

Using vacuum with TGAs and recording balances

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Many applications of Thermo Scientific CAHN TGAs and recording balances require the use of vacuum. Some applications require only partial vacuum while others require high vacuum. Some applications will require pumping of only inert gases while others will require pumping of corrosive gases. Each set of conditions involves different factors that must be considered before selecting the components to be used in the vacuum system. This technical note will try to suggest the various factors and equipment to be considered.

Terminology

Before discussing the various factors, we must define some terms. The unit of pressure used until recent years has been the "torr" which is equal to the pressure that will support 1 mm of mercury. Though torr is still widely used, especially in the English speaking countries, the International Organization for Standardization (ISO) in 1973 declared the new standard unit of pressure to be the "pascal" (Pa). It is defined as 1 newton per square meter. In the vacuum industry, however, the term millibar (mbar) is mostly used to replace the torr unit. In this paper, we will use the term mbar whose value is close to the torr unit. The following table shows the relationship between these vacuum units.

Gas flow mechanisms

When considering vacuum, it is important to understand the mechanism of removing gas molecules from a chamber. In the rough vacuum region, a gas molecule moves from an area of higher pressure to an area of lower pressure due to the higher collision rates with molecules from the higher pressure side. This motion is called "viscous flow". In the high and ultra-high vacuum regions, gas molecules will usually have a mean free path of movement that is greater than the diameter of the exhaust tubing.

mbar	Pa	Torr*	Atm*
1,000	100,000	750	1
100	10,000	75	0.1
1	100	0.75	0.001
0.1	10	0.075	10 ⁻⁴
10 ⁻²	1	10 ⁻²	10 ⁻⁵
10 ⁻ⁿ	10 ⁻⁽ⁿ⁻²⁾	10 ⁻ⁿ	10 ⁻⁽ⁿ⁺³⁾

* Slight amount of rounding involved.

It is customary to divide the large span of pressure into smaller regions for discussion purposes. These smaller regions are defined as:

Name	Pressure (mbar)
Rough vacuum	1000 to 1
Medium vacuum	1 to 10 ⁻³
High vacuum	10 ⁻³ to 10 ⁻⁶
Ultra-high vacuum	10 ⁻⁶ and below

This means that a molecule will usually hit the tubing wall before it will strike another gas molecule. Under these conditions, molecules move in a random order and have only a statistical chance of finding their way to the vacuum pump. This is similar to balls bouncing around a pool table trying to find one of the pockets. This statistical motion is called "molecular flow". The medium vacuum region is the transition region between viscous flow and molecular flow. The flow pattern here is called "Knudsen flow."

If you are designing a system for rough vacuum, you can use tubing as small as ¼ inch as this size tubing will allow viscous flow down to 10.2 mbar. However, for vacuum below viscous flow, much larger tubing is required in order to have a reasonable statistical chance for the gas molecules to find their way to the vacuum pump. Hydrogen and helium have a much greater mean free path and are, therefore, more difficult to evacuate. Using tubing less than 1" (2.5 cm) will greatly restrict your ability to achieve high vacuum in a reasonable time. The vacuum ports on Thermo Scientific CAHN recording balances are 1" in diameter.

Using tube adapters, larger tubing can be used on these ports.

When working in the molecular flow region, a bend in the tubing can greatly restrict the chances of

molecules moving past the bend. A 90° bend can restrict the flow by about 40%. Therefore, two elbow fittings, which is often the case, in the tubing of a vacuum system can restrict the flow by about 65%. Try to set up the vacuum tubing with the minimum of bends.

Basic vacuum components

In this technical note, we will only consider vacuums in the medium and high regions. Rough vacuums only require inexpensive pumps and the technique required is simple. Ultra-high vacuum is beyond the capabilities of the vacuum enclosure supplied with the Thermo Scientific CAHN systems. If you need ultra-high vacuum for your application, the weighing mechanism can be removed from the supplied enclosure and installed in your own vacuum chamber.

The basic components of a vacuum system are:

1. Roughing pump
2. High vacuum pump
3. Rough vacuum gage
4. High vacuum gage
5. Valves
6. Tubing and fittings

Vacuum pumps

Though there are 32 different types of vacuum pumps, this paper will concentrate on the three types most often used with Thermo Scientific CAHN systems. The three types are rotary vane pump, diffusion pump and turbomolecular pump. These types were chosen because they are the work-horses of the scientific lab and are generally available. For most applications, at least one of the remaining types of pumps can be successfully substituted.

Rotary vane pump

The rotary vane pump is used to pump the vacuum chamber down through the rough vacuum region. Because of their primary duties, these pumps are often called roughing pumps. However, most rotary vane pumps also have the capability of reaching into the medium vacuum region. The pump, a mechanical device driven by an electric motor, can capture gases present at the inlet port, compress the gas and exhaust it to the atmosphere at the outlet port (see Fig. 1). Because oil is usually used to seal the chamber walls, small amounts of oil vapor can backstream from the pump. This type of pump can handle some corrosive gases by trapping them in the oil reservoir. However, if a significant amount of corrosive gases or vapors are present, a foreline trap or filter should be used to protect the pump. The oil should be checked in the sight glass whenever the pump is being used. If the level is low, oil should be added. If the oil is discolored or contains liquid globules, it should be drained and replaced with new oil. If the oil is cloudy and contaminated with particulate material, the pump should be flushed before new oil is added. The characteristic gurgling sound

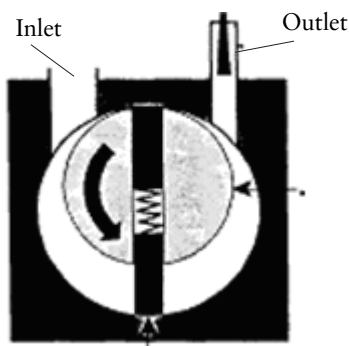


Figure 1: Rotary vane pump

heard when starting a vacuum pump is due to air in the pump. If gurgling develops while pulling a vacuum, it may be due to a vacuum leak or to volatiles which could react with the oil. If you only need rough or medium vacuum, the rotary pump may be all you need. If your application can not tolerate any oil, be sure to place some type of trapping system in the tubing between the pump and the chamber.

Diffusion pumps and turbomolecular pumps are used to bring the chamber down into and below the high vacuum region. Since these pumps can not or should not be used in the rough vacuum region, they are used in conjunction with a roughing pump. The roughing pump brings the chamber down to below 0.1 mbar and then the high vacuum pump is brought on line. The roughing pump is then used to remove the gases captured by the high vacuum pump. In this configuration, the roughing pump is often called the backing pump.

Diffusion pump

The diffusion pump consists of a cylinder shaped body enclosing a jet assembly shaped like a Christmas tree (see Fig. 2). Hot oil vapors stream downward from nozzles located at various levels in the jet assembly. The rapidly moving oil molecules entrap any gas molecules that are present at the top of the pump. These gas molecules are compressed into a small volume near the bottom of the pump where they are removed by the backing pump. The oil vapor is condensed on the air or water cooled walls of the body where it runs down the walls and returns to the oil reservoir at the bottom of the pump. A heater below the oil reservoir vaporizes the oil again for another trip through the pump. The chief working region of the diffusion pumps lies between 10^{-3} and 10^{-7} mbar. In this region, the pump has a high capacity, is vibration free, is very durable, has low maintenance costs and can handle some corrosive gases. However, the aluminum jet assembly can be attacked by some corrosive gases. The disadvantages of a diffusion pump are slow start up time (oil must be heated), a required water supply for cooling (a few are air cooled), required vertical position of the pump, and backstreaming of oil vapor. Most diffusion pumps have

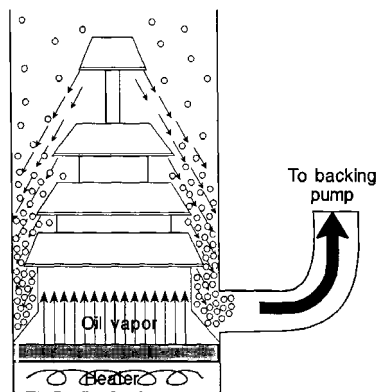


Figure 2: Diffusion pump

a baffle system on top of the pump to greatly reduce the amount of backstreaming. Small traces of oil from the pump, however, are usually found in the vacuum chamber. If a hot diffusion pump is accidentally and suddenly exposed to the atmosphere, considerable amounts of diffusion pump oil can enter the vacuum chamber.

Turbo pump

A turbomolecular (turbo) pump is basically a turbine with rings of blades rotating at 25,000 to 60,000 rpm and up. Gas molecules coming within the range of the outer blades are hit toward the next inner ring of blades. Each ring of blades compresses the gas molecules into a smaller volume and higher pressure. At the last ring of blades, the gas is removed by the backing pump. The turbo pump is sometimes started at atmosphere, together with the roughing pump, so roughing occurs through the turbo as it accelerates. However, though the turbo does not become effective until about 10^{-2} mbar, it can operate down into the ultra-high vacuum region. This pump can usually be operated in any position, has low vibrations, has no oil to back stream (except some that may be on the rotor bearings), is relatively small in size and does not require external cooling. The disadvantages of the turbo pump are high initial cost, costly maintenance when required and susceptibility to damage from corrosive gases, condensable vapors and particulates. Some models can be damaged if they are exposed to ambient pressure while operating at high vacuum. Also, the maximum intake temperature is about 100°C which could be a problem if you are doing a thermo-analytical experiment.

Vacuum gauges

Vacuum gauges have the same problem as the pumps in that there is not one gauge that covers all of the vacuum regions. Therefore, you must use one gauge to monitor the rough vacuum region and another gauge for the medium and high vacuum region. If you are going into the ultra-high region, you will need another gauge.

Pirani gauge

The thermal conductivity (Pirani) gauge is used to measure the rough vacuum region. This gauge uses the principle that thermoconductivity is a function of gas pressure. The gauge has a filament with high resistance/temperature coefficient and is heated by a constant electric current. As the gas pressure is reduced, the heat conducted away from the filament is reduced, causing the filament temperature and resistance to increase. The electronic package associated with the gauge converts the filament resistance to pressure readings. The practical range of the Pirani gauge is 1000 mbars down to 10^{-3} mbar. This gauge has a fast response time and is fairly independent of the gases being measured when the pressure is below 1 mbar. Since the gauge is usually calibrated for nitrogen gas, heavier gases will cause a reading below actual pressure when working above 1 mbar. The reverse is true for the light gases. The disadvantages of the Pirani gauge are the start up time required to heat the filament, errors in readings caused by oils and other organic vapors and the burnout of the filaments.

Penning gauge

The cold-cathode ionization (Penning) gauge is the most popular type for measuring pressures from 10^{-2} to 10^{-6} mbar. The high voltage cathode ionizes the gas molecules which in turn allows a current to flow between the cathode and the anode. The current carried between the cathode and the anode is dependant upon the number of gas molecules present. The electronic package converts the current flow to pressure readings. A Penning gauge has two sources of errors. First, because of the ionic flow of the molecules, the gauge itself acts as a small vacuum pump. If the gauge is at the end of a small diameter tube, it will create and report a higher vac-

uum than that which exists in the sample chamber. You must have large diameter tubing leading to the gauge. Second, the pressure readings are dependent upon the ionization probabilities of the gases being measured. Most gauges are calibrated for nitrogen. For lighter gases, the readings are generally too low. For example, when measuring helium, you should multiply the reading by 7. For heavier gases, the readings are too high. The water vapor readings should be divided by 2.4. These factors are usually given in the instruction manual of the gauge. The advantages of the Penning gauge are cost effectiveness, insensitivity to inrush of air and vibrations and no heatup time required.

The location of a high vacuum gauge is important. Placement of the gauge above the main valve of the pumping station is meaningful only if you are using a short length of large tubing to the chamber. Otherwise, you are only measuring the capability of the pumping station, not the vacuum in the chamber. There can be a drop of two to three decades of vacuum in a four foot length of $\frac{3}{4}$ inch tubing when working with molecular flow. For the most accurate readings, therefore, a high vacuum gauge should be positioned at the vacuum chamber.

Valves

Your system will need at least three valves: a main valve, a roughing valve and a gas-bleed valve. All of these valves should have KF ports (see Vacuum Fittings below).

The main valve isolates the high vacuum portion of the pumping system from the vacuum chamber. It is closed while you are at atmosphere or rough vacuum. It is opened when you have reached medium vacuum. When you have achieved the desired high vacuum and you want to introduce the gases needed for the experiment, you again close the main valve. If the experiment requires the maintenance of high vacuum, the main valve would be left open while running the high vacuum pump. Main valves are usually of the butterfly or the gate type. The butterfly is more often used because it is less expensive, smaller and faster operating. It has a handle that only requires a quarter turn to open and close. A gate valve is used for ultra-high vacuum or when better conductance is required. It is

opened and closed by turning a knob many turns. In any case, the main valve should be equipped with a roughing port. This port provides a bypass of the main valve to the roughing pump during initial pump down. This port eliminates the need for a "T" fitting above the main valve.

The second valve is the roughing valve. Since the rotary pump has the two functions of roughing and backing, the roughing valve allows the switching of the inlet port of the rotary pump to the chamber (usually through a port on the main valve) for initial roughing of the chamber. Then the roughing valve can be switched so the inlet port is backing the diffusion pump. The roughing valve will also have a closed position needed when sealing off the vacuum system from the chamber. If a turbo pump is being used, only an on/off roughing valve is required because the roughing is done through the turbo pump while the turbo pump is accelerating. The third valve is the bleed valve. A simple up-to-air bellows valve can be used to bring the chamber back to atmospheric pressure. However, this valve should be opened slowly. In the molecular flow region, the gas molecules enter the chamber as a beam concentrating its energy at any target in front of the port. When the pressure rises to the viscous flow region, a considerable amount of cooling takes place along with the drag forces involved with rapidly moving air. To allow the injection of special gases into the chamber, a needle valve with a graduated dial should be used. Again, be careful of the gas beam effect present at high vacuums.

Vacuum fittings

ISO has also established a standard used for vacuum fittings. The most common fittings used with recording balances conform to the KF (small flange) standard (see Fig. 3). Though there is a similar American standard, the ISO standard should be the only one considered. ISO has established standard sizes designated as NW (more recently DN) and measured in millimeters of the bore I.D. as follows:

ISO Size	English Size
NW16	3/4
NW25	1
NW40	1 1/2
NW50	2

The KF flange system allows a wide variety of components to be fitted together. The junction between two flanges contains an o-ring held in place by a centering ring. The junction is held together and sealed by a clamp. Almost every piece of vacuum equipment and tubing can now be purchased with a KF flange.

Since most fittings used in vacuum systems are made from type 304 stainless steel, you should be aware that some corrosive gases can effect these fittings. Type 304 is not resistant to sodium and calcium chlorides, hydrochlorite vapors, sulfurous acid compounds and phosphoric and acetic acids. However, it is resistant to most organic vapors. Some fittings are available in aluminum but this metal has an outgassing rate that is five times greater than stainless steel.

Most vacuum fittings designed to operate in high vacuum use Viton o-rings. Viton has a vacuum rating of 10^{-6} mbar and a temperature range from -30 to 175 °C. It is resistant to most chemicals except ketones (MEK, acetone), anhydrous ammonia, low molecular weight esters and ethers, and hot hydrofluoric or chlorosulfonic acids. When using high vacuum, Viton has a very small amount of volatile material, mainly water vapor, but does have some permeability for helium. When trying to achieve maximum vacuum, compress the o-ring as much as possible to reduce the helium leak. However, do not compress the o-ring during bake-out to prevent a permanent set. For more details concerning o-ring material, request Thermo Scientific CAHN paper No. 1686.

Make sure that your fingers are clean when installing o-rings to prevent contamination. Before assembling an o-ring joint, the groove should be cleaned and the ring wiped with a lintless cloth. o-rings should not be cleaned with solvents. When installing o-rings on glass joints, gently roll the o-ring up the joint to the o-ring groove. After installation, inspect the o-ring for any twisting that will cause leaks. A very thin film of good quality vacuum grease can be used on the o-ring. However,

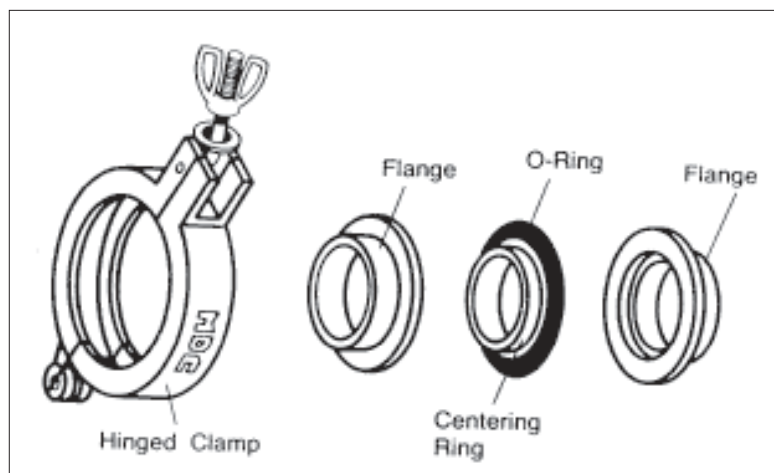


Figure 3: KF Fittings

grease can not overcome poor installation or defective o-rings. As a normal practice, o-rings should be replaced yearly or after being exposed to high temperatures under set load or as a first step when trying to remove a leak. A good supply of Viton o-rings can be purchased with your vacuum equipment since they have a shelf life from 15 to 20 years.

Putting together a vacuum system

The questions to be considered when putting together a vacuum system are:

1. What is the ultimate desired vacuum?
2. Will the gases be corrosive?
3. Will substantial amounts of water vapor be present?
4. Can the application tolerate residual oil?
5. What are the initial investment and operation costs?

If you will only need rough vacuum, you can usually attach the needed valves and gauges to the inlet of the roughing pump. If you are putting together a high vacuum system, you will usually need a stand with a framework on which to mount all of the necessary components. If you buy a complete high vacuum system, the manufacturer will supply all of the components mounted on a stand. If you are not familiar with vacuum technology, you should consider the purchase of a complete system. In the long term, you may save money and a lot of time. However, even if you buy a complete system, the system salesman should carefully review with you the list of components to assure that you are getting the equipment that you need.

In the following discussion of the components used in a vacuum system, we will assume the use of KF NW25 size flanges and tubing. The vacuum take-off adapter used with Thermo Scientific CAHN systems are equipped with this size flange. We also recommend that all fittings will be made from type 304 stainless steel and not aluminum. The diagrams show the suggested positioning of the components. They are not drawn to scale.

In almost every case, a 2 m³/hr roughing pump will be needed to bring the chamber down to 10⁻² mbar where, if needed, the high vacuum pump can be turned on. Since most roughing pumps have a threaded inlet, screw a KF NW25 flange adapter into the inlet. If you only need rough vacuum, attach a butterfly valve to the flange (see Fig. 4), then a "T" fitting to the valve. Attach a Pirani gauge to the side port of the fitting and your chamber tubing to the remaining fitting. If you have condensable vapors or corrosive gases, place a foreline trap between the valve and the pump. This trap will also restrict the flow of oil vapors from the pump to the chamber. If you use a trap, you may need a frame to brace the stack of fittings.

If you need higher vacuum, you will have to add a diffusion or turbo pump. You should use a turbo pump if you can not tolerate oil in your chamber. However, remember that turbo pumps can not handle corrosive gases or particulates and they are expensive. If a slight amount of oil can be tolerated and corrosive gases are present, use a diffusion pump. A diffusion pump will also be required if you have very hot gases entering the pump.

Designing a diffusion pump system starts with roughing pump. Screw in a NW25 flange adapter into the inlet port (see Fig 5). The tubing from the roughing pump goes to the roughing valve which can be switched either to the chamber (through the main valve) or to the diffusion pump. The tubing to the chamber would include a Pirani gauge attached to a "T" fitting. A Penning gauge could be located on top of the main valve or it can be attached close to the chamber for better readings.

To operate this system, start by shutting the main valve and turning off the Penning gauge. Turn on the diffusion pump. Switch the roughing valve so the roughing pump is drawing from the chamber. Turn on the roughing pump. When the pressure is below 0.1 mbar, switch the roughing pump to back the high vacuum pump. Switch on the Penning gauge and open the main valve.

A turbo system, with a 50 l/sec pump, is setup in a similar manner except that the roughing can be done through the turbo pump. An inlet screen should be used to protect the pump from large particles.

Leak detection

The ability to reach and then maintain a good vacuum depends upon the quality of the pumping equipment and the condition of the vacuum chamber and fittings.

Reaching a good vacuum

To reach the needed vacuum, your vacuum pumping equipment must be in good condition. The oils in the roughing and diffusion pumps must be free of contaminating gases and chemicals. All seals must be clean and tight. Before starting the pumping system, it should be sealed off from the chamber and tested to assure that it can reach the desired vacuum. Gauges should also be checked with known gases to determine that they are giving reasonable readings. If the system does not function properly, test the roughing pump first and then test the system as addition components are added.

Maintaining a good vacuum

Vacuum is a very dynamic environment. Once you have achieved a high vacuum, you can not shut the main

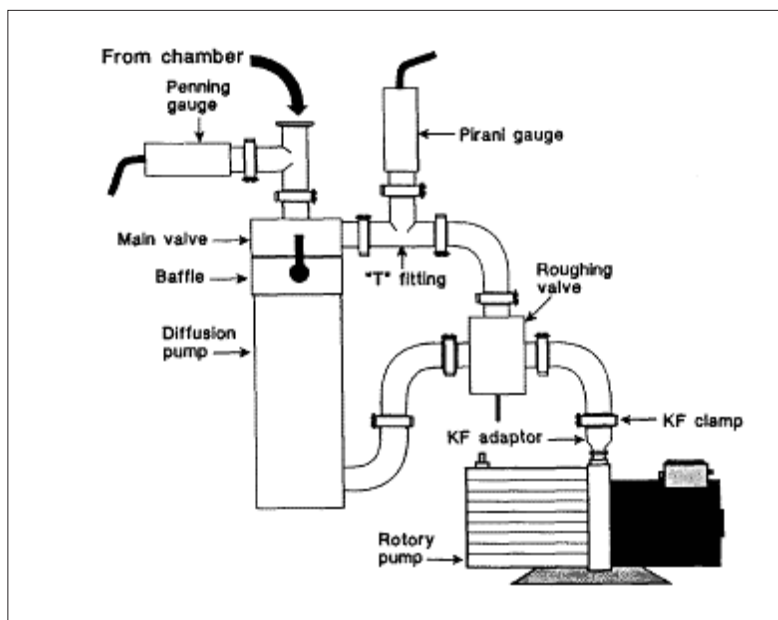


Figure 4: Roughing pump system

valve and expect the high vacuum to hold. First, there will be some leaks that decrease from 10^{-6} to 10^{-3} mbar in > 1 hour is considered to be a very tight chamber. However, any mistakes in technique will reduce the vacuum much more rapidly. Assuming that the o-rings have been installed properly, Viton o-rings will still leak some hydrogen and helium from the air. Even assuming that the o-rings did not leak at all, the walls of the chamber and tubing are continually outgassing. Unless the chamber and fittings have been well baked, most of the outgassing will consist of moisture (see next section). Even if there is no moisture attached to the walls, the walls may outgas some gases depending upon the wall material. Last but not least, contamination carried over from previous experiments is another probable source of outgassing. A 2.5 liter chamber (about the size of a Thermo Scientific CAHN

balance chamber plus sample tubes) that decreases from 10^{-6} to 10^{-3} mbar in > 1 hour is considered to be a very tight chamber. However, any mistakes in technique will reduce the vacuum much more rapidly.

If you can not maintain a vacuum in the rough or medium vacuum regions, you have a serious leak which should be easy to find. If you can not maintain a high vacuum, you may have a small leak or an outgassing problem. Before beginning the actual leak searching, information on the rate of the leakage should be obtained. This information can be obtained by the pressure rise method. Pump down to the desired vacuum and then close the main valve. Record the pressure rise that occurs in 1 hour. Repeat this procedure two or three more times over a period of

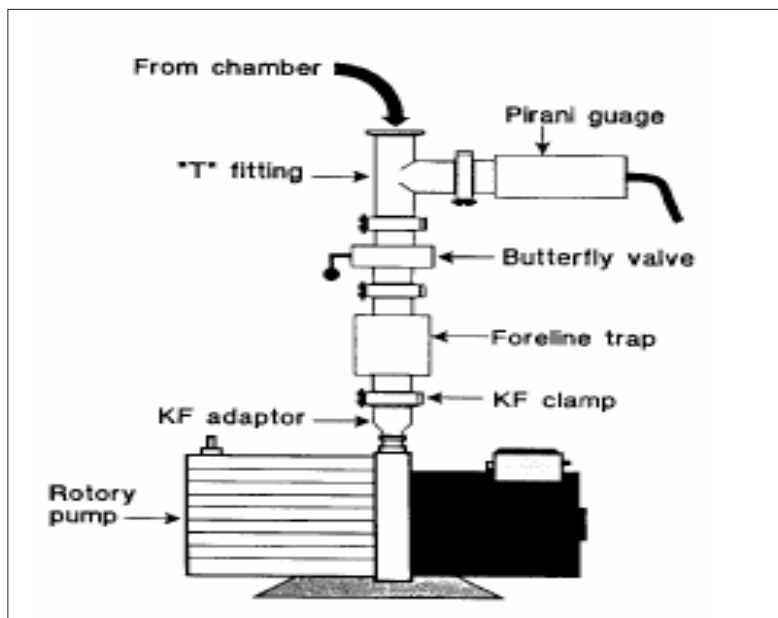


Figure 5: Diffusion pump system

days. If the rate of the pressure rise is constant, you have a leak. If the rate decreases, you have an out-gassing problem.

If you have a leak, check your o-ring joints. The o-rings should be without any twists, clean and evenly seated in the groove or on the centering ring. If they are deformed in any way, replace them. If this does not solve the problem, you may need a leak detection system. These systems can cost more than your vacuum system, so again check your seals.

Moisture: A special problem

Moisture is the number one cause of loss of high vacuum. It is in the air, and it clings to everything you put into a vacuum chamber. Because of the polar nature of water, it can build hundreds of layers of moisture on almost any surface. In high vacuum, moisture will outgas for days. Yet, the same chamber without moisture will pump down in 10 minutes. To use high vacuum effectively, you must make a special effort to remove moisture from the vacuum system and minimize its re-introduction.

Heat is the best method of removing moisture from a surface. It is this procedure that causes most vacuum manufacturers to list the temperature rating of their components. Baking all of the parts of a system at over 100 °C for a minimum of eight hours is often required for high vacuum work. Thermo Scientific CAHN balances have an upper limit of 125 °C while Viton o-rings have a limit of 175 °C. Wrapping the components of the system with heating tape or mantles is about the easiest method of applying heat. Infrared and ultraviolet lamps can also be used. However, you must be careful not to exceed the temperature limits of any of the parts. Another procedure for reducing moisture is to flush the system with dry air or nitrogen before starting the pump down. Flushing for one hour will remove about 75% of the moisture on the surfaces. However, your pumping time will not be reduced by 75% because the remaining

layers are the most difficult to remove and heat may still be required.

Once you have a moisture-free system, the problem now is how to minimize the re-introduction of the moisture during sample loading. The clean surface acquired during the pump down will preferentially adsorb the highly polar water molecules. Instead of exposing a chamber to room air, break the vacuum with dry nitrogen. Even though the chamber will be exposed to the water in the air when the chamber is opened, a number of layers of nitrogen will coat the clean inner surfaces of the system and reduce water adsorption. Since the most tenacious moisture layers are those closest to the solid surfaces, the nitrogen layers on the surface will greatly reduce the time to outgas the moisture. Once the moisture layers are removed, the nitrogen will outgas rapidly. If the system will not be used for some time, flush the system with dry nitrogen and then seal it until it is needed again. Be aware that the sample itself may be a large source of moisture that can contaminate a clean chamber. If possible, it should be heated with infrared lamps then blanketed with nitrogen as it cools before being put into a clean system.

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