.2:**

**Pumping various gas species using cryo-sorption vacuum pumps.**

**Equipment required:** small vacuum vessel or bell jar vacuum system (less than 100 liters internal volume), two or three cryo-sorption vacuum pumps, isolation valves for each sorption pump, a vacuum gauge capable of reading from atmospheric pressure to \(10^{-3}\) Torr, connecting tubulation, lecture bottles of back-fill gases (Helium, argon).

**Procedure:** Assemble the equipment as shown in figure 11.3. Make certain the vacuum connections are secure. If necessary, regenerate the cryo-sorption pumps following the manufacturer's directions. Be careful to avoid contacting the hot surfaces with the skin and keep flammable materials at a safe distance from the sorption pumps during regeneration. Close isolation valves on each pump at the end of the regeneration cycle. When the pumps have cooled to room temperature, and are ready for use, attach a dewar to one pump at a time, fill the dewar with liquid nitrogen, and perform the following experimental measurements:

1. Using only one cryo-sorption pump, measure the time to evacuate the vessel from atmospheric pressure to 50 milliTorr. Close the isolation valve, vent the vacuum vessel to room air and repeat the evacuation, again recording the time to pump to 50 milliTorr. Repeat this process until the time to achieve the specified pressure is unacceptably long, or the pump fails to reach 50 milliTorr. Plot the data as pressure versus time for all the runs performed on a single piece of graph paper, clearly identifying each plot. Calculate the number of Torr-liters pumped during each measurement. Calculate the amount of gas (expressed in Torr-liters) required to saturate the pump. Compare this value to the manufacturer's specifications.

2. Repeat the steps in the first series of measurements, using a fresh cryo-sorption pump, this time initially flooding the vessel with helium. Following
each evacuation, back-fill the vacuum vessel with helium. Plot the data as before and compare the results with the data for pumping air. What conclusions can you draw from this comparison?

3. Repeat the procedure in (2) using argon gas instead of helium. Again plot the data and draw conclusions on the performance of cryo-sorption pumps used to pump these three gas loads.

**Laboratory Exercise 11.3:**

**Parallel and series pumping using cryo-sorption vacuum pumps.**

**Equipment required:** small vacuum vessel or bell jar vacuum system (less than 100 liters internal volume), two or three cryo-sorption vacuum pumps, isolation valves for each sorption pump, a vacuum gauge capable of reading from atmospheric pressure to $10^{-3}$ Torr, connecting tubulation, lecture bottles of back-fill gases (Helium, argon).

**Procedure:** Assemble the equipment as was done for experiment 11.1. Regenerate the cryo-sorption pumps if necessary. Evacuate the vessel by opening all isolation valves simultaneously (parallel pumping). Record pressure as a function of time. Regenerate the cryo-sorption pumps and repeat the experiment, only this time open the cryo-sorption pump isolation valves sequentially (series pumping). Allow each pump to achieve its base pressure before closing its isolation valve and opening the isolation valve to the next pump. Plot the data for both measurements as pressure versus time, carefully labeling each set of data. Mark the plot of series pumping to show the point at which switching from one pump to the next occurred.

How do the two sets of data (parallel versus series) compare? Which configuration produced the fastest initial pumping speed? Which achieved the lowest base pressure? Is your data consistent with your understanding of cryo-sorption pump operation?

**Laboratory Exercise 11.4:**

**Inspection of a compressed helium cryogenic pump.**

**Equipment required:** a cryogenic pump, manufacturer's literature.

**Procedure:** Using manufacturer's literature for the model cryo-sorption pump to be used in this activity. Answer the following questions:

1. What is the capacity of the pump for atmospheric gases?
2. Which gases are pumped on the inlet (80 K) array?
3. Which gases are pumped on the inlet (15 K) array?
4. What is the sorbent material used on the underside of the 15 K array?

5. Locate the temperature gauge and probe. Where is the temperature measured? What is the operating temperature of this component?

6. Locate the radiation shield. Note the color of the shields interior and exterior. Why would the manufacturer intentionally choose these finishes?

7. How is the radiation shield attached to the inlet array?

8. Identify the pressure relief valve on the cryogenic pump body. How does the valve work? Is the pressure relief valve in good operating condition? What are the potential dangers associated with regeneration of this type of vacuum pump?

9. Find the purge gas inlet. How is flow of purge gas controlled?

10. Is the pump fitted with a blanket heater? How is the temperature controlled during regeneration? What is the maximum recommended temperature? What limits the maximum suggested temperature?

**Laboratory Exercise 11.5:**

**Operation of a compressed helium cryogenic vacuum pump.**

**Equipment required:** a compressed helium cryogenic pump with cold head, compressor and charged helium lines, for cryo-pumps with an O-ring seal at the inlet flange: a 1" thick pyrex glass plate having a diameter at least 1" larger than the O-ring diameter; for other flange styles: a pyrex glass viewport to match the inlet flange. An oil sealed mechanical pump, connecting lines and an in-line pressure gauge capable of reading from atmospheric pressure to 1 milliTorr.

**Procedure:** The instructor will assemble the cryogenic pump system. Place the glass plate or viewport on the inlet flange of the cryo-pump (see figure 11.3). Begin flowing cooling water into the compressor, and start the compressor.
Rough pump the cryogenic pump body to approximately 10 milliTorr. Isolate the mechanical pump and record the rate of pressure rise for five minutes. Does the pressure rise indicate the pump is ready for operation? If the rate of rise test indicates the need for regeneration, follow the manufacturers recommendations and the procedure given in this unit to regenerate the pump. If the pump is ready for pumping, begin operation of the cold-head. Observe through the viewport the operation of the cryo-surfaces. Upon completion of the experiment, stop the cold head, turn off the compressor and vent the system (with dry inert gas, if possible).

Discussion:
When in the procedure for this experiment is it acceptable to turn off the mechanical roughing pump? Do you see any advantages to this?

What temperature did you read on the cryogenic pump's temperature gauge?

What does this suggest about the operation of the pump?