Chapter 7: Oil Vapor Diffusion Pumps

It wasn't that long ago when you could walk into any vacuum laboratory and find a vapor diffusion pump on every system. Vapor diffusion pumps were first conceived about 1915-16, and used mercury as the pumping fluid. A decade later, experimenters found that some oils had high boiling points and low vapor pressures and were good pumping fluids. These oils were useful because they remained in the pump indefinitely and allowed lower pressures to be attained without the use of a cold trap. During W.W.II, and again during the 1960's for the space effort, diffusion pumps went through some significant design changes that increased their pumping speed, increased their ability to produce lower pressures, and oils gave way to synthetic pumping fluids. Due to it's simplicity, high performance, and low initial cost, the diffusion pump remains the primary industrial high vacuum pumping mechanism. Applications for this type of pump are found in R&D labs, coatings facilities, manufacturing, and space simulation. When diffusion pumps are used with the correct fluid, traps, and baffle, they can produce pressures to approximately $2*10^{-10}$ Torr.

Theory Of Operation

Diffusion pumps are vapor jet pumps that work on the principle of momentum transfer. This occurs when a heavy, high speed vapor molecule collides with a gas molecule and moves it in a preferred direction through the pump. The bottom of the pump contains an electric heater which is used to heat the pumping fluid to it's boiling point, thus, producing the vapor. This must be done at a reduced pressure. This means that before the diffusion pump is started, it must be "rough pumped" down to an acceptable pressure, typically 100 millitorr. To do otherwise will result in no pumping action and possible damage to the pumping fluid. Once boiling of the fluid has begun, the vapor is forced up the central columns of the jet assembly. It then exits at each downward directed jet in the form of a molecular curtain that impacts the pump body. The pump body is externally cooled so that the fluid will condense on its inside surface and run back down into the boiler. Pump bodies are typically water-cooled, but some are air-cooled. As gas molecules from the system randomly enter the pump (molecular flow conditions), they encounter the top jet. Some of them are impacted and driven on to the next jet. Subsequently, they reach the foreline where they are exhausted to the atmosphere by the mechanical backing pump.

Compression Ratio

The diffusion pump is similar in character to other compression pumps in that it develops a relatively high exhaust pressure compared to the inlet pressure. For most gases this compression ratio may be one million to one (or greater). For example; for an inlet pressure of $2*10^{-7}$ Torr and a foreline pressure of $2.0*10^{-1}$ Torr, the compression ratio would be one million. As far as compression goes, in a mixture of gases, each species may be pumped with different effects. It is possible to have different maximum compression ratios and different flow rates for gases having different molecular weights. For example, the compression ratio for hydrogen will differ greatly from the compression ratio for argon simply because their molecular weights are very different. Also, when the pumped gas has a molecular weight different from air the maximum compression ratio will shift, but the tolerable foreline pressure (critical discharge pressure) remains the same.

Critical Discharge Pressure

The critical discharge pressure of a diffusion pump is the maximum permissible pressure at the foreline during normal pump operation. The expected pumping action of a diffusion pump ceases when the critical discharge pressure is exceeded. That is, the vapor of the discharge stage of the pump does not have sufficient energy and density to provide a barrier for the air in the foreline, thus, this air will flow through the pump in the wrong direction carrying with it the pumping fluid vapor. For most modern diffusion pumps, the maximum allowable foreline pressure is about 0.5 Torr. Diffusion pumps cannot function at all unless the foreline pressure is held below this limit by the backing pump. The most important rule of diffusion pump operation is: Do not exceed the critical discharge pressure! If this single most important rule is observed, then most difficulties associated with diffusion pump operation can be eliminated.

Backstreaming

Backstreaming can be defined as the passage of the pumping fluid through the inlet port of the pump and in the direction opposite to the direction of desired gas flow. However, backstreaming must not be limited to the pump, but must include the trap, baffle, and plumbing as well because all affect the transfer of pumping fluid vapors from the pump body to the chamber. There is a multitude of conditions that can cause backstreaming. The most common are; exceeding the critical discharge pressure in the foreline, exceeding maximum throughput capacity for long periods of time, and incorrect start-up or shutdown procedures. Backstreaming of pumping fluids into your work environment is always considered catastrophic. I know of very few vacuum related processes in which oil contamination is not a disaster! My suggestion to system operators is to know their equipment thoroughly and learn proper operating techniques. Ninety-nine percent of costly backstreaming problems are due to operator error. Finally, equip your system with the appropriate interlocks that will prohibit valve cycling above a specified pressure. This will protect your system whenever it is left unattended.

Baffles And Traps

Baffles have one particular purpose: to reduce the backstreaming of pump fluid into the vacuum chamber. Most baffles are "optically opaque" which implies that their internal geometry is such that light cannot pass directly through them. This insures that a molecule will collide at least once with a surface regardless of the incoming direction. Baffles do impede the flow of pumped gases, but well designed units can retain about 60% of the pumping speed. Baffles are installed directly above the pump inlet and are often used in conjunction with a trap. Water-cooled baffles can reduce the rate of reevaporation of condensed fluid thereby reducing the density of vapor in the space between the baffle and the trap. See illustration for several baffle designs. Cryogenic or refrigerated traps serve two purposes. They act as barriers against the flow of condensable vapors from pump to system; and they also serve as cryopumps for condensable vapors (primarily water vapor) emanating from the system. In typical unbaked systems, water vapor may constitute about 90% of the remaining gas after initial evacuation. Chilled traps increase the pumping speed for water vapor and therefore can in many cases lower the base pressure of your system. I know of two distinct varieties of liquid nitrogen traps. One is a trap that is placed anywhere within the vacuum chamber. This may be a cryopanel, a sphere or cylindrical bottle, or a tubular arrangement acting as a "cold-finger" on which condensable gases will be trapped. The other is of the optically opaque design and is placed between the chamber and the pump inlet. These traps insure that gas molecules collide at least once with a cold surface.

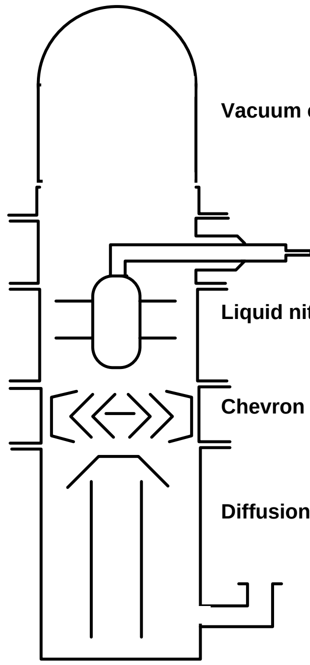


Figure 7.1 Configuration of traps and baffles used on diffusion pumped systems.

Fluids

Many of the pumping fluids used today have been developed within the last 30 years. Up to about 1960, most fluids had a vapor pressure of 10^{-7} Torr or 10^{-8} Torr and the base pressure of the system was limited to that range. The choices of pumping fluids became greater after Hickman publicized the used of polyphenyl ethers which offered exceptional thermal and chemical stability. Operational characteristics of another low vapor pressure silicone fluid (DC705) were also found to be excellent. The use of either of these fluids will permit base pressures of 10^{-9} Torr or 10^{-10} Torr to be achieved. More recently, fluorinated oils have been developed for use in diffusion pumps. These have the added advantage of compatibility with corrosive gases used in some processes.

Ultimate Pressure

Two distinct observations can be made regarding the ultimate pressure of a diffusion pump. Ultimate pressure may be considered to be a gas load or a pressure ratio limit. The pressure ratio limit is usually associated with light gases (hydrogen, helium, xenon). The pumping action of the vapor jets does not cease at any pressure, however low. The ultimate pressure of the pump depends on the ratio of pumped versus back-diffused molecules, plus the ratio of the gas load to pumping speed. Also, the pump itself can contribute a gas load either through backstreaming of pump fluid vapor and its cracked fractions or the outgassing from its parts. In practice, then, the ultimate pressure of a pump is a composite of several elements. The first limit of the ultimate pressure is usually due to the vapor pressure of the pumping fluid, although this limit may not be observed at pressures below 10^{-8} Torr.

Operating Procedures

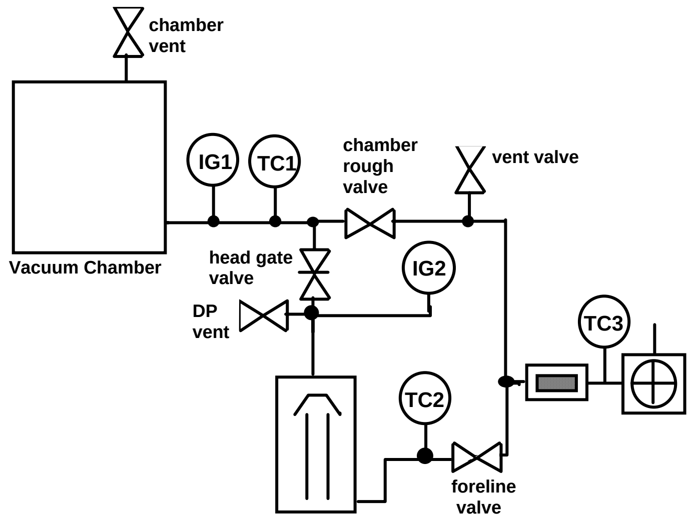
The operation of high vacuum, diffusion pumped systems requires certain care and attention to several items. General cleanliness is extremely important, especially in smaller systems. Remember, if a drop of oil were to be trapped somewhere in your vacuum system, it may take days or weeks to evaporate that drop from your system. Humidity and temperature can be important in view of the constant presence of water vapor in the atmosphere. When your system is opened to the environment, pump down time is significantly longer if the air is humid. The time of exposure is also significant. If possible, the backfilling should be done with nitrogen or argon. For short exposures, this appears to reduce the amount of water vapor adsorption in the vacuum system.
It is extremely important to develop good habits in valve sequencing operations, especially in systems with manual valves. It is useful to have a "map" or schematic of your system on your control panel that shows valve locations and functions. A single wrong operation can result in very costly maintenance to the system. Automatic valve sequence controllers have been used widely for many years, and they all have built in interlocks to prevent accidental opening of the wrong valves. During the evacuation of a vessel, the question arises regarding the proper time to switch from the roughing pump to the diffusion pump. In other words, when should the high vacuum valve be opened? There is no general answer to this question because each system is different with different gas loads and different volumes. In practice, the transfer from roughing to the diffusion pump is made between 50 and 150 millitorr. Below this pressure region, the mechanical pump rapidly loses it's pumping effectiveness and the possibility of oil backstreaming increases. Although the throughput of a diffusion pump is nearly constant when inlet pressures are in the 1 to 100 millitorr range, the initial surge of air into the pump when the high vacuum valve is opened will overload the diffusion pump temporarily. We recommend that the period in which pump inlet pressure is above 150 millitorr be kept as short as possible; i.e., just a few seconds! Without a doubt, you'll have questions on proper diffusion pump operation. There is literature available to help you, and one document we suggest is a Varian Corp. publication written by M. H. Hablanian called "DIFFUSION PUMPS: PERFORMANCE AND OPERATION" which is part of the AVS Monograph Series.

Sample Problems:

7.1 What is generally regarded as the single most important thing to remember about operating your diffusion pumped vacuum system?

7.2 Determine the compression ratio of a typical diffusion pump which has an inlet pressure of $5x10^{-7}$Torr and a discharge pressure of $1x10^{-1}$ Torr.

7.3 Explain what may happen if an operating diffusion pump is accidentally vented through the foreline with air.



Start-Up of Diffusion Pump:

1. Close all valves.
2. Start mechanical pump.
3. Open foreline valve, evacuate DP to ~20 mTorr.
4. Fill DP cold trap (liqiud nitrogen).
5. Turn on DP heater, 15 minute warm-up.
6. Close foreline valve.
7. Open chamber rough valve, evacuate to ~ 100 mTorr.
8. Close chamber rough valve, open foreline valve.
9. Open head gate valve.
10. Turn on ionization gage.

Shut-Down of Diffusion Pump:

1. Close head gate valve, turn off ionization gage.
2. Turn off DP heater, allow 30 minutes to cool.
3. Warm DP cold trap to room temp.
4. Close foreline valve.
5. Vent DP through vent valve.

Laboratory Exercise 7.1:

Observation of diffusion pump operation.

Identify the diffusion pump as you did in exercise 6.1 for the mechanical pump {manufacturer, model, size, capacity, etc.}. Assemble your pumping system so that your diffusion pump is backed by a small mechanical pump (see figure 7.2).

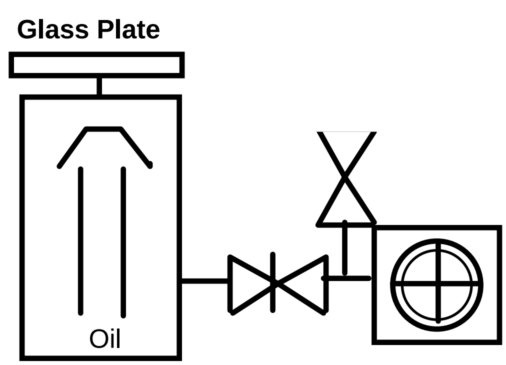


Figure 7.2 Set-up for observation of DP backstreaming.

Place a pyrex view port on the pump inlet and start the mechanical pump. When system pressure is below 100 millitorr, turn on the diffusion pump and observe the formation of oil condensation on the pump side of the pyrex view port. How long does it take for a slight haze to form? a heavy haze?, how long before droplets appear on the view port? Report your observations. What, in your mind, is the sequence of events that cause droplet formation on the view port? If you took a heat lamp and aimed it at the view port, what would you expect to see?

Data table 7.1 Observations for experiment 7.1

Time [minutes]	Observation

Laboratory Exercise 7.2:

Testing and recording your system's base pressure.

Now add the components to make your system look like the one in figure 7.3.

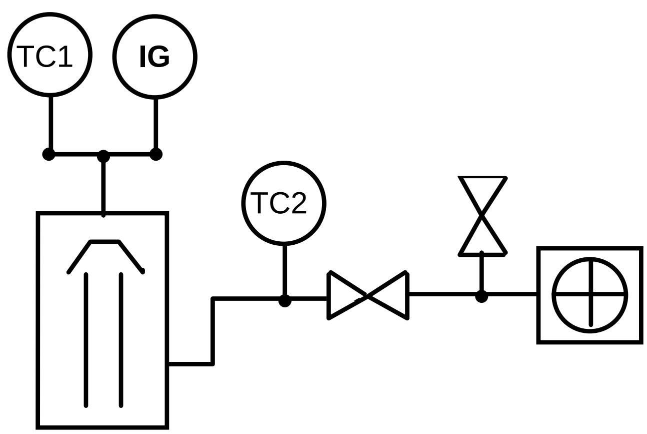


Figure 7.3 Experimental set-up for measurement of DP base pressure.

You'll need a thermocouple gauge at the DP inlet, a pressure gauge on the mechanical pump, and an ionization gauge at the inlet to the diffusion pump. Start the mechanical pump and reduce the pressure in the system to below 100 millitorr. Now, turn on the diffusion pump and allow the pump to reach normal operating temperature. As you progress through this assignment, record the foreline pressure at frequent intervals so that it may be graphed later. Once your pump is working, turn on the ionization gauge and record the steady drop in pressure. You should give the pump a couple hours to reach it's base pressure. Now you have two data sets to record on semi-log graph paper. A table for data entry is provided for you on the following page.

Data Table 7.2 Data for DP experiment 7.2

Time [minutes]	**Mech. pump press. [Torr] {TC2}**	**System press. [Torr] {TC1}**	**System press. [Torr] {IG1}**

<u>Laboratory Exercise7.3:</u>
Measurement of pump down behavior for DP system.

Assemble your system so that a vacuum chamber is attached to the diffusion pump through an isolation valve. You'll need pressure gauges on the chamber and also at the DP inlet. Rough pump the system as you've done previously, and allow the DP to warm up to normal operating temperature. Open the isolation valve and record time versus pressure data until the base pressure is achieved. You can monitor foreline pressure as well, and plot both sets of data on semi-log paper.

Data Table 7.3 Data for DP experiment 7.3

Time [minutes]	Mech. pump press. [Torr] {TC2}	System press. [Torr] {TC1}	System press. [Torr] {IG1}

Laboratory Exercise 7.4:
Measuring pumping speed at constant pressure for a DP system.

Assemble your system similar to the way you did when testing the speed of a mechanical pump. That is, attach a variable leak valve and an atmosphere valve to the chamber and use your burette to find the volume displaced in a specific time period. Since you're using the constant pressure method, you must select several pressures at which to conduct the speed tests. Let's recall the vacuum formula used in the previous exercise:

$$S = \frac{Q}{P}$$

S = pump speed [Liters/sec]
Q = mass flow rate [Torr-Liters/Sec]
P = pressure in vacuum vessel [Torr]

where:

$$Q = \frac{V_A \times P_A}{t}$$

V_A = atmospheric volume [liters]
P_A = atmospheric pressure [Torr]
t = time to leak in V_A [seconds]

We suggest you start somewhere in the mid to high 10^{-5} Torr range. Then pick higher pressures at intervals suitable to the apparatus you have assembled. You will be plotting pumping speed versus pressure. When you graph pumping speed for the diffusion pumped system, be sure to include all information that may be pertinent to the outcome of the test. A table for data entry is provided for you on the following page.

Table 7.4 Data for pumping speed of diffusion pump at constant pressure.

Vessel Press [Torr]	**Atmospheric Volume [liter]**	**Time for V_A [seconds]**	**Q [Torrliters/sec]**	**S_P [liters/sec]**

Answers to Chapter 7 Sample Problems

7.1 Don't exceed the critical foreline pressure!
7.2 200,000.
7.3 The critical foreline pressure will be exceeded, the pump will cease to function, and hot diffusion pump oil will very likely backstream into the vacuum system.