

COMPLIMENTS OF:



Eastern PA (Corporate)
1969 Clearview Road
Souderton, PA 18964

Western PA
30 Industrial Road
Hermitage, PA 16148

California
8606 Live Oak Lane
Fontana, CA 92335

Southeast
108 Progressive Court
Greenville, SC 29611



1983 Clearview Road
Souderton, PA 18964



174 Keystone Drive
Telford, PA 18969



2299 Amber Drive, Suite 130
Hatfield, PA 19440

Basic Vacuum Practice

Third Edition

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1

Vacuum Fundamentals

Here is a list of things we think you should be able to do after reading this chapter:

You should be able to—

1. Use the basic terminology for vacuum technology.
2. Explain what a vacuum is.
3. Give some uses for vacuum.
4. Explain why a vacuum is necessary for some processes.
5. Discuss the two important types of gas flow.
6. Describe the effect of temperature and pressure on a volume of gas.
7. Recognize and use pressure units correctly.
8. Use the term “throughput” to describe vacuum system, pump operation and gas load.

Introduction

In the first part of this chapter, we will introduce you to vacuum:

- What it is
- How it relates to pressure
- How it is produced
- The different types of vacuum
- Where it is used
- Why we need it

Then, you will learn about the way we express very large and very small numbers. You will also learn about temperature as a factor in vacuum work and the types of pressure and how it is measured.

Finally, we will discuss some of the basic concepts used in vacuum work. These are:

- The effects of pressure
- Pressure ranges in vacuum systems
- Some basic laws about the behavior of gases
- Some types of gas flow
- How we measure the work done by vacuum systems

The Nature of Vacuum

What Is Vacuum?

vacuum

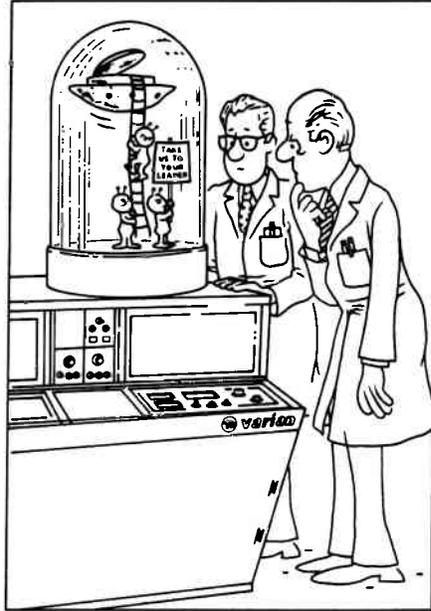
work chamber

The word *vacuum* comes from the Latin “vacua,” which means “empty.” Actually, vacuum is only partially empty space. In a vacuum, some of the air and other gases have been removed from a contained volume. This volume is usually called the *work chamber*. It separates the vacuum from the outside world.

A more practical definition for vacuum is what exists in any contained volume where there is less gas than there is in the surrounding atmosphere.

We shall see that these gases exert a force on the surface area of the container. This force is called *pressure*. We can measure the

pressure in the chamber by comparing it to the atmospheric pressure on the outside. In this way, we can find out how much gas is left in the vacuum.



What About Pressure?

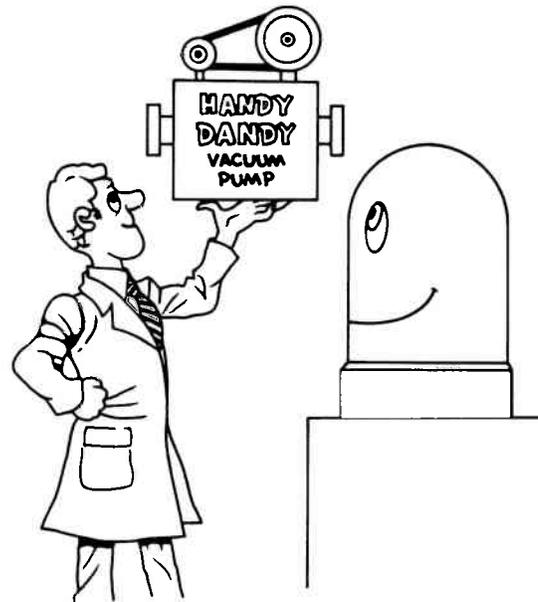
pressure

pressure measurement

Pressure is defined as force per unit area. Gases are composed of small particles. These gas particles are in constant motion. As these particles move around in space, they hit objects. When they hit something, they exert a force, or pressure. We can take a unit of area and measure the number and intensity of particle impacts on that surface. The result is a *pressure measurement*.

How Is a Vacuum Produced?

A vacuum is made by removing air and other gases from the work chamber. We remove the air and other gases by using special pumps, called vacuum pumps.

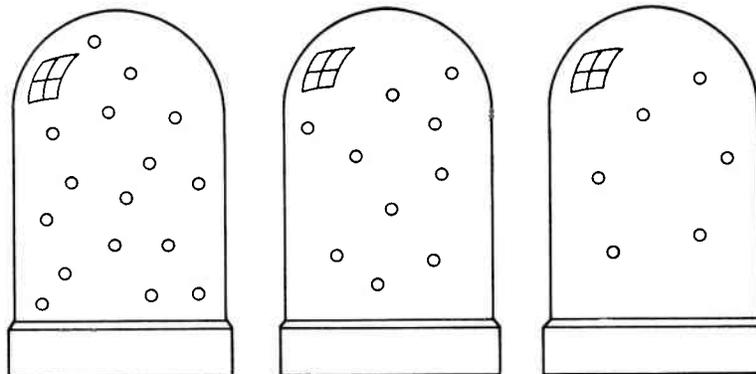


vacuum pumps

There are many, and very different, kinds of *vacuum pumps*. Some of them actually remove the gases. Other pumps trap the gases or change their form. In any case, the pump's job is to take as many gases out of circulation as necessary.

Different Types of Vacuum

There are different degrees of vacuum, called rough vacuum, high vacuum, and ultrahigh vacuum. Which one is used depends on the application. As the chambers below show, the better (or higher) the vacuum is, the less air and gas are present.



GOOD
ROUGH
VACUUM

BETTER
HIGH
VACUUM

BEST
ULTRAHIGH
VACUUM

Where Is Vacuum Used?

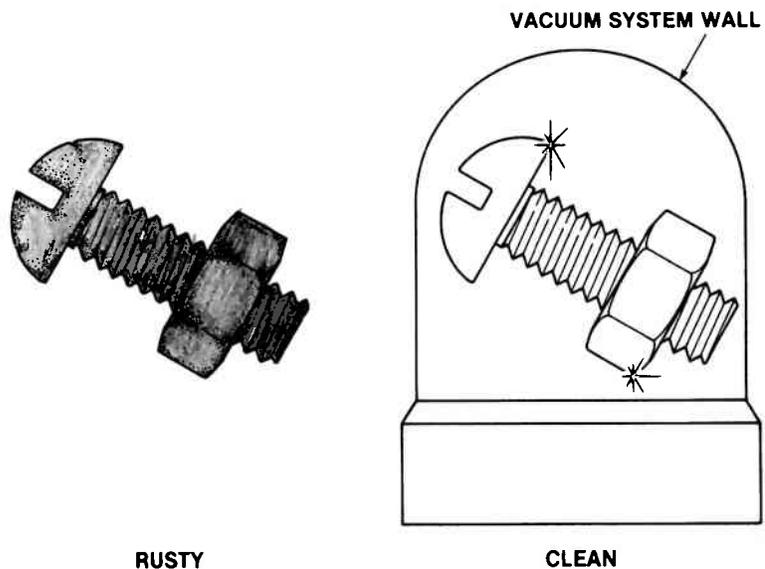
Vacuum is used for many products and processes. Some of them are:

Rough Vacuum	High Vacuum	Ultrahigh Vacuum
Food processing	Tube processing	Space research
Evaporation	Heat treating	Materials research
Freeze drying	Integrated circuit manufacture	Metallurgy
Distillation	Decorative coating	Physics research
Sputtering	Particle acceleration	Surface analysis
Electrical conduction (neon lights)	Chemistry research	Molecular beam epitaxy
	E-beam welding	
	Vapor deposition	
	Ion implantation	
	Insulation (thermal)	

Why Is Vacuum Needed?

We use a vacuum when we need a space that is very *clean*. It must be free of gases that can interfere with what we want to do.

Let's take iron, for example. When iron is left out in air, it reacts with the gases in the air, and the result is rust. This wouldn't happen in a vacuum.



Another example is television. If gases are not removed from a TV tube, the electrons are blocked from reaching the screen—no picture!

The easiest way to define “clean” is to say that everything is contaminated, or dirty, to some degree. It is a matter of how much contamination is present. The less contamination, the “cleaner” something is.

Let’s look at some of this contamination that we are trying to remove.

**DRY ATMOSPHERIC AIR
IS A MIXTURE OF GASES**

Gas	Percent by Volume
Nitrogen	78.08
Oxygen	20.95
Argon	0.93
Carbon Dioxide	0.03
Neon	0.0018
Helium	0.0005
Krypton	0.0001
Hydrogen	0.00005
Xenon	0.0000087

Atmospheric air is a mixture of gases. Over 99% of atmosphere is nitrogen and oxygen. All other gases make up less than 1%.

Water vapor, another common gas, is not listed above because the amount changes with atmospheric pressure and temperature. Water vapor, which varies from 0.6% to 6% by volume, is one of the biggest sources of vacuum contamination, or “dirt.”

Large and Small Numbers

Powers of Ten

powers of ten

You may already be familiar with the *powers of ten*. It is a way of describing very large and very small numbers. It is called exponential notation, scientific notation, logarithmic numbers, or simply “powers of ten.” Powers of ten is a simple, convenient way for writing and working with very large and very small numbers. For example, one million = 1×10^6 , and one-millionth = 1×10^{-6} .

Some Powers of Ten

$1 \times 10^6 = 1,000,000$	$1 \times 10^{-1} = 0.1$
$1 \times 10^5 = 100,000$	$1 \times 10^{-2} = 0.01$
$1 \times 10^4 = 10,000$	$1 \times 10^{-3} = 0.001$
$1 \times 10^3 = 1,000$	$1 \times 10^{-4} = 0.0001$
$1 \times 10^2 = 100$	$1 \times 10^{-5} = 0.00001$
$1 \times 10^1 = 10$	$1 \times 10^{-6} = 0.000001$
$1 \times 10^0 = 1$	

In practice, a number is written as some value from 1 and up to 10, but not including 10. Then, it is multiplied by either a positive or negative power of ten. For example, $7.6 \times 10^2 = 760$.

The small 2 to the upper right of the 10 is called an exponent, or power. The exponent is the number of times the first number is multiplied by 10 (2 in the example above). Where the exponent is a *minus*, it is the number of times the first number is divided by 10.

You may be interested in these very large numbers—

*Approximate number
of particles in:*

Atmosphere	$\approx 10^{40}$
Earth	$\approx 10^{60}$
Visible Universe	$\approx 10^{100}$

and in some very small numbers—

Typical Molecule Diameter	$\approx 10^{-8}$ in.
Bacteria	$\approx 10^{-3}$ in.

Using the Powers of Ten

Let's try a few examples using the powers of ten. But first of all, the rules of the game.

1. *To add or subtract:* First, adjust the numbers to make the exponents the same value. Then, add or subtract.

Adding—No adjustment needed

$$\begin{array}{r} 2 \times 10^{-3} \\ + 3 \times 10^{-3} \\ \hline 5 \times 10^{-3} \end{array}$$

Adding—Adjustment needed

$$\begin{array}{r} 2 \times 10^{-4} \\ + 3 \times 10^{-3} \\ \hline ? \end{array}$$

(continued on next page)

Adjusting gives you:

$$\begin{array}{r} 0.2 \times 10^{-3} \\ + 3.0 \times 10^{-3} \\ \hline 3.2 \times 10^{-3} \end{array}$$

To adjust, we changed the 10^{-4} exponent by *one* power to 10^{-3} . We then moved the decimal in the whole number *one* place to the left.

Here's another example:

$$\begin{array}{r} 2 \times 10^{-5} \\ + 3 \times 10^{-3} \\ \hline ? \end{array}$$

Adjusting gives you:

$$\begin{array}{r} .02 \times 10^{-3} \\ + 3.00 \times 10^{-3} \\ \hline 3.02 \times 10^{-3} \end{array}$$

To adjust, we changed the 10^{-5} exponent by *two* powers—to 10^{-3} . We then moved the decimal in the whole number *two* places to the left.

Subtracting—No adjustment needed

$$\begin{array}{r} 6 \times 10^{-4} \\ - 5 \times 10^{-4} \\ \hline 1 \times 10^{-4} \end{array}$$

Subtracting—Adjustment needed

$$\begin{array}{r} 6 \times 10^{-4} \\ - 5 \times 10^{-5} \\ \hline ? \end{array}$$

Adjusting gives you:

$$\begin{array}{r} 6.0 \times 10^{-4} \\ - 0.5 \times 10^{-4} \\ \hline 5.5 \times 10^{-4} \end{array}$$

2. To multiply: Add exponents.

- a) $10^1 \times 10^2 = 10^3$ b) $10^{-1} \times 10^2 = 10^1$
 c) $(2 \times 10^1) \times (3 \times 10^2) = 6 \times 10^3$

3. To divide: Subtract exponents.

- a) $\frac{10^3}{10^2} = 10^1$ b) $\frac{10^2}{10^3} = 10^{-1}$
 c) $\frac{4 \times 10^3}{2 \times 10^2} = 2 \times 10^1$

4. To raise to a power: Multiply exponent by power.

- a) $(10^1)^3 = 10^3$ b) $(10^2)^2 = 10^4$
 c) $(2 \times 10^1)^3 = 8 \times 10^3$

(continued on next page)

5. To find a root: Divide by root.

$$\text{a) } \sqrt{10^2} = 10^1$$

$$\text{b) } \sqrt[3]{10^6} = 10^2$$

$$\text{c) } \sqrt[3]{9 \times 10^3} = 3 \times 10^1$$

Here is a statement which results from division:

A pressure of 10^{-3} torr is 1 million times greater than 10^{-9} torr.

$$\frac{1 \times 10^{-3}}{1 \times 10^{-9}} = 1 \times 10^6 \text{ (which is 1 million)}$$

Two more examples of division:

$$\frac{10^2}{10^2} = 10^0 = 1$$

$$\frac{3 \times 10^3}{1.5 \times 10^{-7}} = 2 \times 10^{10}$$

Powers of Ten and Number of Decimal Places

When you write the number "1", it is taken for granted that you are really writing "1." with a decimal point to the right.

When the decimal point is on the right side of "1.", it is said to be in the "zero" position and so, $1 \times 10^0 = 1$. If it is one more position to the right, it is in the "one" position, so $1 \times 10^1 = 10$.

Likewise, when the decimal point is on the left side of one, "0.1", it is in the minus one position and $1 \times 10^{-1} = 0.1$. Notice that the minus exponent also means "one divided by that number."
Thus: $1 \times 10^{-1} = 1/10$.

Temperature

We have mentioned temperature already in our discussion. Most of us are familiar with the Fahrenheit (°F) and the Celsius or Centigrade (°C) scales of temperature measurement. In the world of vacuum, we are also concerned with the absolute temperature as well.

temperature

Temperature is a qualitative measurement of energy. The hotter something is, the more energy it contains. Or, if we want to get rid of gases, we could pump the energy out of them until they become frozen. That is, we have lowered the temperature of the gases.

absolute temperature

Calculations of heat and energy do not work well in the Celsius and Fahrenheit scales because of the negative numbers. This is where the *absolute* or Kelvin scale comes in. Let's compare some temperatures and conversion factors.

°F	°C	°K	
212	100	373	Boiling point of water
32	0	273	Freezing point of water
-321	-196	77	LN ₂ temperature
-437	-261	12	Cold head temperature
-459	-273	0	Absolute zero

Conversion factors

$$^{\circ}\text{C} = \frac{5}{9}(\text{F} - 32) \quad ^{\circ}\text{K} = \text{C} + 273$$

$$^{\circ}\text{F} = \frac{9}{5}\text{C} + 32 \quad ^{\circ}\text{K} = \frac{5}{9}(\text{F} - 32) + 273$$

Now let's discuss some information about gases.

Pressure

Earlier we defined pressure. Now, we'll explain the kinds of pressure vacuum is concerned with. We'll also describe how we measure pressure. First, let's look at what a gas is.

What Is Gas?

gas

What is a *gas*? It is a state of matter where the individual particles are free to move in any direction and tend to expand uniformly to fill the confines of a container. The gas particles are very small and freely moving. Some, like hydrogen and oxygen, are very reactive and easily form stable chemical compounds with other gases or elements. Other gases, such as helium and argon, are inert. These are sometimes known as the noble (inert) gases. They do not tend to form compounds.

pressure

All gases have mass and are thus attracted to the earth by the force of gravity. This "ocean" of gas we call "air" has weight. This weight pushing on the earth's surface is called atmospheric pressure. By definition, *pressure* (P) is the force (F) exerted on some particular area (A), such as a square inch, square foot, or square centimeter. Put into mathematical terms,

$$P = \frac{F}{A} \text{ (Pressure = Force per Unit Area)}$$

standard atmosphere

At 45° N latitude and at sea level, the average pressure exerted on the earth's surface is 14.69 pounds per square inch (absolute), or 14.69 psia. When the temperature is 0°C, this 14.69 psia is called a *standard atmosphere* (1 std atm). Gas behavior is usually described with reference to "standard conditions" of temperature and pressure (stp).

Atmospheric Pressure

We use several different pressure scales. Here are four readings, all at standard conditions.

$$14.7 \text{ psia} = 760 \text{ torr} = 1 \text{ std atm} = 101,325 \text{ pascal}$$

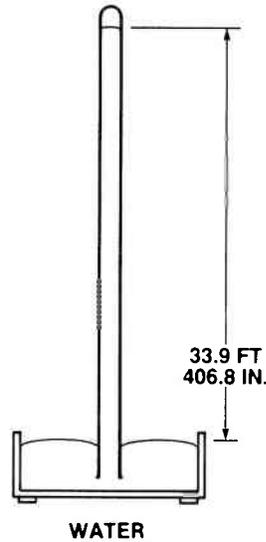
The average atmospheric pressure at sea level (45° N latitude) is 14.7 psia, 760 torr, or 101,325 Pa. Vacuum processes are usually done at pressures much lower than atmospheric pressure. Atmospheric pressure changes with distance above sea level (altitude) and changes in our weather.

AVERAGE PRESSURE AT VARIOUS ALTITUDES

Altitude (Ft)	Pressure (Torr)	Altitude (Ft)	Pressure (Torr)	Altitude (Ft)	Pressure (Torr)
- 1,000	787.87	7,000	586.49	25,000	282.40
- 500	773.83	7,500	575.45	27,500	253.00
0	760.00	8,000	564.58	30,000	226.13
500	746.37	8,500	553.88	35,000	179.33
1,000	732.93	9,000	543.34	40,000	141.18
1,500	719.70	9,500	532.97	45,000	111.13
2,000	706.66	10,000	522.75	50,000	87.497
2,500	693.81	11,000	502.80	55,000	68.889
3,000	681.15	12,000	483.48	60,000	54.236
3,500	668.69	13,000	464.76	70,000	33.662
4,000	656.40	14,000	446.63	80,000	21.010
4,500	644.30	15,000	429.08	90,000	13.208
5,000	632.38	17,500	387.65	100,000	8.356
5,500	620.65	20,000	349.53	120,000	3.446
6,000	609.09	22,500	314.51	140,000	1.508
6,500	597.70				

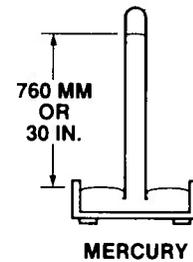
Source: U.S. Standard Atmosphere, 1962 (NASA)

A way to measure the force exerted by the atmosphere was developed in the mid-1600s by Evangelista Torricelli. It consisted of balancing a fluid of known weight against the weight of air. The first fluid used was water. Later, mercury was used. The measurement was made using an instrument called a barometer. We have named a pressure unit, torr, in Torricelli's honor.



$$\begin{aligned} 1 \text{ IN.}^3 \text{ OF WATER} &= .036 \text{ LB} \\ \text{WEIGHT OF WATER} &= 406.8 \text{ IN.}^3 \times .036 \text{ LB/IN.}^3 \\ &= 14.69 \text{ LB} \end{aligned}$$

NOTE: MERCURY IS 13.56 TIMES HEAVIER THAN WATER, SO THE MERCURY BAROMETER WILL BE 13.56 TIMES SHORTER; I.E., $\frac{406.8 \text{ IN.}}{13.56} = 30 \text{ IN.}$



THE BAROMETER

Pressure Measurement

millimeters of mercury
torr
microns
pascal

There are several different scales for pressure measurement. *Millimeters of mercury*, *torr*, and *microns* are all commonly used. *Pascal (Pa)* is the metric unit for pressure measurement and is the international standard.

The following table shows some of the common scales. The values for these scales are all listed at the same pressure— one standard atmosphere (1 std atm).

PRESSURE EQUIVALENTS	
Atmospheric Pressure (Standard) =	
0	psig (gauge pressure)
14.7	pounds per square inch (psia)
760	mm of mercury
760	torr
760,000	millitorr or microns
101,325	pascal
1.013	bar
1013	millibar

Here is a table for the equivalent values for one torr and one millitorr (mtorr).

One Torr =	One Millitorr =
$\frac{1}{760}$ atmosphere	$\frac{1}{1000}$ torr
1 mm of mercury	$\frac{1}{1000}$ mm of mercury
1000 microns or millitorr	10^{-3} torr
10^3 microns or millitorr	0.001 torr
133 pascal	1 millitorr
	0.133 pascal

A conversion table and equivalents for the different measurement scales are provided in the Appendix.

Partial Pressure

total pressure
partial pressure

The *total pressure* of a mixture of gases is the sum of each of the individual gas pressures in the mixture. This is known as Dalton's Law of Partial Pressure. Each individual gas pressure in a mixture is called a *partial pressure*.

PARTIAL PRESSURES OF GASES CORRESPOND TO THEIR RELATIVE VOLUMES

Gas (Air)	Symbol	Percent by Volume	Partial Pressure	
			Torr	Pascal
Nitrogen	N ₂	78	593	79,000
Oxygen	O ₂	21	159	21,000
Argon	Ar	0.93	7.1	940
Carbon Dioxide	CO ₂	0.03	0.25	33
Neon	Ne	0.0018	1.4×10^{-2}	1.8
Helium	He	0.0005	4.0×10^{-3}	5.3×10^{-1}
Krypton	Kr	0.0001	8.7×10^{-4}	1.1×10^{-1}
Hydrogen	H ₂	0.00005	4.0×10^{-4}	5.1×10^{-2}
Xenon	Xe	0.0000087	6.6×10^{-5}	8.8×10^{-3}
Water	H ₂ O	Variable	(5 to 50 torr typically)	Variable

At standard conditions (760 torr, 0°C), each gas exerts a pressure relative to its percent of the total volume: for example, N₂ = 78% = $0.78 \times 760 = 593$ torr.

evaporation
 vapor
 vapor pressure
 condensation
 sublimation

Vapor Pressure

When a liquid or solid becomes a gas, we call that process *evaporation*. The gas produced, we call a *vapor*. It, of course, exerts a pressure. This pressure, we refer to as the *vapor pressure* for that particular material. The act of turning the gas back into a liquid, we call *condensation*. When a solid evaporates to a gas directly, we call that process *sublimation*.

In general usage, vapors are gases that tend to condense back to the liquid state at moderate temperatures and pressures. All substances have a characteristic saturation vapor pressure that varies directly with temperature.

The lower the temperature, the lower the vapor pressure. This is true for all substances.

Water deserves special attention because of its behavior in the vacuum system. It is present in air as a gas in relatively large quantities. In the vacuum system, it is hard to remove condensed water vapor from surfaces at room temperatures.

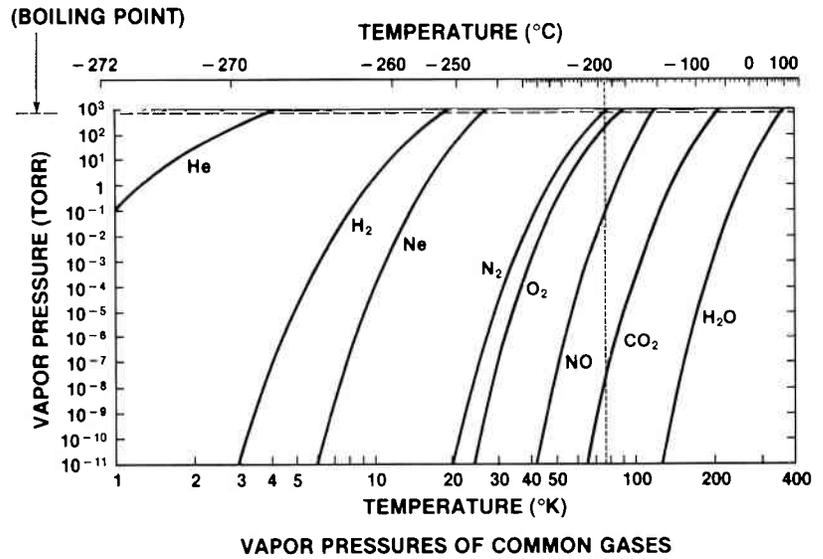
**VAPOR PRESSURE OF WATER
AT VARIOUS TEMPERATURES**

T °C		P Torr
100	(Boiling)	760
50		93
25		24
0	(Freezing)	4.8
-40		0.1
-78.5	(Dry Ice)	5×10^{-4}
-196	(LN ₂)	10^{-24}

VAPOR PRESSURES OF SOME LIQUIDS

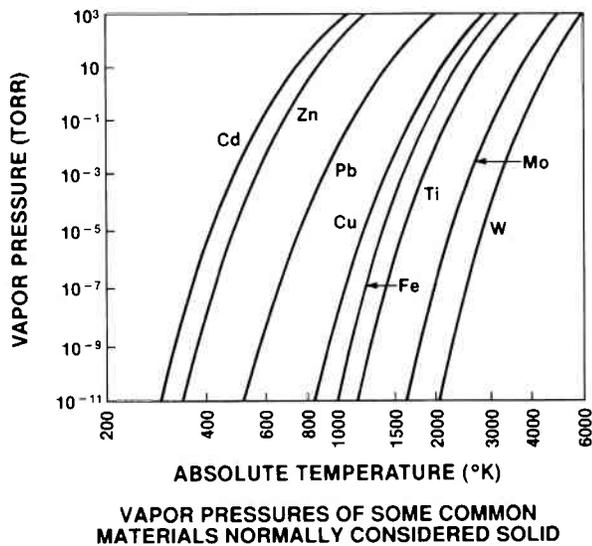
Liquid	Vapor Pressure Torr at 20 °C (68 °F)
Benzene	74.6
Ethyl Alcohol	43.9
Methyl Alcohol	96.0
Acetone	184.8
Turpentine	4.4
Water	17.5
Carbon Tetrachloride	91.0
High Vacuum Pump Oil	10^{-7}

Acetone has the highest vapor pressure of the liquids on this list. It evaporates the fastest of those substances on the list. It releases the most gas into the chamber in a given length of time. High vacuum pump oil is the least volatile liquid on the list. It will take the longest time to evaporate.



When gases become cooled sufficiently, they liquify and/or freeze. These curves give the vapor pressure for selected gases when they are liquids or solids. Curves to the right of the vertical dotted line (77°K, -196°C) indicate low vapor pressures at this temperature. Curves to the left show high vapor pressures at this temperature, which is the boiling point of liquid nitrogen.

Gases at the left side of the chart have high vapor pressures at extremely low temperatures. *Note:* Vapor pressure of all gases is the same at the boiling point in atmosphere (760 torr) even though they boil at different temperatures.

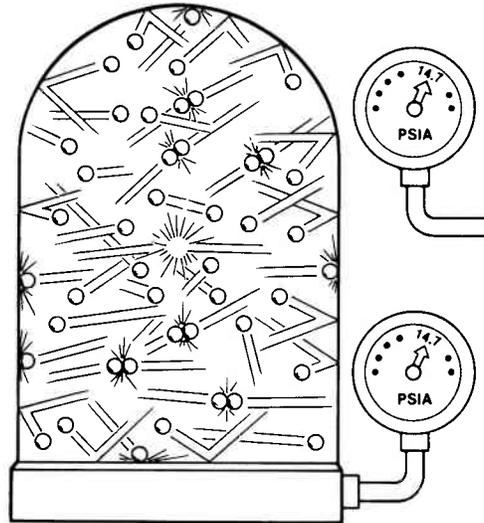


All materials have a vapor pressure, even though it may be very small. Note that, for some of these materials, their vapor pressure may be high enough to be a problem in some vacuum systems.

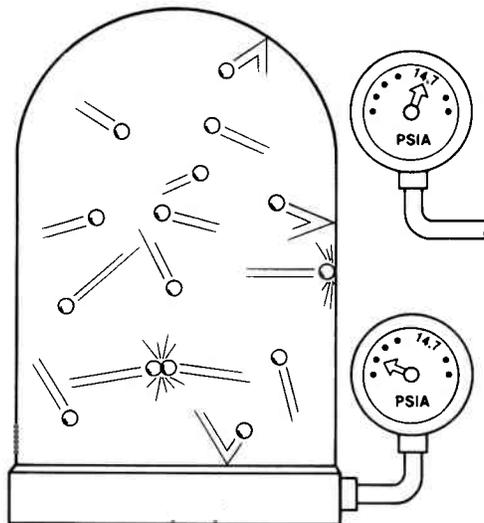
Effects of Pressure

absolute pressure

Before the air and other gases are pumped from the work chamber, constant, high-speed motion makes the particles bump into each other and into the chamber walls. This activity develops a total actual (absolute) pressure of 14.7 pounds per square inch (psia). As we have already seen, 14.7 psia is the average atmospheric pressure at sea level. Therefore, the pressure is the same inside and outside the chamber.



As air is pumped out of the chamber, pressure drops. However, we can never remove all particles from the chamber.



desorption
outgassing

After most of the free-moving gas (sometimes called the volume gas) is removed, there are still other sources of gas entering the system. Gases come off of surfaces in the vacuum system or out of the materials inside the work chamber. This is called *desorption* or *outgassing*.

implode

Vacuum systems can *implode* because of the external atmospheric pressure, causing the walls to collapse inward.

Pressure Ranges

These are the pressure ranges generally used in vacuum work:

rough vacuum

Rough (low) vacuum 759 to 1×10^{-3} torr (approx.)

high vacuum

High vacuum 1×10^{-3} torr to 1×10^{-8} torr (approx.)

ultrahigh vacuum

Ultrahigh vacuum Less than 1×10^{-8} torr

Gas Particles

atoms

Let's talk about the nature of the gases that exert this pressure. They are made from naturally occurring chemical elements. These elements are shown in the periodic table on the next page. These elements are the building blocks of earthly matter. The smallest identifiable part of an element is one of its *atoms*.

nucleus

protons

neutrons

An atom has a dense center portion known as the *nucleus*. This nucleus has particles called *protons* and *neutrons*. The protons have a positive electrical charge. Neutrons are neutral. This electrical charge in the nucleus is different for each element.

electrons

Under normal conditions, the nucleus is surrounded by a number of electrons. *Electrons* have a negative electrical charge. The number of electrons balances the positive charge and this makes the atom electrically neutral.

masses

atomic mass units (amu)

Neutrons and protons weigh the same and make up the bulk of the atom. The atoms of the different elements have different numbers of protons and neutrons. They thus have different *masses*. This means they have different weights (masses). They are classified by their atomic mass or weight. We call this *atomic mass units*, or *amu*.

PERIODIC CHART OF THE ELEMENTS

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molecules

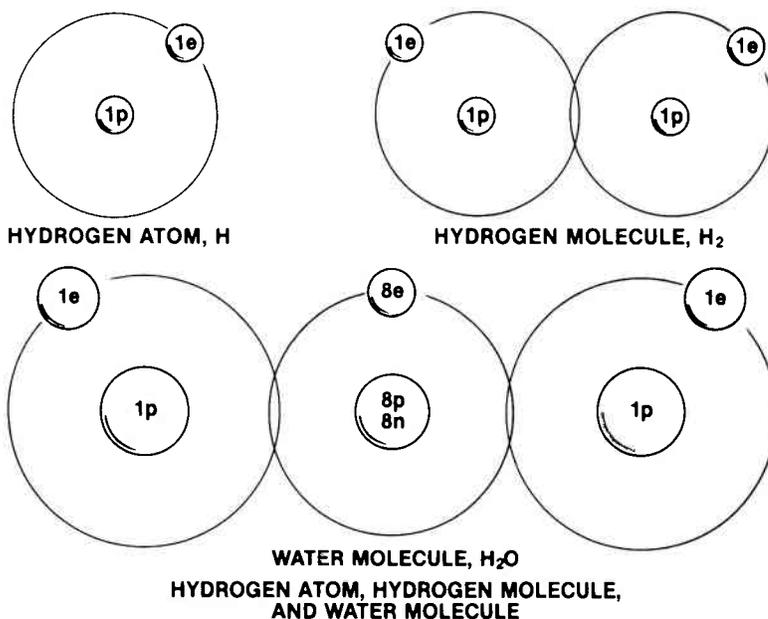
Molecules simply consist of one or more atoms joined together, with definite chemical and physical characteristics.

molecular weight

Molecules are likewise classified by their *molecular weight* (or mass). This is simply the sum total of the individual atomic weights that make up the molecule.

Some of the elements usually exist as gases. Some of these, like hydrogen, nitrogen and oxygen, travel as molecules with two or three atoms bound together.

Some gases are composed of more than one element, such as water.



For instance, the atomic weight of hydrogen (H) is 1 amu. Its molecule is made up of two hydrogen atoms (H₂) so its molecular weight or mass is 2 amu.

The atomic weight of oxygen is 16 amu. Thus, the molecular weight of water (H₂O) is 18 amu. That is the mass of two hydrogen atoms plus the mass of one oxygen atom (1 + 1 + 16).

ion

Under certain conditions, an atom or a molecule can become electrically charged. It is then referred to as an "ion." This process will be considered in more detail in the discussion on "Ionization."

Gas Laws

Let's look at what happens to gases as we use them in our vacuum system. We first assume that gases are perfect—and in general, they are. So we can apply some “laws” to their behavior. Let's look at some of these laws.

Avogadro's Law

mole

Under the same conditions of pressure and temperature, equal volumes of all gases have the same number of particles (molecules, actually). We call this a *mole*.

One mole of any gas has 6.023×10^{23} particles, under standard conditions (760 torr, 273°K), occupies 22.4 liters, and weighs one molecular weight.

Avogadro's Law

We know this as *Avogadro's Law*.

1. How many particles would be in a standard liter?

$$\frac{6.023 \times 10^{23} \text{ particles}}{22.4 \ell} = 2.69 \times 10^{22} \text{ particles}/\ell$$

standard cubic centimeter

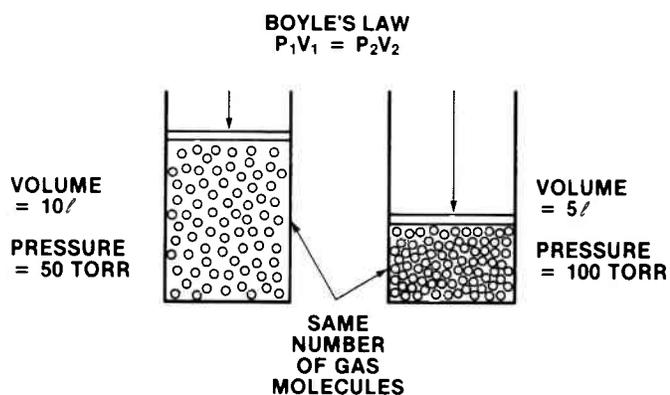
2. How many in a standard cubic centimeter?

$$\frac{6.023 \times 10^{23} \text{ particles}}{22.4 \ell} \times \frac{10^{-3} \ell}{1 \text{ cc}} = 2.69 \times 10^{19} \text{ particles/cc}$$

Boyle's Law

Boyle's Law

Boyle's Law, $P_1V_1 = P_2V_2$, or original pressure times original volume equals new pressure times new volume. Reduce the volume by half, the pressure is doubled. This equation predicts new pressure or new volume whenever the other is changed by any amount, providing that the temperature remains the same.



NOTE: TEMPERATURE HELD CONSTANT

Gas Expansion



Gas expands tremendously under vacuum (from Boyle's Law). This happens to gas absorbed in fingerprints and dirt in general. Water and solvents are also sources of large gas loads. The large volumes these materials produce are a major part of "outgassing."

Suppose you have a chamber which has a volume of 100 l at a pressure of 1×10^{-4} torr. If 1 std cc of gas is suddenly added, what will be the pressure?

Let's use Boyle's Law, $P_1V_1 = P_2V_2$.

Note that we are really calculating a new pressure, not a new volume. Also, the partial pressure of the gas we are adding will add to the gas pressure already there.

$$P_1 V_1 = P_2 V_2$$

For the gas we are adding to the chamber:

$$760 \text{ torr} \times 1 \text{ cc} = P_2 \times 100 \ell$$

Solving for P_2 and converting cubic centimeters to liters:

$$P_2 = \frac{760 \text{ torr} \times 1 \text{ cc}}{100 \ell} \times \frac{10^{-3} \ell}{1 \text{ cc}}$$

$$P_2 = 7.6 \times 10^{-3} \text{ torr}$$

Now the total pressure in the container is the sum of the pressure there (1×10^{-4} torr) plus the pressure from the gas we added (7.6×10^{-3} torr).

$$\begin{aligned} P_{\text{Total}} &= P_{\text{Chamber}} + P_2 \\ &= 1 \times 10^{-4} \text{ torr} + 7.6 \times 10^{-3} \text{ torr} \\ &= 7.7 \times 10^{-3} \text{ torr} \end{aligned}$$

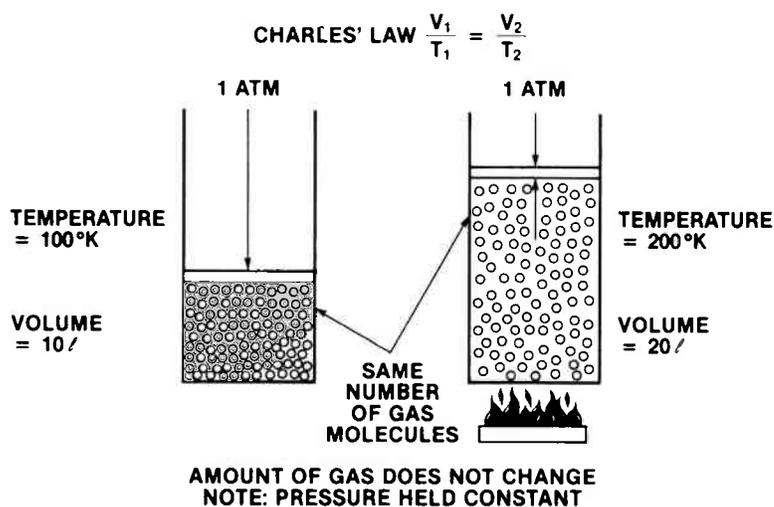
We see that the 1 cc of gas at atmospheric pressure contributed much more to the pressure in the chamber than the gas already there!

Charles' Law

Charles' Law

Let's look at what happens to the volume of gas as we change the temperature. As we cool a gas, its volume gets smaller. If we heat the gas, its volume increases. We call this *Charles' Law*. The equation looks like this:

$$\frac{V_1}{T_1} = \frac{V_2}{T_2}$$



Charles' Law states that if the absolute temperature is doubled, the volume of gas is doubled providing that the pressure is unchanged.

Law of Gay-Lussac

If Charles' Law is examined carefully, a more specific relationship develops.

If the temperature of a volume of gas at 0°C is changed by 1°C, the volume will change (plus or minus) by 1/273 of its original value. This is *Gay-Lussac's Law*. Thus:

$$V = V_0 + \left(\frac{^{\circ}\text{C}}{273}\right) \times V_0$$

Rearranging this equation gives us:

$$V = V_0 \left(1 + \frac{^{\circ}\text{C}}{273}\right)$$

Lord Kelvin used this relationship to develop the absolute temperature scale.

Gay-Lussac's Law

General Gas Law

general gas law

We can combine these laws to get a *general gas law* (Boyle's and Charles' combined):

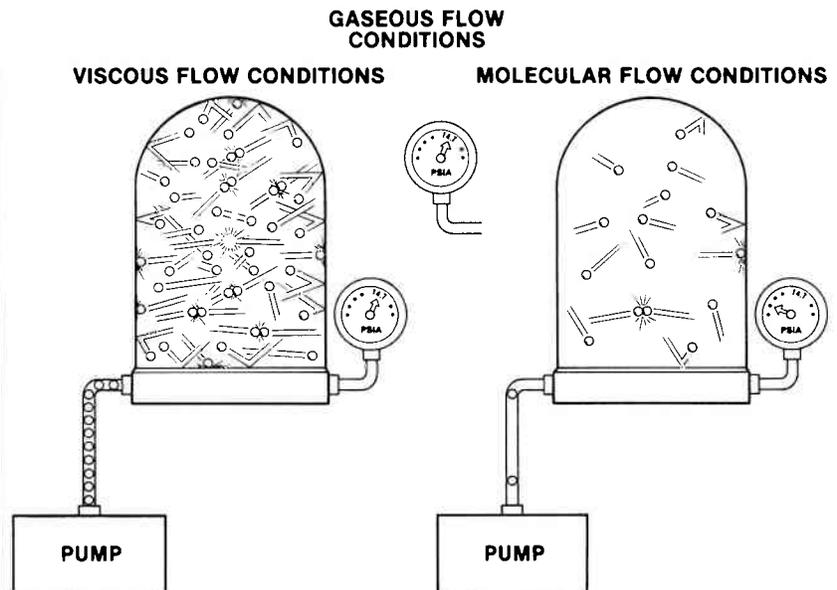
$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$$

The general gas law combines pressure, volume, and temperature in a single equation.

Note: The temperature in Charles' Law and the general gas law is expressed in the absolute scale, or degrees Kelvin; to convert from °C to °K, add 273° to °C; thus, 100°C + 273° = 373°K.

Gas Flow

Since we want to move gas molecules out of the vacuum chamber, we should know how gas flows. Of the many types of gas flow, we will discuss two kinds: viscous flow and molecular flow. Both types of flow have to do with how tightly molecules fill a space.



viscous flow

Viscous Flow

Generally, gas molecules occupying a space at a pressure greater than 1×10^{-2} torr act very much like a fluid, so this is called *viscous flow*. In the viscous flow range, the molecules are constantly bumping into each other. The molecules are so closely packed together that as our vacuum pump moves some of them out of the chamber, others will rush to fill up that empty space.

In viscous flow conditions, molecular movement is predictable. When a molecule is hit or hits a surface, we can predict its movement after impact with reasonable accuracy.

Because the molecules are tightly packed and move predictably, we can use smaller diameter hoses and tubulations for rough pumping operations.

Viscous flow conditions will generally allow us to move great quantities of molecules per unit time from one place to another.

Molecular Flow

molecular flow

Molecular flow occurs when the molecules are so far apart that they no longer have any influence on each other. Their motion is strictly random. This occurs at low pressures where fewer molecules are present.

Depending on the pressure, a gas molecule might travel inches, feet, or even miles before it strikes another molecule. This means we can't depend on molecular interaction to push or start a flow pattern.

In the molecular flow range, molecular movement is unpredictable. This is why we have such large inlets in high vacuum pumps.

The use of large inlets increases the probability that one of these randomly moving molecules will move into the pump.

In molecular flow, the molecular motion is explained by kinetic theory, which uses statistics (chance) to describe the condition.

The difference between viscous flow and molecular flow does not depend upon the pressure alone. It also depends upon the

dimensions of the vacuum container (pipes, chamber, etc.). Basically, it depends upon the mean free path and whether it is longer or shorter than the container dimensions. Let's take a look at what is meant by "mean free path."

Mean Free Path

mean free path

As we lower the pressure in the vacuum chamber, the amount of space between the gas particles increases. The particles bump into each other less frequently. The average distance a particle moves before it bumps another particle is the mean free path.

MOLECULAR DENSITY AND MEAN FREE PATH

	7.6×10^2 Torr (atm)	1×10^{-3} Torr	1×10^{-9} Torr
# mol/cm ³	3×10^{19} (30 million trillion)	4×10^{13} (40 trillion)	4×10^7 (40 million)
MFP	2×10^{-6} in.	2 in.	30 mi

At atmosphere, the mean free path is extremely short, about two millionths of an inch. Under vacuum, fewer molecules remain, and the mean free path is longer. Its length depends on the number of molecules present, and therefore on the pressure. The mean free path for air can be estimated from the relationship:

$$\text{Mean Free Path} = \frac{5 \times 10^{-3} \text{ torr cm}}{P_{\text{torr}}}$$

From this, we can see that as the pressure gets lower, the mean free path gets longer. Likewise, as the pressure gets lower, there are fewer molecules of gas present, so there is less chance of them running into each other.

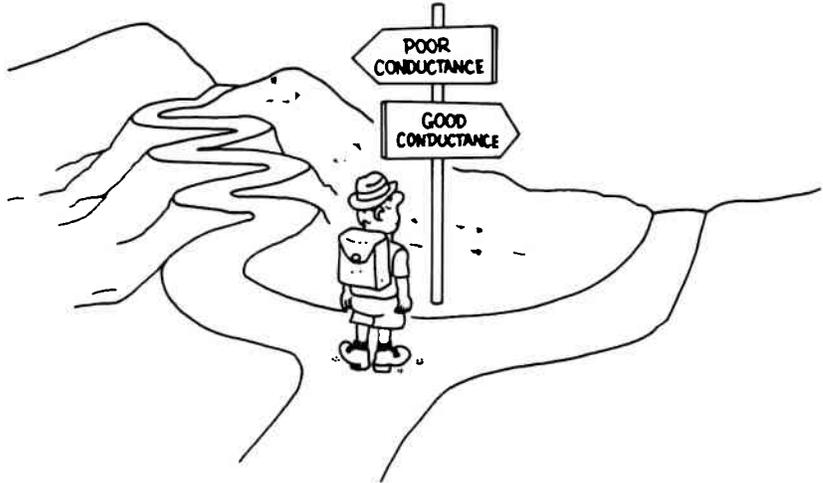
gas density
(*molecular density*)

In 1 cc of gas at standard conditions (760 torr at 0°C), there are about 3×10^{19} gas molecules and the mean free path is about 2×10^{-6} cm (a few millionths of an inch). At 1×10^{-9} torr, there are about 4×10^7 molecules/cc, and the mean free path is about 30 miles or 50 kilometers. The number of molecules per unit volume (in this example cubic centimeters) is called the *gas density*.

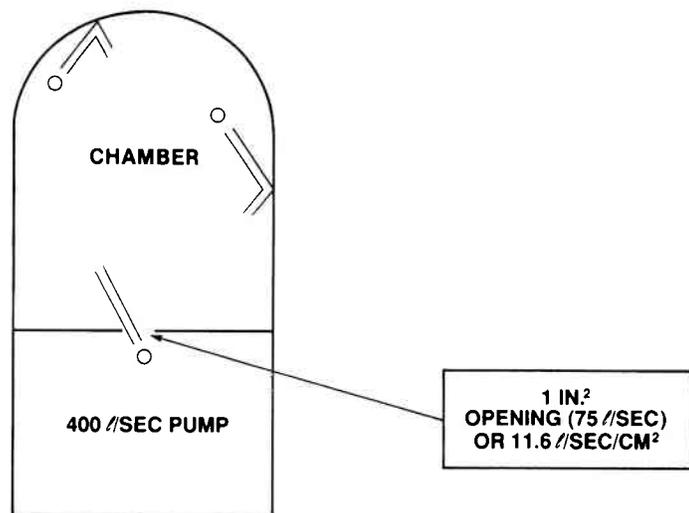
Conductance

conductance

When we talk about moving a gas through a vacuum system, we use the term conductance. *Conductance* is the ability of an opening or pipe to allow a given volume of gas to pass through in a given time. It is expressed in such units as liters per second, cubic feet per minute or cubic meters per hour.



In molecular flow, a good conductance path is wide and short. It has few turns, thus allowing free gas flow. In viscous flow, these conditions are not so important. This is because the molecules tend to push one another along under the influence of a pressure difference.



MOLECULAR FLOW

In the molecular flow range, a 1 in.² opening has a 75 //sec conductance. The pump speed, in this case 400 //sec, is really 75 //sec as far as the chamber is concerned because the mole-

cules must go through the hole before they can be pumped. To improve system performance, the conductance must first be improved. (Make the hole bigger!) To repeat: In the molecular flow range, a pump works only when molecules migrate into the pump by chance.

Conductance in Viscous Flow

The volume of gas that can flow per unit of time through a pipe under viscous flow conditions is related to the *fourth* power of the pipe diameter and is inversely related to the length of the pipe.

For example, if you use a pipe with a diameter twice that of the pipe presently being used, it will allow 2^4 or sixteen times as much gas to flow through it, assuming that the length of the pipe is the same.

Now let's compare this to molecular flow conditions.

Conductance in Molecular Flow

The volume of gas that can flow per unit of time through a pipe under molecular flow conditions is related to the cube of the diameter and inversely to the length of the pipe.

Using the same pipe as in the viscous flow example, doubling the diameter of the pipe will, at most, allow 2^3 or eight times the flow for the same length of pipe.

In either case of viscous flow or molecular flow, making the pipe shorter will increase the flow of gas through the pipe. Please note that these are gross statements that are subject to all kinds of qualifications.

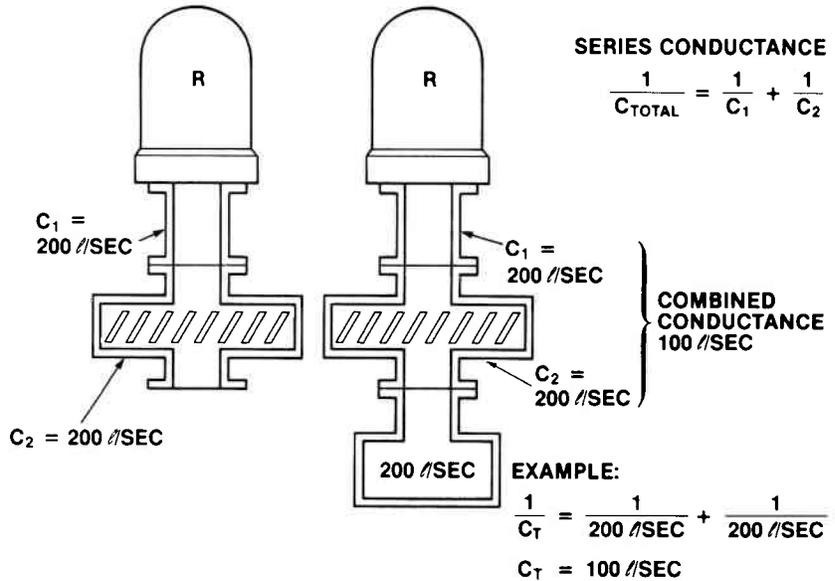
transition range

There is another region where we approach molecular flow, but the flow is not really viscous either. This region is called the *transition range*. There is another set of calculations to be used for the transition range, but we will not discuss them in this text.

Please see the appendix if you need further details on how to calculate conductances.

Series Conductance

When you place components in series in a vacuum system, the total conductance is less than the smallest of the conductances in series.



Let's look at the result (point "R" at the vacuum chamber) of adding a pump (shown in the drawing) with a speed (S) of 200 l/sec to the combined 100 l/sec series conductance.

The pump can be represented by another conductance of 200 l/sec in the line. In this case, we call the total conductance, R, the combined pipe conductance, C, and the conductance of the pump, S. So:

$$\frac{1}{R} = \frac{1}{C} + \frac{1}{S}$$

If we play with this a bit, we get:

$$R = \frac{CS}{C + S}$$

Thus:

$$R = \frac{\frac{100 \text{ l}}{\text{sec}} \times \frac{200 \text{ l}}{\text{sec}}}{(100 + 200) \text{ l/sec}}$$

$$= 66.6 \text{ l/sec}$$

Thus our 200 l/sec pump is effectively delivering only one-third of its speed to pump the work chamber.

What would be the effect of changing to a 2,000 ℓ /sec pump?

Going through a similar calculation:

$$R = \frac{\frac{100 \ell}{\text{sec}} \times \frac{2,000 \ell}{\text{sec}}}{(100 + 2,000) \ell/\text{sec}}$$

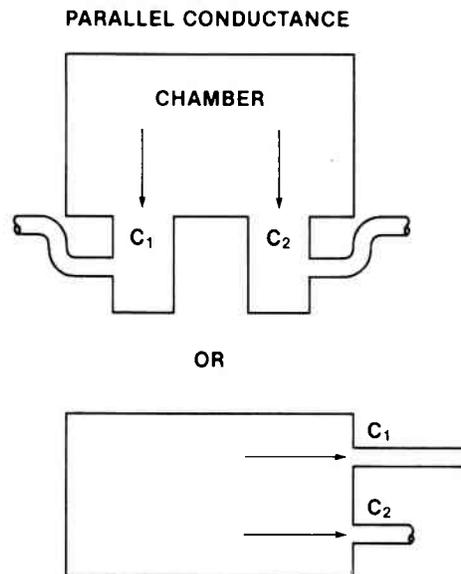
$$= 95 \ell/\text{sec}$$

An amazing improvement of 29 ℓ /sec! (But only about 5% of its speed is being used!)

conductance-limited

“The pump is no better than the pipe!” Remember that we cannot pump the gas until it reaches the pump. We are *conductance-limited* here.

Parallel Conductance



In parallel conductance,

$$C_{\text{total}} = C_1 + C_2$$

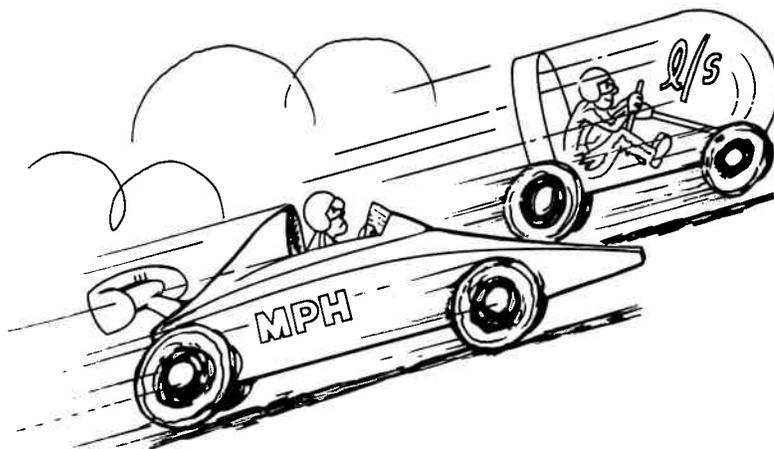
We simply add parallel conductances to get the total.

Let's go on, now, to see what kind of work our vacuum system does.

Measuring Work Performed by Vacuum Systems

pumping speed

We have many different ways to measure things. We may measure the performance of our car by how fast it will go, for instance. We measure how fast a vacuum pump is by stating its *pumping speed*. (More explicitly, by its volumetric pumping capacity.)



Pumping speed is rated in liters per second, or cubic feet per minute. Pumping speed alone does not tell us what we want to know about vacuum system work. We are really interested in getting the molecules out—not in pumping an “empty volume.” Our liters per second or cubic feet per minute tells us nothing about how many molecules are being removed from our vacuum chamber.

throughput

Air is easily removed from a chamber at high pressures. After a short while, not much gas is left, so pumping speed doesn't tell us what we want to know about vacuum system work per unit time. We need a new term: *throughput*, which is vacuum pumping capacity. The throughput tells us how many molecules we are pumping. Let's look at this in more detail.

gas load

Quantity of Gas

The amount of gas present in a system is determined by multiplying the pressure (torr) times the volume (liters). This tells us the actual number of gas molecules contained in a particular enclosure. We also call this the *gas load*. The usual units are torr-liters or pascal-liters.

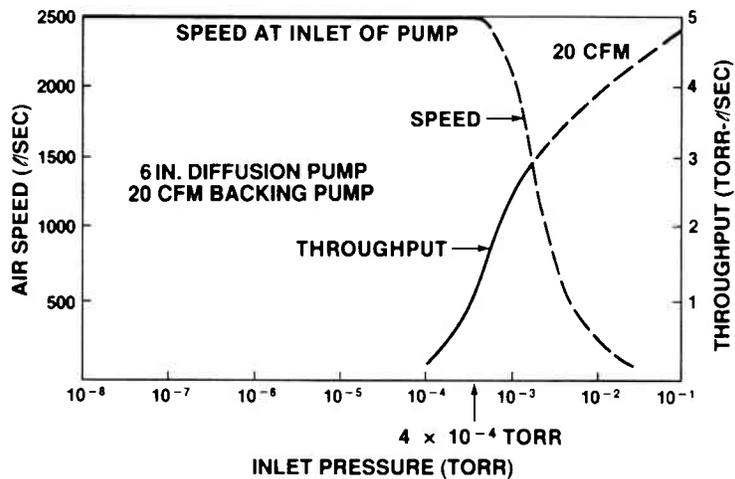
In vacuum, we are interested in how much work a pump has to do to transfer a mass of gas from one place to another. Pressure times the volume (PV) is a measure of this work. A point of even more interest is the time it takes to do this work, or (PV/t), which is the capacity of a pump. This is called the *throughput*. Typical units for throughput are torr-liters per second.

Throughput

$$Q = PS$$

Throughput is called Q . So throughput (or Q) equals pump speed times pressure per unit time, or quantity of gas flow. This more realistically helps define pumping work. (The lower the pressure, the less gas remains.) Speed in liters per second, times pressure, in torr, gives us Q in *torr-liters per second*.

Since pumping speed (S) is volume per unit time (V/t), then $Q = PV/t$. Torr-liters per second are the common units. You could also use pascal-liters per second or atm ft³/minute if you so desired. Let's look at a typical use for the pumping speed and throughput.



DIFFUSION PUMP SPEED
AND THROUGHPUT CURVES

Q = pressure times pump speed per unit time. At a pressure of 4×10^{-4} torr and a rated pumping speed of 2,500 ℓ /sec, we find

$$Q = 2.5 \times 10^3 \frac{\ell}{\text{sec}} \times 4 \times 10^{-4} \text{ torr}$$

$$= 1 \text{ torr-}\ell/\text{sec}$$

Pick out additional points on the horizontal axis of the chart to see how Q is affected as the pumpdown continues. Note that the throughput will continue to decrease as the pressure decreases. This is true even though the pump speed is constant. The throughput is telling us that it is getting harder to remove molecules because there are fewer of them per unit volume.

How $Q = PS$ Is Used

If the Q in a system is 1 torr- ℓ /sec and we had a 1,000 ℓ /sec pump, what pressure could be reached?

$$Q = PS$$

$$\frac{1 \text{ torr-}\ell}{\text{sec}} = P \times \frac{1,000 \ell}{\text{sec}}$$

Now, let's solve for P:

$$P = \frac{1 \text{ torr-}\ell}{\text{sec}} \times \frac{\text{sec}}{1,000 \ell}$$

$$= 10^{-3} \text{ torr}$$

or

If you wanted a pressure of 1×10^{-8} torr, what pumping speed is needed (assuming the same Q as above, 1 torr- ℓ /sec)?

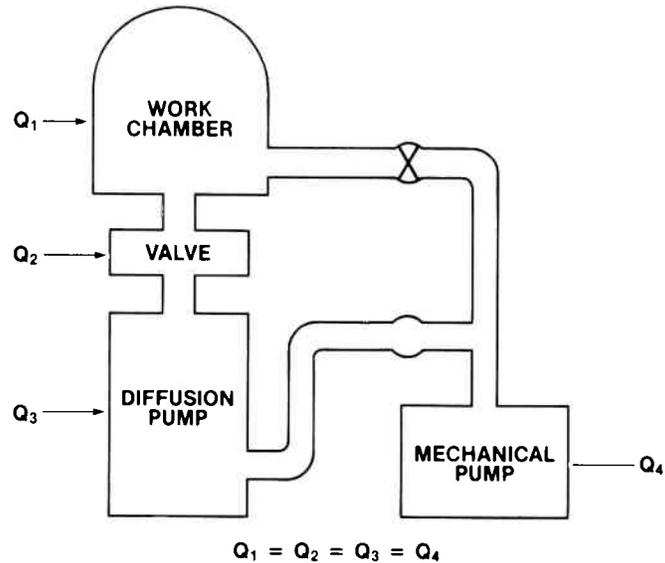
$$\frac{1 \text{ torr-}\ell}{\text{sec}} = 1 \times 10^{-8} \text{ torr} \times S$$

Solving for S:

$$S = \frac{1 \text{ torr-}\ell/\text{sec}}{1 \times 10^{-8} \text{ torr}}$$

$$= 1 \times 10^8 \ell/\text{sec}$$

That's ridiculous! 100 million ℓ/sec ($1 \times 10^8 \ell/\text{sec}$)! Nobody makes a pump that big— so what do you do?? We will have to change something— most likely our expectations!



In steady-state or equilibrium conditions, throughput is the same at one end of a vacuum system as it is at the other. Speeds and pressures may vary from point to point, but they combine to give the same throughput through all of the system. (This is important when selecting pumps to work together— more on this point later.)

Gas Load

Another name for Q , the throughput, is gas load. Where does the gas load, " Q ," come from? It comes from several places: leaks, outgassing, and contamination are all contributing to the gas load.

In the preceding problem, if we could fix the leaks, heat the surfaces, use the right materials, and clean up the system, the gas load, Q , could probably be reduced to about 1×10^{-5} torr- ℓ/sec . Let's calculate the needed pump speed for this (remember, we want to pump to 1×10^{-8} torr):

$$\frac{1 \times 10^{-5} \text{ torr-}\ell}{\text{sec}} = 1 \times 10^{-8} \text{ torr} \times S$$

Solving for S:

$$S = \frac{1 \times 10^{-5} \text{ torr-}\ell/\text{sec}}{1 \times 10^{-8} \text{ torr}}$$
$$= 1 \times 10^3 \ell/\text{sec}$$

That's more like it! This is a much more realistic value for the pump speed.

“Q” and Power

As mentioned before, Q is work per unit time or power. As such, it can be expressed in watts. If you want more throughput (power), you need more electricity (power). That is, you need higher power heaters and motors. The conversion is

$$7.50 \text{ torr-}\ell/\text{sec} = 1 \text{ watt} = 1,000 \text{ Pa } \ell/\text{sec}$$

For example, a 1000 ℓ/sec pump operating at 5×10^{-5} torr is 0.05 torr- ℓ/sec or much less than one watt of power.

Summary

Vacuum is an environmental condition produced in a suitable container. It is produced by reducing the number of gas particles per unit volume to below that which exists on the outside. The removal of gas particles is done by means of devices known as vacuum pumps. The number and temperature of the gas molecules in the container are responsible for the force exerted on the walls of the container. The force per unit area is called the pressure. The standard atmospheric pressure is 14.69 pounds per square inch absolute. This standard pressure is what air molecules would exert on the surface of the earth at 45° N latitude on a clear day at sea level.

The word *vacuum* comes from the Latin “vacua,” which means “empty.” Actually, the container is only partially empty, and varying degrees of emptiness are required for various types of work. Complete emptiness is not attainable. In other words, there is no such thing as a “perfect vacuum.”

We have examined in some detail the basic vacuum concepts and how materials and contamination affect getting a desired pressure. We have also seen how the hardware and plumbing can limit effectiveness of your pumps.

Vacuum is a valuable tool in many areas of science and industry. Its applications continue to grow.

So, let's get deeper into the pumps and hardware.

2

Roughing Pumps

Here is a list of things we think you should be able to do after reading this chapter:

You should be able to—

1. Give the three major pressure ranges of vacuum pumps.
2. List the types of pumps in each of the pressure ranges.
3. Describe the major components of each roughing pump type.
4. Explain how the major types of roughing pumps work.
5. Describe the place of these pumps in vacuum system use.
6. Describe in general how these pumps are maintained.

Introduction

It is important to choose the right pump for your vacuum system. A number of pumps are used in vacuum work. Some remove the gases from the chamber; others trap gases or change their form. Their ranges of operation differ, and usually no single pump develops the degree of vacuum needed. Therefore, the pumps are used in various combinations.

While some pumps seem quite ordinary, others pump in strange ways, using chemical or electrical methods. Some vacuum pumps merely capture and store the gases.

In this chapter, we will explain how one of the three general categories of vacuum pumps—roughing pumps—works and the general ways they are used. We will also discuss the major roughing pump components and how the pumps are maintained. And we will give an overview of how they fit into vacuum systems. System operation is explained in more detail in chapter 7.

First, however, we will describe vacuum pumps in general and their pressure ranges, and how pumps are teamed up.

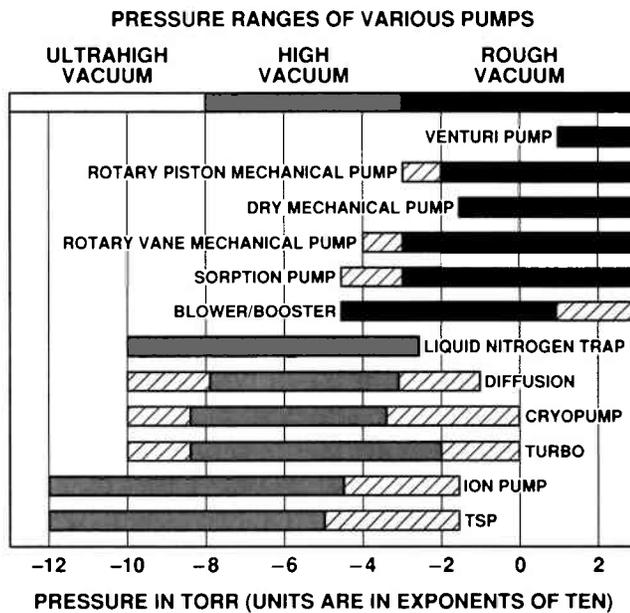
Pressure Ranges of Pumps

We cannot expect one pump to take a system from atmosphere to the high or ultrahigh vacuum range. Two or more pumps must be used to reach and sustain a particular pressure.

As you can see in the chart on the next page, each type or kind of pump has a useful operating range. At higher pressures, some might not operate or might even contaminate a system. Some pumps can cause contamination if they are exposed to pressures below their operating range. Usually, a pump will perform well if it is used within its operating pressure range. If the process pressure and system base pressure requirements are both in the rough vacuum range, a single roughing pump may be enough. Often, however, this is not the case.

useful operating range

A pump usually has a higher pressure limit where it will begin pumping, and a base pressure (or ultimate pressure), which is its lower limit. The pressure range between these two extremes is the pump's *useful operating range*.



This chart shows typical operating ranges for a variety of vacuum pumps. The full range is broad, going from atmospheric pressure down to 10^{-12} torr. The range is divided into rough vacuum, high vacuum, and ultrahigh vacuum operation. Baking the system is usually required to reach UHV range.

The solid bars indicate the normal *steady-state* operating range. The diagonally cross-hatched part of the bars indicate extensions of the normal operating range under *transient and special* conditions or may require the use of a specially-designed pump.

To summarize:

- *Roughing Pumps*: 760 torr to 1×10^{-3} torr

Rotary Piston Oil-Sealed Mechanical Pump
 Rotary Vane, Oil-Sealed Mechanical Pump
 Dry Vacuum Pump (oil-free)
 Sorption Pump
 Venturi Pump
 Booster/Blower Pump

- *High Vacuum Pumps*: 1×10^{-3} torr to 1×10^{-8} torr

Oil Diffusion Pump
 Cryotrap and Baffles
 Mechanical Cryopump
 Turbomolecular Pump

- *Ultrahigh Vacuum Pumps*: 1×10^{-8} torr and lower

Titanium Sublimation Pump (TSP)
 Ion Pump
 Non-Evaporable Getter Pump (NEG)

How Pumps Are Teamed Up

Vacuum pumps are teamed up. Usually no single pump develops the needed degree of vacuum. Therefore, pumps are used in various combinations. Typical combinations are shown in the following chart.

Roughing Systems	High Vacuum Systems	Ultrahigh Vacuum Systems
Mechanical Pump	Mechanical Pump Cryotrap or Baffle Diffusion Pump	Sorption Pump Cryotrap Ion Pump
Dry Vacuum Pump	Trapped Mechanical Pump	Titanium Sublimation Pump
Mechanical Pump	Mechanical Cryopump	Venturi Pump Sorption Pump
Booster/Blower Pump	Mechanical Pump Turbomolecular Pump	Cryotrap Ion Pump Titanium Sublimation Pump

Now, let's move on to roughing pumps. We will discuss only roughing pumps in this chapter; other pumps are described in later chapters.

Roughing Pumps

Rotary Piston Oil-Sealed Mechanical Pump

Rotary Vane, Oil-Sealed Mechanical Pump

Dry Mechanical Vacuum Pump (oil-free)

Sorption Pump

Venturi Pump

Booster/Blower Pump

The roughing pumps we'll consider in this chapter are the rotary piston mechanical pump, rotary vane, oil-sealed mechanical pump, the dry mechanical pump, the sorption pump, the Venturi pump, and the blower/booster pump.

Rotary Piston Mechanical Pump

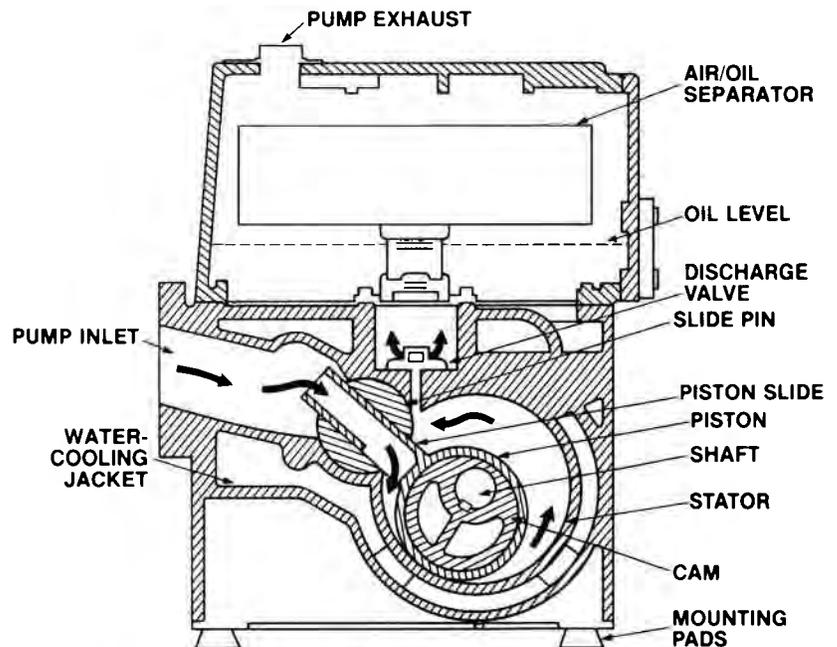
The rotary piston oil-sealed mechanical pump pumps gases by compressing them mechanically and expelling them to the atmosphere.

It is used when roughing pressures are needed, there is a fairly high incidence of particulates at the pump inlet, and large volumes of gas must be pumped quickly. It is available in pumping speeds as large as 1,000 cubic feet per minute (cfm) or more.

Components

The rotary piston pump has a hollow cylindrical piston with an eccentric cam driving it. It is contained in a cylindrical stator chamber. The piston has a sliding valve on it. The cylindrical chamber has an exhaust valve submerged in low vapor pressure oil. This oil also lubricates the pump and seals the space between the rotor and stator.

How the Pump Works

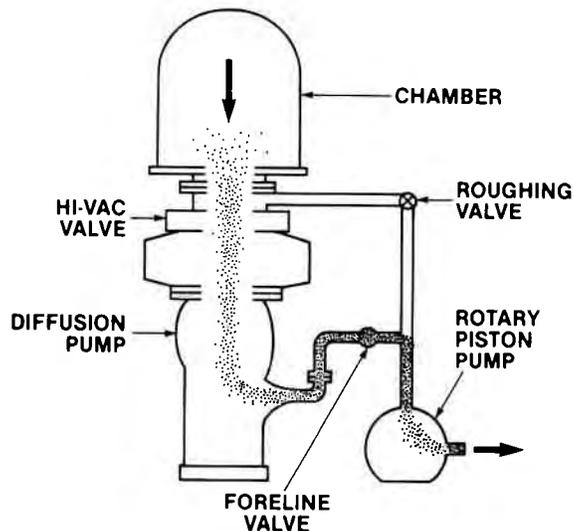


As the eccentric cam turns, it rotates the piston inside the stator. Thus, gases that come in through the sliding valve are compressed by the piston and forced out through the discharge valve and exhaust port. At first, this may seem to be a complicated way of doing things. In fact, however, this is one of the most rugged of the industrial vacuum pumps. Properly maintained and used,

pumps of this type have been in operation for twenty years or more. Because of the mechanisms involved, these pumps are usually belt-driven. The heat of compression as well as the heat of mechanical friction may be removed by a water cooling jacket. Friction between the rotor and stator is not a problem in this pump, because two or three thousandths of an inch clearance exists between the rotor and stator. This limits its ultimate pressure to about 10 mtorr.

Vacuum System Use

These pumps are made in one-stage or two-stage models. They can be used as a roughing pump or as a forepump to exhaust high vacuum pumps.



Maintenance

The best indicator of proper performance, in most pumps, is to see if it can reach its normal ultimate or base pressure. This is done by isolating the pump inlet from any significant gas loads while measuring the inlet pressure with a reliable gauge. This, in effect, checks the oil condition (vapor pressure), physical damage or wear as well as air leakage. Doing this on a regular basis will aid in troubleshooting.

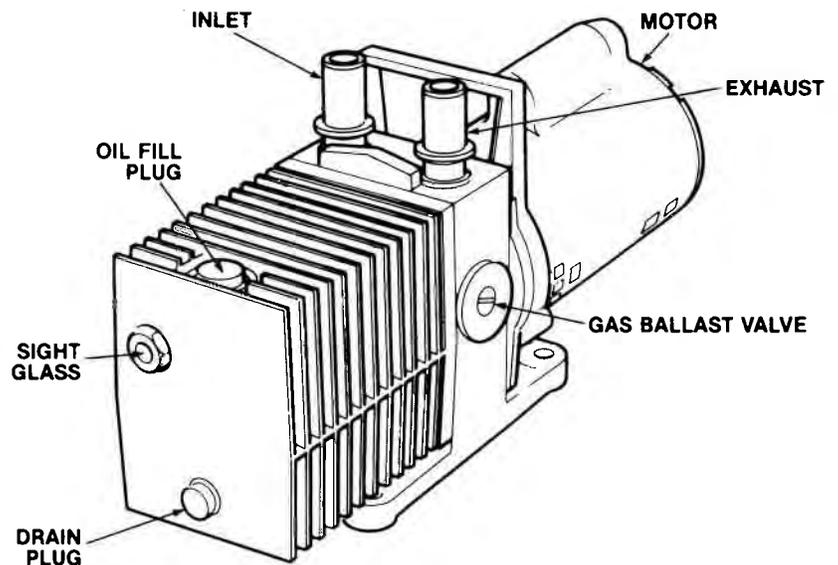
A progressive rise in ultimate pressure, each time the pump is checked, usually indicates a low oil level, a breakdown of pump oil or a buildup of condensable vapors such as water vapor in the oil. If adding oil to the correct level does not help, one or more changes of oil should be done, but only if the pump is at normal operating temperature. If frothy, white (milky) oil appears in the oil-level sight glass, opening the gas ballast valve for an extended period of time (half an hour or more) may slowly purge the oil of condensable vapors.

The drive belts on belt-driven pumps should be regularly checked for proper tension and wear. On multiple belt pumps, if one belt is worn, replace all drive belts in matched sets.

Follow the manufacturer's instructions to change oil and to gas ballast properly.

Rotary Vane, Oil-Sealed Mechanical Pump

The rotary vane, oil-sealed mechanical pump removes gases by compressing them to a point slightly above atmospheric pressure. It then expels the gases to the outside world. It is used to produce roughing or forepressures lower than the rotary piston pump. Due to the friction caused by the sliding vanes in this type of pump, the largest size available is about 150 cfm, the smallest is less than 1 cfm.

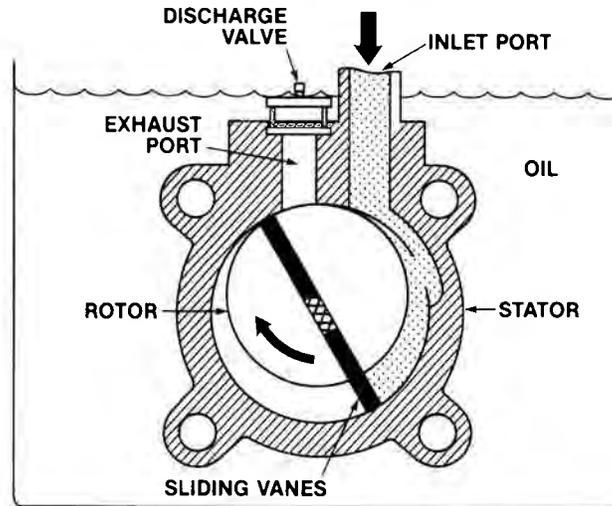


The pump module is immersed in an oil bath. This oil is purified to remove high vapor pressure contaminants. The oil serves the following purposes:

1. Cools the pump.
2. Lubricates.
3. Seals against atmospheric pressure.
4. Opens second-stage exhaust valve at low inlet pressures.

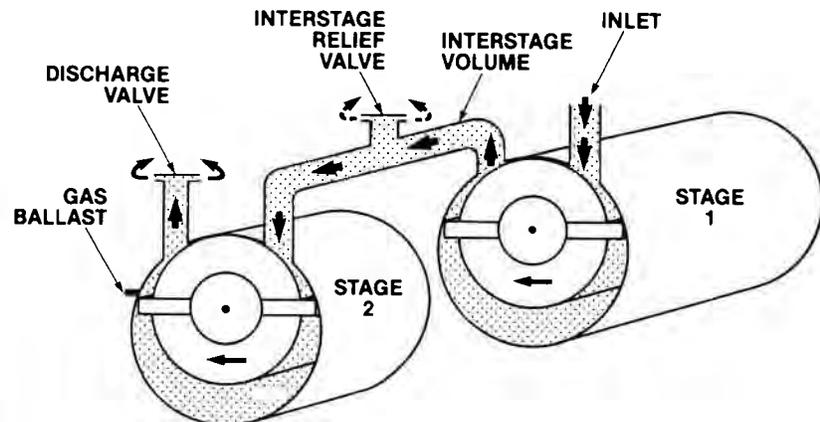
Components

An oil-sealed mechanical pump includes a housing, or stator, an offset rotor with spring-loaded vanes, an intake port and an exhaust port equipped with a discharge valve. It may also have a ballast valve. The pump rotor may be driven by a belt-drive mechanism or it may be directly coupled to the drive motor. Belt-driven pump speeds range from 250 to 400 rpm. Direct drive pumps usually run at 1,725 rpm. Most pumps have two stages to produce better vacuum. This view of a mechanical pump shows the inlet and exhaust ports of one stage.

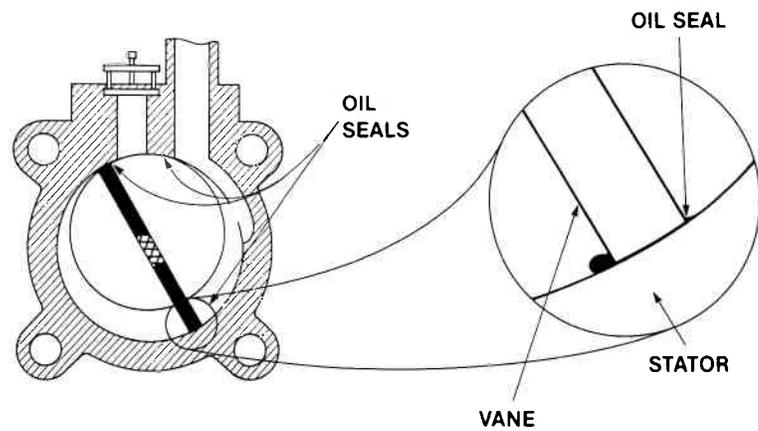


ROTARY OIL-SEALED MECHANICAL PUMP MODULE

How the Pump Works



Gases from the chamber enter the inlet port. They are swept around by vanes, and compressed. Compression builds up pressure to overcome atmospheric pressure. The spring-loaded discharge valve is opened. The air is then expelled to atmosphere. These pumps can remove over 99.9% of the air from the chamber.



A thin film of oil makes the final seal in these pumps. Therefore, base or ultimate pressure is partly determined by the vapor pressure of the oil. If the oil becomes loaded with water, or other impurities, these contaminants increase the vapor pressure. Then it is impossible to reach satisfactory low pressures. The oil is a very important part of your mechanical pump. Dirty pump oil is usually the reason why the pump is not performing well.

gas ballast

Use of the gas ballast feature of the mechanical pump may help to clean up the dirty oil if condensable vapors such as water are the problem. Opening the *gas ballast* valve allows a quantity of air to be admitted during the compression cycle. This causes the exhaust valve to open each and every cycle, sweeping out condensable vapors before they condense inside the pump.

When an oil-sealed mechanical pump is operated at low pressure, it tends to backstream oil vapor into the roughing line. This oil will migrate into the vacuum system and may contaminate the process. There is even more backstreaming if molecular flow conditions are present in the roughing lines. Remember that molecular flow happens when either the pressure is low enough or the diameter of the pipes is small enough.

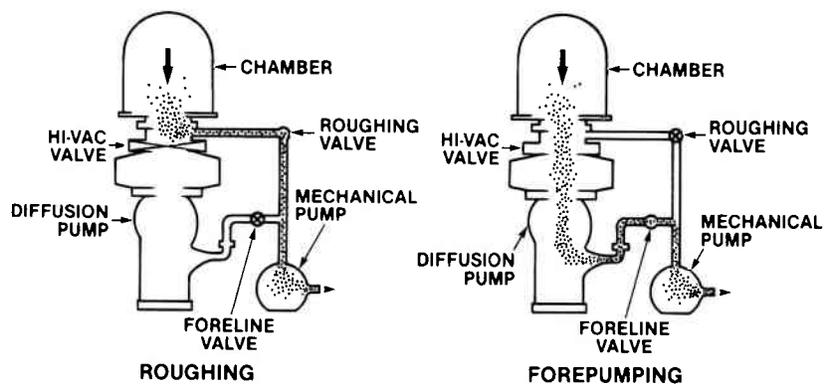
Oil migration can be controlled by using proper traps in the lines. One such trap is the molecular sieve type discussed in the maintenance section. Remember, if you can smell oil in the vacuum chamber, it is most likely coming from the mechanical pump.

forepump

backing pump

Vacuum System Use

Mechanical pumps are generally used two ways. They are used to rough pump the chamber. They may also work with vacuum pumps such as diffusion, blower and turbomolecular pumps. This is because these pumps can't discharge directly into the atmosphere. Instead, they must exhaust into a mechanical pump. The mechanical pump further compresses the gases, then expels them to the atmosphere. When mechanical pumps are used to exhaust another pump, they are called a *forepump* or *backing pump*.



Maintenance

Regular oil changes are an important part of good pump maintenance. The oil level and the oil color should be checked regularly. If the oil is frothy or milky in color, the pump may be kept running while the gas ballast valve is kept open for about one half hour or the oil may be changed.

Over time, pump components will wear out and eventually the pump will need to be rebuilt. Minor or major overhaul kits are available for this purpose from most manufacturers.

When the chamber must be kept free of oil, a useful addition to the roughing pump is a *molecular sieve trap*. It prevents mechanical pump oil *backstreaming*. The trap will restrict the gas flow (less conductance), however. These traps must be periodically baked to 250°C to remove the accumulated oil and water vapor.

There are also other types of traps which are used. Typically they are packed with brass, copper, stainless steel or glass wool. Don't forget that all traps become a *source* of oil if they are neglected. The traps must be regularly cleaned, or replaced.

molecular sieve trap
backstreaming

All oil-sealed vacuum pumps we have talked about have oil in them for a number of distinct functions: as a lubricant for sliding surfaces, as a sealant to prevent back leakage of compressed gas into the inlet part of the pump, as a coolant of internal rotors (oil is continuously circulated in and out of the pumping mechanism by a variety of ingenious methods), as a filler of "dead" space under the discharge valve, and as a hydraulic fluid to operate certain valves. All of these functions are necessary for obtaining satisfactory performance.

The most basic requirements are: a more or less constant pumping speed at a wide range of inlet pressures, ultimate pressure near 1 mtorr (without auxiliary traps), tolerance for a certain amount of water vapor and, of course, reasonable longevity and cost. A variety of such pumps are available in very practical, reliable models. However, the presence of the oil which assures the satisfactory performance also produces certain disadvantages. Among these are: the possibility of contaminating the vacuum system by backstreaming oil, solution of pumped gases in the oil which limits the ultimate pressure and the possibility of chemical reactions between the pumped gases and the oil. In recent years, certain processes using corrosive gases have been producing considerable difficulty in operation of the oil-sealed vacuum pumps. Now let's look at a pump with *no* oil.

Dry Vacuum Pump

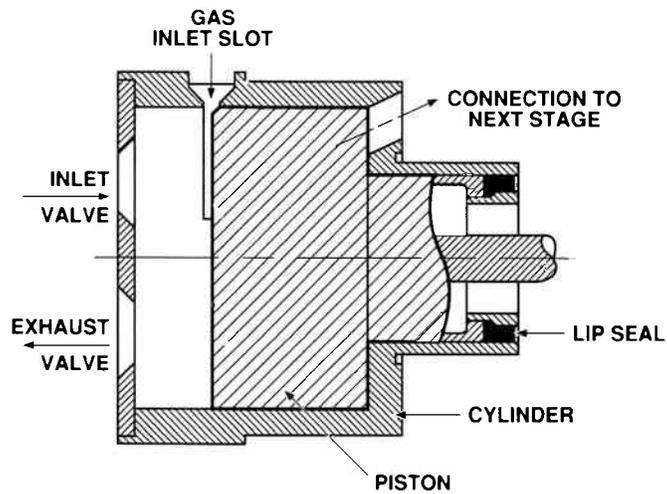
A dry vacuum pump removes gases by a simple compression stroke of a piston in a cylinder. Four stages of compression are used.

The pump is totally oil-free, making it well-suited for use on vacuum systems with ultraclean operating requirements.

The use of composite nonmetallic materials on the moving surfaces allows the pump to work without the use of sealing or lubricating fluids. Thus, the oil backstreaming found with rotary vane, oil-sealed mechanical vacuum pumps is eliminated. Dry pumps need no conductance-limiting foreline traps.

Components

Dry pump components are much the same as those found in a simple engine—a series of pistons and cylinders with appropriate valves for inlet and exhaust. The first two pistons work in parallel; from there on the gas is compressed, exhausted to the next stage and compressed again until the pressure is just a bit more than atmospheric pressure.



Pistons

A typical piston is shown above. The stepped design has a few advantages. The smaller diameter at the atmospheric side helps to reduce the atmosphere leakage because of the smaller outer area of the seal.

The step provides a convenient arrangement for connection to the next stage and a convenient location for the final exhaust valve.

All of the cylindrical surfaces of the piston are lined with special low-friction polymeric material (essentially reinforced polytetrafluoroethylene but without glass, metal, or graphite fillers).

At the end of the piston is a lip seal that helps to minimize the atmospheric leakage into the pump.

Dual Valve System

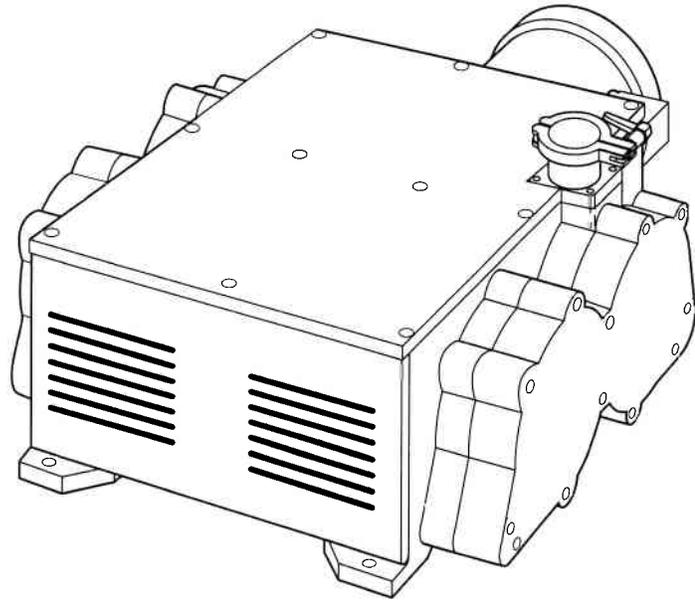
When the inlet pressure is high, the two valves placed at the top of the cylinder work in the normal fashion as in an ordinary compressor; that is, they open and close due to pressure of the air which develops as the piston moves in and out. However, when the pressure becomes too low to open the valves, the inlet valves remain closed. The gases flow through the slots in the cylinder which are uncovered at the end of the piston travel. The discharge valves are forced open by soft pads on the face of the piston which bump the valve at the end of the stroke. Only the final exhaust valve at the end of the last stage is opened by the pressure of slightly compressed air, compressed only to the point of overcoming the force of the valve spring.

How the Pump Works

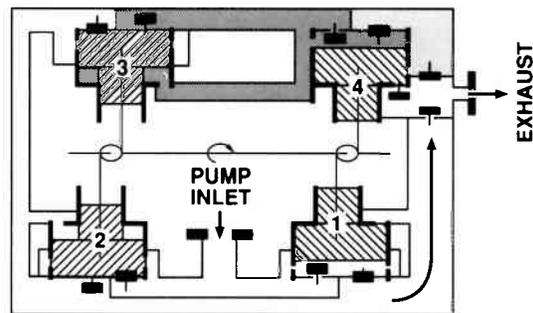
Operation

A reciprocating piston design was chosen primarily because it allows a convenient method for cooling the internal moving parts. In the rotary pump, it is very difficult to cool the rotor without

using some heat transfer medium. In addition, it is difficult to keep the shaft seals sufficiently cool because their sliding friction surface areas are rather small.



In the reciprocating piston design, the inside of the piston is exposed to air and the atmospheric seals slide over a large area which is periodically exposed to room temperature air. In addition, all moving parts, bearings, connecting rods, and the main shaft are also in atmospheric air.

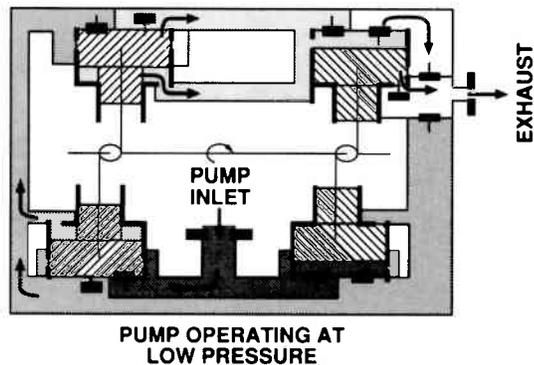


**PUMP OPERATING NEAR
ATMOSPHERIC PRESSURE**

The four pistons are arranged in pairs 180° apart. The first two are connected in parallel simply to obtain a higher pumping speed at high pressure. The third piston is used as the second stage of compression and the fourth piston has two compression stages, one in front and one in the back.

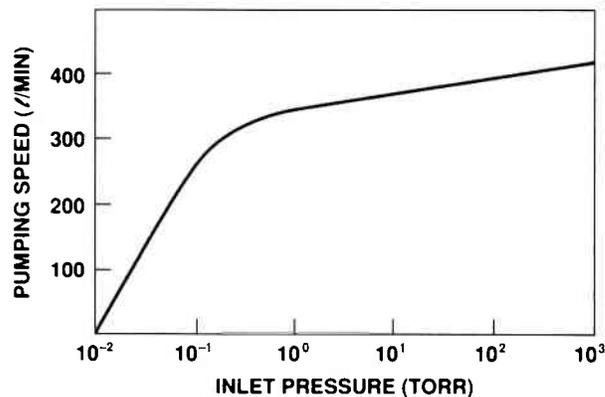
The backs of the first three pistons are always pumped by the pistons of the next stage. This reduces the amount of atmospheric leakage and improves the compression ratio in each stage.

Additional interconnections shown in the pump schematic serve to reduce the amount of gas subjected to compression. That is, when the inlet pressure is high, most of the gas is exhausted through intermediate valves rather than passing through all four stages. In other words, when the inlet pressures are high, the pump functions as a single-stage pump. This reduces the amount of power needed to operate the pump and the degree of heating. Thus, a 20 cfm pump requires only a one horsepower motor (at 1,200 rpm).

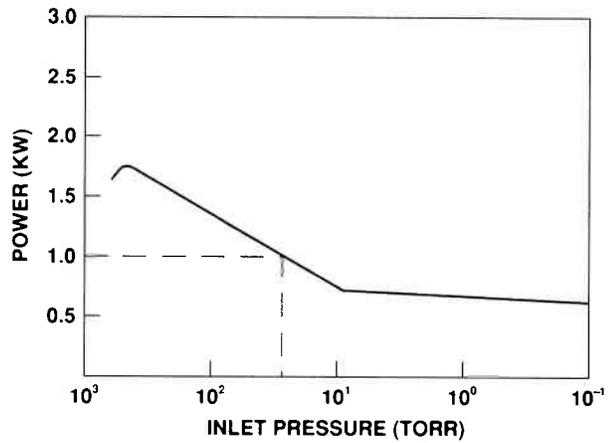


The dry pump has two interstage exhausts and, in addition, the flow into the *last cylinders* is proportioned by controlling the conductances of ducts.

Performance

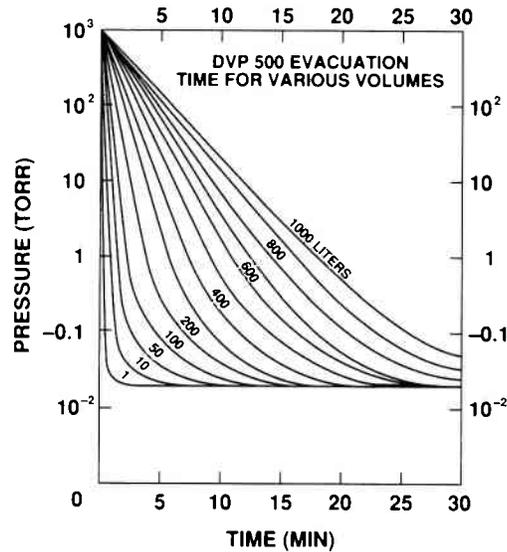


The basic pumping performance of the dry pump is shown in the form of a traditional pumping speed curve. The shape of the curve is very similar to the typical curves obtained with oil-sealed pumps. The gradual decline in speed toward the lower pressure is due to the changes in volumetric efficiency and the switching of the flow from the regular valves to the inlet slots. The final decline toward 10 mtorr is due to residual leakages which produce the limiting compression ratio.



The power requirement at various inlet pressures (or, in other words, at various flow rates) is shown above. The shape of the power curve depends on specifics of design, such as stage matching and the interstage exhaust valve system, but the general relationship is not unlike the one obtained with the conventional pumps. The peak demand occurs typically anywhere between one third and one atmosphere, depending on design.

It is evident from the power curve that for continuous operation and a one horsepower motor, the inlet pressure should be limited to a 30 torr maximum. For evacuation of large chambers, a one horsepower motor is sufficient to handle about a 1,000 liter volume. The evacuation process of various chambers is shown in the figure below.



Vacuum System Use

The ideal use for the new pump is pre-evacuation of vacuum systems which then are pumped by cryopumps, turbo pumps or ion pumps or any other oil-free high vacuum pump. These applications are described in chapter 7, Systems.

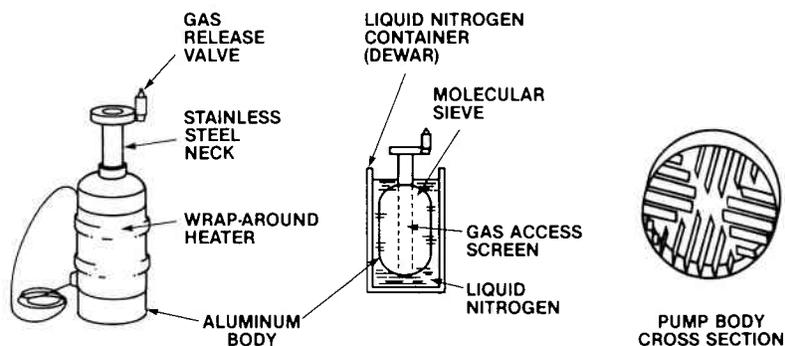
Maintenance

The pump does not need any maintenance under normal conditions for a period of approximately one year. Depending on severity of use, replacement of piston liners and some bearings may be required.

Sorption Pump

This pump operates on a totally different principle than those we have previously discussed. That is, there is no mechanical compression. The sorption pump is a capture pump. That is, it pumps gases by trapping or capturing them within the molecular sieve material. It is usually used for roughing ultrahigh vacuum systems where there can't be any oil— not even a trace.

Components



molecular sieve

The sorption pump has an aluminum body with internally extruded heat transfer fins (see cross section). The pump is filled with a very porous material known as *molecular sieve*. A pressure relief tube is mounted on the connecting flange. The pump is placed in a dewar containing liquid nitrogen (LN_2).

The LN_2 dewar may be either expanded polystyrene or stainless steel. The polystyrene is a solid foam-like material and the stainless steel dewar is double-walled and evacuated like a thermos bottle. Both provide good thermal insulation properties to keep the LN_2 from boiling away too rapidly. The liquid nitrogen cools the pump body to about $-196^\circ C$. This helps the pumping process. Let's see how it works.

cryocondensation

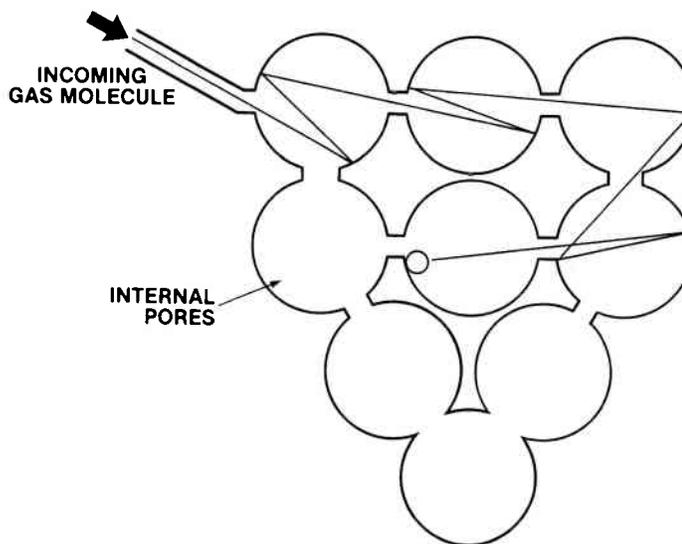
How the Pump Works

Gases are trapped on cold surfaces in two ways—by cryocondensation and by cryosorption.

Cryocondensation is the trapping of gases that are condensable at liquid nitrogen temperature (-196°C). The cold trapping surfaces in the pump are the molecular sieve material. The porous molecular sieve provides acres of surface area. By chilling the material with liquid nitrogen at -196°C , the pumping or trapping action is very effective.



You're familiar with condensation!

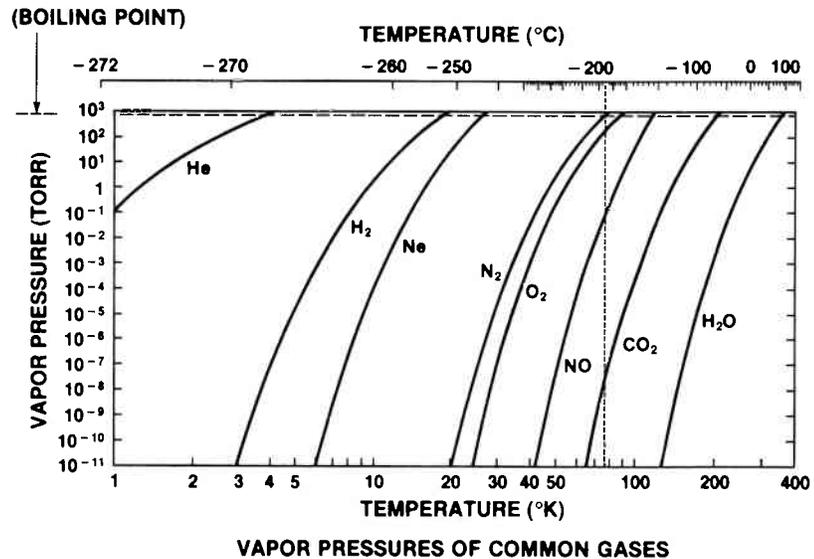


cryosorption

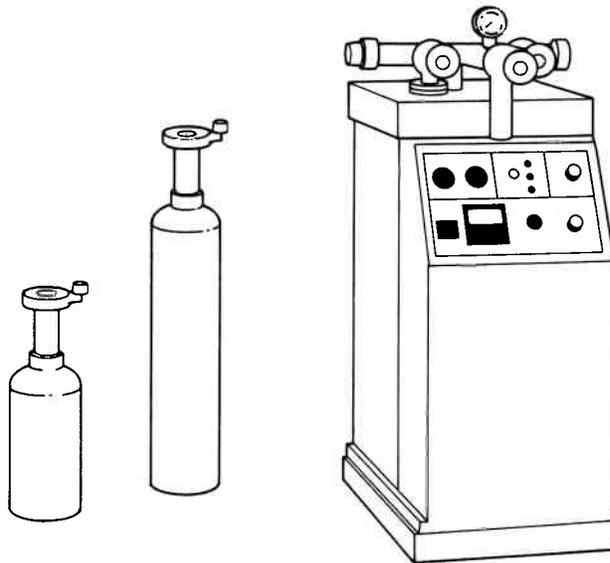
Cryosorption is the trapping of gases not readily condensed or pumped at LN_2 temperature. Cryosorbed gases are trapped in the pump's molecular sieve material.

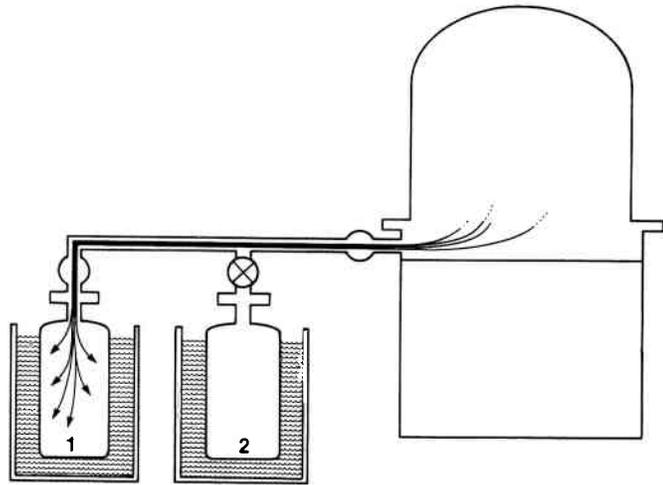
The molecular sieve material is very porous (giving tremendous surface area). Gases that are not readily condensed at LN_2 temperature bounce around within the sieve, losing heat. They are finally trapped (cryosorption). Other gases such as hydrogen, helium and neon are never truly trapped by either condensation or sorption. If we try to pump too long or to too low a pressure, these gases will tend to wander back out of the pump.

The vapor pressures of the incoming gases are reduced because of the cold temperature. Those that condense have greatly reduced vapor pressures, as shown in the following chart.



Variations on the Varian sorption pump include a double-sized pump, and an automatic roughing system. The automatic system includes a Venturi pump and an array of five triple-sized sorption pumps.

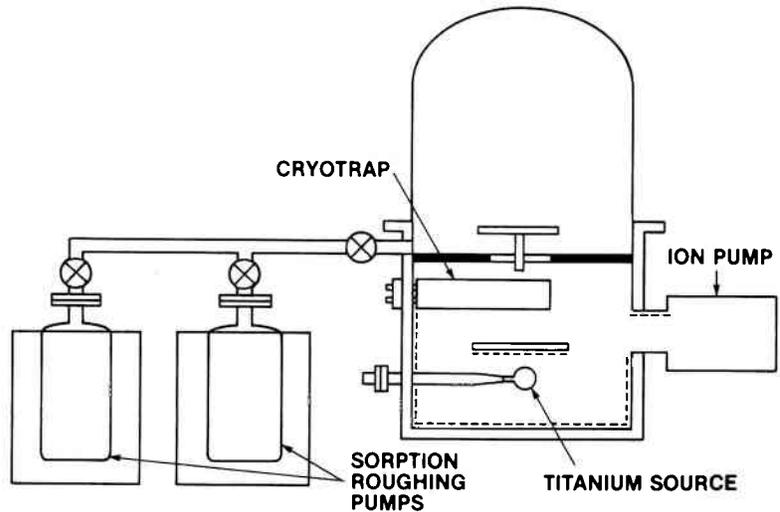




The actual operation goes something like this:

The pump body is pre-chilled for about 20 minutes. Then the valve is opened and pumping begins. The inrush of air carries all gases into the pump. Condensable gases, of course, are trapped on the cold fins and sieve material. Gases that are not condensable at liquid nitrogen temperatures are pumped by sorption or can begin to migrate back into the chamber. When two pumps are used, the first is valved off at a relatively high pressure to prevent back-migration of pumped gases. The second pump then takes over to continue rough pumping. Ultimate pressures of 10 mtorr or less are possible.

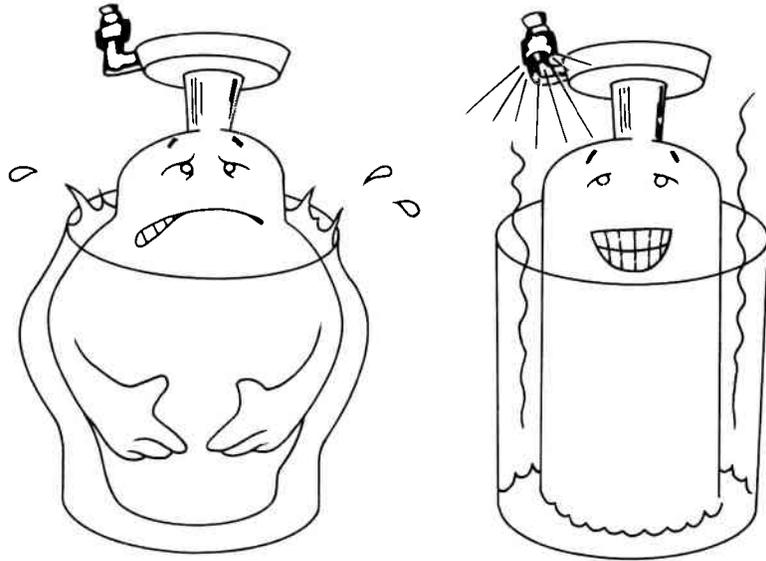
Vacuum System Use



Sorption pumps are very clean. They have no moving parts and do not use oils in their operation. For this reason, they are typically used in ultrahigh vacuum systems. This system configuration includes sorption roughing pumps, a titanium sublimation pump, and an ion pump.

Maintenance

Because the pump is a storage pump, it will eventually get full. Pumping will slow down and then stop. Because of this, the pump must be emptied or “regenerated” before the pumping action stops.



regeneration

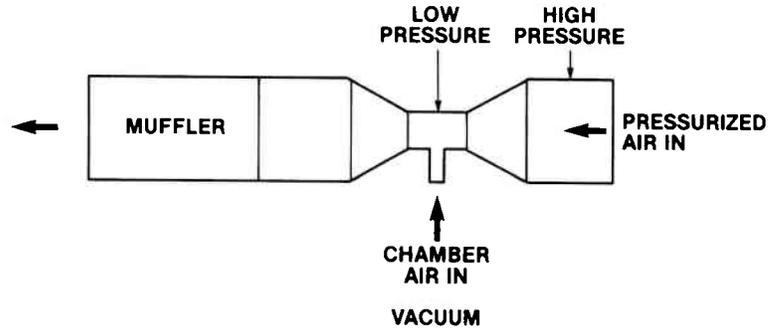
Regeneration consists of warming the pump. As the liquid nitrogen boils away, the pump and sieve material warm up. Now the trapped gases come off the surfaces of the pump and sieve material, build up pressure, and escape through the pressure relief valve. Water vapor, which does not escape so readily, must eventually be driven off by electrically heating the pump body. Typically, the pump is heated to around 250°C. It is very important not to obstruct the pressure relief valve in any way. Otherwise, dangerous pressures could build up in the pump.

For good sorption pump maintenance, the sieve material should be changed once a year. At that time, the inside of the pump and its components should be cleaned. Early degeneration of the sieve due to contamination will cause the pump to require more frequent cleaning.

Venturi Pump

The Venturi pump pumps gases by creating a low-pressure volume into which chamber gases are drawn and expelled to atmosphere. It is usually used along with sorption pumps to extend the time between regenerations.

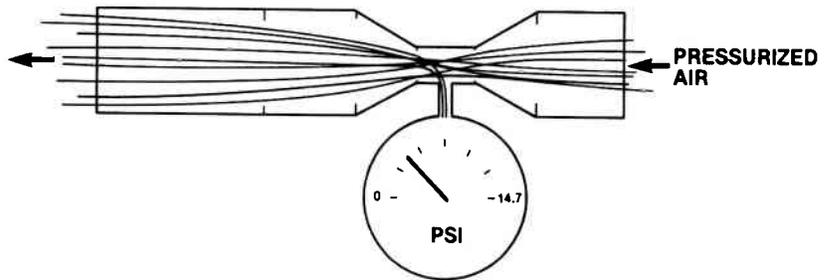
Components



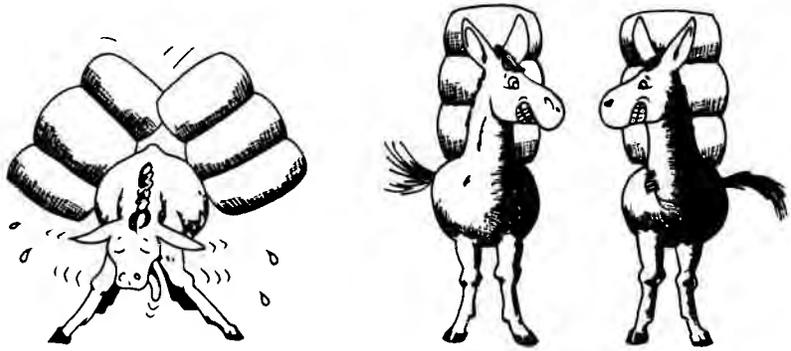
A Venturi pump is simply a tube with a pinched midsection. The tube has an inlet from the work chamber, a pressurized air inlet, and an exhaust. A muffler at the tube exhaust reduces the noise level during pumpdown.

How the Pump Works

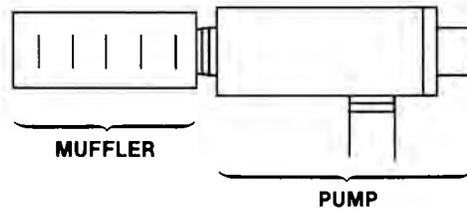
The pinched section of the Venturi tube causes an increase in the speed of the incoming pressurized air. These fast-moving air molecules tend to remove gas molecules from the small side opening. More molecules rush into the side opening from the chamber, only to be removed also.



Pressurized air at about 60 psig entrains air from the work chamber and exhausts it through the noise muffler. The chamber is evacuated to about 60 torr, then a sorption pump continues rough pumping. It's a very rough roughing pump, but it does offer this advantage:



The Venturi pump takes some of the load off the sorption pump. It removes about 90% of the air. Therefore, saturation of the sorption pump doesn't occur so readily, and regeneration is required less frequently.

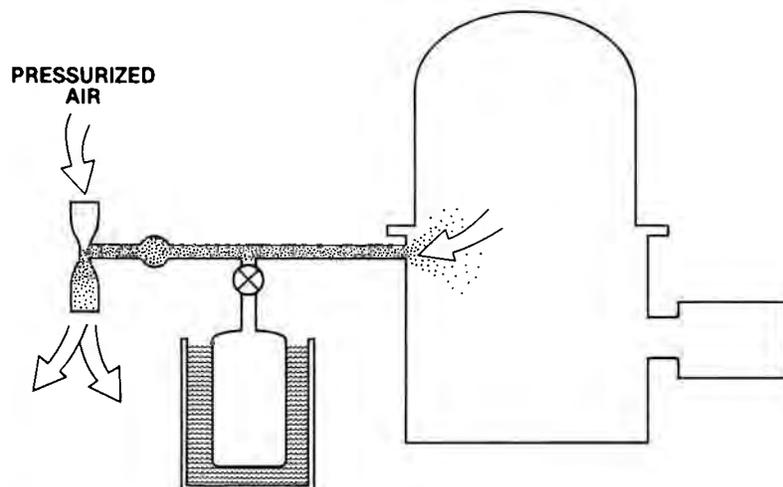


The Venturi pump is quite small, about 5 inches long and 1 inch in diameter.

Vacuum System Use

All you need to do to "operate" it is to connect a high-pressure air line to its air inlet and open the valve. It's not even necessary to connect the line with any kind of permanent coupling.

Next, you simply close off its valve to prevent outside air from re-entering the chamber, and remove the source of pressurized air.



Maintenance

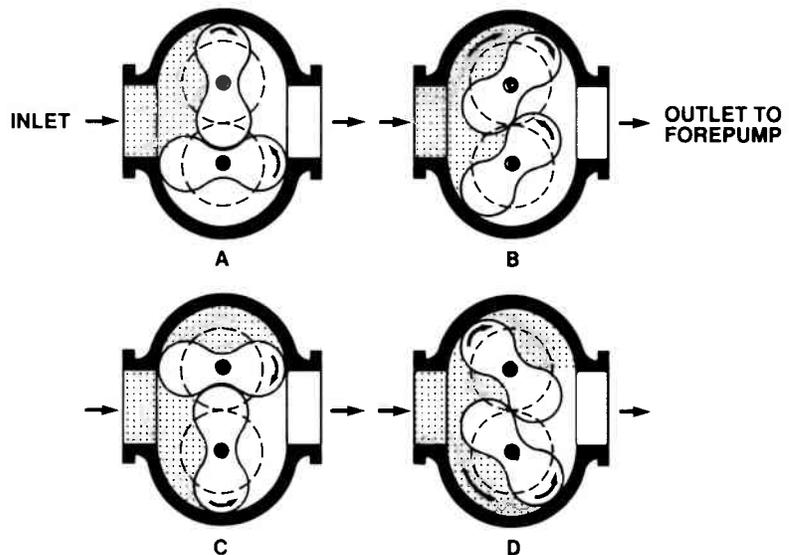
The Venturi pump is simple, and is virtually maintenance-free. However, be sure that the air used is properly filtered. Also, the muffler should be periodically checked for particulates or debris that might restrict gas flow.

Blower/Booster Pump

The blower or booster pump (sometimes referred to as a "Roots™" pump) is a high throughput, low-compression pump. This pump is usually used on systems where a large volume of gas must be pumped. It is also used with a mechanical pump to forepump large diffusion pumps, turbomolecular pumps, or even other Roots type of pumps.

Components

The pump consists of two figure eight-shaped rotors or lobes mounted axially on parallel shafts, as shown in the drawing below. These rotors are synchronized by gears to prevent physical contact and damage, and rotate in opposite directions. This rapidly displaces gas from the inlet to the outlet.



How the Pump Works

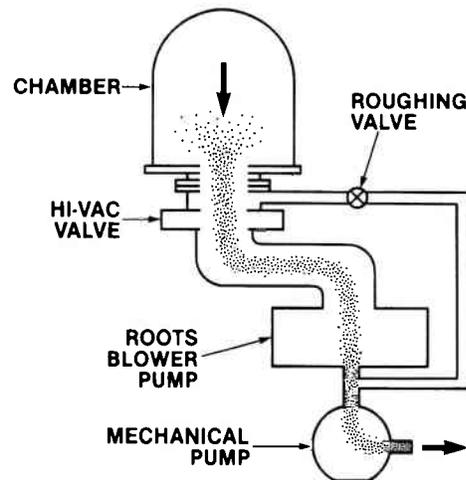
These rotors are designed so that while spinning, they approach each other and the housing within several thousandths of an inch. Rotor speeds vary from 2,500 to 3,500 rpm. Because of the high speeds and close tolerances of the rotating lobes, booster pumps are usually not started until roughing pressures of about

10 torr have been reached. The typical blower “windmills” at atmospheric pressure, producing much heat and very little pumping action. They are most useful between the 10 torr to 10^{-4} torr pressure range. They are always backed by a mechanical pump as a result. Operating at high pressures will cause heating and expansion of the lobes. This can result in damage to the pump. No oil is used to seal the stator/rotor gap. Oil is used in the forevacuum section of the pump to lubricate the gears and bearings located there.

Vacuum System Use

Operating procedure consists of turning the mechanical pump on, then the blower. Usually the mechanical pump has lowered the pressure sufficiently for the blower to begin pumping by the time the blower has reached operating speed. A bypass valve around the blower is sometimes used for high-pressure roughing.

Blowers are commonly used where large volumes of gas need to be pumped. They are used when the lowest pressure needed is 10^{-4} to 10^{-5} torr. They also are used to help the mechanical forepump or backing pump maintain a low pressure and help reduce the possibility of oil backstreaming.



Maintenance

The mechanical components and external drive mechanisms must be kept properly lubricated and cleaned. Consult the maintenance information provided by the manufacturer.

You have seen that there are several different kinds of roughing pumps, each with some advantages and also some disadvantages. Some are much cleaner than others. Let's go on now to high vacuum pumps, where we will see some different ways to produce a vacuum.

3

High Vacuum Pumps

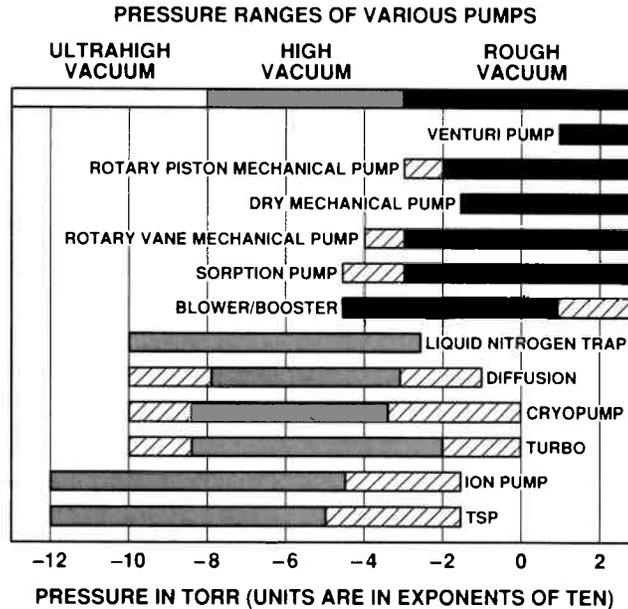
Here is a list of things we think you should be able to do after reading this chapter:

You should be able to—

1. Describe the major components of each type of high vacuum pump.
2. Explain how the major types of high vacuum pumps work.
3. Describe the place of these pumps in vacuum system use.
4. Describe in general how these pumps are maintained.

Introduction

In this chapter we will explain how high vacuum pumps work and the general ways they are used. We will also discuss the major pump components and how the pumps are maintained. We will give an overview of how they fit into vacuum systems. System operation is explained in more detail in a later chapter.



This chart shows typical operating ranges for a variety of vacuum pumps. See the beginning of chapter 2 for a brief discussion of pressure ranges of pumps.

The liquid nitrogen trap is not really a pump but is often used with other high vacuum pumps to collect condensable vapors such as oil or water vapor. The other three high vacuum pumps operate marginally or briefly above their normal range.

The high vacuum pumps covered in this chapter are:

Oil Diffusion Pump

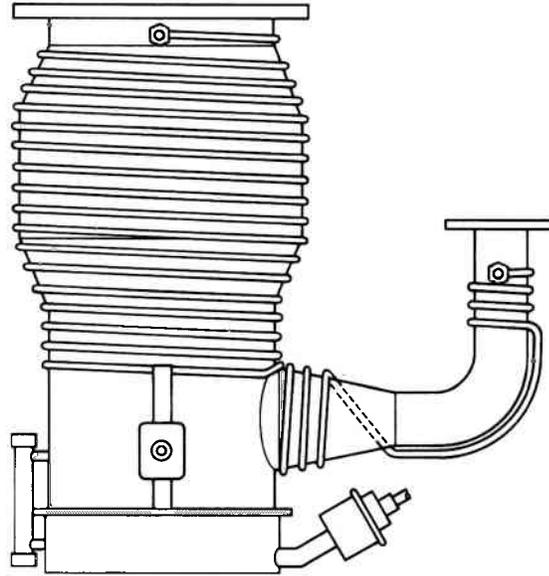
Baffles and Traps

Turbomolecular Pump

Cryopump

Oil Diffusion Pump

The oil vapor diffusion pump is a compression type of pump that has long been the workhorse of the high vacuum industry. It is generally used where throughput for heavy gas loads is important.



The diffusion pump will not operate at or exhaust to atmospheric conditions. It begins to work at 10^{-3} torr, after the mechanical pump has exhausted most of the air. It therefore must have a backing pump, or forepump, to operate. It, like other high vacuum pumps, cannot successfully operate at atmospheric pressure.

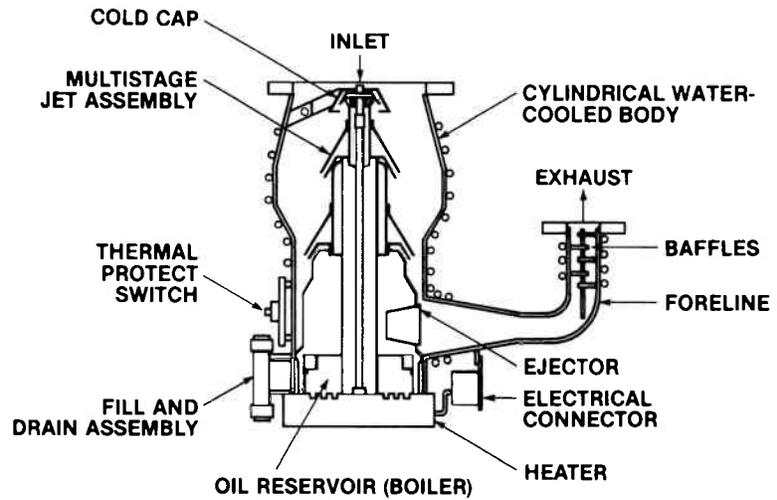
Components

jet assembly
cold cap

The heart of the diffusion pump is the multistage jet assembly. The jet assembly is a group of concentric cylinders. These cylinders are capped to leave small openings through which vapor can be deflected down and out toward the pump walls. A *cold cap* may be mounted on top of the jet assembly. This cap helps keep pump fluid vapor out of the chamber.

Other important parts are the water- or air-cooled body and a heater mounted at the bottom. Inside is a reservoir of oil. Many pumps also have a fill and drain assembly and thermal protect switch. The inlet is at the top, and the exhaust is through the foreline. (Early diffusion pumps used mercury as a motive fluid. These pumps are still manufactured today for use in special applications.)

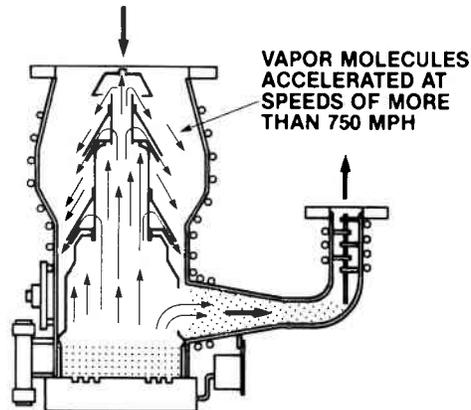
There are no moving parts in this pump.



How the Pump Works

Pump Operation

The diffusion pump works by heating the pump fluid to its boiling point. The vapors travel upward inside the jet assembly and exit through the jet nozzles. In fact, they are accelerated downward through the jet nozzles. The vapor molecules travel very fast and can actually reach supersonic speeds!

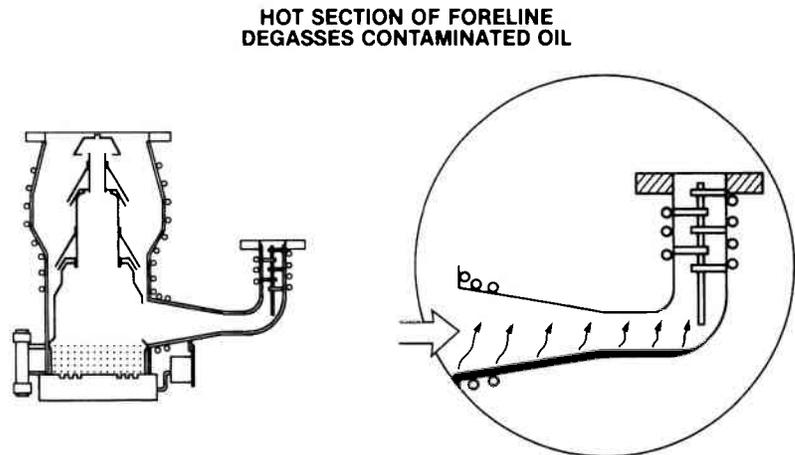


These vapor streams are directed toward the outer walls of the pump. The walls are typically cooled by water. When the vapor hits the cooled walls, it condenses back into a fluid. This fluid then flows downward into the pump boiler for reboiling.

The actual pumping of gases happens when the large, heavy, high-speed oil vapor molecules hit gas molecules. The gas molecules are knocked downward and compressed by the movement of the vapor jet stream.

The gas molecules are thereby compressed in several stages to higher pressures. They are finally pumped away through the foreline by the mechanical pump.

In the pumping process, some of the gas is trapped, or entrained, in the fluid. This trapped gas could be re-released above the pump by the top vapor jet and re-enter the chamber. To minimize this effect, the lower portion of the pump body and foreline are kept hot in order to help drive the gas out of the contaminated fluid. The released vapors are then removed by the mechanical forepump.



In addition to the vertical jet assembly, modern pump designs incorporate an ejector stage. This stage helps to move gas molecules out of the pump body and into the foreline. This action allows higher pressures in the foreline, which in turn allow the mechanical forepump to efficiently remove the compressed gas molecules. Pumps with ejector stages usually have a baffle in the foreline to prevent oil vapor loss to the forepump.

fractionation

Fractionation

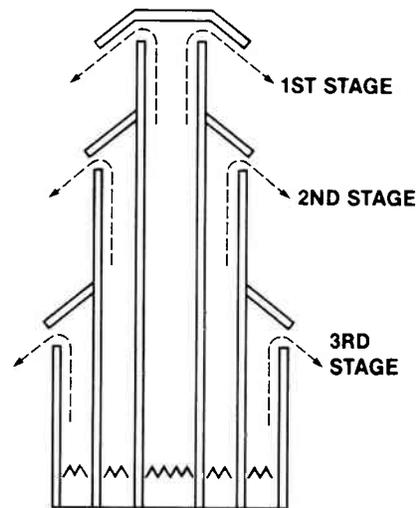
Some diffusion pumps are designed to use a simple fractional distillation process that helps to purify the condensed fluid. This process is called *fractionation*.

What happens is this:

Condensed pump fluid vapor returns to the boiler from the cooled walls. In its course from the outer edges to the center of the boiler, its temperature rises rapidly and new vapor is continuously formed. Diffusion pump fluid slowly decomposes and produces contaminants, or lighter fractions. Over long periods of

time, enough of these fractions may build up to reduce the effective pumping speed and require draining and refilling the pump with new fluid. Here is where the special construction of the boiler and jet assembly is put to use. If the boiler and jet assembly are designed to separate fractions as they are formed, these fractions will be pumped away with the other gases. The purity of the original fluid is thus maintained. The large surface area of a grooved boiler plate distributes heat to the oil more uniformly to minimize the forming of the bad, lighter oil fractions.

So, in this design, the vertical tubes of the jet assembly will separate the first-stage vapors from the others. This design ensures that only the purest fluid reaches the center of the boiler and is forced out of the top jet. This in turn ensures the best vacuum conditions above the first stage, which is the part of the pump closest to the chamber. Less pure fluid vapor is forced out of the lower jets.



backstreaming

Backstreaming

Backstreaming occurs with all diffusion pumps. By definition, it is that small amount of pump fluid vapor that goes in the wrong direction toward the chamber. Various methods are used to reduce the amount of backstreaming fluid reaching the chamber. The table on the next page shows how cryotrap and other components added to the diffusion pump can affect the backstreaming rate. You might also note the decrease in pump speed as a more effective device for controlling backstreaming is used.

This data sheet for a 2,400 ℓ /sec diffusion pump illustrates some of the characteristics of the diffusion pump. Note that efforts to decrease the backstreaming rate slow the pump speed significantly.

Combinations	Typical Applications	Pumping Speed (ℓ /sec) (air)		Backstreaming Rate* (mg/cm ² /min) (DC-705)		Ultimate Pressure** (torr)	
		With Cold Cap	With Mexican Hat	With Cold Cap	With Mexican Hat	Using DC-704	Using DC-705 or S-5
Pump only • highest speed • lowest cost	 General purpose Vacuum furnaces	2,400	1,600	5×10^{-4}	$<1 \times 10^{-4***}$	$<5 \times 10^{-8}$	$<5 \times 10^{-9}$
Pump and Low-Profile Baffle • high speed • clean • fast cycles (valved)	 Metallizing Protective coatings Vacuum furnaces	900	Water-Cooled Baffle normally not necessary when Mexican Hat is used.	$<1 \times 10^{-4***}$	$<1 \times 10^{-4***}$	$<5 \times 10^{-8}$	$<5 \times 10^{-9}$
	 10^{-4} to 10^{-7} torr range	800	Mexican Hat is used.	$<1 \times 10^{-4***}$	$<1 \times 10^{-4***}$	5×10^{-8}	5×10^{-8}
Pump and Cryotrap • high speed • very clean • fast cycles (valved) • long LN ₂ duration	 Thin-film deposition Optical coatings Electronic coatings Solid-state research Molecular beams	1,050	870	1×10^{-7}	1×10^{-7}	5×10^{-9}	2×10^{-9}
	 10^{-6} to low 10^{-8} torr range	900	750	1×10^{-7}	1×10^{-7}	2×10^{-8}	2×10^{-8}

*Backstreaming rates (near ambient temperatures) vary directly with vapor pressure of fluid.
 **Approximate values at inlet of pumping combination. System ultimate depends on chamber design, type of seals, and process outgassing. Mild bakeout is usually required to reach 10^{-8} torr levels.
 ***Too small to be measured by standard collection method of American Vacuum Society.

The combination given below reduces pumping speed by approximately 40% (550 ℓ /sec vs. 900 ℓ /sec for valved combinations.) This system may provide added protection against accidental exposure of the diffusion pump to pressures exceeding the normal operating range.

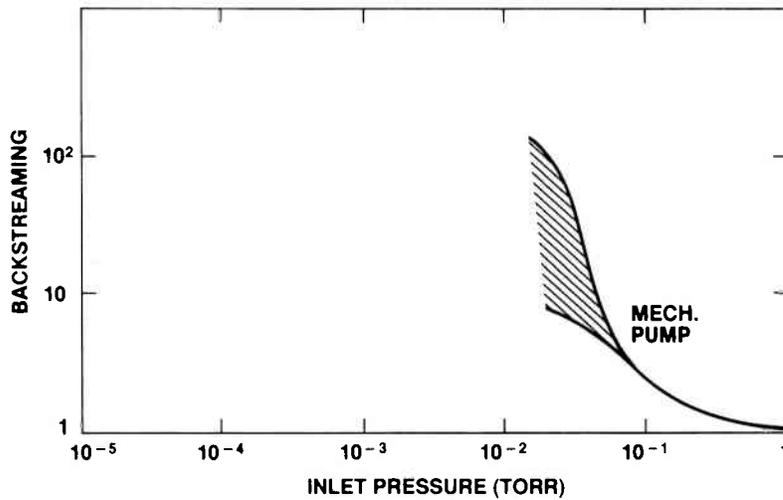
Pump, Low-Profile Baffle, Cryotrap and Slide Valve • extremely clean • extra protection	 Thin-film deposition Optical coatings Electronic coatings Solid-state research Molecular beams 10^{-6} to low 10^{-8} torr range	800	Water-Cooled Baffle normally not necessary when Mexican Hat is used.	$<1 \times 10^{-7}$	$<1 \times 10^{-7}$	$<2 \times 10^{-8}$	$<5 \times 10^{-9}$
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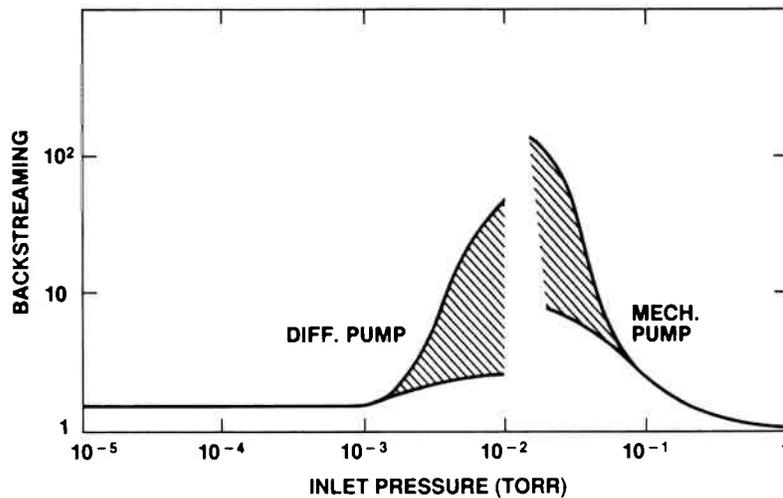
If excessive backstreaming occurs, a major system cleanup may be necessary. Large amounts of oil vapor may end up in the chamber. This results from various causes— sudden pressure bursts in the chamber; opening the high vacuum valve at high pressures; loss of the mechanical forepump as a result of perhaps a broken drive belt; loss of power; and, in some cases, improper valving sequence which overpressures the foreline. This is not backstreaming but is dumping— caused by improper operation or malfunction of the system.

Another source of oil is the mechanical pump or forepump. When the mechanical pump operates in the molecular flow range, the oil is free to migrate out of the pump into the lines and from there into the chamber. The figures on the next page indicate the likely pressure range where the problem is greatest.

BACKSTREAMING VS. PRESSURE CURVES



RELATIVE AMOUNT OF BACKSTREAMING OIL FROM A MECHANICAL PUMP



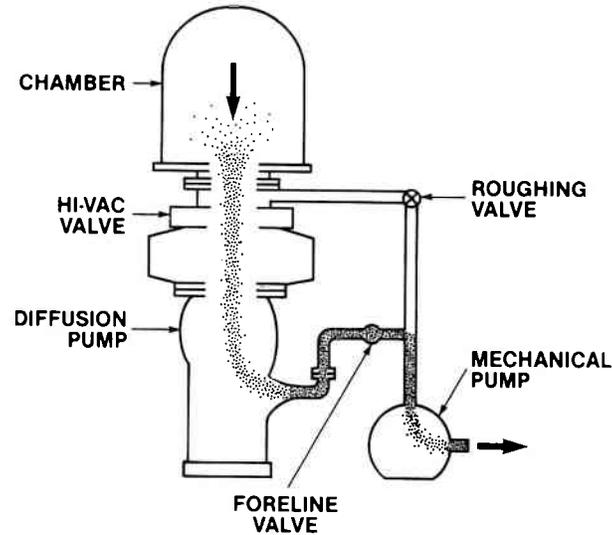
PUMPING AND LUBRICATION FLUID BACKSTREAMING IN THE TRANSITION ZONE BETWEEN DIFFUSION AND MECHANICAL PUMPS

Notice that the mechanical pump backstreams oil more at lower inlet pressures. It reaches its lowest practical limit below 10^{-2} torr (10 mtorr).

By comparison, the diffusion pump backstreams more at its upper limit and typically fails completely above 10^{-2} torr.

A diffusion pump can't exhaust directly to atmosphere.

Instead, it compresses the incoming gases to the millitorr range and exhausts them into another pump. The mechanical pump then compresses the gases further, and expels them to atmosphere.



If an operating diffusion pump is exposed to atmosphere for any length of time, severe oxidation or breakdown of the fluid can occur. Sometimes, in extreme cases, a fire or explosion in the pump may result. The contaminants created in these cases will severely hinder pumping speed and operation due to the gas loads they will contribute to the system. A diffusion pump at atmospheric pressure acts just like a pan of hot oil on a stove. Leave it too long— it will thicken, harden or burn!

Pressure in the foreline *must* be kept below the maximum tolerable foreline pressure, or critical forepressure.

Maximum Tolerable Foreline Pressure

Maximum tolerable foreline pressure is a measure of the ability of the diffusion pump to pump gases against an external pressure. If this pressure in the foreline is exceeded, pump vapors will be forced into the chamber or high vacuum valve in great amounts. Naturally, the presence of these “contaminants” above the pump will increase pumpdown times and raise the system base pressure.

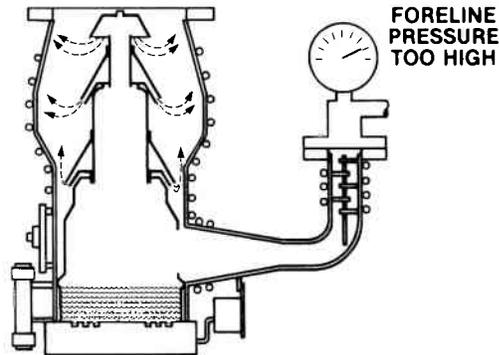
Critical forepressure is another term used to describe maximum tolerable foreline pressure.

If the foreline pressure becomes too high, the fluid vapors blow backward or upward. Now, instead of pumping, the diffusion pump contributes to the contamination in the chamber from the

maximum tolerable foreline pressure

critical forepressure

diffusion pump fluid being forced to go in the wrong direction. A common expression for this is “dumping” the pump. For this reason, a proper-size mechanical pump is needed to help the diffusion pump hold the foreline pressure at an acceptable level.



A reduction of the normal pumping speed (actually throughput)—caused, for example, by a reduction in heater power to the boiler—will cause a corresponding reduction of the maximum tolerable forepressure.

Pumping speed of diffusion pumps is usually rated in liters per second. This speed is constant below 1 mtorr. Of course, at lower pressures, a liter of gas contains fewer molecules. Thus the pumping process slows down. Finally, the lowest (ultimate) pressure is reached when the number of gas molecules removed per second is matched by the number of molecules released into the chamber which were not previously there in the gas phase—from tiny leaks and desorption from interior surfaces.

Selection of Forepump

The mechanical forepump must be able to handle the gas load the diffusion pump is compressing into its foreline. That is, the Q of the mechanical pump must match the Q of the diffusion pump.

The equations below show how to calculate the proper mechanical pump size that will keep the pressure below the critical forepressure.

$$Q_{(MP)} = Q_{(DP)}$$

But $Q = SP$ (Substituting SP for Q)

$$SP_{(MP)} = SP_{(DP)} \quad (\text{Then divide both sides by } P_{(MP)})$$

$$S_{(MP)} = \frac{SP_{(DP)}}{P_{(MP)}} \quad (\text{Solving for } S_{(MP)})$$

Consider a 2,000 ℓ /sec diffusion pump speed at 1 mtorr (inlet pressure). According to the manufacturer's specification sheet, the maximum tolerable forepressure is 400 mtorr. Then,

$$S_{(MP)} = \frac{(2,000 \ell/\text{sec}) (1 \text{ mtorr})}{400 \text{ mtorr}} = 5 \ell/\text{sec}$$

Mechanical pumps are not generally specified in liters per second (ℓ /sec) but, rather, in cubic feet per minute (cfm). To convert cfm into ℓ /sec, remember that there are 60 seconds in 1 minute. And also remember that there are 28.4 liters in 1 cubic foot (ft^3). Then,

$$S_{(MP)} = \frac{5 \ell/\text{sec} \times 60 \text{ sec}}{28.4 \ell/\text{ft}^3} = 10.6 \text{ cfm}$$

This design, of course, would prevent us from valving off the foreline for any reason while the diffusion pump is operating. We would also be extremely vulnerable to dumping the diffusion pump due to improper valving. It would certainly serve us to operate the diffusion pump with the foreline pressure far below the critical value in order to assure proper operation. In practice, we specify a maximum foreline pressure well below the maximum permitted. So let's reduce the forepressure we will allow to a maximum of 150 mtorr and recalculate:

$$S_{(MP)} = \frac{(2,000 \ell/\text{sec}) (1 \text{ mtorr})}{150 \text{ mtorr}} = 13.3 \ell/\text{sec}$$

$$S_{(MP)} = \frac{13.3 \ell/\text{sec} \times 60 \text{ sec}}{28.4 \ell/\text{ft}^3} = 28 \text{ cfm}$$

So, let's go buy a pump that is between 28 and 35 cfm.

Diffusion Pump Fluids

The fluid vapor pressure largely determines the ultimate pressure attainable by a diffusion pump at its inlet. Therefore, diffusion pump fluid is formulated to possess very low vapor pressure at operating temperatures.

Currently, the commonly used fluids for producing clean high vacuums are DC-704™, DC-705™ (silicone-based) and Santovac 5™ or Convalex 10™ (polyphenyl ethers). These tables show the properties of several diffusion pump fluids.

SOME DIFFUSION PUMP FLUIDS

Trade Name	Chemical Name	MW (ave)	P _v at 25°C (Pa)	Viscosity at 25°C (mm ² /s)	Boiler Temp. at 100 Pa (°C)
Convoil®-20	Hydrocarbon	400	5×10^{-5}	80	210
Octoil-S®	Bis (2-ethyl-hexyl) sebacate	427	3×10^{-6}	18.2	220
Invoil®	Dioctylphthalate	390	3×10^{-5}	51	200
Dow Corning®-704	Tetraphenyl-tetra methyl trisiloxane	484	3×10^{-6}	38	220
Dow Corning®-705	Pentaphenyl-tri methyl trisiloxane	546	4×10^{-8}	175	250
Santovac 5®	Mixed 5-ring polyphenylether	447	6×10^{-8}	2400	275
Fomblin® Y VAC 25/9	Perfluoropoly-ether	3400	9×10^{-7}	190	230

Characteristics of Diffusion Pump Fluids

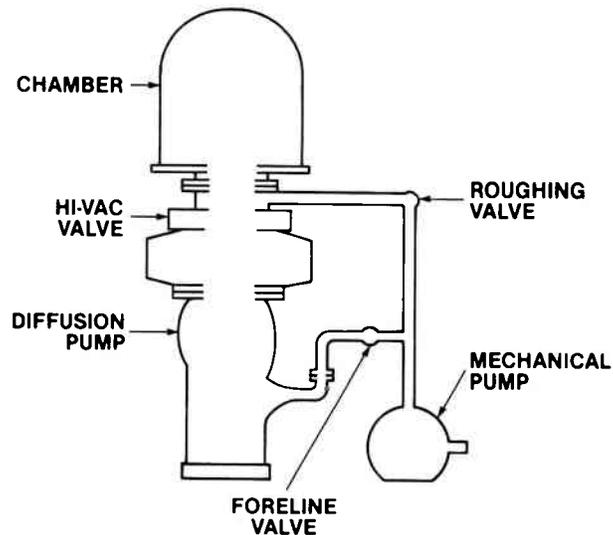
- (1) Hydrocarbon Oils
 - (a) low cost
 - (b) decompose on air exposure
 - (c) o.k. for gauges and ion sources
- (2) Silicone Compounds
 - (a) moderate cost
 - (b) good on air exposure
 - (c) bad for gauges and ion sources
- (3) Polyphenyl Ether
 - (a) high cost
 - (b) good on air exposure
 - (c) o.k. for gauges and ion sources
- (4) Fatty Esters
 - (a) low cost
 - (b) poor on air exposure
 - (c) o.k. for gauges and ion sources
- (5) Halogenated Compounds
 - (a) high cost
 - (b) oxygen compatible
 - (c) may be decomposed by Lewis acids

Brands of Diffusion Pump Fluid

- (1) Hydrocarbon Oils
 - (a) Apiezon A, B, C
 - (b) Litton oil
 - (c) Convoil-20
- (2) Silicone Compounds
 - (a) DC-704
 - (b) DC-705
 - (c) Invoil 940
- (3) Polyphenyl Ether
 - (a) Santovac 5
 - (b) Convalex 10
- (4) Fatty Esters
 - (a) Octoil and Octoil-S
 - (b) Butyl Phthalate
 - (c) Amoil and Amoil-S
 - (d) Invoils
- (5) Fluoroether Polymers
 - (a) Krytox
 - (b) Fomblin

Vacuum System Use

Diffusion pumps are quite widely used for high vacuum systems. In fact, they are the most common type of pump used. We will discuss the actual operation and valving of a diffusion pump system in the chapter on vacuum systems.



Maintenance

If a diffusion pump is properly operated, maintenance is simple.

Whenever the pump is taken apart, used O-ring seals should be replaced. Old or contaminated fluid should be drained from the pump. Many companies now recycle their diffusion pump oil. Special precautions must be taken when the pump has been used to pump toxic or caustic gases because these materials will be present in the pump and its fluid in perhaps dangerous concentrations.

All components should be disassembled. Internal pump surfaces and components must be cleaned as directed by the manufacturer. Any oxidized fluid deposits should be removed as directed. The components should then be rinsed (also as directed) to remove solvent films and minimize condensed water vapor.

New O-rings should be lubricated and installed. The pump should be reassembled, filled with new, clean fluid and reinstalled in the system.

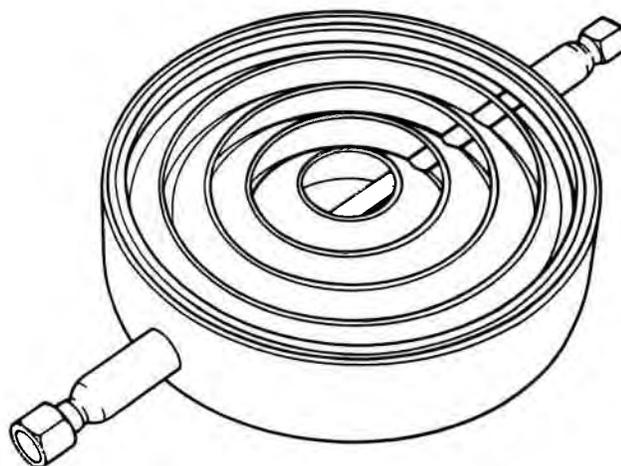
Baffles and Traps

Earlier, we saw how the cryotrap can be used to reduce backstreaming. The baffle can also be used for the same purpose. Let's go on now to discuss baffles and traps in more detail.

Water-Cooled Baffle

Water-cooled baffles aren't designed to be pumps. However, baffles are used above the diffusion pump inlet to condense backstreaming pump vapors before they reach the chamber. In some applications, water-cooled baffles serve to remove (or reduce) excessive heat loads generated in a process which might adversely affect pump performance.

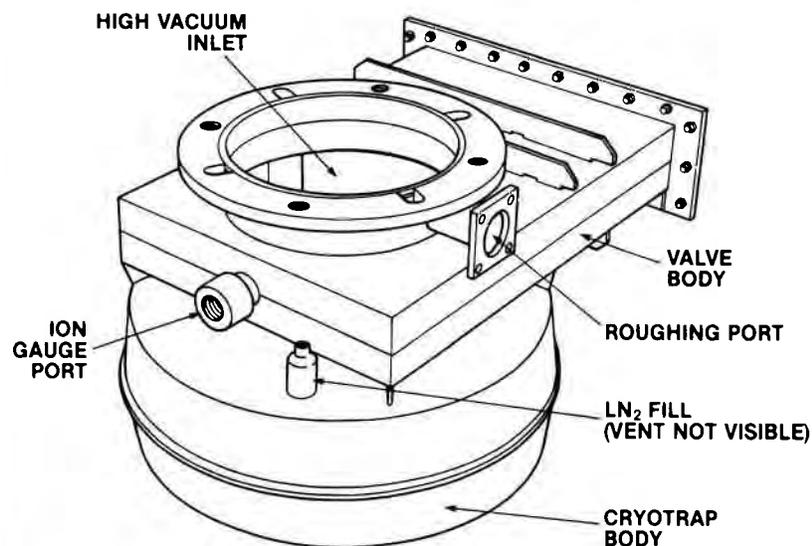
Baffles are composed of arrays of fins that are optically dense. That is, you cannot see through them. These arrays are cooled by continuous water flow through or about their internal components.



Baffles, however, make a useful contribution to the effectiveness of diffusion pumps. (Refer to the chart titled "Backstreaming" in the Diffusion Pump section to see how baffles can reduce diffusion pump backstreaming.)

Cryotrap

Cryotrap also aren't designed to be pumps. They do act as a selective pump for certain gases—namely, water vapor, carbon dioxide and most solvents. Cryotrap also restrict pump fluid backstreaming while giving reasonable conductance figures. A cryotrap is often combined with a high vacuum valve in a single high conductance unit. (See table.)



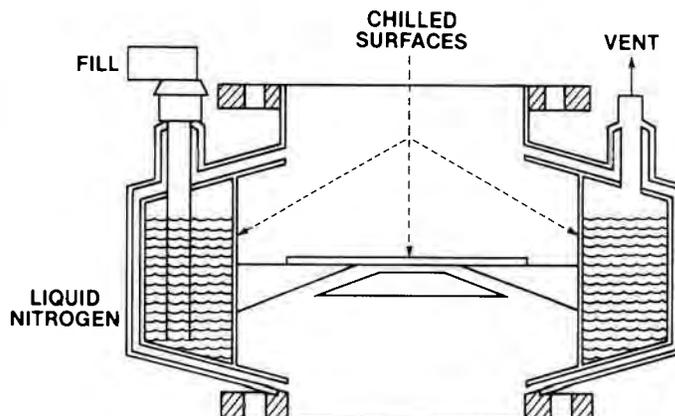
Components

A cryotrap, or *liquid nitrogen cold trap*, has a liquid nitrogen reservoir and various baffling surfaces. The reservoir is insulated from the environment by an evacuated space. The LN_2 boils off to atmosphere through a vent port. Since LN_2 boils at -196°C , the trap's internal surfaces are extremely cold.

liquid nitrogen cold trap

open-loop refrigeration system

The cryotrap can be called an *open-loop refrigeration system*, since the coolant vents to atmosphere. An example of a closed-loop refrigeration system is the mechanical cryopump.



How the Cryotrap Works

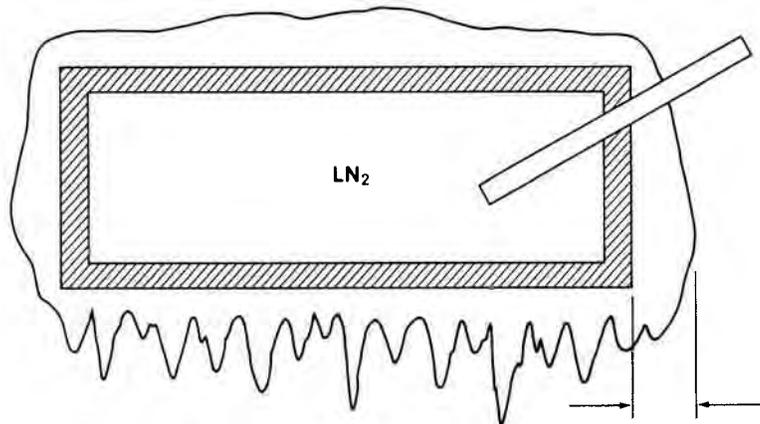
Cryotrap pump gases or vapors by freezing, or condensing, them on their chilled surfaces. This cryocondensation effectively removes them from the work chamber. However, some gases don't condense at liquid nitrogen temperature, or -196°C .

The table shows that water vapor is trapped very effectively at -190°C . However, other gases are not trapped at all, since their vapor pressures are quite high at this temperature. Therefore, other means must be used to pump noncondensable gases.

VAPOR PRESSURES OF SOME GASES AT -190°C

Gas	Approximate Vapor Pressure (Torr)
Water (H_2O)	10^{-22}
Argon (A)	500
Carbon Dioxide (CO_2)	10^{-7}
Carbon Monoxide (CO)	760
Helium (He)	760
Hydrogen (H_2)	760
Oxygen (O_2)	350
Neon (Ne)	760
Nitrogen (N_2)	760
Solvents	Very Low

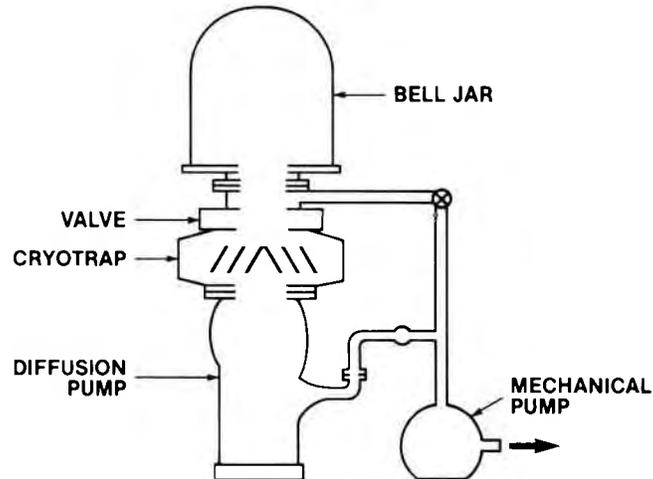
LN_2 traps should be filled after roughing pressures near 50 mtorr have been reached in the area where they are being used. At this pressure, much of the water vapor in the chamber volume has been removed. The mean free path of the remaining gases is long enough to create an insulating vacuum. If exposed to pressure above 100 mtorr, heavy frost builds up on the cryotrap. This frost insulates the chilled surfaces and thus reduces the pumping action.



FROST BUILDUP ON CRYOGENIC SURFACES CAN ACT AS AN INSULATING LAYER, MINIMIZING OR DEFEATING TRAPPING EFFICIENCY.

Instead, the chamber must be rough pumped to remove the bulk of the condensable gases. Then, the cryotrap provides continuous trapping of small but significant amounts of the remaining condensable gases.

Vacuum System Use



Typically, cryotrap are teamed with diffusion pumps (or other high and ultrahigh vacuum pump systems). Cryotrap have also been used in roughing lines to minimize mechanical pump oil migration toward the chamber. In addition, they have been used with helium leak detectors for the same purpose. For these applications, however, a molecular sieve trap is usually better.

It is important to remember that if a cryotrap is not kept properly filled, the baffle surfaces will warm up. This rise in temperature will release condensed gases. The result will be a rise in system pressure, further rise in temperature, and potential contamination of the area or component we are trying to protect. To prevent this contamination, the valve between the work chamber and the cryotrap must be closed if warmup possibility exists.

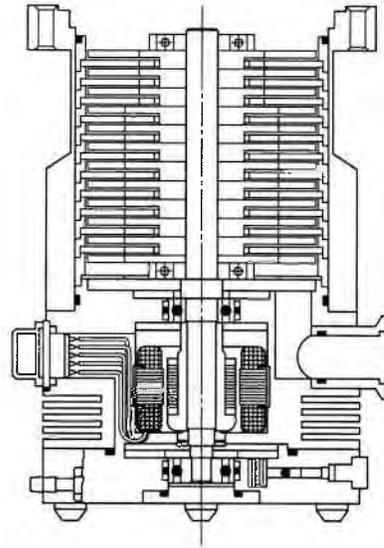
Maintenance

The cryotrap needs to be replenished with LN_2 frequently, before warmup starts.

The cryotrap requires little maintenance other than periodic cleaning of internal surfaces according to manufacturer's directions.

Turbomolecular Pump

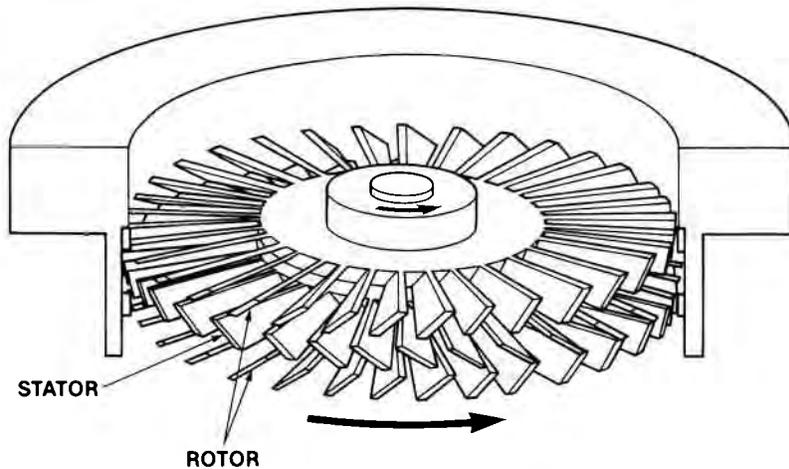
Turbomolecular (turbo) pumps are very clean mechanical compression pumps. They pump by using a high-speed rotating surface to give momentum and direction to gas molecules. They operate smoothly and contribute little vibration to the operating system. They are the only purely mechanical vacuum pump that can reach pressures of less than 5×10^{-10} torr without using traps. (Metal gaskets, and a mild bakeout of the vacuum system are necessary to reach this pressure.)



When operated correctly, turbo pumps are highly reliable and clean. Because they can operate from steady state inlet pressures as high as 10^{-2} torr to below 5×10^{-10} torr, turbos are used in a wide variety of applications. They are ideal for uses where a vacuum relatively free of hydrocarbons is a must. Turbo pumps offer the advantages of a fast start-up to full pumping speed and a clean, hydrocarbon-free vacuum. They are ideal for all but the most stringent applications (such as molecular beam epitaxy, surface analysis) where even the most miniscule hydrocarbon background cannot be tolerated.

Like the diffusion pump, the turbo pump cannot exhaust directly to atmosphere. Usually a rotary mechanical pump or dry vacuum pump is used to forepump the turbo.

Components



The turbo pump is mainly composed of rotating and fixed disks. These are called rotors and stators, respectively. The rotor disks are arranged alternately with the stator disks. On each disk are blades. A disk may have from 20 to 60 blades. The number of blades on a disk, the blade length, width, spacing and rotational speed determine its ability to pump gases.

Each rotor and stator disk can be called a compression stage. A pump may have as many as ten to forty stages. The rotor is driven by a motor capable of reaching speeds from 9,000 rpm to 90,000 rpm, depending on the size of the pump. The motor is typically powered through a special power supply. Compressed gases are expelled from the pump via a foreline which must be evacuated by some type of forepump.

The primary source of vibration of a turbo pump is the residual imbalance of the rotor assembly. This imbalance causes an acceleration in the radial direction of the pump rotor, appearing as a displacement of the inlet flange "side to side." Typically, this displacement is of the order of 0.02 microns (2×10^{-8} meters) and is inconsequential for the majority of turbo pump applications.

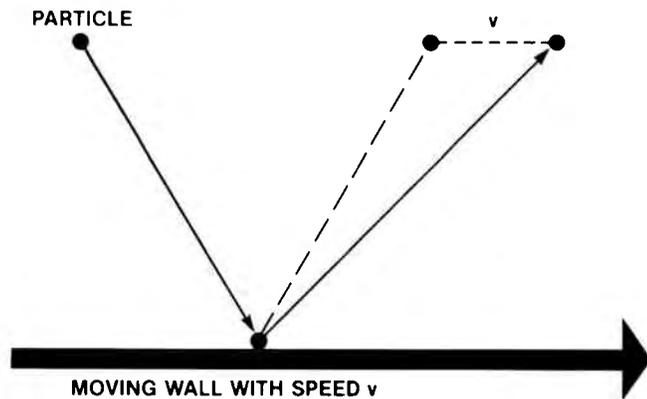
For vibration sensitive applications such as scanning electron microscopes or focused ion beam systems, vibration isolators are available that reduce the vibration level by an additional factor of ten to thirty times.

With turbo pumps, the vibration is at a relatively high frequency (near the controller output frequency) and is much easier to isolate than the low frequency vibration present in other types of mechanical pumps.

How the Pump Works

Pump Operation

When a gas molecule strikes a moving surface, it keeps its own speed. It also picks up a little more speed and a different direction from the contact with the moving surface. By this process, the movement of molecules can be directed, and pumping takes place.



PRINCIPLE OF THE TURBOMOLECULAR PUMP

The turbo pump works much like this; however, it adds blades to the moving surface plus a close-coupled stator. The stator also has blades. Each blade, when moving, will give some momentum to the gas molecules it hits. Each rotor blade, then, acts as a molecular pump. The result? Much greater momentum, speed, and direction are given to gas molecules entering this pump.

In the molecular flow range, the gas particles collide much more often with the moving blades than with each other. The effect of moving blades on the gas particles is highest in the molecular flow range. Pumps operating on this principle are called molecular pumps.

On the stages closest to the inlet, the blades have a large angle so as to pump at a faster rate, because more "open" space allows more access to the chamber. The blades closest to the foreline have a small angle for greatest compression. This works to move the gases from the inlet into the foreline. It also works to keep the gas and oil molecules in the foreline from making their way to the inlet.

Pumping Speed

Turbo pumps typically operate at speeds ranging from 9,000 rpm to 90,000 rpm. For any given turbo pump, variations in the rotational speed will strongly affect the pumping performance.

The pumping speeds and compression ratios achieved with a turbo pump are related to the rotational speed. Unless a manual switch for "low-speed mode" is made by a user, full rotational speed is achieved at pressures lower than 1×10^{-3} torr (1 micron).

The pumping speed of the turbo pump is directly proportional to its rotational speed. For example, if a turbo pump has a rated speed of 300 ℓ /sec (normal rpm) and is switched to a "low-speed mode" that reduces rotational speed to 70% of normal, the turbo pump will pump at $70\% \times 300 \ell$ /sec, or 210 ℓ /sec. The "low-speed mode" allows operation at pressures of several hundred millitorr. This is useful in applications such as sputtering, which require a gas backfill into the millitorr pressure range.

Most manufacturers of turbo pumps give pumping speed specifications for nitrogen, helium and hydrogen.

The pumping speed for a gas species is a function of the velocity ratio of the tip speed of the rotor blade to the thermal velocity of the molecule being pumped. Helium and hydrogen molecules have high thermal velocities, in excess of 1,000 meters/sec, compared with nitrogen molecules which move at approximately 450 meters/sec. This large difference in molecular thermal velocities is why separate pumping speed specifications are given for helium, hydrogen and nitrogen. For other air gases such as oxygen, argon and carbon dioxide, the pumping speed for a typical turbo pump is within 10% of the nitrogen specification.

Compression Ratio

The compression ratio of a turbo pump is equal to the foreline pressure divided by the inlet pressure for a particular gas species. It is an exponential function of the molecular weight of the gas and the rotational speed of a particular turbo pump. The major operational significance of the compression ratio of a turbo pump is that it determines the cleanliness of the vacuum system.

The compression ratio of a turbo pump is a function of the molecular weight of the gas being pumped. In a well-baked UHV system, the compression ratio for hydrogen will limit the ultimate pressure achieved in the system. This is because the residual gas in a baked-out system is typically over 85% hydrogen. The ultimate pressure (P_{ult}) is determined according to the formula:

$$P_{ult} = \sum \frac{Q_i}{S_i} + \sum \frac{P_{2i}}{K_i}$$

where Q_i is gas load (for each gas species from outgassing),

S_i is pumping speed for each gas species,

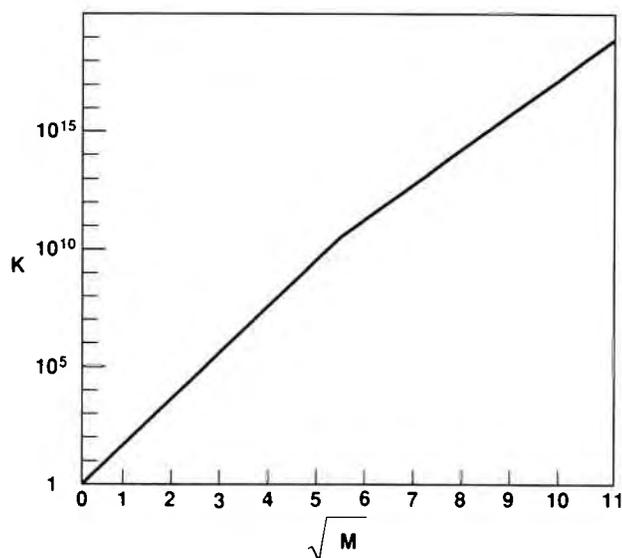
K_i is compression ratio for each gas species,

P_{2i} is partial pressure at the exhaust for each gas species.

With an oil-sealed mechanical pump as the forepump, the foreline partial pressure of hydrogen is approximately $2-5 \times 10^{-7}$ torr due to the hydrogen produced by cracking of the mechanical pump oil vapor. With typical turbo pumps having a compression ratio of 10^3 for hydrogen, the system pressure will contain a hydrogen partial pressure of 2×10^{-10} torr to 5×10^{-10} torr.

To obtain a lower ultimate pressure, into the 10^{-11} torr range, the turbo pump should be backed with another, smaller turbo pump which is then backed by a mechanical pump. This configuration is usually only used when the lowest possible hydrogen partial pressure must be achieved.

Mechanical pump oil typically has a molecular weight of at least 100. From the chart, the compression ratio, K, for this mass is 10^{15} .



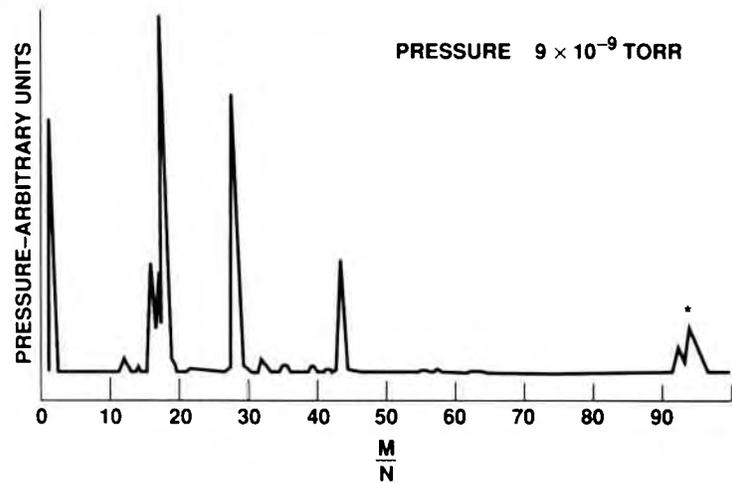
COMPRESSION RATIO (K) VS.
SQUARE ROOT OF THE MOLECULAR MASS

If the forepump is producing a foreline pressure of 1 mtorr (0.001 torr), then the partial pressure of hydrocarbon gas (mechanical pump oil) at the inlet of the pump is

$$= \frac{10^{-3} \text{ torr}}{10^{15}}$$

$$= 10^{-18} \text{ torr}$$

This is why turbo pumps produce virtually hydrocarbon-free vacuum. Only the most sensitive of analytical equipment can detect this level of hydrocarbon vapor.



MASS SPECTRUM OF UNBAKED TURBO PUMP SYSTEM

*RHENIUM PEAK (FROM FILAMENT IN RGA)

Pump Base Pressure

Ultimate or base pressure is defined as the lowest pressure measured in the standard test dome within 48 hours after the prescribed bakeout is finished, per international test procedures (DIN Norm #28428).

To achieve the lowest base pressure in a system then, it is necessary to bake out the system and the turbo pump. Many people accept the pressure reached without a bakeout as their base pressure.

Care must be taken to ensure the temperature of the turbo pump never exceeds the manufacturer's maximum allowable temperature at the inlet flange, typically 80°–120°C.

Most manufacturers supply heating mantles to give the turbo pump inlet a mild bakeout. The chamber can be baked out with strip heaters or a small clamshell oven.

Potential Problems

The most common failures of turbo pumps are due to particulates, lack of bearing lubrication, and shock.

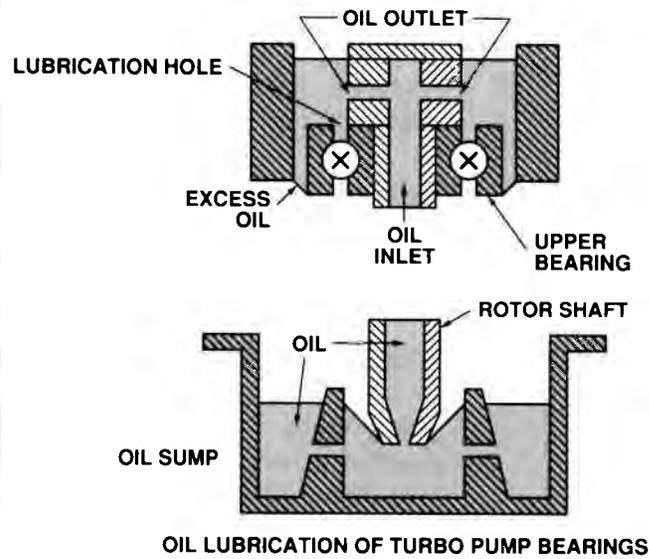
Lubricating Systems

There are a variety of methods used to deliver lubrication to the bearings. The most common are described below.

Circulating Oil

Oil is drawn up the shaft during operation and ejected over the bearings. This method provides a continuous flushing of the bearings, removing any particulates that have accumulated, and provides a continuous flow of lubricating oil. The oil also helps to cool the bearing.

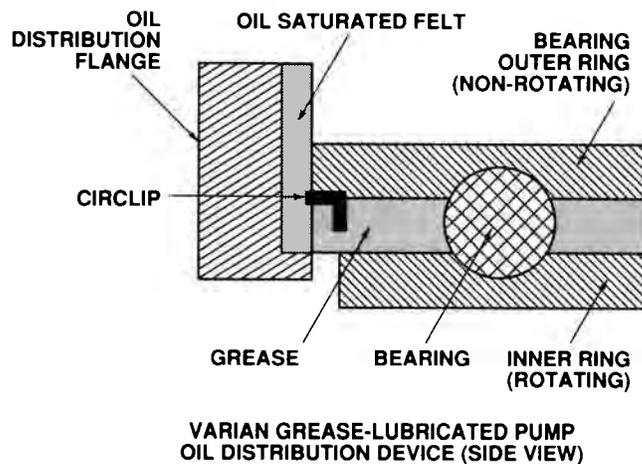
Circulating oil is the most reliable method of bearing lubrication; however, it requires a vertical mounting of the pump and water cooling. In addition to superior reliability, it allows a visible indication of the oil quality and quantity through a transparent oil sump.



Grease-Lubricated Pumps

In this method, the bearings are packed in grease, which contains a lubricating oil. Periodically, oil must be injected into the pump lubrication port to replenish the consumed oil.

Advantages of this method are that the pumps can be mounted in any orientation and usually can be air-cooled. Disadvantages are that the status of the lubrication cannot be determined as with the oil-lubricated pumps.



Shock

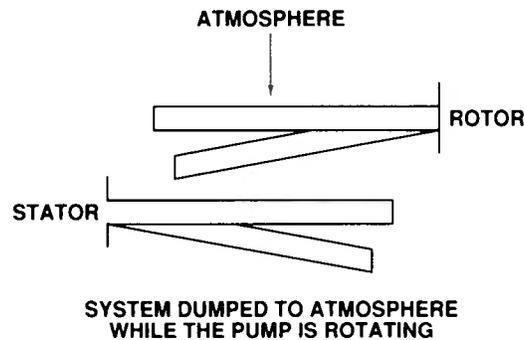
Since a turbo pump incorporates a precisely balanced high-speed rotating turbine, it should never be moved or jolted while it is in operation. This will help prevent a catastrophic crash (rotating rotor touching a stator and destroying the turbine).

There are two other modes of shock damage that should be avoided. One mode is that the system dumps to atmosphere while the pump is rotating. The second is improper venting. Let's look at these modes in further detail.

System Dumps to Atmosphere While Pump Is Rotating

Most well-designed turbos can withstand infrequent dumps to atmosphere without suffering a pump crash, although it is certainly recommended that one avoid this whenever possible!

The pump blades will flex when a gas load is dumped into the pump. If the rotor and stator blades come in contact with each other under these conditions, a catastrophic failure mode will occur. This is "fondly" called a crash. Proper valving procedures will help prevent this occurrence.

*Venting*

One should always vent the turbo pump while the rotor is still spinning.

If the pump is still in rotation when vented, rotational lift (like the way a helicopter blade produces lift) will minimize the loading on the bearings. If the balls in the bearings are not rotating, "dimples" can result as the impact on the balls permanently deforms the bearing race. Once this occurs, bearing failure can occur at any time due to the damaged bearing surface.

The proper location for the vent valve is at the turbo inlet, or at an interstage vent port, if available. Venting through the foreline is not recommended, as accumulated mechanical pump oil vapors can be forced into the high vacuum area of the pump by the inrushing air, eventually migrating to the system chamber if done

repetitively. This is the prime cause of contamination in a turbo pump vacuum system.

See the chapter on System Operation for more detail on how to vent a turbo pump properly.

Additional Concerns

Turbomolecular pumps must be protected against mechanical damage as well as against the loss of cooling water because the pump is a high-speed device with considerable stored energy. If a large, solid particle enters the rotor or a bearing seizes, serious damage may be done to the pump.

A splinter shield or screen located at the pump inlet adequately protects the rotors and stators from physical damage with some loss in pumping speed. Some pumps are available with side inlet ports. Water cooling is preferred in both oil-lubricated or grease-packed bearings. Proper cooling is necessary to remove heat from the bearings and to extend bearing life.

Turbomolecular pumps will give reliable, trouble-free operation if they are adequately lubricated and protected against cooling-water failure, power failure, mechanical damage, and excessive torque.

Backstreaming of mechanical pump oil can occur. Stopping the pump with the forepump operating and the work chamber under vacuum will result in rapid backstreaming of oil vapors from the foreline to the clean side of the pump. To minimize this backstreaming, you should always vent the turbomolecular pump from the inlet during shutdown.

The pump should be vented with dry gas in such a way that it will flow toward the foreline through at least a portion of a rotor and stator assembly. Oil vapors in the foreline are then flushed away from the high vacuum chamber. The pump must never be vented from the foreline because oil vapors will be forced back toward the pump inlet and the high vacuum chamber.

Vacuum System Use

Most system designs use the turbo pump coupled with a high vacuum valve. This design is similar to a diffusion pump system using a common roughing and foreline pump. It is possible, however, to rough pump a chamber right through the turbo; in this case, the turbo will gain speed as chamber pressure is reduced.

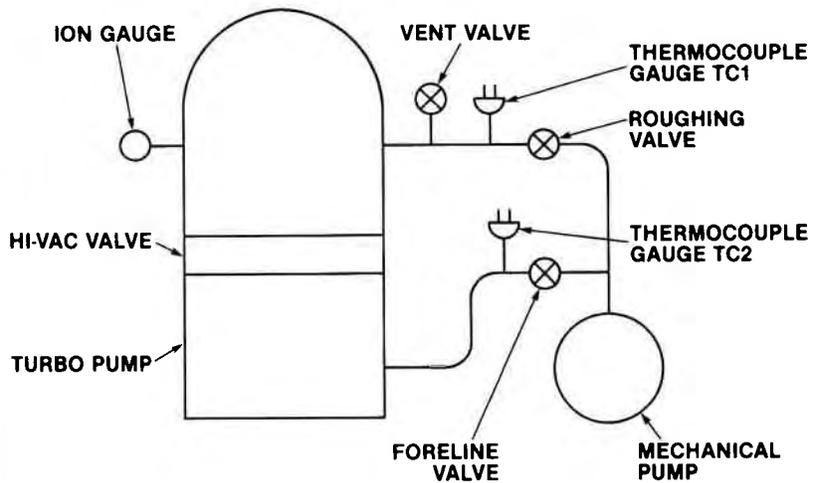
This type of system design shouldn't be used if the work chamber needs to be vented to high pressure very often, or if rapid pump-down to low pressure is required.

Turbo pumps can also be placed in series with cryotrap to promote high-speed pumping of water vapor and other condensables.

It is desirable to use a high vacuum isolation valve to isolate the turbo pump if roughing the chamber through a separate roughing line, similar to the way diffusion pump systems are manifolded.

This is especially true on fast cycling applications such as load locks, where it is desired to cycle the load lock at a rate approaching the start-up time of the turbo pump. To achieve the fastest cycle time, it is necessary first to rough the chamber through a roughing line and then transfer to the turbo pump (which is rotating at full speed). As a general rule of thumb, it is advisable to use a valved system if the chamber is going to be repetitively cycled from atmosphere to vacuum to atmosphere, with a total cycle time of less than ten minutes.

A valved system as shown here should also be used when evacuating a large vacuum chamber. Roughing the chamber through the turbo pump will be slower, since the turbo pump will have rather large conductance losses due to the small exhaust port on the turbo pump.

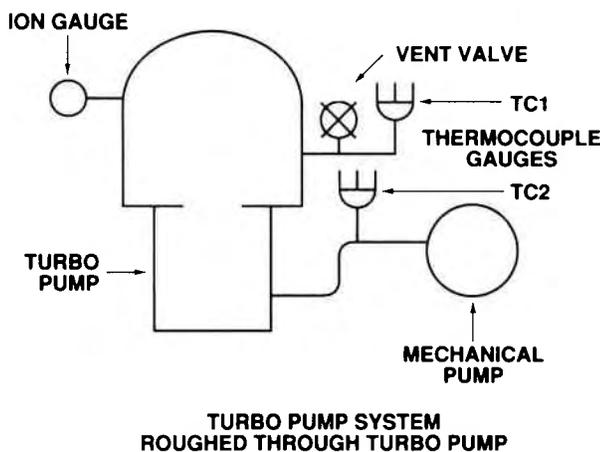


TURBO PUMP SYSTEM

Turbo pumps allow great flexibility in the choice of vacuum manifolding. For moderately sized chambers, the turbo pump can be mounted directly to the chamber without a high vacuum isolation valve. In this case, the turbo pump is cycled from atmosphere to high vacuum along with the chamber, and the chamber is roughed through the turbo. Usually, the turbo pump and

mechanical pump are started and stopped simultaneously. Most modern turbo controllers have provisions for switching the mechanical pump contactor on and off with single switch control of both mechanical pump and turbo pump from the front panel.

Alternatively, if the chamber is larger and cannot be roughed through the turbo pump to several hundred millitorr within the turbo pump start-up time, a delayed start can be employed by using a thermocouple gauge set point to delay starting the turbo pump until the pressure falls into the high millitorr range. The advantage of roughing through the turbo is that a simple system with minimal valving can be built. A disadvantage is the increased time required to reach high vacuum pressure levels.



Maintenance

If the pump and/or vacuum system become contaminated with forepump oil or bearing oil, it may prevent pumping below 10^{-5} torr. The pump and system should be cleaned with the appropriate solvents. Contact the pump manufacturer for the proper solvents and procedure.

Bearing wear is expected. Due to the precision balance and tolerances involved, bearings are normally replaced by factory-trained personnel. Turbos must generally be rebuilt every 2 to 5 years, if operated continuously. Typical turbo pump maintenance includes periodic changes of the lubricating oil for circulating oil-lubricated pumps, and relubrication by injecting the lubricant via syringe for grease-lubricated pumps. The manufacturer's recommended intervals should be followed. If an oil-lubricated pump is used, the oil should also be changed if it becomes discolored.

The manufacturer's relubrication or oil change schedule must be followed to ensure reliable lubrication of the rotor bearings. A lack of lubrication will result in bearing seizure and failure of the pump.

When the turbine speed drops to less than 60% of its nominal speed, gas or oil molecules can make their way from the foreline, past the inlet, and into the chamber. This can contaminate the pump and the product or process. Typical causes can be loss of power to the motor or controller or motor failure. Another cause can be an overpressure condition in either the chamber inlet or pump foreline.

The worst situation for a turbo pump is a hardware "crash." This is usually caused by debris falling into the pump inlet and touching the rotor blades. A "crash" literally destroys the internal pump stages. The pump must usually be replaced after this experience.

Contamination of the turbo pump rotor blades may occur, especially if the foreline is frequently air-released, as this will force mechanical pump oil vapor into the turbo pump. When contamination occurs, it will usually be evident by extended pumpdown times or an inability to reach accustomed base pressures. If this occurs, the pump and system should be cleaned with appropriate solvents. Typically, manufacturers recommend lowering a turbo pump inlet down into a solvent bath, making sure that the solvent does not reach the pump motor or bearings. Consult the manufacturer or turbo pump manual for explicit instructions and solvent to use.

As with any high-speed rotating bearing, wear will occur and require bearing replacement and rebalancing. Due to the precise balancing of the turbo pump required to achieve long bearing life, bearing replacement is usually done by factory-trained personnel.

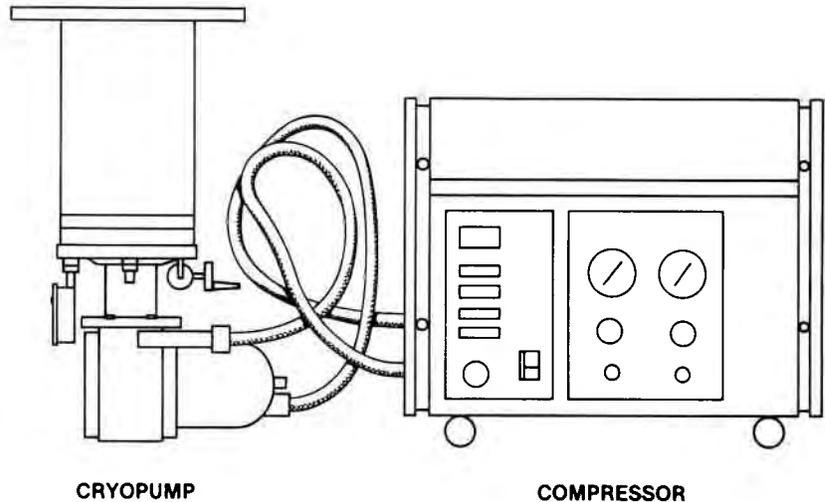
It is impossible to accurately predict bearing life due to such variations as cycling frequency, loading due to atmospheric dumps, and lubrication quality.

The bearings should be replaced when there is an increased level of noise and vibration. Worn bearings generate more noise which is more harsh in tone. You may also note an increased vibration level in your sensitive instruments.

Normal bearing replacement should be scheduled after 15,000 hours of operation.

Cryopump

The cryopump (also called mechanical cryopump) is unique in that it pumps by getting gases so cold that they freeze out and are stored, or captured, in the pump. It is extremely clean— it uses no oils, and has no moving parts in vacuum. It also has very high throughput. The cryopump is used in the high vacuum range in industrial applications where hydrocarbons cannot be tolerated. Models are available for UHV use as well.

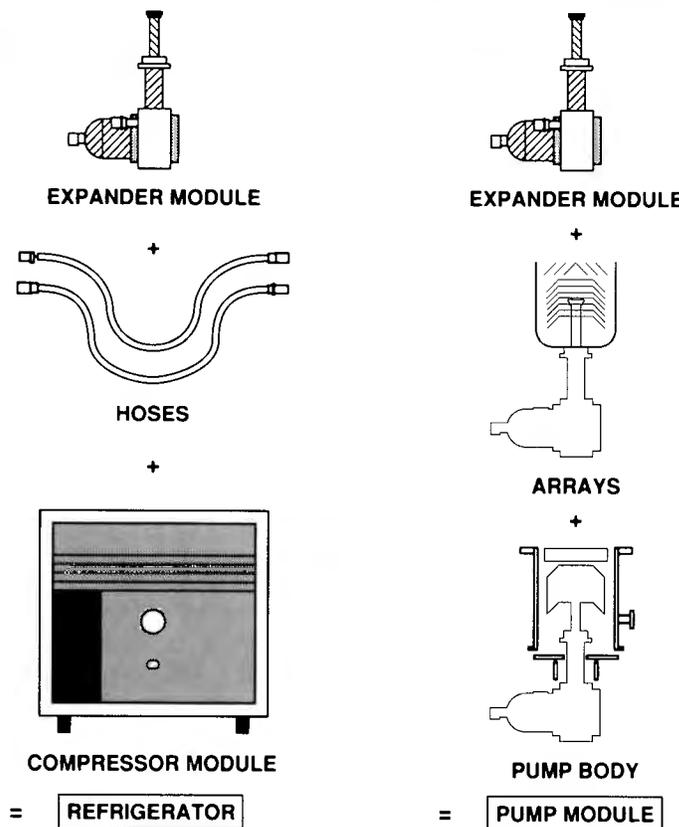


closed-loop refrigeration system

A mechanical cryopump is made up of two main components: a gaseous helium compressor and a pump consisting of a cold head, baffle, pump body and sometimes an integral high vacuum valve. The compressor and cold head operate much like a household refrigerator, except that much lower temperatures are reached. The cryopump is a *closed-loop refrigeration system*, since the coolant does not vent to atmosphere.

Components

A mechanical cryopump consists of a pump module and a compressor module. These two modules are connected by flexible hoses to form a closed-loop refrigeration system. The main components of the pump module are the expander module, arrays and pump body. The compressor module, expander module and the flexible hoses that connect them form a refrigerator. Gaseous helium is circulated between the compressor and expander. Sometimes an integral high vacuum valve is included with the pump module.



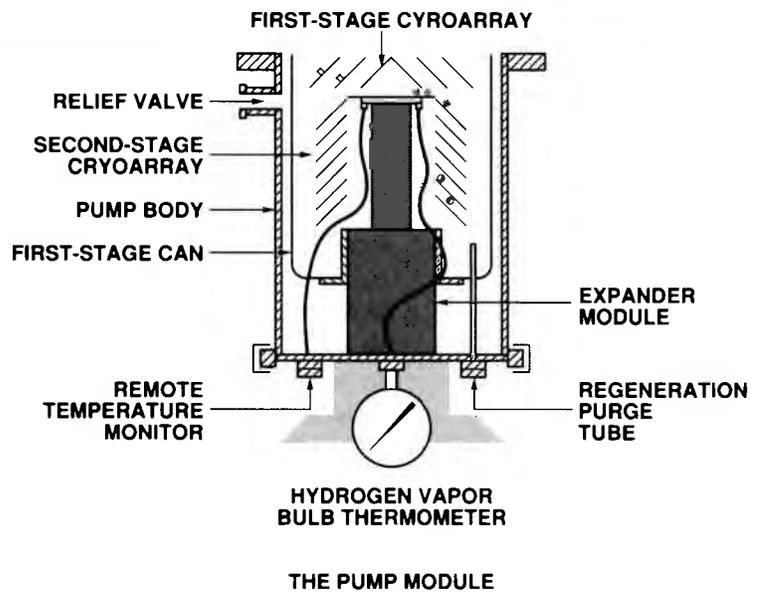
Pump Module

The pump module consists of the following major components: the expander module, the first- and second-stage cryoarrays, the pump body, the second-stage temperature monitors (remote sensor and/or hydrogen vapor bulb thermometer) and the pressure relief valve.

The expander is where refrigeration is produced. High-pressure helium gas is supplied by the compressor. This gas is expanded in two stages to produce cryogenic temperatures. The actual operating temperatures of the two stages will vary, depending on thermal and gas loads that are imposed. Typically, the first stage operates between 50° and 80°K, and the second stage, or cold head, operates between 10° and 20°K.

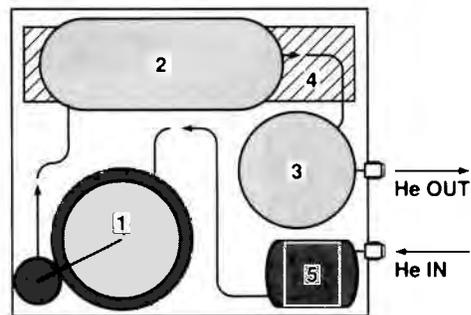
The cryoarrays are the pumping surfaces which are cooled by the expander. It is on these cryoarrays that gases from the vacuum chamber are condensed or adsorbed.

The pump body forms the vacuum-tight envelope which attaches the pump to the vacuum chamber.



Compressor Module

The compressor module supplies high-pressure, high-purity, room-temperature helium gas to the expander module. The compressor module consists of an air- or a water-cooled, oil-lubricated compressor capsule, a two-stage filtering (coalescer and adsorber) system which removes vaporized oil from the high-pressure helium stream, a surge volume to reduce the returning pressure fluctuations, an interlock system to prevent damage to the compressor, and a power supply to run the compressor and expander.



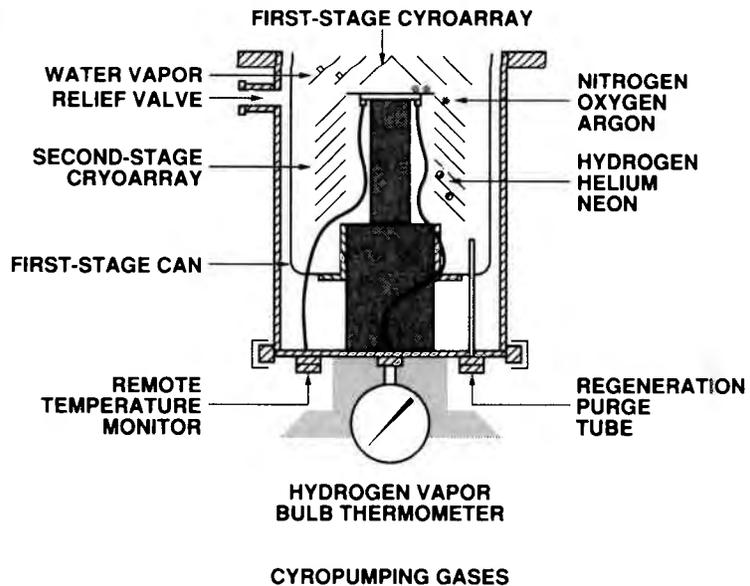
1. Compressor Capsule
2. Coalescer
3. Adsorber
4. Power Supply
5. Surge Volume

THE COMPRESSOR MODULE

How the Pump Works

In cryopump operation, helium is compressed, and gives up its heat to the surrounding walls of the compressor. This heat is removed by water or air cooling, which cools the helium. The cooled, compressed helium then goes to the pump cold head. In the expander at the cold head, a valving system allows the helium to expand. The expanded helium now takes in heat from the cold head and baffle array. This chills the cold head and baffle array to around 12°K and 70°K, respectively.

These chilled surfaces pump gases from the chamber. Chilled surfaces, in a vacuum, pump gases in two ways: The gases are either condensed (called cryocondensation) or adsorbed (called cryosorption) on the cryoarrays.

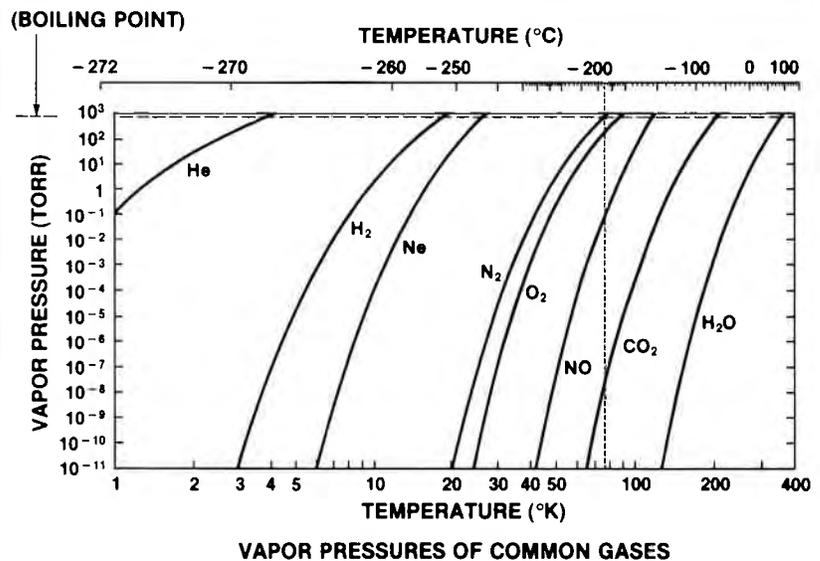


cryocondensation

Cryocondensation

The cryopump pumps most gases the same way a household refrigerator "pumps" water vapor. That is, it condenses the gases to solids. This process is called *cryocondensation*.

The likelihood that most gases will stick to a surface in an ice-like condition at less than 20°K is quite high. At this temperature, the combination of partial pressures of most gases is around 10^{-11} torr or lower.



Most gases are condensed on the first- and-second stage cryoarrays. The first-stage array is cold enough to pump water vapor by cryocondensation. The second-stage array is cold enough to pump nitrogen, oxygen, argon and most other gases by cryocondensation.

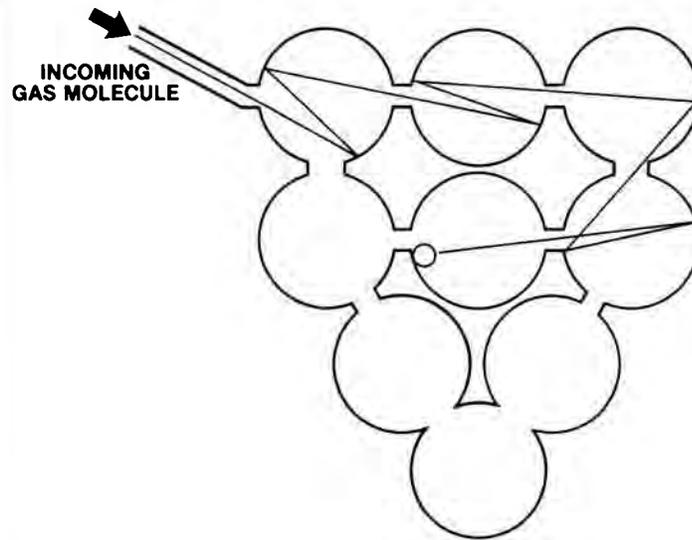
Cryosorption

The arrays are not cold enough to pump helium, hydrogen and neon by condensing them. So, we use another method to remove them. This method is called *cryosorption*.

Unlike cryocondensation, cryosorption is a surface-related phenomenon: the greater the available surface area at cryogenic temperatures, the more likely that gas molecules will stick to it. Although most gases are frozen or condensed between 12°K and 20°K, three gases are still very active at these temperatures: helium, hydrogen and neon. If we did not have some way to remove them, their partial pressures would continue to rise—perhaps to a point where the total system pressure would be unacceptable.

A very porous material is attached to the second-stage (coldest) cryoarray. This material is usually activated charcoal. Helium, hydrogen and neon are trapped on the maze-like structures and spaces of the charcoal. This is similar to a sponge soaking up water vapor at room temperature. This process is called cryosorption.

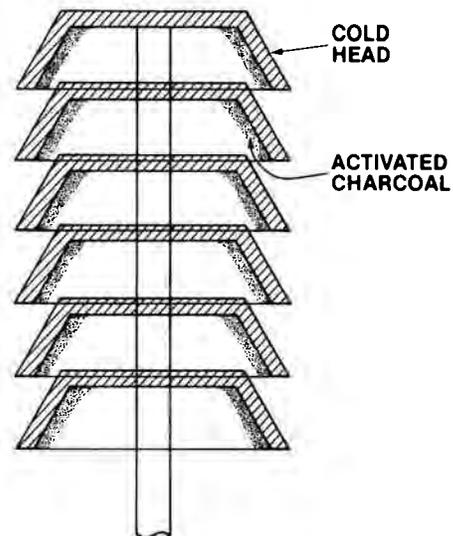
cryosorption



CRYOSORPTION

The use of porous material ensures three things:

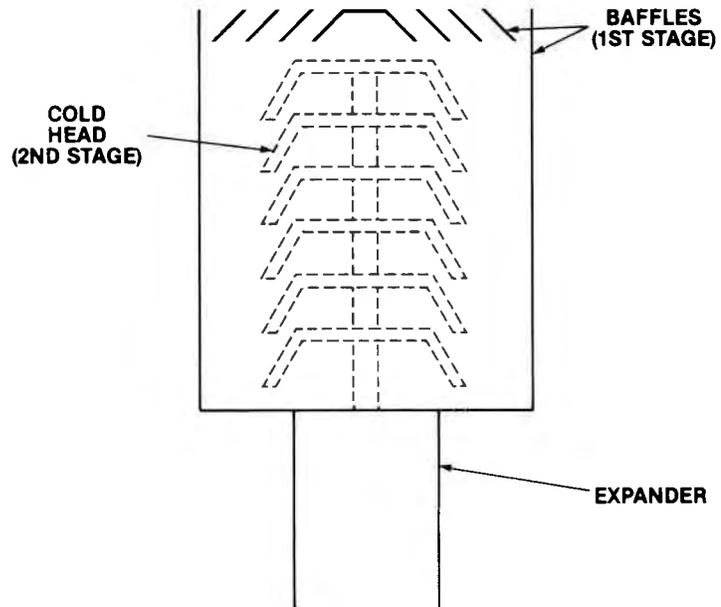
1. Tremendous surface area is available on which to trap gases. The charcoal has an enormous surface-to-volume ratio: about 8,000 ft²/cc.
2. Gases progressively lose thermal energy as they bounce around, and finally stick to these surfaces.
3. It is likely that once inside the "maze," the hard-to-trap gases will not wander out of the pump.



CHARCOAL PLACEMENT

Activated charcoal is bonded to the inner surface of the cold head or second-stage cryoarray. Since the charcoal gives plenty of

surface for helium, hydrogen and neon, it can also trap the easier-to-pump gases. Therefore, some means are required to keep the charcoal for the hard-to-pump gases only.



To keep the charcoal surfaces available for the harder-to-pump gases, the outer surface of the cold head is left uncoated. This provides adequate surface on which to condense the easy-to-pump gases. The charcoal is therefore less accessible to the easy-to-pump condensable gases. Finally, a baffle or first-stage cryo-array is placed around the cold head to limit the conductance to the charcoal surfaces.

The baffle also protects the much colder cold head from sudden temperature changes. Such sudden changes can be caused by gas loads and radiated heat from the pump body surfaces. This ensures that the gases trapped on the charcoal surfaces will not be released in the system inappropriately.

Start-Up

WARNING

The pressure relief valve is designed to prevent built-up pressure. Never alter, restrain, or remove the pressure relief valve.

Trying to start the cryopump at atmospheric pressure is like leaving the door open on your (household) refrigerator— the unit runs fine, but you are trying to cool down the whole room!

So, after we connect the power and water to our cryopump, the first thing to do is get the air out of the cryopump body. That is, we rough out the pump, using a well-trapped or an oil-free mechanical vacuum pump. This rough vacuum insulates our refrigerator from the "hot" air outside the pump—very much like a thermos bottle. Now the pump surfaces can get cold.

We then start chilldown. We valve off the roughing pump. (It will be used later for rough pumping the vacuum chamber.) We then turn on the compressor and let the pump chill down to operating temperatures. We can check this process by watching the temperature of the pump at either of two places. We can check either the hydrogen bulb thermometer or the solid-state sensor remote display—whichever is provided on the system.

Once the second-stage array is cold (below 20°K), our pump is ready to go to work. When the pump is operating, we can indeed treat it much like a refrigerator—let it run.

Crossover

Once the cryopump has cooled to operating temperatures (second stage less than 20°K), it has reached its full pumping speed and its capacity for retaining all gases. It is ready to be valved into the system. This is called *crossover*.

Cryopumps have the unique and desirable ability to pump larger-than-usual gas loads when being valved into the system. This is called the impulsive throughput capability of the pump. This provides shorter roughing times by allowing the roughing pump to be valved off at high pressures. It also reduces the probability of roughing pump oil migrating into the chamber.

The crossover pressure is the pressure in the vacuum chamber at which the roughing pump is valved off and the cryopump is valved in without adversely affecting the pump, the pressure or the process. The crossover pressure is high for cryopumps. It can be calculated by dividing the maximum impulsive throughput by the chamber volume:

$$\text{Crossover Pressure (torr)} = \frac{\text{Maximum Impulsive Throughput (torr-liters)}}{\text{Chamber Volume (liters)}}$$

You can find the impulsive gas load the cryopump can handle in your cryopump data sheet.

Steady-State Operation

After the cryopump is properly valved into the process chamber, the cryopump operating temperature should be maintained below 20°K for optimum performance. At chamber pressures less than 1×10^{-4} torr and/or when the high vacuum valve is closed, the second-stage temperature should normally be 14°K or less. For

crossover

regeneration

steady-state operating parameters such as pump speed, capacity and throughput, check your cryopump data sheet.

Regeneration

Cryopumps capture gases rather than compressing and expelling them, and they will eventually be filled up. We “empty” them by warming them to room temperature or above with heated, dry nitrogen gas. This process is called *regeneration*.

Regeneration, or emptying the cryopump, must be done periodically because the pump, like most refrigerators, will fill up with “frost,” so “defrosting” or regeneration is needed.

Regeneration Process

Remember, a cryopump captures and retains gases on its internal cold surfaces— it has a storage limit. When the level of stored gas approaches capacity, pumping speeds lessen. When that happens, the cryopumping surfaces need to be emptied or regenerated.

Regeneration is a process whereby the captured gases are released and expelled. In effect, the pumping surfaces are renewed for further service. For cryopumps, the regeneration process consists of four steps:

1. Isolate the pump from the process chamber and turn off the compressor.
2. Warm the pump while purging with dry, perhaps heated, nitrogen (maximum temperature 120°C). The stored gases will be flushed from the pump through the pressure relief valve. If the pump has been used for toxic or caustic gases, precautions must be taken to vent the relief valve to a scrubber or other approved manner. As the pump is being regenerated, the sieve traps can be heated (baked out) to regenerate them. This should continue until the second-stage cryoarray reaches room temperature: 290°-295°K.

No gauges should be placed on the pump body to read the pump pressure. During regeneration, the gauge may furnish a source of ignition to the potentially explosive gas mixture (hydrogen and oxygen) in the pump chamber.

3. Evacuate the pump with a roughing pump to remove the remaining gases and to create an insulating vacuum between the arrays and the pump body.
4. Chill the pump to operating temperature, then place it back into operation.

Please refer to the operator’s manual for the details of regeneration for your particular cryopump.

Tips on Regeneration

Here are some tips for proper regeneration. These tips should be used to help keep your cryopump in good operating condition.

- *When to regenerate.* For a cryopump system, find out the length of time to reach an unsatisfactory base pressure or operating pressure. Routinely regenerate at 60% of that time. For example, if it takes 20 working days before a system pressure becomes unacceptable, regenerate once every two weeks.
- *The number of hours between regeneration cycles can also be calculated* in the case of a continuous gas flow of a known gas species.

A = duration of operation with a continuous gas flow (hours)

B = gas flow (sccm)

C = Varian cryopump capacity for the particular gas species being pumped (std liters)

$$A = \frac{16.6 \times C}{B}$$

Example: For a sputtering application (Varian FS-8 pump) of continuously flowing argon gas at 70 sccm, the duration of continuous operation would be:

$$A = \frac{16.6 \times 1,500 \text{ std liters}}{70 \text{ sccm}} = 356 \text{ hours}$$

- *Pressure relief valve.* The pressure relief valve opens at 7–9 psig and is located just below the top of the cryopump body. **Never defeat or limit this valve.**
- *Use of the purge tube.* Purging with heated (100°–120°C) nitrogen at the required pressure (15 psig) will insure that the second-stage array will be kept clean of water vapor; also, the nitrogen purge will insure dilution of any toxic gases collected in the pump.
- *Don't shorten regeneration time.* Follow the operator's manual for proper regeneration procedure and times.
- *Regenerate the sieve trap.* Regenerating the sieve trap prior to regenerating the cryopump will assure maximum protection from roughing pump oil.

Sieve Trap Use/Regeneration

When an oil-sealed mechanical roughing pump is used, a sieve trap is needed to prevent roughing pump oil from migrating back into the cryopump and the work chamber. A sieve trap is basically a canister filled with molecular sieve material. It is installed into the roughing manifold. The sieve material consists of small, porous granules with a very large effective surface area—approximately 8,000 ft²/gram.

The migrating roughing pump oil and other contaminants are filtered out by the trap. When the trap becomes saturated with oil or water vapor, it becomes ineffective as a filter and needs to be regenerated. Regeneration consists of heating the trap to about 250°C, while roughing and purging the trap with dry nitrogen at a pressure of about 2 torr. The nitrogen sweeps the evolving contaminants toward the roughing pump.

The cryopump roughing line sieve trap should be isolated between two valves after this bakeout to protect it and the cryopump from contamination. The valves are only opened during cryopump roughing so the trap will stay clean during normal system operation. The trap will not be exposed to the constant operation of the system roughing pump. (Note that the chamber roughing line sieve trap is not usually isolated in this fashion. The frequent cycling of the chamber makes this impractical. Thus, the roughing line trap should be regenerated with greater frequency.)

As a rule of thumb, the sieve trap should be regenerated just before the cryopump is regenerated. In automated systems, the microprocessor controls the regeneration procedures. The sieve trap bakeout is part of that cycle. The general procedure for regeneration of the sieve trap is as follows:

1. Isolate the sieve trap from the cryopump/work chamber.
2. With the roughing pump on and the gas ballast valve open, set up a nitrogen flow to raise the trap pressure to 2 torr through the trap. Turn on the bakeout heater(s) for the sieve trap(s).
3. When the sieve trap has reached 250°C (about 20 to 30 minutes after turning the heater on), maintain the temperature and continue to purge and pump on it for 45 minutes.
4. Turn off the bakeout heater.
5. Continue pumping and purging the trap for about 1 hour or until it has cooled.
6. Turn off the nitrogen flow and isolate the clean trap from the mechanical pump.

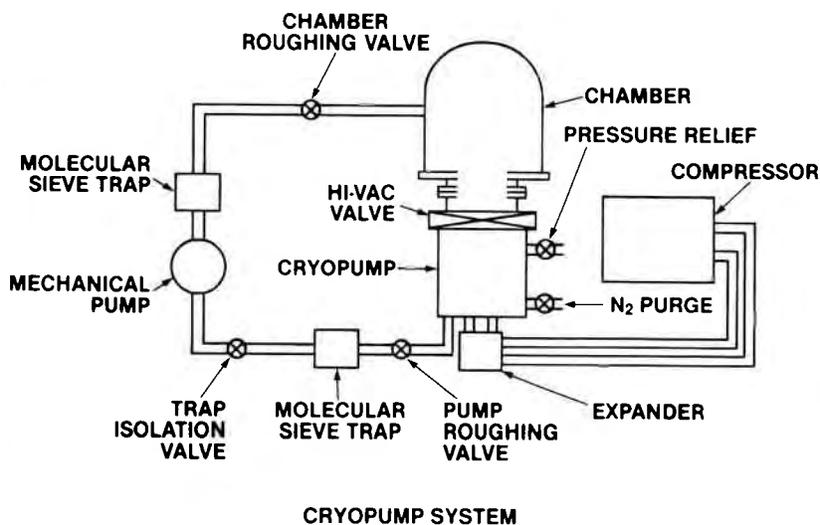
Shutdown

The cryopump can be shut down by simply closing the high vacuum isolation valve and shutting off the compressor. However, warm-up time to room temperature can be quite long (from 12 to 24 hours). Also, without proper regeneration, water vapor condensed on the first stage can migrate to the second-stage charcoal and decrease charcoal capacity.

Therefore, it is best to warm up the pump as described under "Regeneration." Warm the pump while purging with dry, perhaps heated, nitrogen (maximum temperature 120°C). The stored gases will be flushed from the pump through the pressure relief valve. This should continue until the second-stage cryoarray reaches room temperature: 290°–295°K. Once this temperature has been reached, shut off the purge gas heater (if used), close and turn off the nitrogen supply. This will ensure a dry atmosphere in the pump until further use.

Vacuum System Use

Mechanical cryopumps are currently gaining in popularity. They are used on vacuum systems wherever an ultraclean high vacuum and high throughput are required. Their use is limited by high heat processes and the presence of great quantities of hard-to-pump gases like helium, hydrogen and neon.



Maintenance

Only cryopump maintenance procedures that can safely be performed in the user's facility are described here.

Most maintenance performed at the user's facility is not on the high-pressure portion of the cryopump. Any problem relating to the pressurized helium system of the expander, hoses or compressor (except for recharging the compressor) should only be checked at a service center. When the cryopump does not operate as specified, refer to the pump operator's manual. When the cause of the problem has been found, the operator's manual indicates whether maintenance and/or repair can be done immediately or at a service center.

As-Required Maintenance

You may perform the following maintenance items when they are required.

1. Clean pressure relief valve.
2. Replace cryoarrays.
3. Recharge refrigerator with high purity (99.995% or better) helium.
4. Replace the hydrogen bulb thermometer.
5. Replace the solid-state temperature sensor.
6. Clean and replace the Aeroquip fitting hose gasket.

If the charcoal-covered arrays on the cold head have been contaminated with mechanical pump oil, they must be removed and replaced with new arrays, and all internal pump surfaces cleaned.

The pressure of the system helium supply must be maintained. It should be checked only when the pump is at room temperature (chilled gases take up less volume). If the pressure is low, the system should be charged only with ultrapure helium—99.995% or better. Severe pump damage can occur if the wrong grade of helium is used or if the helium supply is contaminated with oil, water, grease, etc. Follow manufacturer's recommendations for proper charging techniques.

For more detailed maintenance, such as removing and cleaning pump surfaces, more detailed training beyond the scope of this course and manual is necessary.

Log Book

Check the vacuum system and cryopump on a regular basis for normal operating parameters. Record these in a log book. This should include such items as:

1. Base pressure
2. Operating pressure
3. Helium pressure
4. Expander temperature
5. Water flow
6. Cryopump regeneration cycle time
7. Sieve trap regeneration cycle time
8. Time when compressor adsorber was last changed

This is by no means a complete list of items to place in your log book, just a minimal list of items which will help in maintaining and troubleshooting your cryopump.

Regular Maintenance

The following procedures should be done on a regularly scheduled basis. Enter the date and time these procedures are done in your log book for future reference.

Cryopump Regeneration

Regenerate the cryopump. The gas load generated by your process determines how often you should regenerate. Cryopumped serial coaters are typically regenerated once a week. Ion implanters, perhaps every two weeks, or monthly.

Sieve Trap Regeneration

Regenerate the sieve trap. When automatic regeneration controllers are used, sieve trap regeneration is a part of the automated pump regeneration procedure. If hydrocarbon contamination of your vacuum system is a concern, regenerate the sieve trap(s) every time you regenerate the cryopump. *Remember:* Cryopumps and oil vapors are not compatible!

Pump Parameters Check

Check and record pump parameters weekly. These are listed above under "Log Book."

Adsorber Capsule Change

Change the compressor module adsorber capsule as specified by the operator's manual: Varian's original VK™ and Cryostack™ adsorbers after 9,000 hours, and thereafter every 13,500 hours; HV, VS, and FS series every 13,500 hours.

Molecular Sieve Change

Change the molecular sieve material and solvent clean the sieve trap body once a year. Follow the procedure in the operator's manual. Periodic sieve trap maintenance is important if you must keep all hydrocarbons out of your system.

4

Ultrahigh Vacuum Pumps

Here is a list of things we think you should be able to do after reading this chapter:

You should be able to—

1. Describe the major components of each type of ultrahigh vacuum pump.
2. Explain how the major types of ultrahigh vacuum pumps work.
3. Describe the place of these pumps in vacuum system use.
4. Describe in general how these pumps are maintained.

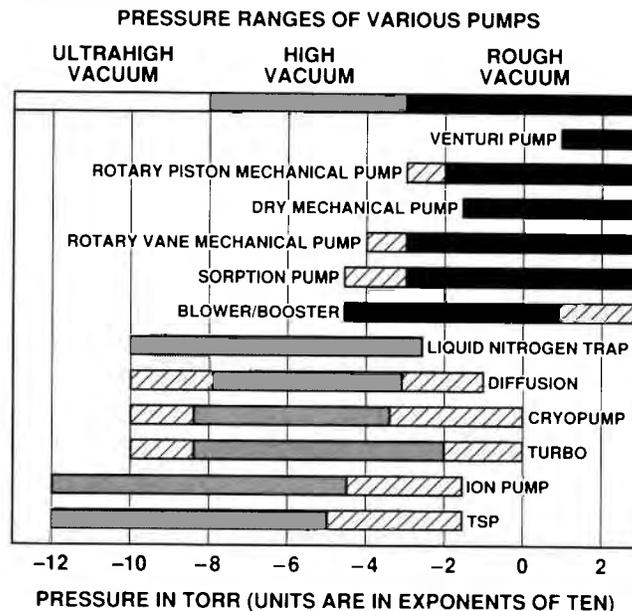
Introduction

You have seen some unique varieties of high vacuum pumps—pumps with no moving parts, pumps that aren't really considered pumps, and pumps that get quite hot or quite cold! But we aren't done yet. There's more to come in ultrahigh vacuum pumps!

bakeout

We should also state here that any high vacuum pumps that we have discussed in the previous chapter can operate in the UHV region. Generally what distinguishes UHV from other vacuum uses is, of course, the lower pressure. Another distinction is the need to *bakeout* the system to accelerate the release of gases and the need to minimize the number of permeable membranes (O-rings) in order to minimize the gas load on the pumps. The pumps that we will discuss in this chapter work very well for UHV systems—they also could be and indeed are used as high vacuum pumps as well.

In this chapter we will explain how ultrahigh vacuum pumps work and the general ways they are used. We will also discuss the major pump components and how the pumps are maintained. We will give an overview of how they fit into vacuum systems. System operation is explained in more detail in a later chapter.



This chart shows typical operating ranges for a variety of vacuum pumps. See the beginning of chapter 2 for a brief discussion of pressure ranges of pumps.

Operating the ultrahigh vacuum pumps above their normal operating range usually results in a very short operating life of the pump.

The ultrahigh vacuum pumps discussed in this chapter are:

Titanium Sublimation Pump
Non-Evaporable Getter Pump
Ion Pump

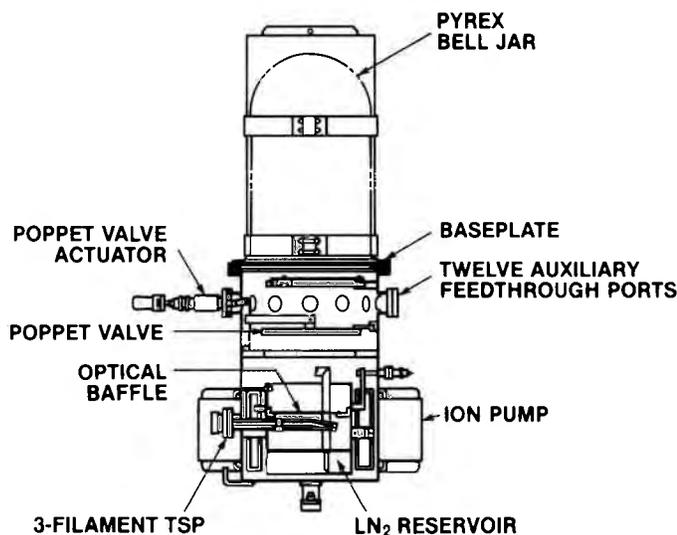
The titanium sublimation, non-evaporable getter and ion pumps are ultrahigh vacuum devices. They operate on completely different principles from those we've seen thus far. Their pumping is done mainly by chemical combination or by electrical means.

Titanium Sublimation Pump

gettering

The titanium sublimation pump is another type of gas capture or storage pump. In titanium sublimation pumping (TSP), titanium pumps gases by chemically reacting with the gas molecules, forming solid compounds. This reaction removes the molecules from the vacuum system. This method of removing gases is called *gettering*. Pumps that chemically react with gases are called getter pumps.

TSP pumps are used in vacuum system applications where no hydrocarbon or other vapors or contaminants can be tolerated; where a large pumping area or sump which is well baffled from the chamber is available; and where the presence of titanium will not interfere with the product or process.



Components

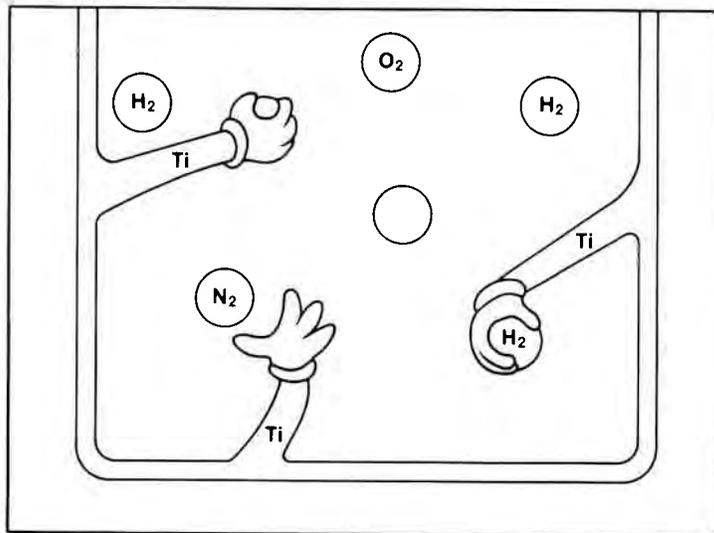
A titanium sublimation pump consists of a source of titanium, a source of heat, and surrounding surfaces on which a titanium film can be deposited.

How the Pump Works

Pump Operation

Like many substances, titanium can skip the liquid state. When properly heated, it *sublimes*. That is, it goes directly from the solid to the vapor state. This vapor deposits on the surrounding surfaces.

sublimes



Chemically, titanium is a very reactive substance. The deposited film combines readily with many gases, changing them into solid compounds such as titanium hydrides, oxides and nitrides. This is a form of chemical pumping. When the film of titanium becomes saturated, a fresh film is deposited, and the chemical pumping action resumes.

TYPICAL PUMPING SPEEDS PER SQUARE INCH OF TITANIUM
SUBLIMATION SURFACE FOR VARIOUS GASES
(Liters per Second per Square Inch)

Surface Temperature	H ₂	N ₂	O ₂	CO	CO ₂	H ₂ O
20°C	20	30	60	60	50	20
-195°C	65	65	70	70	60	90

Pumping Speed

Typical TSP pumping speeds for room temperature and LN₂ temperature are shown on the previous page. As can be seen, TSP pumping speeds improve significantly at LN₂ temperatures. This is due to the improved sticking coefficients. (A sticking coefficient is related to the amount of time available for a chemical reaction to occur as the gas molecules hit the titanium-covered sump walls.) The colder temperatures mean molecules will spend a bit more time on the wall surface. This, therefore, increases the time in which a chemical reaction can occur.

TSP pumping is very effective on reactive gases such as nitrogen, oxygen, hydrogen, carbon monoxide, carbon dioxide and water vapor. These are all common gases in vacuum systems.

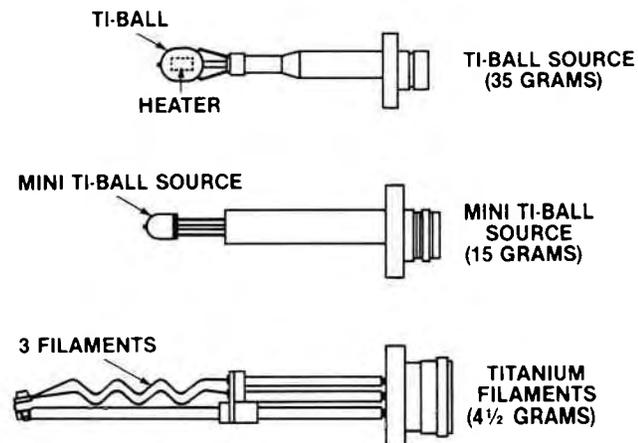
TSPs will not pump inert gases or methane very well. If operated above 10⁻³ torr, two things generally happen:

1. Oxide and hydride formation on the surface of the titanium source will work against sublimation.
2. The titanium that does manage to leave the source will tend to react with gas molecules before it can be deposited on the walls. This usually results in particulates, or "flakes." Although the flakes normally settle at the sump floor, they may be stirred up to become a process contamination source during the next roughing cycle.

Continuous deposition of titanium is unnecessary, and, in fact, is wasteful. A film of titanium can pump for extended periods at low pressures. If pumping doesn't improve following deposition of a fresh layer of titanium, it's likely that the previous layer wasn't fully "used up." The operator should watch the system gauges. After a titanium deposition, he should wait until the pressure drops and stabilizes before activating the titanium source again.

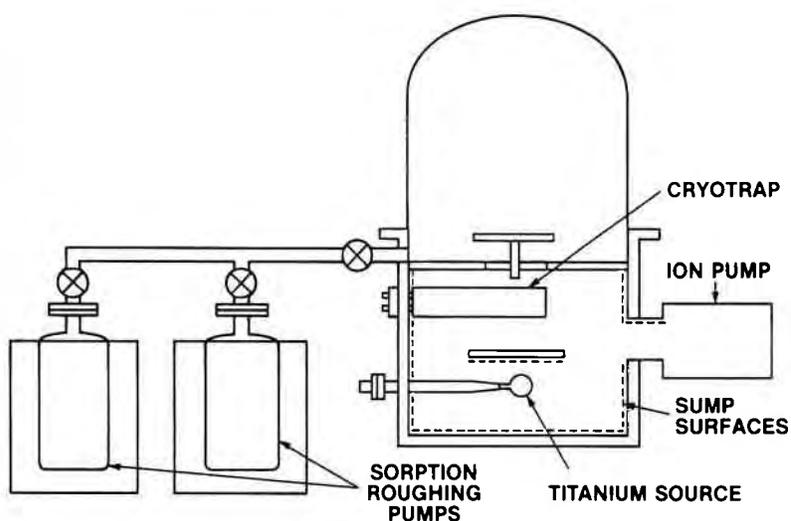
The Titanium Source

The titanium source is a compact unit; most models are mounted on a 2-3/4 in. ConFlat® Flange.



There are various titanium sources. Varian manufactures a three-filament source, a Ti-Ball Source, and a mini Ti-Ball Source. The Ti-Ball Source produces about 750 watts of heat at full power and 200 watts on standby power; the other two sources produce about 380 watts at full power. The mini Ti-Ball standby power is 95 watts; the filament source is simply turned off when not operating. The heat radiated to nearby surfaces may cause excessive outgassing in small volumes.

Vacuum System Use



Typically, UHV systems require an ultraclean environment. Because of this, TSP pumping is often used together with ion pumps and LN_2 traps or baffles. These systems are usually rough pumped by sorption pumps.

Maintenance

Titanium sources must be replaced when the titanium available for sublimation has been exhausted or when the internal heating coil (in the "ball" sources) has failed. Eventually, the sump surfaces are heavily coated and begin flaking. The sump must then be carefully cleaned.

Non-Evaporable Getter Pump

The non-evaporable getter (NEG) pump is just what its name says— a pump that reacts with gases to trap them on and within its surfaces. NEG pumps are typically used along with ion pumps to assist in pumping hydrogen at UHV (i.e., 10^{-9} torr and less). One of its advantages is that the NEG will release its gas load when heated to high temperatures (350°C). This makes it, for the most part, a regenerable pump much like a sorption or cryopump. This is in contrast to the TSP, which cannot be regenerated— we simply evaporate another layer of fresh titanium surface over the used surface.

Components

The NEG is an alloy containing zirconium, vanadium and iron (Zr-V-Fe) which is placed on a constantan strip and then formed into a cartridge for use. The cartridges may be placed in the system chamber or inside of a larger-size ion pump.

How the Pump Works

Pump Operation

The NEG is a getter pump, which means that it will pump chemically reactive gases, just as the TSP does. The pump speed for most reactive gases, such as O_2 , N_2 , CO, decreases as the NEG surface fills up with the gases. The speed for hydrogen, however, remains relatively constant even when the pump is “full” of other gases. This is a great advantage at UHV pressures because the largest gas load is usually hydrogen. Thus the NEG pump can effectively double the pumping speed compared to the ion pump alone.

The NEG is essentially pumping all the time at room temperature, and when using one, it must be regenerated after installation into the system. There is no action that needs to be taken to “turn it on.” It will need to be periodically regenerated in order to keep pumping effectively.

Regeneration

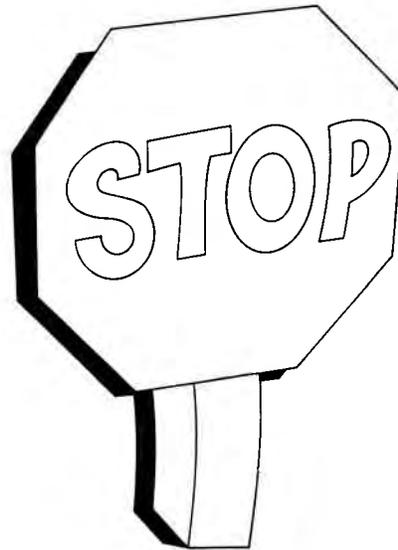
Regeneration consists of turning on the heater, the heater being the constantan strip which holds the alloy, and heating it to 350°C for approximately 1 hour. A very convenient time to do this is as a part of the bakeout cycle during pumpdown. The gases evolved from the NEG are repumped by the ion pump, the TSP, or a roughing pump at higher pressure.

Vacuum System Use

NEG pumps are used in ion pumps. Refer to the section on ion pumps later in this chapter for ion pump vacuum system use.

Maintenance

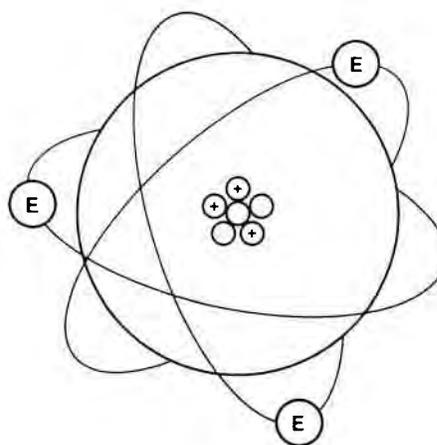
No maintenance is required of the NEG other than regeneration. After thirty or more regeneration cycles, the pump will probably need to be replaced with a fresh NEG pump. For typical UHV work, NEG life is several years because the system is kept at UHV conditions for long periods of time.



Review of the Nature of Gases

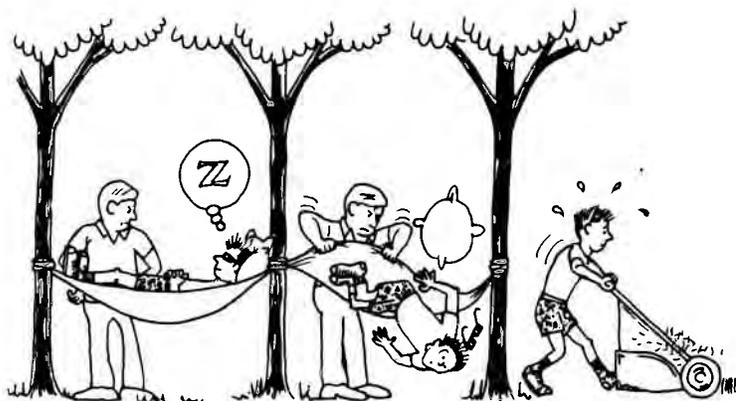
Before continuing, a few basic facts about the atomic and molecular nature of gases should be reviewed. Some of this information was discussed in chapter 1. These facts will be useful for understanding how ion pumps operate. Let's review them briefly here.

An atom is the smallest particle of matter that can exist and still retain the basic characteristics of the material or element from which it came. Molecules are simply one atom or two or more atoms joined together; many gases exist as molecules.



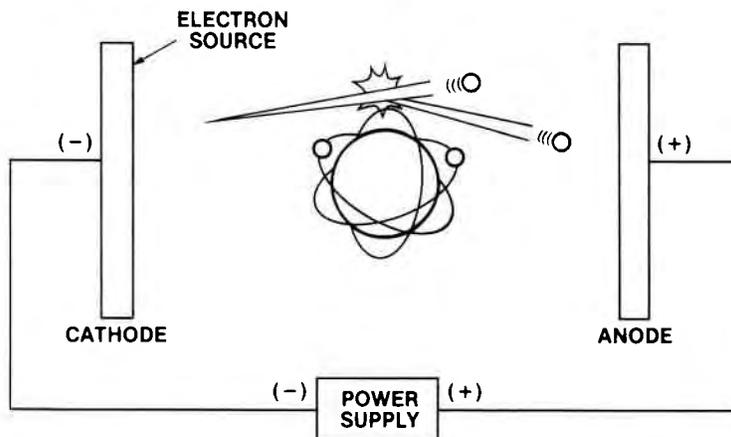
ion
ionization

Atoms and molecules normally have an equal number of protons (positively charged particles) and electrons (negatively charged particles). The neutrons in the nucleus contribute to the weight (mass) of the atom but not to the charge. The atoms are thus neutral, or electrically balanced. If this balance is upset, useful work can be produced. If we remove electrons from the atom, we have made a positively charged atom or molecule we call an *ion*. This process of creating ions is called *ionization*. We can put these charged particles to work because we can direct their motion using a magnetic or electrical field.



Ion Pump

Let's now make our ion pump by connecting two electrodes to a high-voltage supply. Electron flow will be from cathode to anode as in this drawing. Ions will carry current from anode to cathode. Fewer ions than electrons will be produced so that we can say that the current through the pump is the "ion current."



sputtering

In this drawing, a free electron is attracted to a positively charged anode. On the way to the anode, it collides with a neutral atom, ionizing it. Now two electrons are free to continue toward the anode, increasing the probability of still further ionization. The positively charged ions are then accelerated toward the negatively charged cathode. They may impact the cathode with such force that they stick to the cathode material, and are thereby pumped. As one gas molecule is driven into the cathode, one or more molecules of the cathode is usually released from this surface. This process is called *sputtering*.

The ion pump is also a gas capture pump. It is not designed to pump heavy gas loads. For this reason, it is not generally used alone in high-production applications. Instead, it is more often used in research and analytical applications where there is no need to repeatedly and rapidly cycle the work chamber to atmosphere. When combined with a TSP, it also provides adequate pumping for these applications.

Ion pumps are clean operating devices. They are electronic devices which use no moving parts or oils. It is possible to achieve pressures in the 10^{-11} torr range, with overnight bakeout of the system. The bakeout process drives residual gas off walls. This gas is then pumped by the ion pumps.

In research and analytical applications, the ion pump's cleanliness, bakeability, low power consumption, vibration-free operation and long life make it the pump of choice for most ultrahigh vacuum uses.

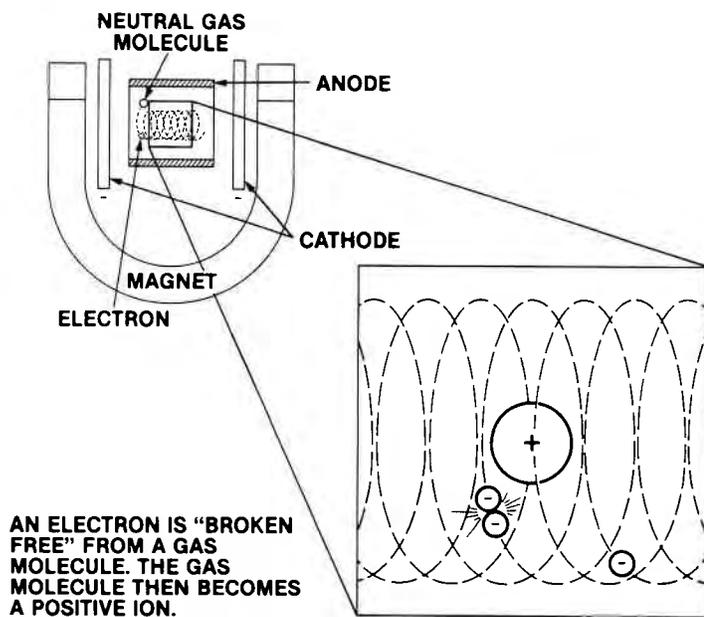
Ion pumps come in various sizes. A small appendage ion pump is used not for pumping down, but for maintaining vacuum conditions in operating devices such as transmitting tubes.

Larger pumps can be used to evacuate small chambers, or several can be connected in parallel with other ion pumps to pump down larger chambers.

Components

A basic ion pump cell consists of two titanium cathodes and an anode. All are placed between the poles of a strong permanent magnet.

How the Pump Works

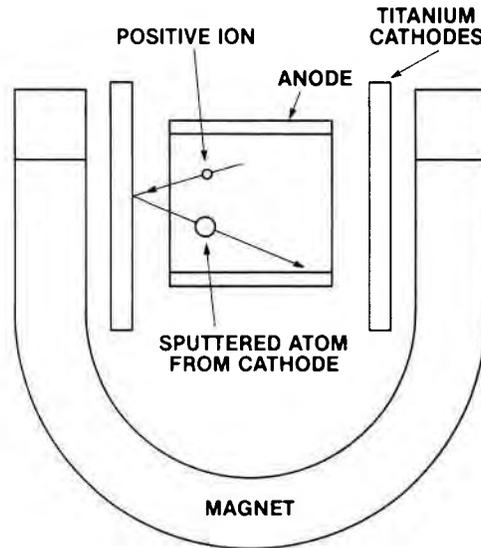


Pump Operation

The magnetic field forces the free electrons to travel in long helical paths instead of straight lines. This increases the probability of collision with molecules on their way to the positively charged anode. This, in turn, increases the ionization probability, and therefore the amount of useful pumping action that can be performed by the pump.

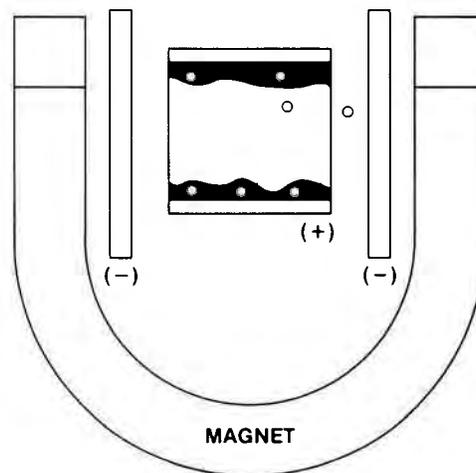
cold cathode discharge

Because of the action of the magnetic field, the electrons do not easily come in contact with the anode. As a result, a "cloud" of electrons is formed in the anode area. This electron cloud becomes fairly stable during pump operation. The electron density is high enough for efficient ionization of gas molecules. Therefore, a hot filament electron source is not needed. So, the name for this process is *cold cathode discharge*.



The positively charged ions, which are relatively heavy particles, are accelerated into the negatively charged titanium cathodes. This impact causes sputtering, or chipping away of the titanium cathode material.

Sputtered titanium deposits onto the internal structure of the pump. There it is available for chemical combination with gas molecules to convert them to solids. Thus we have the needed pumping action.



In addition, a second pumping action takes place. Some of the ionized molecules impact the cathodes with enough force to become buried in them. This burial prevents them from re-combining and becoming a free gas again.

Still another pumping process occurs in the case of hydrogen, which diffuses directly into and reacts with the cathode plate. Also, neutral molecules in the anode regions can literally be buried or "plastered over" by the sputtered cathode material. Complex molecules may also be split in the discharge to smaller, more readily pumped molecules.

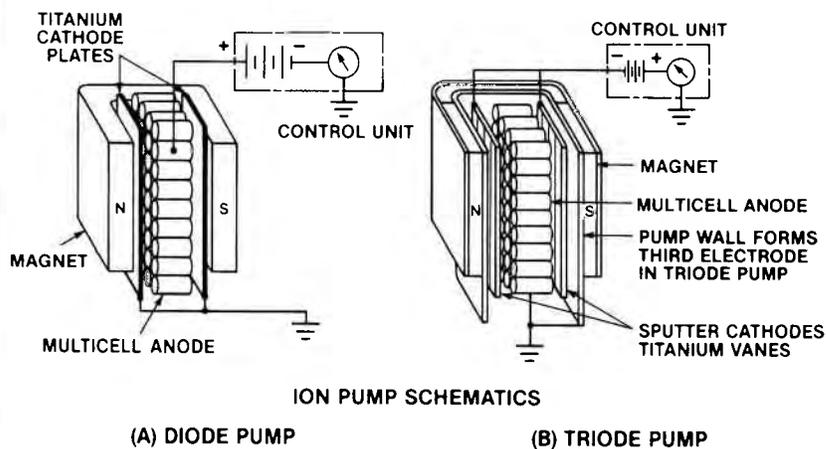
There is a problem with the pump design we have described (also called a diode configuration). Some of the buried molecules can be released again into the vacuum system. This re-release can be caused by heating of the cathodes or reduction of cathode material due to sputtering. It can also be caused by a molecule or atom being physically separated from the sputtered film.

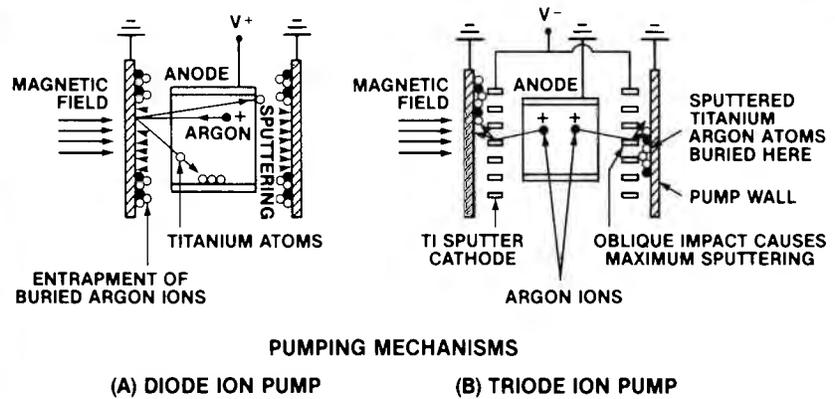
Pumping Characteristics of Different Configurations

Ion pumps are available in different design configurations. Each design has its own special pumping characteristics.

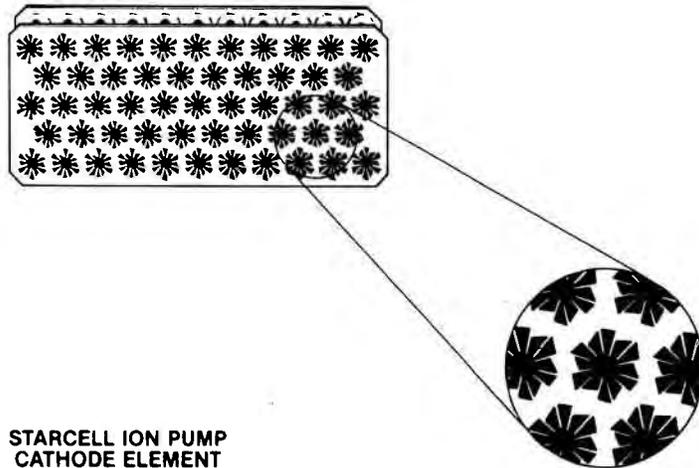
In the diode pump, as we have seen, the ions strike the cathode plate and react with the sputtered titanium.

The triode pump, which is a variation on the diode pump, improves inert or noble gas pumping. Titanium cathodes are in the form of grids. Ions sputter titanium onto the pump walls. This angled impact sputters more titanium than in the diode model and thus furnishes more material for argon or noble gas burial. Because of the electrical arrangement of the pump components, the glow discharge that happens in "starting" the diode pump is typically confined in the triode pump. As a result, the triode pump can be started at slightly higher pressure.



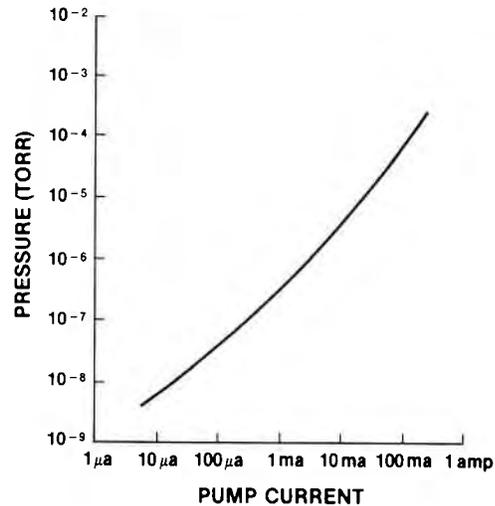


The StarCell™ ion pump provides more noble gas pumping than a triode pump and a stability which is not attainable with a diode element. The StarCell™ pump has an anode structure and two cathode plates. Radially symmetrical, finned cells in the cathode plates are concentric with the anode cells. This increases the probability of higher rebound energy noble gas molecules—an improvement in the efficiency of the noble gas pumping mechanism. Its useful life is greater than diode- or triode-type pumps.



Other Characteristics

The ion pump is self-regulating. At the higher pressures, where much ionization takes place, more current flows. At low pressures, less current flows. This characteristic current drain can be used to measure the pressure, or degree of vacuum achieved with the pump. This feature eliminates the need for an ion gauge on the system.



Ion pumps are long lived; the lower the pressure, the longer the life. Once they begin pumping, they quickly lower the pressure to the long-life region. As long as they are not pumping against a leak, they will last for years. Ideally, ion pumps should be started at pressures approaching 10^{-5} torr. At higher pressures, the plasma discharge that is generated minimizes pumping speed and reduces cathode life. A more common and practical approach is to sorption rough the pump to less than 10^{-2} torr before applying the ion pump power. At very low pressures, the time taken to begin the ionization process may be excessively long.

TYPICAL DIODE PUMP SERVICE LIFE

Pressure (Torr)	Life (Hours)
10^{-3}	20
10^{-4}	200
10^{-5}	2,000
10^{-7}	200,000 (over 20 years of constant operation)

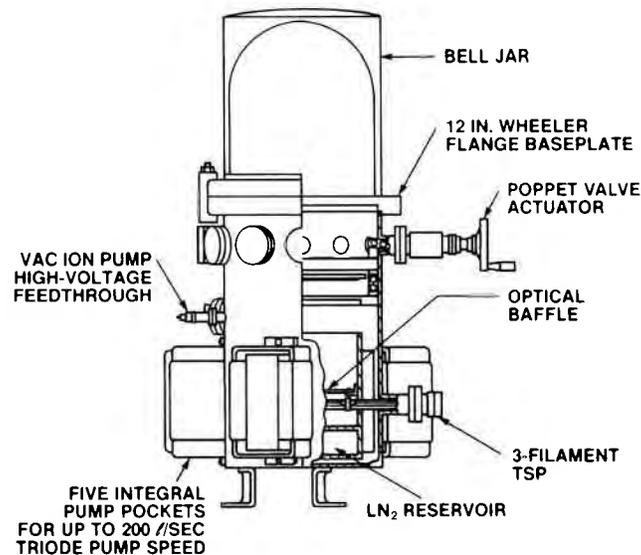
Life (Pumping N_2 at 10^{-6} Torr)

Triode	35,000 hours — approx. 4 years
Diode	50,000 hours — approx. 6 years
StarCell	80,000 hours — approx. 9 years

Vacuum System Use

Ion pumps are typically used in systems which demand ultra-clean, ultrahigh vacuum. This type of vacuum system is pumped to high vacuum or lower pressure and then kept in that condition

for long periods of time. A load-lock chamber is often built on the system to allow access to the chamber without bringing the chamber back to air. Typical uses are for electron microscopes, mass spectrometers, and surface analysis, to mention a few.



Maintenance

Very little maintenance can be performed on ion pumps other than an occasional bakeout. When pumping eventually deteriorates to the point where operating pressures can no longer be attained, pump replacement or sometimes anode/cathode assembly replacement is necessary.

Summary

We have discussed the pressure ranges of vacuum pumps and the major types of pumps in each range. By now, you should be familiar with the different types of vacuum pumps—what their major components are and how they work. You have also learned how they are placed in vacuum systems and some general maintenance information.

Let's go on now to gauges. These are major vacuum components that tell you what is going on inside your vacuum system.

5

Gauges

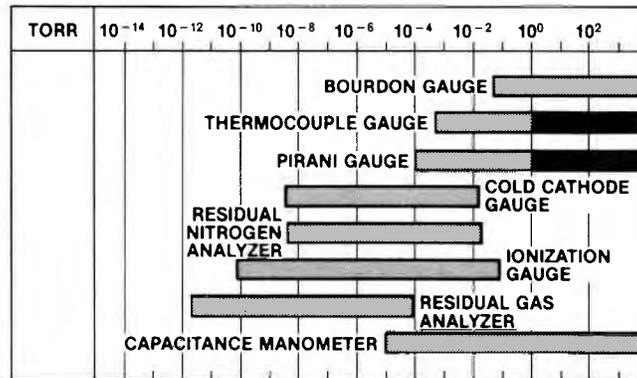
In this chapter, you will learn to do the following:

1. List the commonly used vacuum gauges.
2. Give the pressure range in which each of these gauges operates.
3. Explain how these gauges work.
4. Describe how these gauges are maintained.

Introduction

Gauges are an important part of your vacuum system. You rely on them to tell you what is happening inside the system. If you have worked with vacuum systems, you know that you must read several gauges— not just one. The pressure range is too great for any one gauge to read. The various gauges all have limited useful ranges. As a result, we must use several different types of gauges in order to read all the pressure ranges. Many gauges measure pressure. Only the commonly used ones are covered here. For example, the Bourdon, thermocouple, ionization, and cold cathode gauges, residual nitrogen analyzer, capacitance manometer, and residual gas analyzer. This chart shows typical pressure ranges of the gauges.

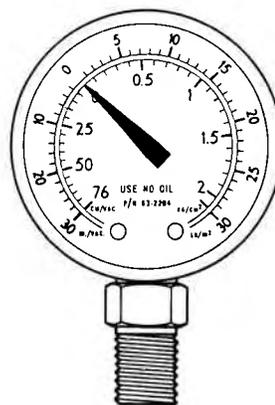
TYPICAL GAUGE PRESSURE RANGES



The gauges cover the entire operating range of all vacuum pumps covered in the previous chapters. We will start with rough vacuum gauges and work our way to the high vacuum gauges.

We would like our gauge to respond to total pressure— force per unit area— in order to be able to read the pressure in our vacuum system. We will see that for most vacuum gauges, the actual force per unit area is not what is measured. The gauge senses some other property. This reading is then converted to a pressure reading. Let's look at some rough vacuum gauges now.

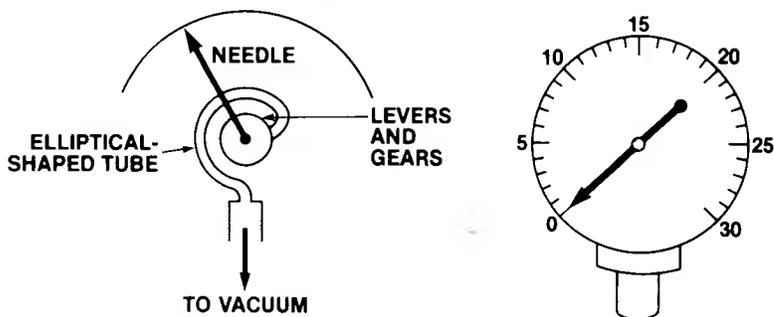
Bourdon Gauge



The Bourdon gauge is rugged and reliable. It can be used for measuring pressures above and below atmospheric pressure. Notice that it resembles a vacuum pressure gauge used in automotive tune-ups—same principle!

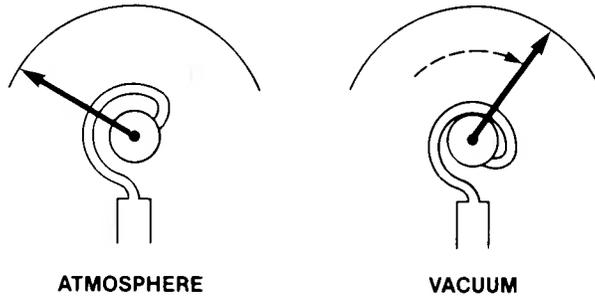
Generally, the accuracy of the Bourdon gauge is not high, but it is simple and reliable. At the lower end of its range, it really is not very sensitive. It stops reading around 1 to 0.1 torr.

How the Gauge Works



The Bourdon gauge measures relative pressure. Here's how it works. A tube in the Bourdon gauge is bent into an arc. One end is open to the vacuum chamber. Its other end is sealed and connected to an indicator needle. The meter scale is calibrated in inches of vacuum: zero is atmospheric pressure, and 30 inches corresponds to the low end of its range. This, of course, means that it is reading the relative pressure, or gauge pressure. It is comparing the pressure inside the tube to the pressure outside the tube. The gauge does respond to changes in pressure; but these

are the changes inside relative to the pressure outside. Since the pressure outside is always changing, by as much as $\pm 10\%$ from the average pressure, its accuracy is only moderately good.



The inside of the tube is open to the chamber, and the outside is at atmosphere. As the chamber is pumped out, the pressure difference between the inside of the tube and the outside of the tube causes the curvature of the tube to change. A system of levers and gears moves the needle according to the change in the bend of the tube.

Maintenance

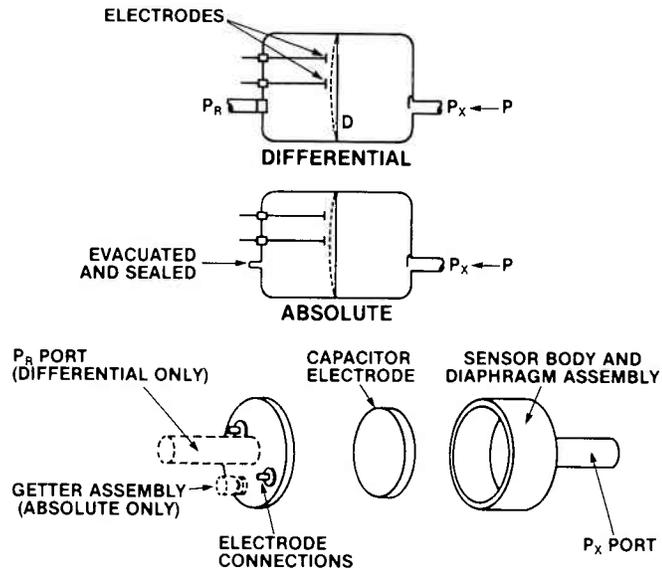
Bourdon gauges typically do not get any maintenance. The more expensive varieties may have mechanical adjustments that can be made. In general, the gauge is replaced rather than repaired.

Capacitance Manometer

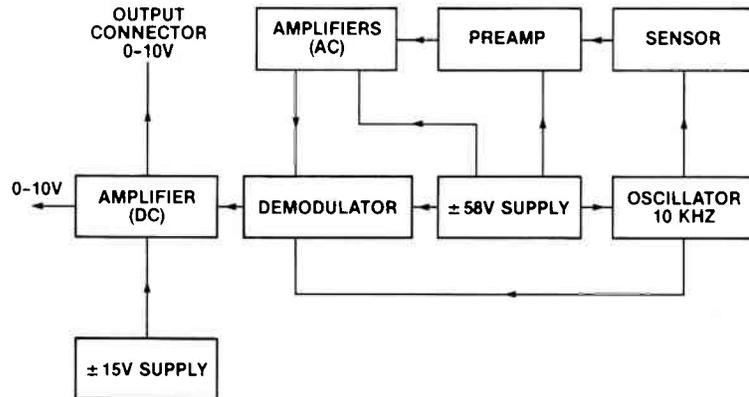
The capacitance manometer is another pressure gauge which can be used in the rough vacuum range. It is capable of measuring the absolute pressure or relative pressure, depending on the gauge model used. It does respond to the total pressure. It is not sensitive to changes in gas mixture as are many other gauges.

The sensing unit contains a tensioned metal diaphragm, one side of which is exposed to the gas whose pressure is to be measured. For absolute pressure measurement, the other (reference) side contains an electrode assembly placed in a sealed high vacuum reference cavity. Since the electrodes in the absolute pressure gauge are not exposed to the gases being measured, this gauge is not affected by oil or water vapors, or by corrosive or other chemically-active process gases.

How the Gauge Works



The diaphragm deflects with changing pressure—force per unit area— independent of the composition of the measured gas. This causes a capacitance change between the diaphragm and the adjacent electrode assembly. The capacitance change is sensed in an oscillator circuit and converted to a frequency change proportional to the diaphragm deflection.



This frequency change, in turn, is converted in the unit to be displayed as the pressure reading. The sensor unit may be constructed of materials such as inconel and stainless steel, allowing the gauge to be used with corrosive gases.

This gauge is sufficiently accurate (about 1% of reading) and precise that one can worry about the effects of temperature changes (Charles' Law) on the pressure readings. The sensor head may be placed in a constant temperature oven as a result.

This gauge is often used as a flow controller because of its fast response (milliseconds) to pressure changes. If you desire to use a capacitance manometer over a wide range, you may need several units. The gauge is constructed to read over three or four orders of magnitude. If you wish to read from atmosphere (7.6×10^2 torr) into the high vacuum range (1×10^{-5} torr), that is seven orders of magnitude. Therefore, you need several different gauge units. These gauges can be constructed so that pressures from 10,000 (10^5) torr to 10^{-5} torr may be sensed, but any particular gauge is limited to about four orders of magnitude of that range. Below 1 mtorr (10^{-3} torr), the accuracy falls dramatically.

Maintenance

The capacitance manometer may receive more maintenance than many gauges because of its ability to read accurate and precise pressure values. It may periodically be taken to the calibration lab for a check against some standard gauge. When it is used in dirty or corrosive gas systems, the sensing side of the gauge head may be flushed with an appropriate solvent.

Overpressuring the gauge (20% over full scale) may shift the reading or permanently damage it. An isolation valve is often used to prevent this.

Heat Transfer

Well, we are finished with “real” pressure gauges. The rest of the gauges we will be discussing sense some property of the gas present in our vacuum system and convert it to a pressure reading. One of these properties we will be using is the thermal conductivity of gases. Let’s take a minute to discuss the concept of heat transfer.

You have probably learned somewhere that heat can be transferred three ways: conduction, convection, and radiation. Let’s look at these three ways on a molecular scale.

conduction

To transfer heat, or energy, by *conduction*, molecules need to touch a surface or another molecule in order to transfer the heat. This principle is used in gauges between 2 torr and 10^{-3} torr.

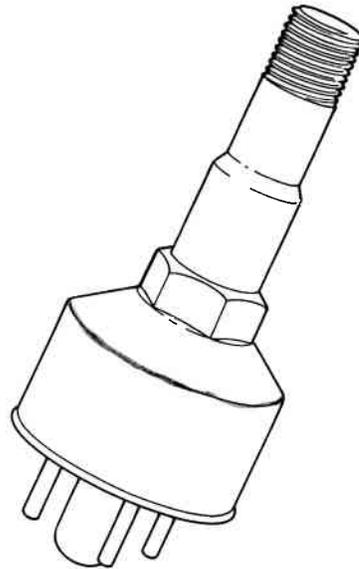
convection

To transfer heat by *convection*, we need massive numbers of molecules flowing. Your hot-air furnace heats by convection. Some gauges use this principle between 760 torr and 2 torr but are generally less accurate in this pressure range.

radiation

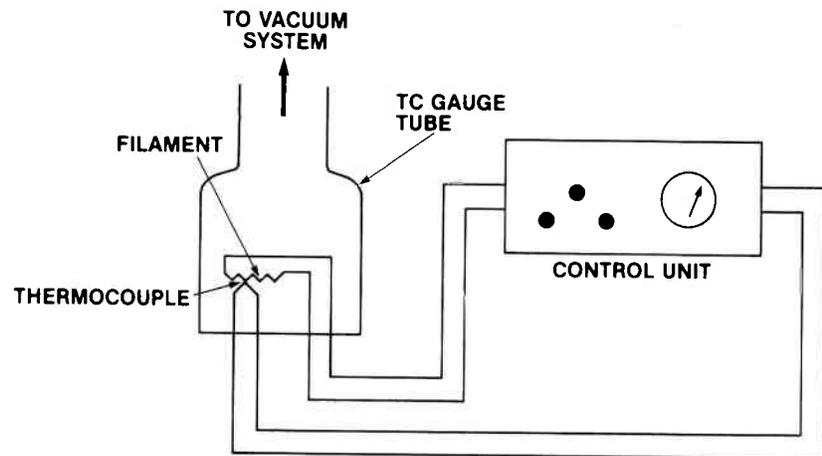
To transfer heat by *radiation*, we need light energy. Not the kind of light that you see, but typically infrared light. The heat you feel when standing in front of a fireplace is mostly the radiated heat. No gas molecules need be involved; that is, radiation is independent of the number of gas molecules present. Radiated heat is the only way to transfer heat inside of a vacuum system at high vacuum. There are insufficient molecules present to provide heat transfer by either conduction or convection. Now, let's go on to discuss gauges that depend on heat transfer to work.

Thermocouple Gauge



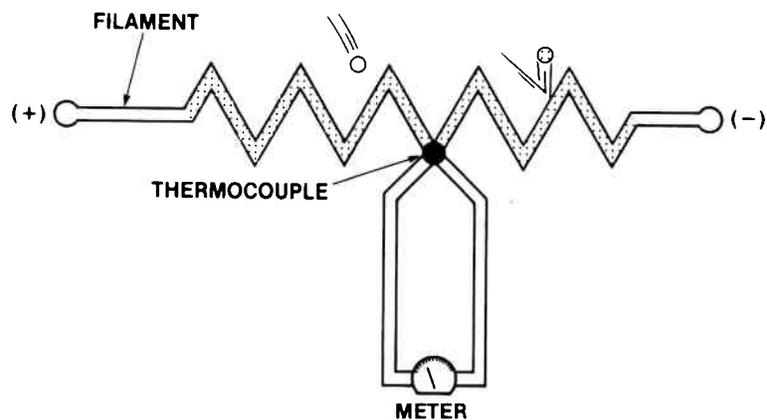
The thermocouple, or TC, gauge is another rugged, simple instrument. It is used to measure pressures in the rough vacuum range. It does its work well under less than ideal conditions. The TC gauge measures temperature and converts it to a pressure reading. Many modern thermocouple gauges have been modified to use convection as well as conduction principles. This effectively extends their useful range to atmosphere. It is typically considered as a very approximate device. Let's take a look at how it works.

How the Gauge Works



A thermocouple gauge consists of a gauge tube and control unit. Within the gauge tube is a heated filament. Spot welded to the filament is a thermocouple that measures the temperature of the hot wire. The meter is calibrated in pressure units, not in temperature.

THERMOCOUPLE GAUGE PRINCIPLE



At atmospheric pressure, there will be many molecular collisions with the heated filament. The gas molecules conduct heat away from the filament. The amount of heat removal can be related to the amount of gas in the chamber. At higher pressures, with lots of molecules, much heat will be conducted away from the wire. Therefore, the wire will be at a lower temperature (cooler). When we pump away the gas, there are fewer molecules to collide with the wire. The wire is therefore at a higher temperature (hotter).

There is not a linear relationship between wire temperature and pressure, so the pressure scale on your TC gauge is not linear. The gauge stops responding at about 1 mtorr (10^{-3} torr) because the heat loss through radiation is now the largest factor. The heat lost through radiation is also constant. Therefore, the gauge reads "zero." Compared to other gauges, the TC gauge has a slow response time. This is because the wire must have time to heat up or cool down as the pressure changes. Some newer gauges speed up the response time by operating the gauge at constant temperature and measuring the change in current required to hold the temperature constant.

Maintenance

If the sensing unit, or gauge head, gets dirty, it may be cleaned with an appropriate solvent. Most people will simply discard the TC gauge and install a new one in its place.

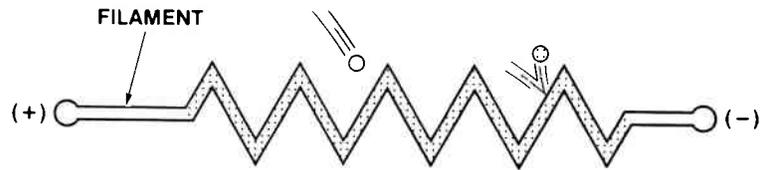
Whenever you clean or replace a TC gauge, it should be adjusted to read the proper values. To do this, you expose the gauge head to a pressure of 10^{-4} torr or less and adjust the control unit to read zero on the pressure gauge. If for some reason you cannot obtain a pressure below 10^{-4} torr, then install a "good" gauge and set the system gauge to read the same pressure.

Please check the operation manual for your particular unit for adjustment instructions, because they do vary in detail.

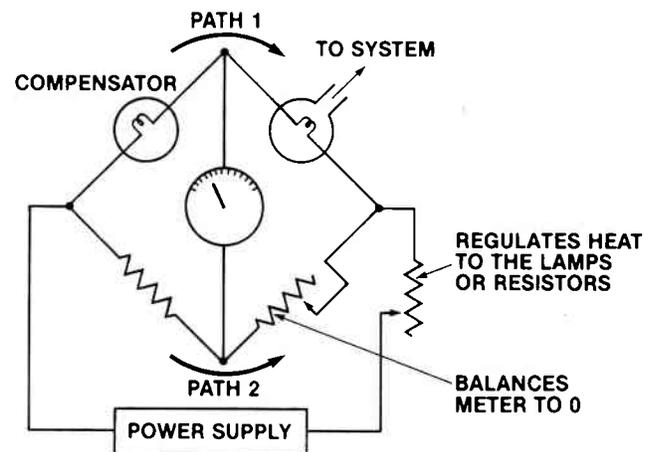
Pirani Gauge

The Pirani gauge operates similarly to the thermocouple gauge. It will read pressures best from about 2 torr to 0.001 torr. The appeal of this gauge is based mostly on its faster response time and range. Because of its more complex design, it is larger and more costly than the thermocouple gauge.

How the Gauge Works



In the Pirani gauge, gas molecules also conduct heat away from a hot filament. In this gauge, we depend on the change in resistance as the temperature changes. The temperature change causes a change in the filament resistance. The filament is part of a bridge circuit that drives the pressure meter. A simplified explanation of this circuit is given below.



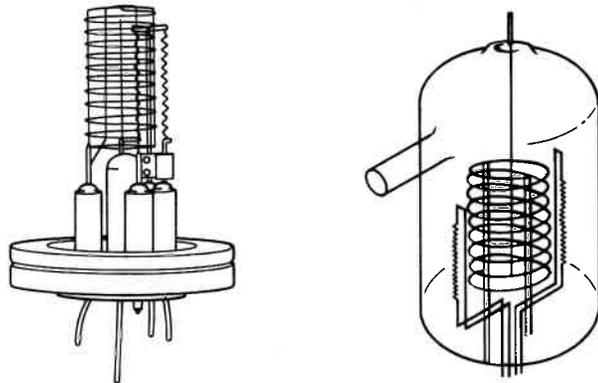
In a balanced bridge circuit, the current flow in Path Number One equals the current flow in Path Number Two. A meter, placed in the center as shown, indicates zero. When heat is conducted away from the filament, its resistance is changed, and this balance is upset. This unbalance develops a voltage difference at the meter connections, and current flows through it. Again, the meter is calibrated in pressure units. The hotter the filament, the more resistance it has. The compensator, which is a very similar filament, remains at a constant temperature and pressure. Therefore, its resistance is relatively constant. Ambient temperature changes do affect the gauge significantly below 10^{-3} torr.

Maintenance

Pirani gauge maintenance includes adjusting and cleaning. A Pirani gauge can be adjusted. Please consult your manual for the gauge to determine the proper procedure.

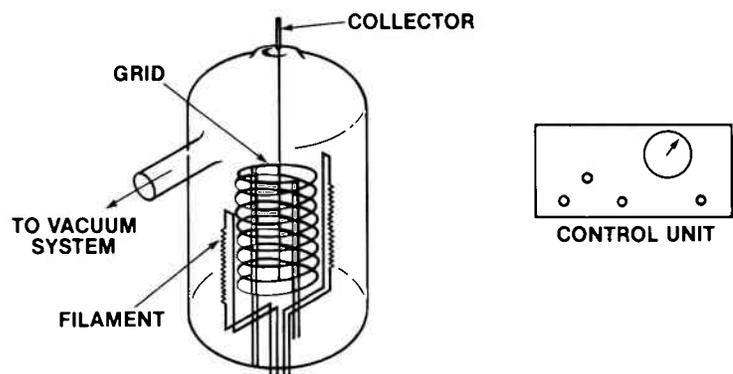
Ionization Gauge

The ionization gauge works on yet another property of molecules. They use the property that if you can energize an atom or molecule, it may lose an electron and become charged. These charged molecules (ions) can be attracted and "counted" as they pick up an electron to become neutral again. This is very similar to the way the ion pump works. Let's look at how this is accomplished as we discuss the gauge.



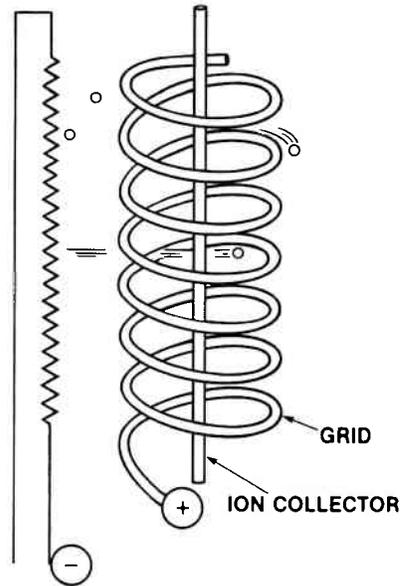
The ionization gauge is perhaps unique in that it can be used over a pressure range spanning eight orders of magnitude (10^{-3} to 10^{-11} torr). It is commonly used over seven orders of magnitude (10^{-3} to 10^{-10}) and expected to be within $\pm 20\%$ of the correct value over that range. This makes it the most widely used gauge for high vacuum work. Models include both glass and metal envelopes as well as "nude" gauges which mount directly in the chamber.

How the Gauge Works

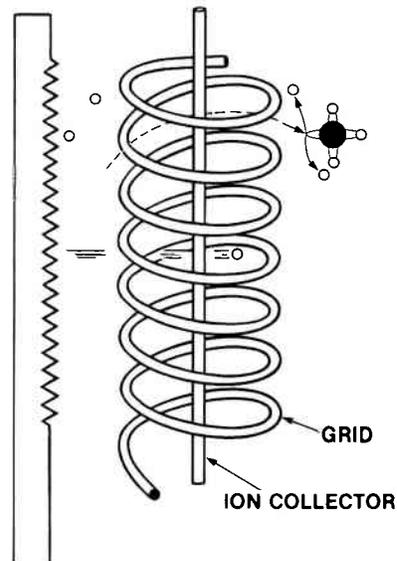


hot cathode ionization gauge

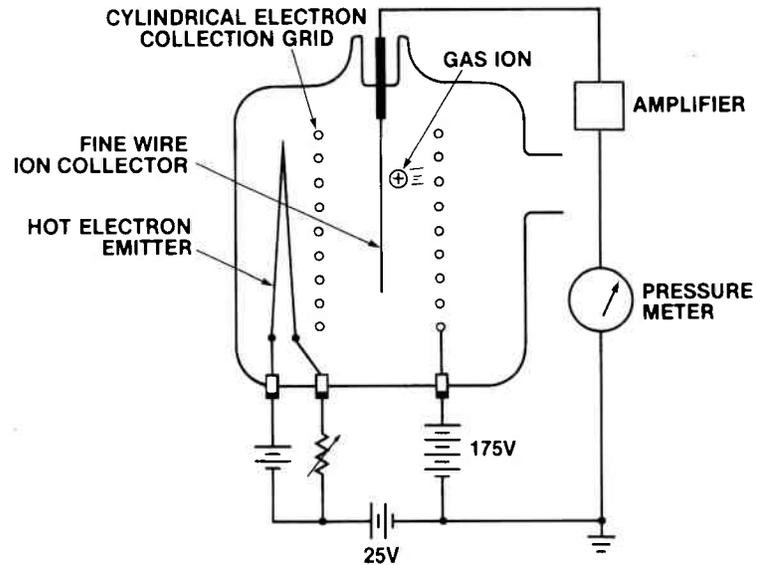
One *ionization gauge* has a hot filament, a grid and an ion collector. A control unit provides power, amplification, and metering. The hot filament supplies ionizing electrons. The grid attracts these electrons. The ion collector attracts the ions and gives up electrons as ions are neutralized. This process creates a small "ion current" which is then amplified.



Let's look at the process in more detail. Electrons emitted by the hot filament are attracted to the positive grid. However, many electrons miss the loosely wound grid and swing past it several times before finally striking it. Because of the large number of electrons emitted by the filament, a fairly constant "electron cloud" is present about the grid in the gauge tube.



On their long, round-about flight to the grid, electrons may collide with gas molecules, ionizing them and releasing more electrons. The longer the flight of these electrons, the greater the chance of collision. Therefore, a stronger, more usable signal is produced.



The positively ionized gas molecules are attracted to the collector. This produces an ion current proportional to the pressure in the chamber. To get meaningful pressure readings, the sensitivity of the gauge must be known, and the emission current must be well regulated. The "sensitivity" selector on the controller must be set the same as the rated sensitivity of the gauge tube. If this "matching" is not done, you will get inaccurate pressure readings.

The sensitivity of the ion gauge is obtained from the relationship

$$P = \frac{1}{S} \times \frac{i_p}{i_e}$$

where P is pressure,

S is gauge sensitivity,

i_p is the positive ion current,

i_e is the emission current.

As you can see from the ratio i_p/i_e , there are two variables, not one. If the emission current varies, it will cause a variation in the ionization current and therefore the pressure. Most manufacturers of ion gauges go to great pains to insure that i_e is constant. Varian uses a patented Ratiomatic™ circuit to directly measure the ratio of the ion current to the emission current. This allows small variations in the emission current to occur without affecting the gauge reading.

The ion gauge has limits like any of the other gauges. The x-ray limit determines the low end of the ion gauge range. Low-energy

x-rays are created when electrons strike the grid. They are always being produced. Those x-rays that strike the collector release photoelectrons and cause a constant error signal. This x-ray current is insignificant until about 10^{-10} torr. There, the value of pressure-related ion current approaches the x-ray current. To read low pressures, we must make a correction for the x-ray current. Doing this makes it possible to read pressures below 10^{-11} torr.

If you look at the manual for your ion gauge, you will find a table of correction factors. Some modern gauges allow you to put a correction factor or two into the control unit. These correction factors have to do with the fact that some molecules are easier to ionize than others. They will therefore give an incorrect pressure reading. The gauge unit is calibrated using nitrogen gas (or perhaps air). If you now use a different gas in your vacuum system, you should apply the proper correction factor to get "correct" pressure readings. You can, of course, do as many people do and "use the pressure that works."

Maintenance

Ion gauges can sometimes be cleaned using appropriate solvents, though we do not advise it. Most people will use the degas control as a cleaning procedure. The degas heats up the grid so that it bakes the walls of the gauge tube. This baking drives any molecules collected on the walls back into the vacuum system where they may be pumped away. Degassing a tube that is contaminated with silicone-based pump oils permanently affects it.

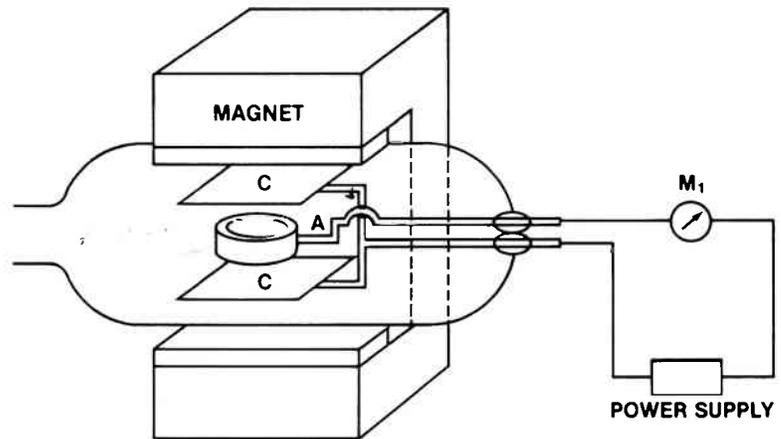
Attempts to clean a gauge tube are not always successful. But if you choose to clean your gauge head with solvents, be certain to thoroughly clean and dry the gauge before installing it. Operating the degas function, which heats the grid, can cause enough outgassing of volatile solvents to create a plasma discharge. The resulting "short" could cause severe electrical shock or death if the system and controller are not adequately grounded. Be sure the gauge has thoroughly dried before reinstallation and subsequent operation.

The ion gauge will need periodic adjustments of the control unit. Refer to the manual for your specific gauge as to how to carry out the procedure. The gauge unit in some installations will require calibration. This involves checking the gauge head and control unit versus a standard or known gauge. The gauge is adjusted to indicate the same pressure as the standard. It is then typically "certified." The National Institute of Standards and Technology will now provide this service for a fee.

Cold Cathode Gauge

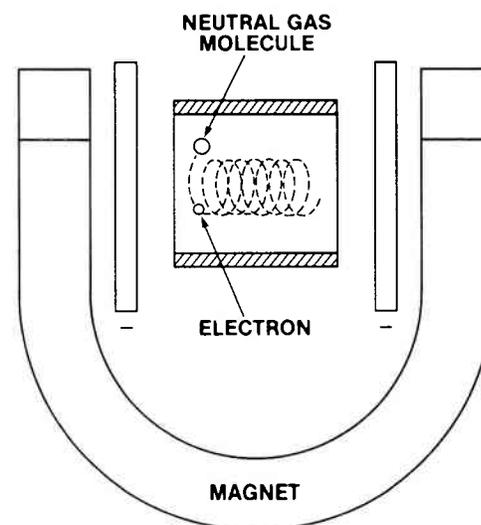
cold cathode ionization gauge

Another high vacuum gauge is the *cold cathode ionization gauge*. It also depends on the ability to ionize molecules.

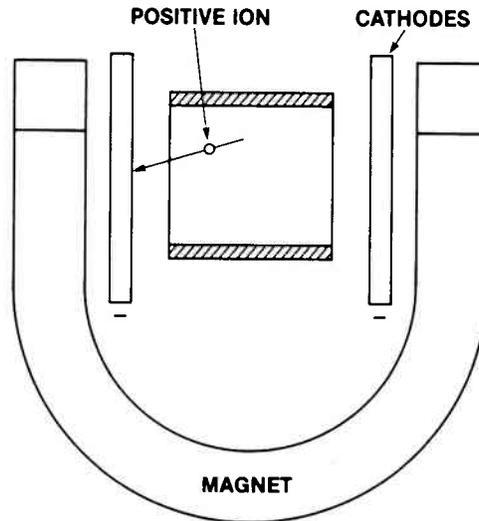


A cold cathode gauge consists of a gauge tube and control unit. The gauge tube has a central anode and two cathodes or a cylindrical cathode. A strong permanent magnet surrounds these elements. Its accuracy is approximately $\pm 50\%$ of the true pressure.

How the Gauge Works



A dc voltage of about 2,000 volts attracts electrons in the gauge tube to the positively charged anode. The magnetic field forces the electrons into long helical paths. This increases the probability of electron-molecular collisions. The collisions produce ions. This, in turn, produces a glow discharge.



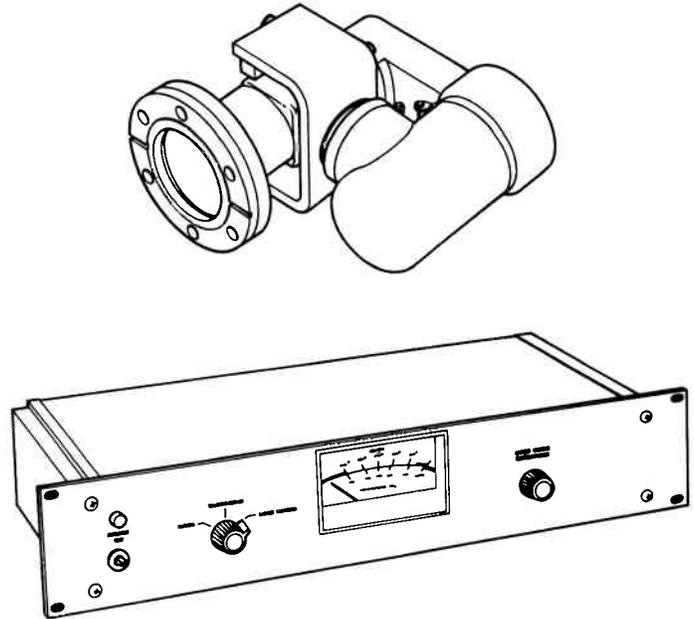
The positive gas ions are now attracted to the negatively charged cathode. The resulting ion current is measured and calibrated in units of pressure. (Note the similarity between this gauge and the ion pump—the cold cathode gauge is an ion pump with non-gettering cathode material.)

The gauge extinguishes when the pressure (gas density) gets too high (about 10^2 torr) to support a gas discharge. It also extinguishes when the pressure gets too low (about 10^{-8} torr) to support a discharge. Most cold cathode gauges are constructed so that they may be taken apart for cleaning. An advantage of this gauge is that there is no filament to burn out. This makes it a more rugged gauge that takes more abuse but is less accurate.

Maintenance

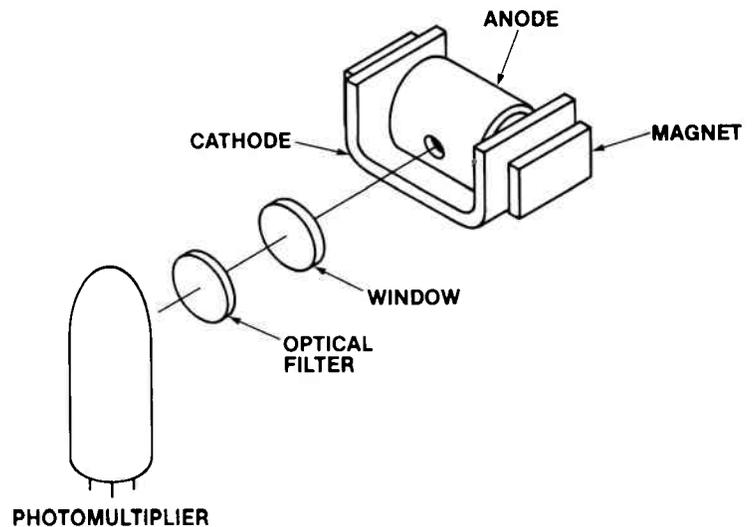
The cold cathode gauge usually does not need calibration or adjustment. It depends completely on the ion current being proportional to the pressure. It is quite rugged because of its all-metal construction. An adaptation of the cold cathode gauge is the residual nitrogen analyzer, which we'll look at next.

Residual Nitrogen Analyzer



The residual nitrogen analyzer (RNA) reads the total pressure and the nitrogen partial pressure. It operates in the high vacuum range.

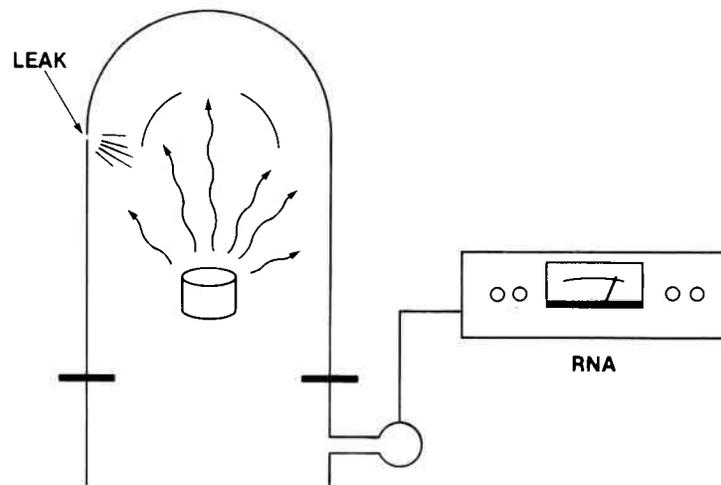
How the RNA Works



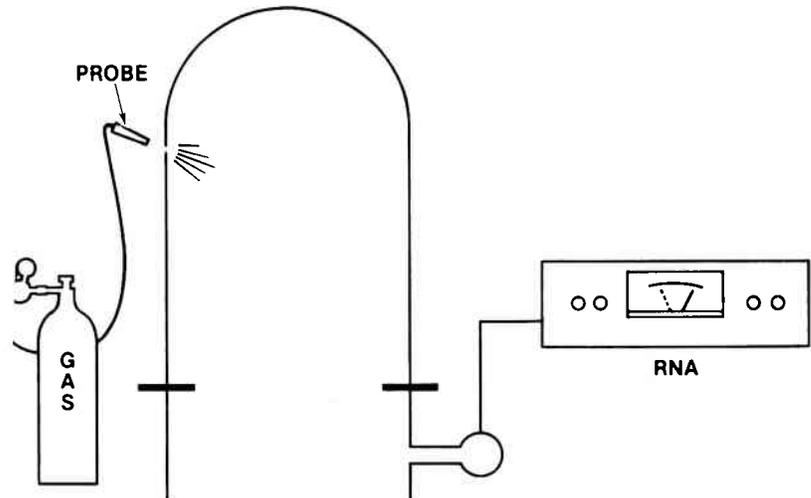
The RNA is a cold cathode gauge that also includes a window, an optical filter, and photomultiplier tube. Ionization produces light. This light has various colors, depending on the gases producing it. The filter passes nitrogen light and blocks the light from other ionized gases. Thus, the gauge reads only the nitrogen pressure. In the "normal" mode, this cold cathode gauge also reads the total pressure. We can use this to help us in determining the condition of our vacuum system.

Gases are pumped at different rates. Nitrogen is relatively easy to pump, so its percentage in a pumped-out system is lower than it is at atmosphere. A leak, on the other hand, admits an abundance of nitrogen. The RNA, which is tuned to nitrogen, senses this abnormality, and identifies an air leak.

For example, the percentage of nitrogen remaining in a normal evacuated leak-free system at 5×10^{-6} torr may be about 10%. An air leak can change this percentage to about 80%. A low nitrogen reading upsets the normal balance in the other direction, and is a good indication of outgassing or an internal leak in the system. The gauge is a simple way to diagnose a system problem. It answers a very important diagnostic question— does the system have a real leak or a virtual leak?



For example, coating material builds up with time, outgasses and raises the pressure. The RNA can tell the difference between outgassing, which may be still within acceptable limits, and an air leak, which is not.



The RNA also can be used to pinpoint the location of a leak. When a gas probe using any gas except nitrogen passes over the leak, the percentage of nitrogen is again changed. This change can be used to establish the location of the leak.

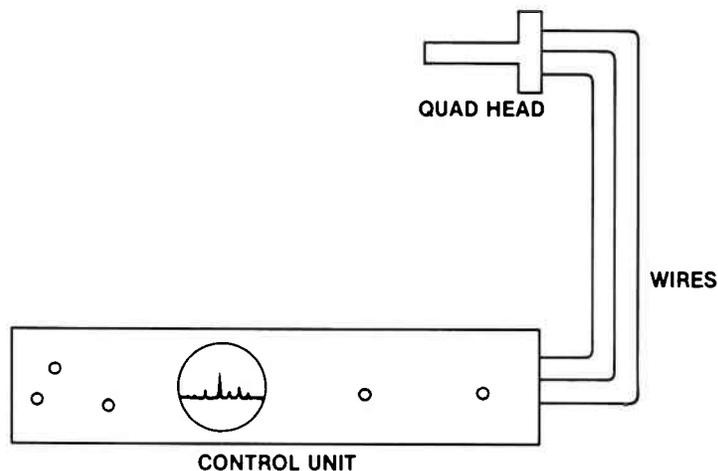
Maintenance

The RNA, being essentially a cold cathode gauge, requires the same maintenance. In addition, the window, filter and photo tube assembly may need attention periodically.

Residual Gas Analyzer

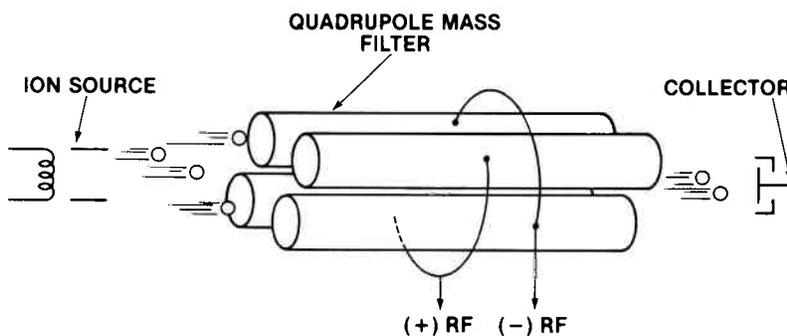
partial pressure

Another gauge that measures *partial pressure* is the RGA, or the residual gas analyzer. This instrument measures the partial pressure of each gas present in the vacuum system as well as the total pressure. It operates in the high and ultrahigh vacuum ranges. It is sometimes used to sample gases at higher pressure (above 10^{-4} torr), but the gauge head must operate at high vacuum.



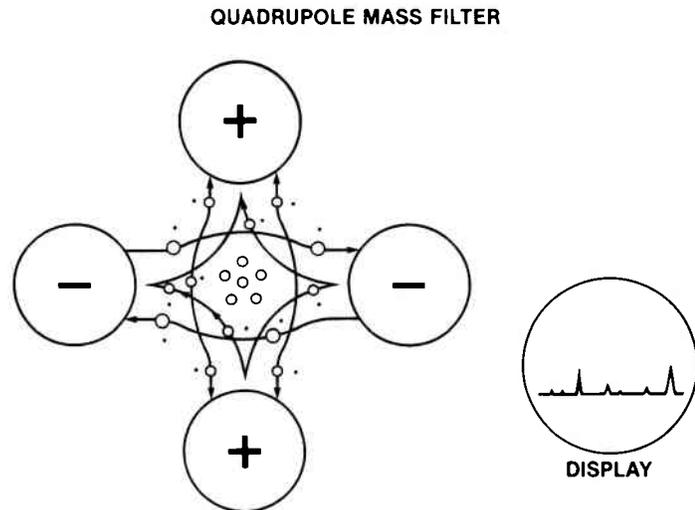
One type of residual gas analyzer includes a quadrupole sensing head and control unit. The control unit has a computer-controlled screen that displays the ion current signals of the gases remaining in the chamber. Some of the older units use an oscilloscope to display the signals.

How the RGA Works

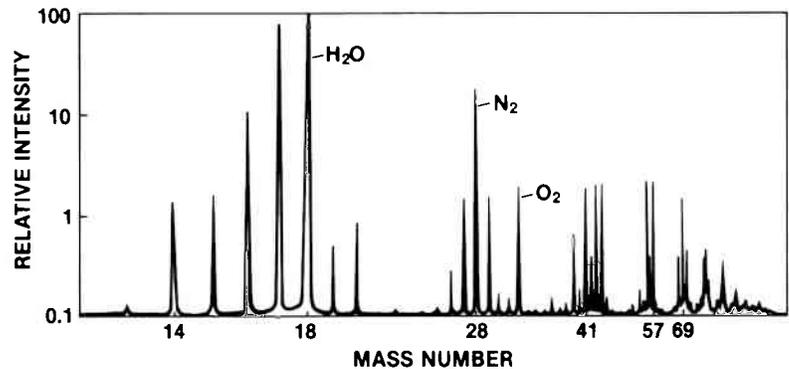


The residual gas analyzer separates, identifies and measures the partial pressures of residual, or remaining, gases in an evacuated chamber. The gases produce peaks in the display. The position of the peaks identifies the gases producing them. The partial pressures of the gases are measured by the heights of the peaks. Total pressure can also be measured.

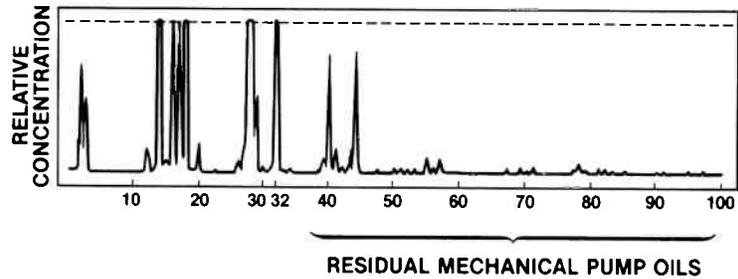
A quadrupole RGA has a sensing head that includes an ion source, quadrupole mass filter, and a Faraday cup collector. Ionized gas molecules are injected into the mass filter. The ions that have the correct mass-to-charge ratio pass through the filter to the Faraday cup collector. These ions produce signals that are in proportion to the number of ions that get through the filter.



The quadrupole mass filter is an array of two pairs of metal rods having equal and opposite rf and dc voltages. For a given set of voltages, only ions of a given atomic weight, or mass, pass through the filter. Ions above and below this mass are grounded on the rods. Then, by progressively changing the voltages on the rods, other ions are allowed through the filter in order. Thus, the various gases are separated and identified. Because this is an electrical process, it can occur quite fast, so that the instrument can display a wide range of masses. Typically, this display capability is 1-100, 200, or 300 mass units.



In this illustration, we can see that the vacuum system has an air leak by the large amount of nitrogen, oxygen and water vapor shown in the display. The instrument can then be used to find the leak by switching to detect helium only, and the system is probed with helium. When the probe is placed near the leak, helium enters the system and is quickly displayed. This pinpoints the location of the leak. Other gases may be used to probe for leaks if the RGA is properly tuned to that gas.



The residual gas analyzer is also useful for determining other types of contamination, such as water leaks or excessive out-gassing from dirty components. (Vapors from backstreaming pump oils can also be identified with this instrument.)

Maintenance

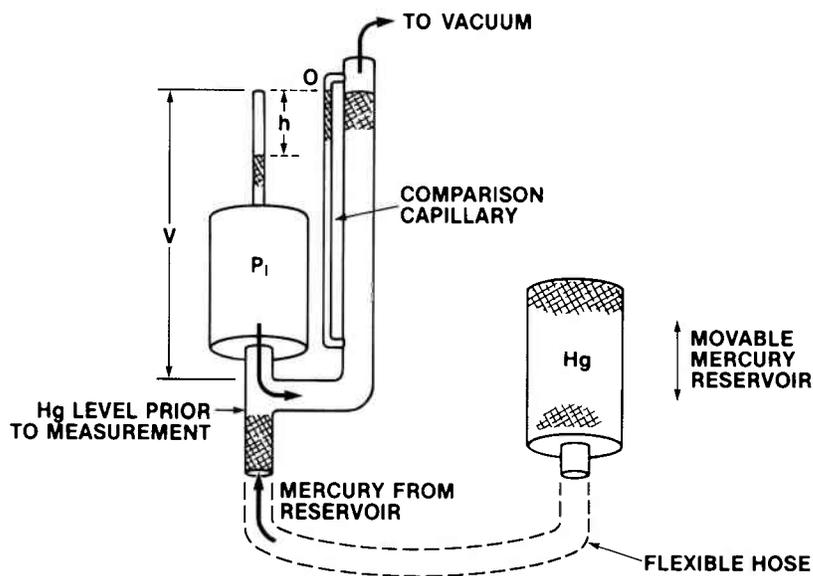
The RGA is a fairly complex and expensive instrument. Please consult your operation manual to determine the required maintenance.

Gauges Used for Calibration

In order to know what the pressure is in your vacuum system, you must measure it. But how do you know that your gauge is correct? Most of us take the manufacturer's word until we are forced to consider that it might be the gauge that is the problem.

To check the accuracy of a gauge, we need a known pressure. We must use an independent gauge to measure the pressure. This independent gauge is usually called a calibration gauge. A calibration gauge may range from one that you keep clean and use as your comparison gauge to a gauge that can be traced to the National Institute of Standards and Technology. Let's look at a few gauges that are used for calibration purposes. We will talk about the McLeod gauge and the spinning rotor gauge.

McLeod Gauge



A McLeod gauge depends upon Boyle's Law for perfect gases. We use a large glass bulb and a small capillary tube with mercury to compress the gas at low pressure into a small volume at a higher pressure. A scale is attached so that we do not have to calculate the pressure from Boyle's Law each time. Because the gauge is made of glass and must use liquid mercury, it is rather fragile. If you had one on your production floor, it's broken by now! They are generally kept on a shelf in the "calibration lab" and used only there. The gauge suffers from several problems:

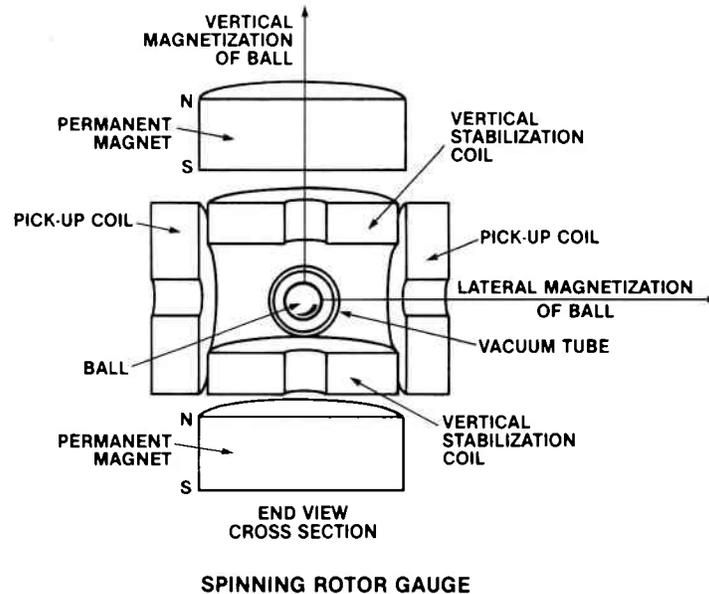
- It is fragile and is filled with mercury.
- It takes several minutes to get one reading.
- If the vacuum system contains a condensable gas (water vapor, for instance), the gas will condense during compression. The gauge reads incorrectly as a result. Therefore, drying filters must be used, and kept clean in order to get "correct" pressure readings for noncondensable gases.
- The mercury vapor is potentially toxic.
- It cannot measure rapidly changing pressures.

For many years, it has been the best "standard" gauge available, even with its problems.

To use the gauge, you connect it to your system and pump the gauge down. Be careful not to place the mercury bulb so that mercury can go into the vacuum system! When you think the gauge is pumped down (a problem), raise the mercury bulb slowly

until it rises to the mark in the sidearm. Read the pressure on the scale. Lower the mercury to expose the gauge to the system pressure and try again.

Spinning Rotor Gauge



A newer gauge is now in the process of being accepted by the National Institute of Standards and Technology as a transfer standard gauge which they may certify. This is possible because the principle on which the gauge works can be related through calculation to basic laws of physics. Its name says exactly what it is—a spinning rotor.

You may recall from some experience that one of the problems with cars is the friction due to air. It's this property of air that is used in the gauge. A ball is magnetically suspended in a small chamber to eliminate all sources of friction except air friction. It is made to spin or rotate while suspended. If there are gases present in the chamber, the ball will slow down due to the impacts from molecules in the chamber. The rate at which it slows down is directly proportional to the gas pressure (number of impacts). All we need do then is to very accurately measure the rate at which the ball slows down, and calculate the pressure as a result. This calculation is, of course, done in the gauge control unit.

The manufacturer of this gauge states an accuracy of "1% of the reading plus or minus 3×10^{-8} torr from 10^{-2} to 5×10^{-7} torr." While you will not be using this gauge as a routine pressure gauge, your gauges may be calibrated using this gauge.

Other Calibration Gauges

The capacitance manometer may be used as a calibration gauge, generally in the rough vacuum range. It, in turn, could be calibrated using the spinning rotor gauge. You may keep one or more gauges in your tool box, just to replace a gauge on the system to see if they are about the same reading.

Summary

We have discussed various types of gauges used to measure pressure from the rough to ultrahigh vacuum ranges. You have learned what these gauges do, how they work, and how they are maintained.

Now that we've covered two major components of vacuum systems, pumps and gauges, let's move on to look at the hardware components and materials used in vacuum systems.

6

Vacuum Materials and Hardware

When you have completed this chapter, you will be able to:

1. Describe the basic materials used in vacuum work.
2. Understand the common joining techniques used in the construction of vacuum systems.
3. Describe the hardware components usually found in typical vacuum systems.

Introduction

Modern industrial vacuum equipment enables the manufacture of many different products. It enables processes such as vacuum skin packaging and vacuum drying. It also makes it possible to do many kinds of chemical processes, analytical work, and thin-film coating. These are only a few examples of the work done in vacuum systems. Of course, we must have the pumping mechanisms to produce high-purity vacuum environments. Let's now look at another key factor in vacuum equipment: the hardware components and materials used in vacuum systems.

Vacuum Purity Levels

It will help to have another look at what we are doing when we want to use a vacuum system. Here's a summary of some information about vacuum for you.

Pressure		Gas Density	Mean Free Path	
Torr	Pa	Molecules/cc	Meters	English
760	10^5	3×10^{19}	6×10^{-8}	2.5×10^{-6} in.
0.76	10^2	3×10^{16}	6×10^{-5}	2.5×10^{-3} in.
7.6×10^{-3}	1	3×10^{14}	6×10^{-3}	2.5×10^{-1} in.
7.6×10^{-6}	10^{-3}	3×10^{11}	6×10^0	21 ft
7.6×10^{-8}	10^{-5}	3×10^9	6×10^2	2100 ft
7.6×10^{-10}	10^{-7}	3×10^7	6×10^4	40 mi

Let's add two more numbers to compare to this table: About 10^{15} molecules can cover one square centimeter (cm^2) of surface area only one layer (a *monolayer*) thick, when the molecules are at room temperature. At a pressure of 10^{-6} torr, it takes about 1 second to deposit one monolayer of molecules on the surface.

$$10^{15} \text{ molecules/cm}^2 \quad 1 \text{ sec at } 10^{-6} \text{ tor}$$

Now, we can look at these facts and make some use of them. Let's suppose we want to put down a few layers of our own choosing on a surface. What pressure would we need to use in order to put down what we wanted— not just whatever was flying around in the chamber? Every second there will be another layer coating our surface at 10^{-6} torr. We better work fast. If we take a long time to lay down our layers, we will need to work at very low pressure.

For example, MBE (molecular beam epitaxy) processes are carried out at ultrahigh vacuum levels (10^{-9} torr) in order to allow us to put on a pure coating. At 10^{-9} torr, it takes about 10^3 sec to lay down a monolayer! Now, we can work more slowly and still make sure that the layers that we want to put on our surface are indeed placed there.

We can look at these facts another way: Let's compare the purity of our vacuum to the purity of something that you perhaps know a little about. Let's use ultrapure helium, sometimes called "five nines" helium (meaning 99.999% pure), as our example.

Most people think of this as being very pure. To get an equivalent level of purity in our vacuum chamber would require that we remove all but ten parts of every one million parts that were originally present. Looking at our table, we see that one millionth of atmospheric pressure is about $760/10^6$, or about 7.6×10^{-4} torr – barely into the high vacuum range! It's about 1 mtorr!

Even at 1 mtorr, our vacuum chamber has too many molecules (too much "dirt") to carry out some of the processes that we perform in a chamber. In fact, at a typical high vacuum working level (10^{-6} torr), we are in the parts-per-billion purity level. Ultra-high vacuum gets us into the equivalent of parts-per-trillion purity level (one part per 10^{12}).

You can see that we can indeed get a very clean working environment by using a vacuum system. This may also help you to see some of the problems that go along with using a vacuum system. Let's go on now to look more in detail at some of those problems.

How Gases Are Pumped

One obvious problem is to get rid of all those gas molecules in our vacuum chamber. Let's look at what happens as we pump down.

Our roughing pump removes more than 99.99% of the air or gaseous contaminants from the chamber. We know that this is adequate to perform some vacuum processes but not those requiring high purity.

As we begin pumping at atmospheric pressure, we are pumping out the air. This air is sometimes called the volume gas. As we continue to pump, we begin pumping the molecules which have been fastened loosely to the walls. After these are gone, the remaining gas load is mostly water vapor. In fact, 75% to 95% of it is water vapor.

When the chamber is vented, the water vapor clings to all surfaces and to its own layers. Water can build up layers about 100 molecules thick on surfaces! After the volume gas is removed, the water begins to come off the surfaces, and makes up most of the remaining gas load.

As we continue to pump down to lower pressures, the character of the gas load changes. Other gases begin to make up the major portion of the gas load, as you can see in this table:

RESIDUAL GAS LOADS	
Pressure (Torr)	Major Gas Load
Atm	Wet air
10^{-3}	Water vapor (75%-95%)
10^{-6}	H ₂ O, CO
10^{-9}	CO, N ₂ , H ₂
10^{-10}	CO, H ₂
10^{-11}	H ₂

As we continue to pump, the mean free path of the gas molecules becomes longer and longer. We have gone from the viscous flow range into the molecular flow range. Remember that in molecular flow, pumping occurs only when the molecules randomly move into the pumps of their own accord. We now need high vacuum pumps.

base pressure

The vacuum system can be pumped to lower and lower pressure as long as the throughput of our pump is greater than the gas load. When the throughput equals the gas load, the pressure stabilizes. We have reached the *base pressure* for our system. Reaching base pressure means that in the high vacuum range, the following happens: the system pressure goes down only as long as the gases make it into the pumps faster than they come off the walls and other surfaces; then, when the gases come off the surfaces as fast as they make it into the pumps, the pressure for that particular system will go no lower.

outgassing

virtual leak

real leak

When you find that you cannot “pump down” to the typical pressure for your system, you may be seeing the effect of an increased gas load. We call this *outgassing*, or a *virtual leak*, or perhaps just a leak. Why the increase in the gas load? A dirty system—more molecules on the walls to be pumped off. (It may also be because of a *real leak*—a crack or hole in the system.)

We have briefly reviewed the purity levels that we achieve in a vacuum system and the way that gases are pumped. Let's now go on to consider how these are affected by the materials that we use in a vacuum system. So, in this next part of our discussion, we will touch on a few important properties of the materials that go into a vacuum system.

Materials

*thermal expansion
coefficient*

Materials used in vacuum work often need to withstand wide changes in temperature. This is because the equipment is often baked to drive gases off the chamber surfaces and into the pumps. In other cases, chilling to very low temperatures helps to produce a good vacuum. Some equipment is exposed to both high and low temperatures.

Materials change in size when their temperatures change. This size-to-temperature relationship is called the *thermal expansion coefficient*. These changes in size are different for different materials.

Materials with different expansion rates are often joined to each other in vacuum equipment. During bakeout, the materials change in size at different rates. This causes strains at the points where they are joined.

The strain distorts the joints and can result in—you guessed it—leaks! Unless, of course, something is done ahead of time to prevent them. We'll continue this part of our discussion later.

Another major problem in vacuum performance is the outgassing rate of the materials in the system. A rough surface has a larger surface area for gases to stick to than a smooth surface does.

The problem is that this kind of sticking is only temporary. The partially trapped gas slowly comes off the walls after roughing is complete, making it difficult to achieve good vacuum performance. Also, some materials just naturally outgas more than others.

So, we want the materials used in our vacuum system to have several characteristics:

- Wide temperature tolerance
- Similar thermal expansion rates
- Low outgassing rate

There are other characteristics that may be of importance to us. We may need an electrical conductor or insulator, high strength, a thermal conductor or insulator, a non-magnetic quality, elasticity, low volatility, low chemical reactivity, radiation resistance, and probably others as well. With these characteristics in mind, let's look at some of the materials used in vacuum systems.

Stainless Steel

Stainless steel (or SST), 304 SST in particular, is widely used. It is a high-strength material that stands up to the wide temperature changes experienced in vacuum work. We need the high strength to withstand the air pressure trying to collapse our vacuum container.

Also, SST doesn't oxidize easily, so its surface remains smooth. This means that it doesn't produce large surfaces for gases to stick to. Therefore, it doesn't outgas much. It may be joined by welding or brazing.

A large work chamber made of "easily" machined 304 SST will usually have many ports welded to it. It may have pieces attached by brazing as well. Some stainless steels are non-magnetic, which is important in some applications.

Copper

Our chamber may have OFHC™ copper used as gasket seals or as plumbing to carry materials in and out of the chamber. (OFHC means oxygen-free, high conductivity.) Because of its careful refining process, OFHC copper contains very little oxygen. Therefore, it doesn't outgas much.

OFHC copper can also take wide changes in temperature. This is important because copper is often baked at very high temperatures, then chilled to very low temperatures. It is a very good electrical and heat conductor.

Brazing and welding are common methods of joining copper to copper and copper to other materials. OFHC copper is an ideal gasket material because it is relatively soft. Also, it contains very few microscopic leak paths (micropipes), which would prevent production of high and ultrahigh vacuum levels.

The ability of copper to conduct heat makes it an excellent choice for cryogenic applications. Copper is used in liquid nitrogen traps and cryogenic pumps. It is not as inert (non-reactive) a material as we might like. As a result, we usually nickel-plate copper to improve its chemical resistance. We use copper to handle large heat loads, as in cooling a sputtering gun or evaporative source.

Of course, copper is widely used because of its electrical conductivity. We use it to get any amount of electrical energy into our vacuum system.

Ceramics

We use ceramic materials (alumina, in particular) to contain electricity. Ceramics have excellent insulating properties, both for electricity and heat. We routinely braze the ceramics to the other materials in the system.

Ceramics are fragile but have great compressive strength. Their thermal expansion rate (also called coefficient of expansion) is very low.

Kovar

We use an intermediate material, such as Kovar™, to join glass to metal and ceramic to metal. Kovar has a coefficient of expansion which is between that of ceramics and stainless steel. By brazing the Kovar to the ceramic, then the Kovar to the metal, we get a vacuum-tight seal that remains leak-free even when exposed to extremes in temperature. Kovar is the trade name for an alloy composed of 54% iron, 29% nickel and 17% cobalt. It is magnetic.

Elastomers

Elastomers are materials that are flexible but not compressible. As such, they are very good to use for gasket seals. Because they are soft, they fill the gaps between mating surfaces and make leak-free joints. Their elasticity, or the ability to spring back to their original shapes, makes them reusable in most cases.

The permeability of elastomers can be a problem in some systems, particularly UHV systems. In these systems, the additional gas load caused by permeation causes problems. Elastomers in

general are quite permeable to helium. Leak checking with helium can give slow indications of small real leaks when the source of helium is actually helium that is slowly diffusing through the seal into the system.

Buna-N™ is a common elastomer that is used because of its resistance to helium permeation and because it is inexpensive. It works very well in seals that are not going to be heated above 80°C. It is essentially a synthetic rubber material.

Viton™ is an excellent elastomer which is widely used for O-rings, valve seals, bonnet gaskets and chamber L-gaskets. It outgasses very little, so it can be used for both high and ultrahigh vacuum work. Viton will withstand temperatures up to about 150°C.

Polyimide™ is substituted for Viton where higher temperature tolerance is required. Polyimide will remain elastic up to about 200°C. It is a stiffer material than most elastomers. Therefore, it requires higher sealing pressure to assure leak-free operation. It is also more resistant to radiation than most elastomers. But it has the disadvantage of absorbing water.

Silicone compounds also withstand high temperatures, have poor outgassing rates and are quite permeable to helium and water. They are used in vacuum furnace work because of their temperature tolerance.

Teflon™ is also a good elastomer. However, it is also a plastic-like material, meaning that when it is deformed it tends to remain deformed. We call this "cold flow." When we try to use Teflon as an O-ring, it flows slowly out of the seal, even at room temperature. This, of course, results in a leak at the seal. Teflon is widely used to seal pipe thread joints and ferrules in flexible couplings. The threads help hold the Teflon in place and therefore stay leak-free. Teflon is quite permeable to helium. It can withstand temperatures to about 150°C.

Viton, Polyimide and Teflon are all fluoropolymers. They should not be overheated or burned due to the possibility of toxic gas production.

We have seen quite a collection of materials here. These are, of course, not all the materials that are used in vacuum systems. Special processes may require materials that must meet quite different or special requirements.

Our requirements for vacuum systems in general expose some materials to very low temperatures, others to very high temperatures, and some materials to both. We must allow for the changes in the size of materials when their temperature changes.

We must also be concerned about the surface texture and how it will affect the outgassing rate into the system. Remember that the gases stored or adsorbed on the walls become our major problem under molecular flow conditions.

We may be quite concerned about the vapor pressure of solids under the conditions present in our vacuum system. Recall the graph of vapor pressure of the elements from chapter 1; the graph suggests that there are elements such as lead, zinc and cadmium which may have a vapor pressure which is too high for vacuum system use. This means that brass parts and cadmium-plated screws are not to be used in a high vacuum system. Their vapor pressure will be high enough to prevent us from reaching operating pressure, particularly if we will be using high temperatures in the process.

The same consideration should be used in determining what organic materials should be (or not be) used inside the vacuum system. Materials with high vapor pressure will cause higher gas loads. This typically means that the system will be unable to reach the desired pressure.

Joining Techniques

We have already mentioned several joining techniques in talking about materials. Welding and brazing are very commonly used in construction of vacuum systems.

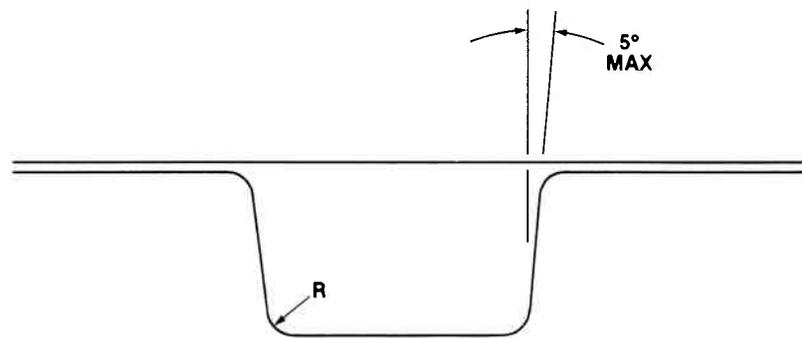
When we need to be able to take part of our system apart and put it back together leak-tight, we use flanges or couplings to join the system components. Let's take a look at the various types of flanges that can be used.

Flanges

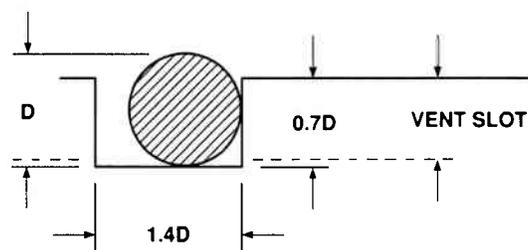
Flanges enable us to connect (join) the system parts in a reasonable and convenient manner. They also make it possible to quickly connect feedthroughs for purposes of controlling and monitoring system operation, and to maintain the system when trouble occurs.

Elastomer-Sealed Flanges

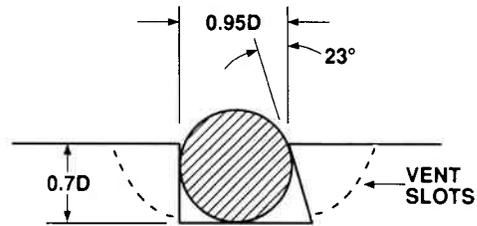
Elastomer-sealed flanges are used where there is little objection to the use of an elastomer, mostly based on temperature considerations and, perhaps, outgassing.



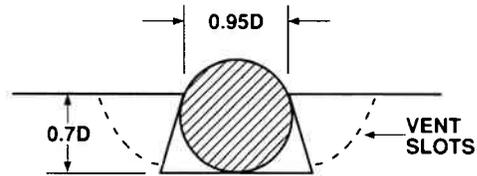
The groove in the flange for the O-ring should have sides that slope outward to a maximum of 5°. The groove should also have a radius on the inner corners equal to about two-tenths of the O-ring diameter. The surface finish of the seal area should be at least 32 microinches. The outer edges should be smooth, to avoid scratching the O-ring when making the seal.



The depth of the groove provides for deformation of the O-ring to about 70% of its unsqueezed diameter. This gives enough elastomer material to make the seal without overstressing the O-ring, but not so much as to force it out of the groove where it might be pinched or cause excessive outgassing. A vent slot is usually machined across the face of the groove to eliminate trapped volumes and for leak detection.



A. DOVETAILED O-RING GROOVE

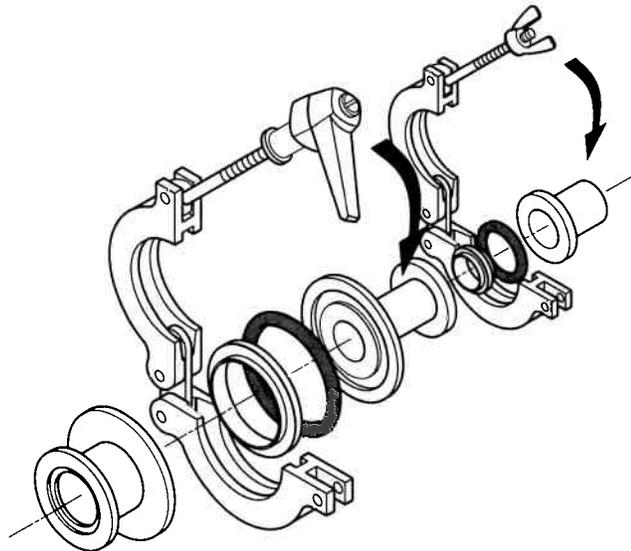


B. DOUBLE DOVETAILED O-RING GROOVE

DOVETAILING OF O-RING GROOVES

The O-ring groove can be dovetailed to help retain the O-ring. You may also see fully dovetailed O-ring grooves in cases where a gas flow process might otherwise blow the O-ring out of the groove. Dovetailing, or keystoneing, is also useful for retaining the O-ring against the force of gravity. The O-ring groove must be relieved to prevent pockets of partially trapped gas from becoming sources of virtual leaks.

Another popular type of elastomer flange is the KF™ flange. As marketed by Varian, it is known as the KLAMP-FLANGE™.



KF FLANGE ASSEMBLIES

The flange is of standard ISO 2861/1 design, consisting of two symmetrical flanges, a center ring to support and position an

O-ring, and a clamp that allows assembly without any tools. KF flanges are quite convenient to use in rough and high vacuum systems.

Here are some suggestions you will find useful when working with O-rings:

1. When preparing to make a flange connection, be sure to clean and dry the groove and the flat mating surfaces. Check the sealing surfaces for scratches that cross the seal area.
2. Lightly lubricate the O-ring with a vacuum grease such as Apiezon-L™. Then, wipe off most of the grease with lint-free paper before making the connection. Keep in mind that the O-ring makes the seal, not the grease. The grease makes the O-ring slip and helps it to conform to its groove.
3. Don't apply a lot of helium to an O-ring when leak checking. You will get a small, slowly increasing signal as the helium permeates the O-ring and goes into the vacuum chamber.
4. If you reuse an O-ring, visually inspect it to make sure it has no small cross-wise cracks or nicks that might leak. If it has swollen because it has been exposed to solvents or excess heat, do not reuse it. It is best practice to replace used O-rings with new ones.
5. Leak checking O-ring sealed flanges with solvents affects the O-rings (acetone is an example). The solvent slowly works into the O-ring and on into the system, causing an outgassing problem. The O-ring may even tend to dissolve in the solvent and become gummy and sticky.
6. O-rings will absorb water and will cause outgassing of water vapor. Baking the O-rings will minimize this problem.

Let's go on now to discuss another variety of flanges, the metal-sealed variety.

Metal-Sealed Flanges

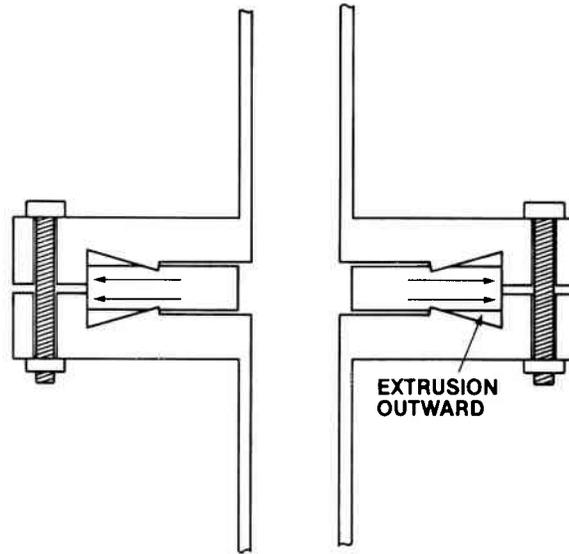
Metal-sealed flanges accomplish the same task that elastomer-sealed flanges do. They do have certain advantages over elastomer seals, however. They may be used at much higher temperatures—up to 500°C. They have low outgassing rates. They are more expensive to install than elastomer-sealed flanges.

The Varian ConFlat™ flange has become the vacuum industry's standard flange. Let's take a more-detailed look at this flange and the features that have made it a reliable and dependable flange.

The process starts with the selection of the 304 stainless steel. Either cross-forged or electroslag remelted (ESR) steel is used.

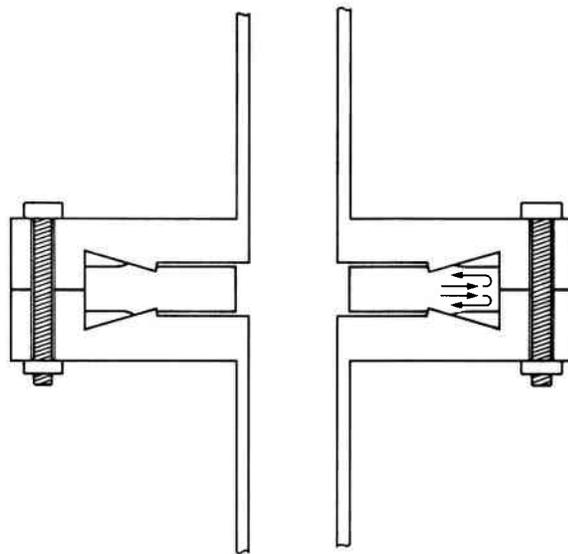
Care is used in selecting the material to eliminate leak paths caused by inclusions or micropipes in the material.

The design has built-in, long-term reliability because it captures the gasket. This prevents the gasket from flowing away from the seal area, even under the most extreme temperature changes. Let's look at the design of the flange, particularly the knife edge.



PARTIAL SEAL

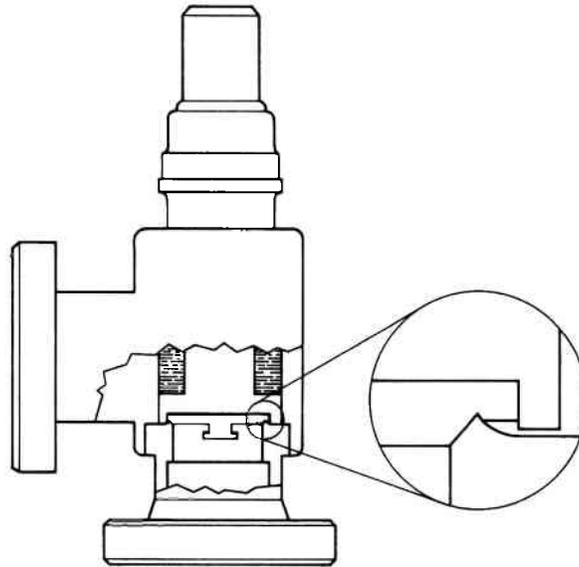
As the flange knife edge begins to bite into the copper gasket, the gasket material flows outward until it butts up against the flange supporting surfaces. At that point, additional outward movement is no longer possible. This keeps the material from flowing away from the sealing surfaces.



COMPLETED SEAL

As the flanges are bolted face-to-face, the gasket material is actually forced inward. This situation develops a tremendous pressure at the sealing edges. In fact, this gasket-capturing geometry develops close to 200,000 lb/in.² where the knife edges and gasket come together.

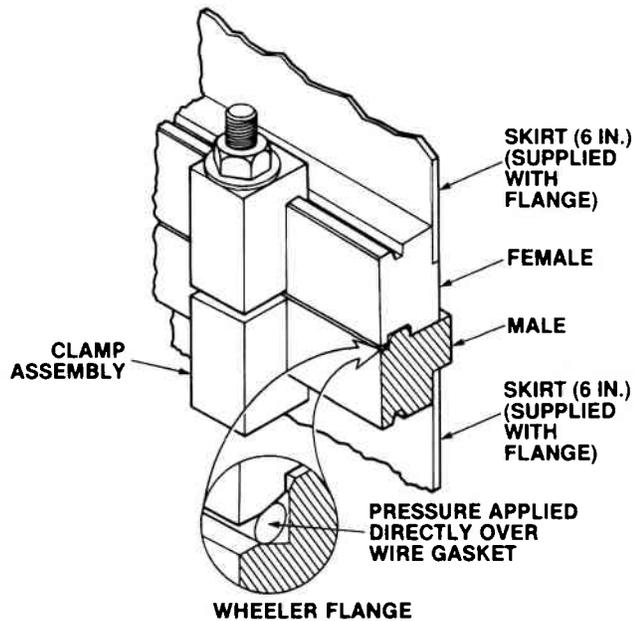
The ConFlat flange seal-capturing geometry is so reliable that it is used in many metal-sealed devices, such as valves and compression ports.



ALL-METAL VALVE

The figure shows an all-metal valve, made entirely of stainless steel and copper. The sealing surface inside the valve has a knife edge which cuts into the copper button to seal the valve closed. A valve of this sort can be baked to 450°C if necessary.

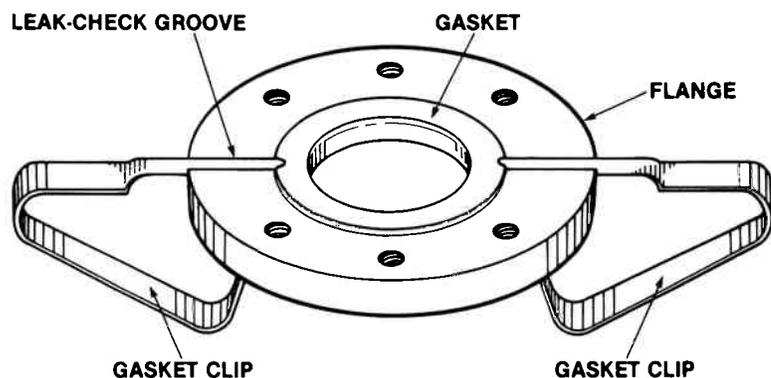
Another variation of the all-metal flange is the Wheeler™ flange, which is shown at the top of the next page.



This flange is used for larger diameter seals. It seals by forcing a copper wire into a compressed area under pressure, much like the ConFlat flange. The force is applied with large C-clamps. Wheeler flanges may be used reliably up to 450°C.

Here are a few suggestions to think about when using all-metal flanges:

1. The knife edges of fixed flanges are protected because they are set back behind the flange faces. The knife edges are exposed on the rotatable flanges and can be damaged if they are not handled properly. Protect the knife edges by covering them with plastic caps. Never place them on a bench or surface where they may be scratched.
2. The grooves milled across the flange mating faces are helpful. They can serve two functions: (1) for a quick leak check of the flange joint, just spray a little helium into the slot when leak checking; and (2) they will also allow the use of gasket clips to hold the gasket in place while you bolt the flanges together.



3. The bolts used on the ConFlat flanges have a twelve-point head on them. This is not done to frustrate you. It indicates that a high-strength steel has been used for that bolt. A good hardware supplier has the 1/4 in. or 5/16 in. twelve-point wrenches you need to hold these bolts while you tighten them. Lubricate these bolts to prevent them from seizing up (galling). We recommend a high-temperature lubricant such as Fel-Pro, C-100 or C-5A.
4. Don't reuse the copper gaskets. The time spent replacing a leaky used gasket that you just installed is much more expensive than the price of a new gasket.
5. Examine the knife edge of the flange, especially on rotatable flanges, to see if there are any nicks or scratches across the knife edge. The general rule is that if you can feel the scratch with a fingernail, it will leak. The flange should not be used. Some scratches may be burnished out.
6. Tighten the flanges metal-to-metal to insure a good seal and that any mismatched forces are carried through the flange faces, not the gasket.

Let's go on now to look at other joining techniques.

Cold Welding

Cold welding, or pinch-off, is a common method of sealing OFHC copper tubes such as those on ion pumps and vacuum tubes. After the components are baked and pumped out, the connecting tubes are pinched off. This crushes the tube walls together so tightly that a leak-tight seal is made. This keeps the devices under vacuum.

When you open a pinched-off seal, use a tube cutter. Don't use a hacksaw or some other cutting tool that generates particles. The particles will be drawn into the sealed-off device as the seal is opened. Remember that the copper particles are good conductors, and may land so that they will short out the device (of course, at the worst possible time).

Brazing

Brazing makes good vacuum joints. Brazing is a high-temperature soldering technique that is done in a hydrogen-filled furnace. The hydrogen atmosphere prevents oxidation at the joints, needs no flux, and allows for careful temperature control. You may have brazed something using a torch for heat. This is also a common process. But unless it is very carefully done, it results in strains which will develop into leaks. It may also leave flux residue which will outgas excessively.

Components that will be brazed are prepared by assembling them in a jig or holder with the brazing material placed between the parts. The brazing material is in the form of wires or gaskets.

They are then placed in the hydrogen furnace under very closely controlled temperature conditions. This is done so that no local heating and stresses occur where the parts are joined. This helps to keep the joints leak-free.

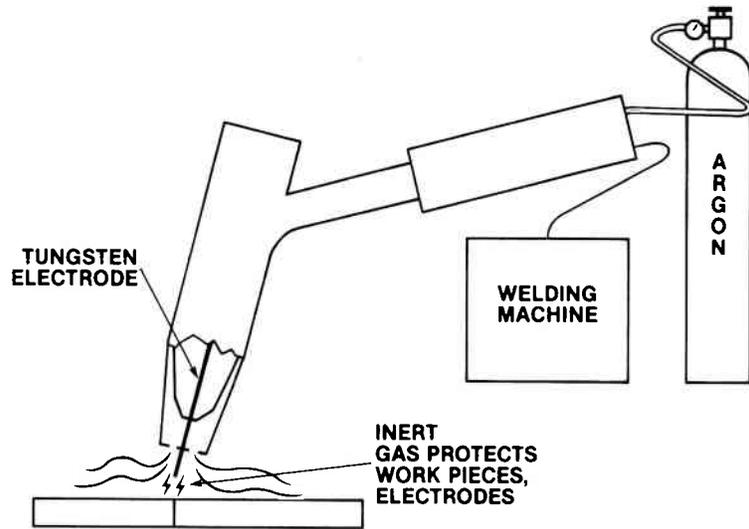
Gold or copper-based alloys are generally used as braze materials. It is possible to do multiple-step brazing by using alloys of varying composition and therefore different melting points.

Brazed joints may be quite strong. However, the material itself may have problems. We commonly use brazing to join ceramics to Kovar™ to stainless steel. The ceramic material is quite sensitive to shocks and tends to crack easily. Treat your brazed joints with care; you will have fewer leaks as a result.

TIG Welding

Tungsten-inert gas (TIG) welding is a widely used joining technique. It is a form of arc welding that joins parts by fusing them together without the use of filler materials or flux.

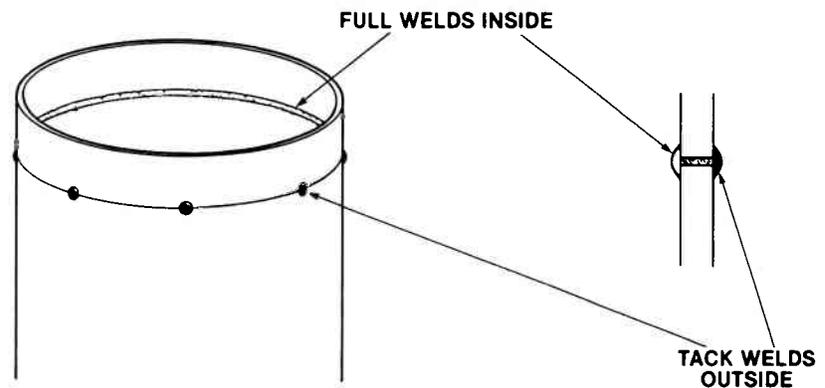
A TIG welding torch concentrates extremely high power at the joints. This makes it easy to weld stainless steel, which melts between 1,450°C to 1,550°C. TIG is also good for joining molybdenum, which melts at 2,620°C.



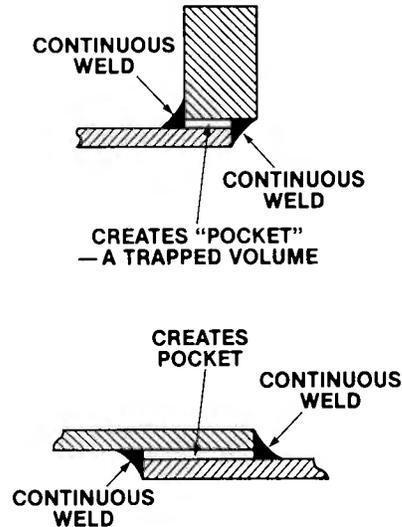
**TIG (TUNGSTEN INERT GAS)
OXIDATION-FREE WELDING**

During the welding process, an inert or non-reactive gas, such as argon, floods the welding torch and the joints. The gas protects both the material and the joints from oxidation. This protection allows the welding of materials, such as aluminum, which tend to oxidize very easily if not protected from the oxygen in the air.

When welding parts together for vacuum usage, the weld should be made from the vacuum side of the joint whenever possible. Through welds, welds that penetrate through the material, are quite desirable, but not always possible.



When the full weld is made from the inside but an outside weld is desired for structural strength, the outside weld should be a tack weld, not a complete weld. This makes leaks easy to find and fix. It also prevents virtual leaks that occur because of a trapped volume between the two weld joints.



We'll talk more about leak detection in situations like this when we discuss vacuum systems.

Glue-Like Materials

There is another joining technique that we should discuss briefly— that is the use of glue-like materials, specifically Torr Seal™. Torr Seal is a low vapor pressure resin which was originally designed to fasten things inside of a vacuum system. It is usable over the temperature range from -45°C to 150°C . The low vapor pressure is due to the fact that it contains no solvent. It does not outgas into your system as it dries. It also has found wide use as a leak sealant.

There may be other joining methods used in the vacuum industry; we don't claim to have covered all of them. However, brazing, welding, and flanges cover most of the techniques. Let's go on now to discuss some of the common components that are used in vacuum systems.

Components

We have already discussed several components such as pumps and gauges. Let's look now at some of the other components that are commonly found in a typical vacuum system. Valves and feedthroughs will be our major focus.

Feedthroughs are used to get something from the outside world into the vacuum system. Typical examples are electrical, optical, water, instrumentation, high voltage, fluid, and motion feedthroughs, just to mention a few.

Although we will concentrate our discussion on these components, they do not cover all possibilities, just the more common varieties.

Valves

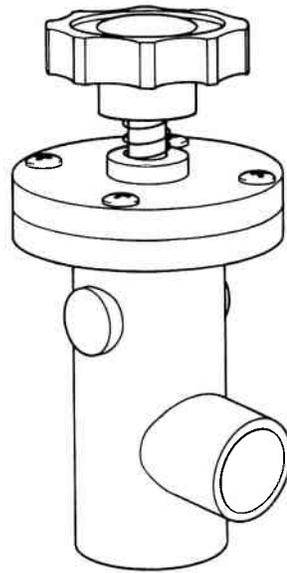
A variety of valves are manufactured for various vacuum requirements. Each of these takes into consideration factors such as operating vacuum levels, degree of cleanliness needed, need for bakeout, and materials construction.

We will separate valves into several types based first on whether they are elastomer-sealed or all-metal; then, whether they are small or large valves.

Valves also come in hand-operated, pneumatically-operated, and solenoid-operated varieties. Small valves will be defined as valves with inside diameters less than 2 inches. Large valves will be defined as valves with more than 2 inches inside diameter. Valves can also be classified as right-angle, tee, straight-through, back-to-air, variable-leak, gate and slide valves.

Let's look first at elastomer-sealed small valves.

Elastomer-Sealed Small Valves



These valves use various elastomers to make a reliable, leak-tight interface between the valve seal and the seat. Elastomers are also sometimes used to seal the actuating shaft from the outside environment.

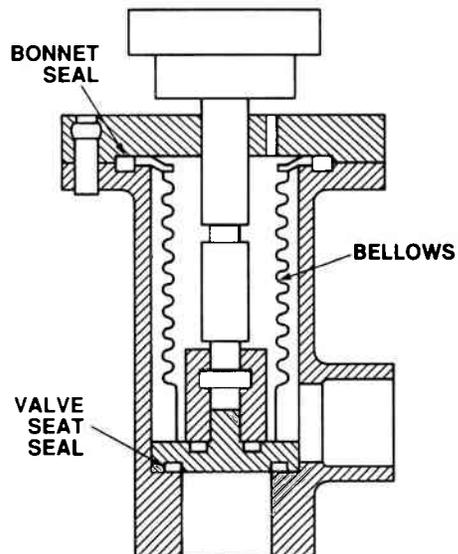
dynamic seal

bonnet

One variety of valve used for some rough vacuum applications is the O-ring-sealed variety. The seal on the shaft of the valve is a *dynamic seal*, that is, it moves. Therefore, it is subject to a lot of wear and leakage during operation. The portion of the valve above the opening is called the *bonnet*. This type of valve requires much maintenance and attention because of the shaft seal.

bellows-sealed valve

This dynamic seal problem can be avoided through the use of a *bellows-sealed valve*.



static seals

The valve shown at the bottom of the previous page is an elastomer-sealed bellows valve. All O-ring seals are *static seals*, meaning that they do not move. These valves are much more reliable than the O-ring shaft-sealed valve.

This type of valve has bellows made of brass, aluminum or stainless steel. The price, of course, varies accordingly. The choice of material depends upon the use of the valve.

The stainless steel bellows is generally used in high and ultrahigh vacuum systems. An example where a stainless steel bellows is not chosen is on valves used with sorption pumps. The hot steam that is produced during regeneration corrodes the stainless steel bellows rather quickly. Inconel bellows are therefore recommended rather than the stainless steel bellows.

Viton is generally the elastomer of choice in valves, although other elastomers are also used. Polyimide finds use in special applications requiring higher temperatures or better chemical resistance.

Valve maintenance is not needed often, but valves can cause problems if not properly cared for. Here are some suggestions to help you with your elastomer-sealed valve maintenance:

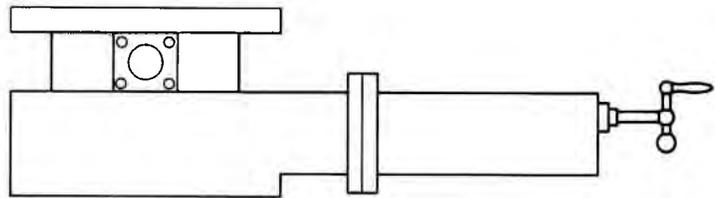
1. Look at the seals whenever you do maintenance on a valve. Inspect the seals to see if they have been warped, or are nicked or scratched.
2. The pistons on pneumatically-actuated valves need to be lubricated yearly. Most pneumatically-actuated valves are of the air-open, spring-closed variety. Be careful when disassembling the valve so that it doesn't come apart during disassembly.
3. Lubricate the piston on your pneumatic valves with a lubricant recommended for use with compressed-air lines. Please remember that this is not exposed to the vacuum system. This lubricant, which works fine for compressed-air pistons, will cause big problems if used inside your vacuum system.
4. Inspect the bellows for dents or cracks. The brass variety is easy to dent. The dent will cause the bellows to work-harden and crack at that location. You should replace it while you have it apart on the bench.
5. Lubricate the O-rings with a vacuum grease such as Apiezon-L™ or other lubricant as required by your process. Remember that only a very thin film is needed.
6. The valve should be leak-checked on a helium mass spectrometer leak detector. Don't forget to check the bellows as well as the valve seal and bonnet seal. Leak checking before reinstallation on the system can save a lot of disassembly/reassembly time should a leak be present.

Elastomer-Sealed Large Valves

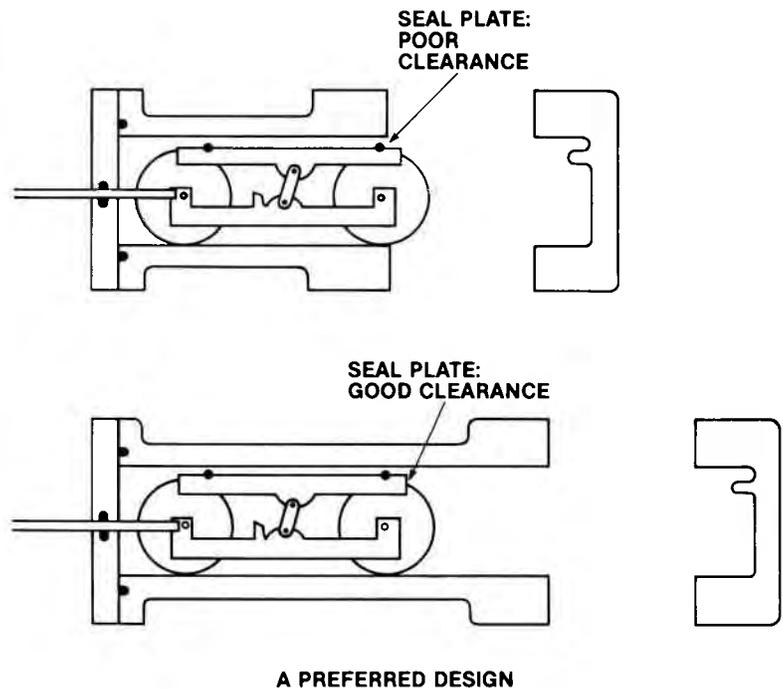
Most industrial applications require isolation of the work chamber from the system pumps. Many processes require that the chamber be alternately cycled from vacuum to atmosphere. Without a valve between the chamber and pump, the cycle time might be too long, or even physical damage to the pump or system components might result.

Valves for these uses are usually of the sliding gate or swing gate design. Typical port diameters of this type are 4, 6, or 8 inches, although much larger valves are available for specialized pumps and applications.

Since the seals in these valves are usually made of Viton O-rings, heat ranges and operating pressures are about the same as those for small elastomer-sealed valves. The valve bodies are usually made of cast aluminum or stainless steel.



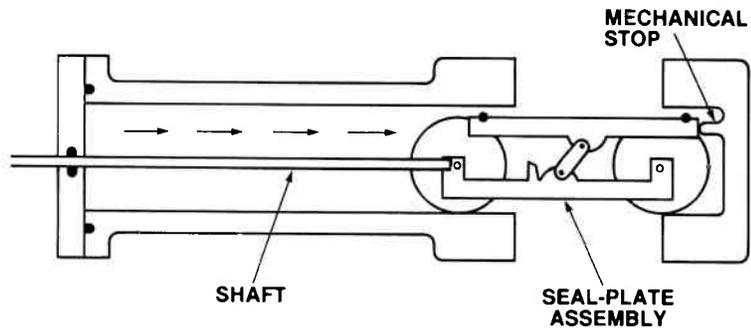
The seal plate opens and closes in a gate fashion. That is, the seal plate drops and retracts from the port. In some designs, the plate does not fully clear the port and, therefore, doesn't give maximum conductance. Also, debris can fall on the seal and cause leaks.



A preferred design completely clears the ports.

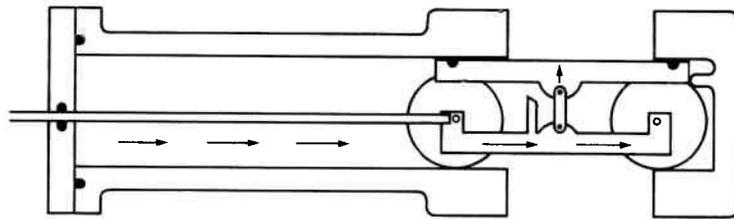
How the Valves Work

The valve closes when the shaft drives the seal-plate assembly into the gate until a mechanical stop prevents further forward motion.

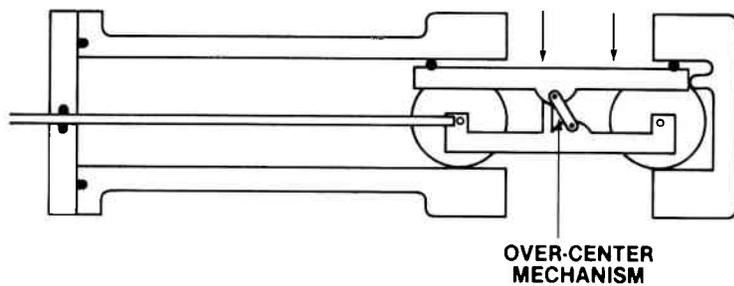


During the forward motion, grease and other debris get into the vacuum environment. And, the longer the stroke, the greater the contamination.

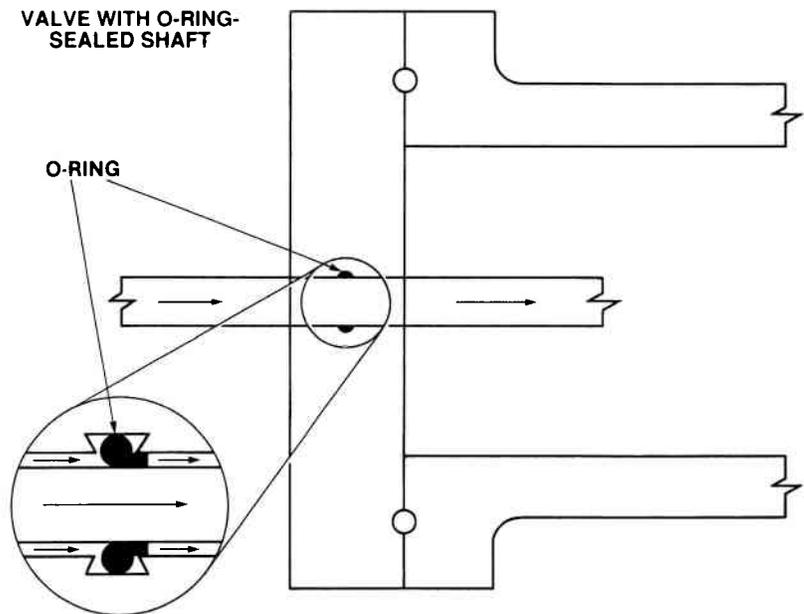
After forward motion stops, further driving motion moves the seal plate up into the sealed position.



The over-center mechanism and second mechanical stop insure that the seal plate is positively locked in the sealed position.

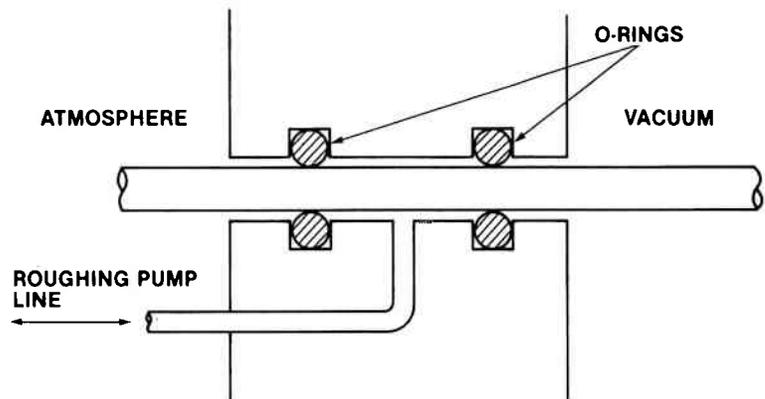


The valves are often air-operated and close with considerable speed and force. It is important to remember to disconnect both air and electricity when working on them.



Seals

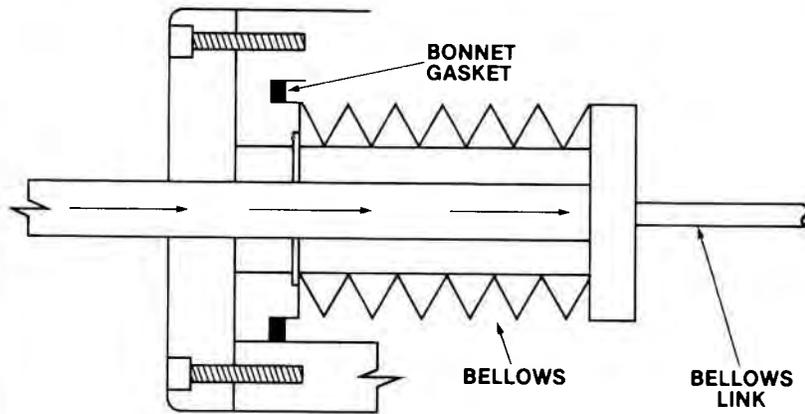
In a valve having an O-ring-sealed shaft, the seal is usually rolled and scuffed. This seriously reduces its life.



VALVE WITH DOUBLE O-RING SEAL

The shaft in the type of valve shown above is often equipped with a double O-ring seal. This double seal provides better separation between the vacuum chamber and atmosphere. This also creates a trapped volume which may result in a virtual leak. The volume between the two O-rings may also be connected to a roughing pump. This is to improve vacuum separation of the work chamber even further. When leak checking a double shaft seal such as this, the line to the rough pump is disconnected. Then both the outer and inner seal can be checked by inserting helium into the space between the O-rings.

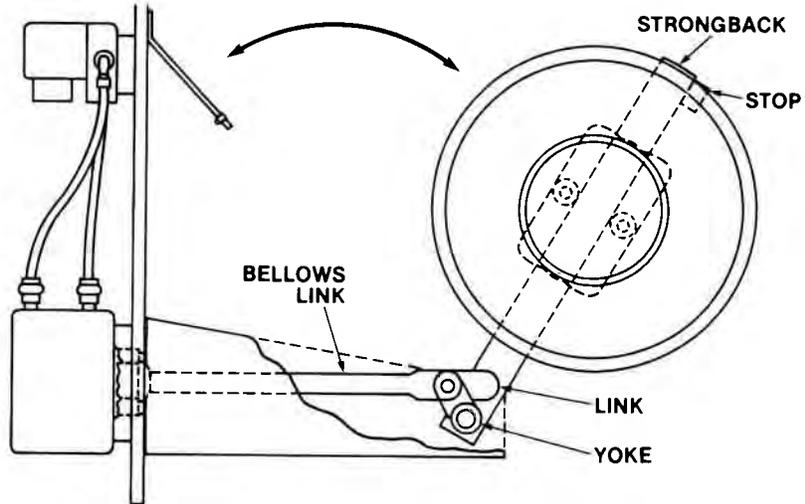
To eliminate contamination problems of the sliding gate valve design, bellows made of brass or stainless steel are often used.



**BELLOWS ACTUATOR ELIMINATES
O-RING ROLLING, SCUFFING**

Bellows-sealed actuators eliminate O-ring rolling, scuffing, and wear problems. An O-ring bonnet gasket seals the bellows, but it's a static seal. Therefore, it isn't rolled, scuffed, or worn. The bellows flexes to transmit the valve-actuating motion.

The extension of the actuator piston exposed to vacuum may be welded directly to the bellows. It is called the bellows link. The bellows is limited to the distance it can extend. Because of this, large valves that use this component are designed to "swing" the sealing plate into and out of position.

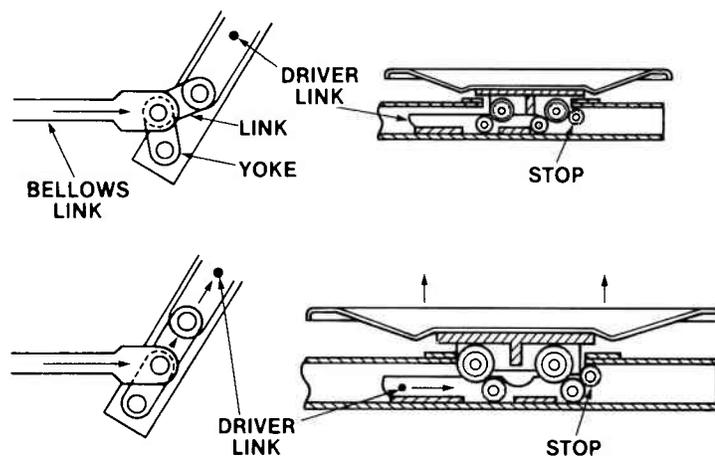


HIGH VACUUM VALVE

An Example of an Elastomer-Sealed Large Valve

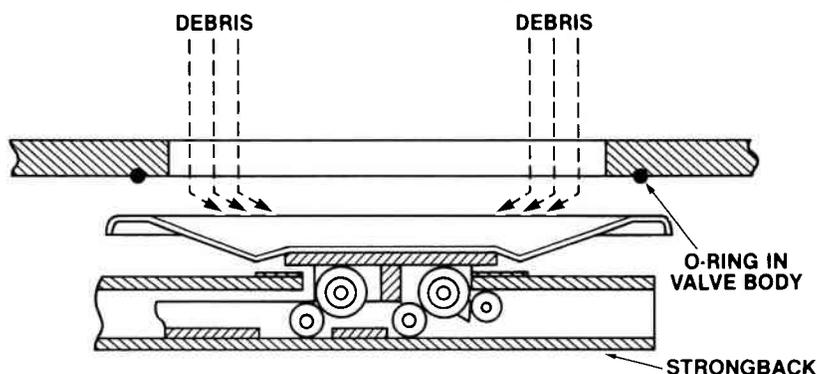
A modern large valve design uses a short-stroke, bellows-sealed actuator. The short stroke is made possible by a swinging gate-type mechanism. The short stroke gets the maximum life out of the bellows because it doesn't get flexed very much. And yet, the

port is completely cleared, giving maximum conductance. The valve body is stainless steel rather than cast aluminum. All internal bearing surfaces are dry-lubricated. This gives them a very low outgassing rate and contributes to leak-tight operation. They can be baked to 150°C in the open position and 125°C closed. Higher temperatures in the closed position can degrade the O-ring seal, causing leaks.



SEALING ACTION

In operation, the bellows link drives the mechanism to a mechanical stop. Notice the relationship of the bellows link to the link yoke. Also notice the driver link and rollers with respect to the seal-plate rollers. Once the first mechanical stop is made, further actuation spreads the link and yoke. This drives the driver link forward, into the strongback. This in turn drives the seal plate upward, into the sealed and locked position.



The conical shape of the seal plate causes process debris to fall away from the seal area. Also, the seal is located in the valve body instead of in the seal plate. This location further minimizes the possibility of seal leaks.

Maintenance

The valve should be in the open position for maintenance and cleaning. The air and electrical lines should then be removed.

Next, the whole actuator sealing-plate assembly should be removed by taking off the body flange and its O-ring.

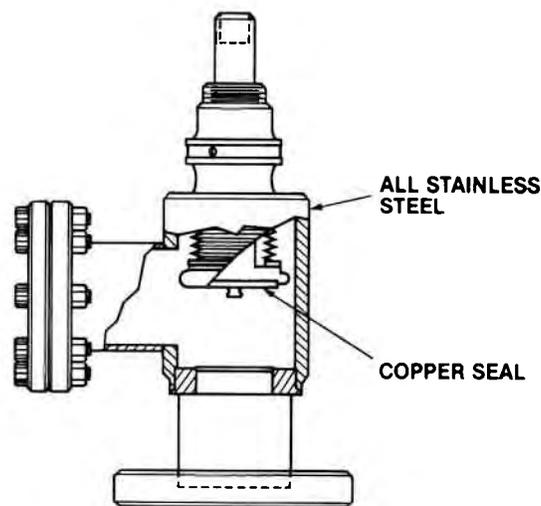
The body flange O-ring is held in position in its location over a large, rectangular O-ring retainer. Reassembly of this flange can be time-consuming if the O-ring continues to slip off of its retainer during the bolt-on process. Bolting on is made much easier by using a seal positioning tool.

Once out of the valve body, the mechanism can be cleaned, adjusted, or repaired. Full removal of the valve and valve body from the vacuum system is usually required to perform proper cleaning and refurbishing. See your instruction manual for proper maintenance procedures.

The cleaning procedure is similar to that listed for small elastomer-sealed valves. Do not attempt to spray cleaning solutions or solvents into the valve body or port area while the valve is on the vacuum system. Serious pump or system contamination could result.

Metal-Sealed Small Valves

These small valves are 3/4 in. to 2-1/2 in. in diameter. The construction of these valves is entirely metal. There are no elastomers used. The seal is accomplished using a copper gasket. Therefore, metal-sealed valves provide reliable seals under repeated bakeout conditions.



Varian all-metal valves use the gasket-capturing design discussed in the flange section. This insures that the gasket material won't flow away from the seal area, even under bakeout temperatures of 450°C.

Metal-sealed valves are used in ultrahigh vacuum systems or for high-purity gas systems. Because of the UHV requirements, these

valves are baked. The temperature at which they keep their sealing integrity is usually related to their size and seal design.

The initial sealing torques range from about 1 to 13 ft-lb to maximums of 6 to 46 ft-lb, again depending on valve size. See instruction manual for applicable torques.

These valves, when operated at room temperature, will perform well up to 100 cycles. When baked out to maximum temperatures, however, valve seal life is about one order of magnitude less. The sealing torque must also be increased after each bakeout.

Pressure ranges of these valves are usually from about atmosphere to 10^{-11} torr. Typical leak rates are less than 10^{-10} std cc/sec.

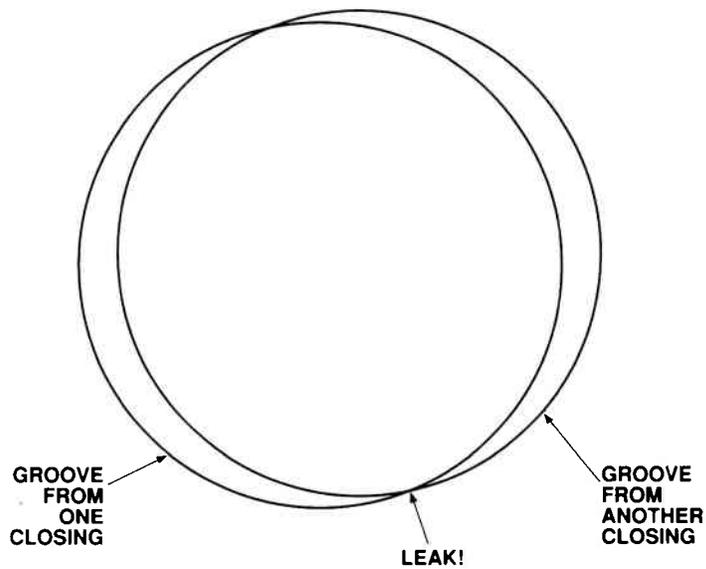
The drive mechanisms must be lubricated with an appropriate high-temperature grease after *each* bakeout. This prevents galling of the threads and early wear of the valve components.

Here are some suggestions for maintenance of all-metal valves:

1. They require more sealing force than elastomer valves. Support the valve so that you do not bend or kink the plumbing attached to the valve when you open or close the valve.
2. After baking at high temperatures, the threads on the valve need to be lubricated to prevent galling. Use a suitable high-temperature lubricant such as Fel-Pro™ C-100.
3. A new seal needs to be installed after a maximum of 300 closures. While the procedure is quite simple, you may need to remove the valve from the vacuum system to carry out the seal change. The valves require a torque wrench to increase the torque by about 1/2 ft-lb per closure. Keep a closure log!

All-Metal Large Valves

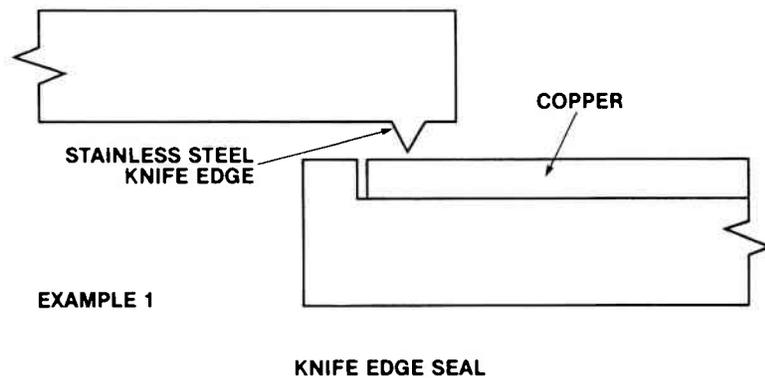
Now, let's consider large metal-sealed valves. Large metal-sealed valves are normally used in UHV applications. Three important aspects are considered in the design of large metal-sealed valves. These valves require much higher sealing forces than elastomer-sealed valves. Also, for some applications, the operation must be greaseless. Still further, it's essential that the seal plate be moved exactly into the same position every time the valve is closed.



This drawing shows how the knife edge grooves can produce vacuum leaks where the grooves crisscross if the seal-plate position isn't exactly maintained. If the grooves do crisscross, a leak can be avoided by using greater sealing pressure each time a closure is made. This limits the life of the gasket, however.

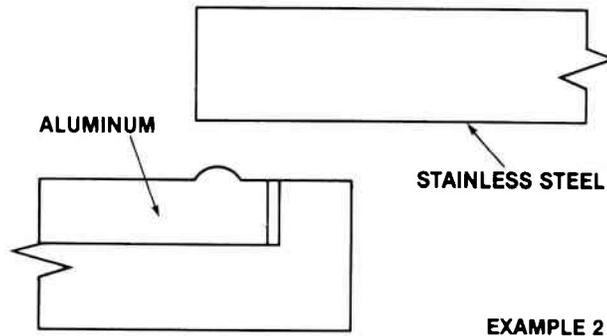
Main Seal Design

The main seal design of metal-sealed valves has taken a number of different forms.



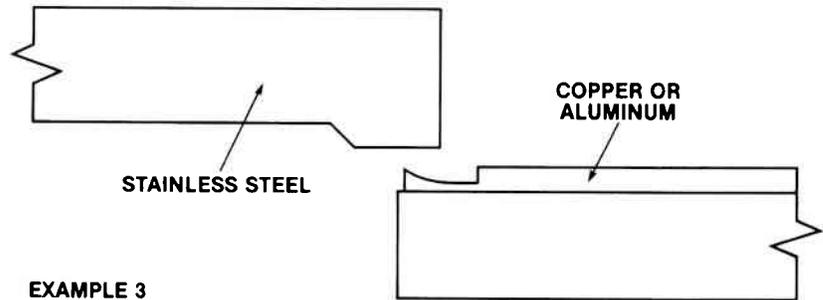
In example 1, a stainless steel knife edge is pressed into an OFHC copper plate. Since the knife edge penetrates the copper gasket, it is essential to reposition in precisely the same location on subsequent cycles. The slightest error in location produces a new groove pattern in the gasket material. This, of course, results in a leak. Leaks can be avoided when positioning errors exist. However, a large increase in sealing force is necessary.

This particular seal design lacks a capturing ability. Therefore, creeping of the gasket material also requires increasingly greater sealing forces. This limits the life of the seal.

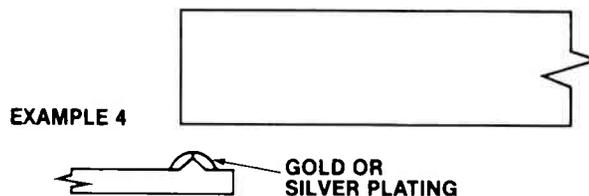


COINED SEAL

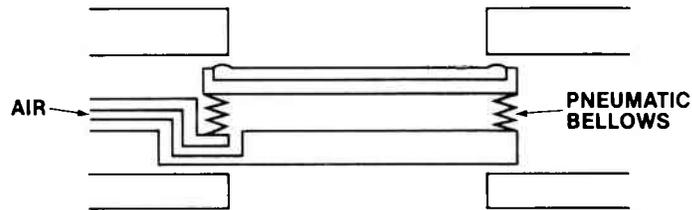
In example 2, an aluminum gasket with a ridge is pressed against a flat stainless steel seat. This is in effect a coined seal. There is enough capturing in this design to produce good seals. Also, this type of seal is insensitive to position repeatability (providing the aluminum is soft enough to avoid denting the seat, and the seat is flat and smooth). However, this capturing effect is lost under high-temperature conditions, especially when the valve is in the closed position.



Example 3 is still another improvement of the metal-seal design. The relatively thick main body of the gasket helps to provide a capturing effect. Also, positioning errors are not so critical, and the life of this design is extended. The valve can be baked to 400°C in the open position. However, no bakeout specifications are published for the closed position.



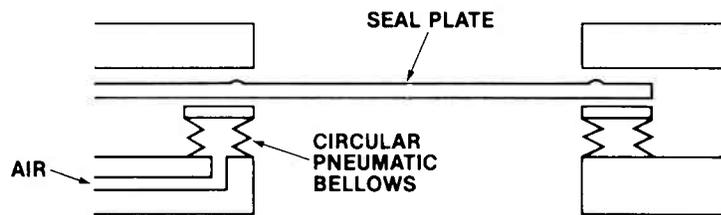
Example 4 uses gold and silver plating on a stainless ridge on the seal plate. The thinness of the plating material prevents extrusion, and positive seals can be made repeatedly. There is some sensitivity to position errors. The very thinness of the plated material means the seal can be affected by particulate matter. This seal design is long-lived, and can be baked in both the open and closed positions.



PNEUMATIC BELLOWS ARRANGEMENT

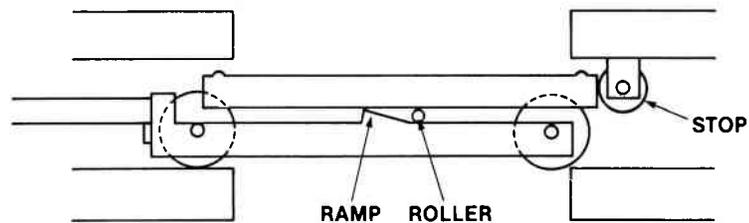
Sealing Force Requirement

The sealing force requirement can be met using a pneumatic bellows arrangement. This construction, however, is heavy, and it is difficult to position the assembly precisely.



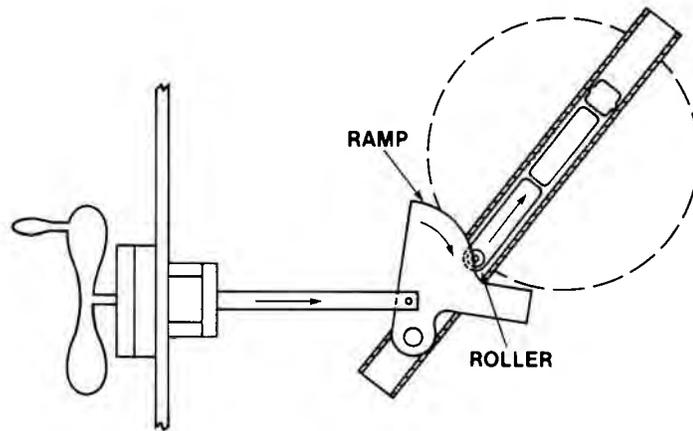
THIN, LIGHTWEIGHT SEAL PLATE

A variation of the bellows actuator is an arrangement that uses a thin, lightweight seal plate. The actuation of the seal plate is controlled from outside the valve body (for purposes of cleanliness). However, this arrangement makes it hard to keep up position repeatability.



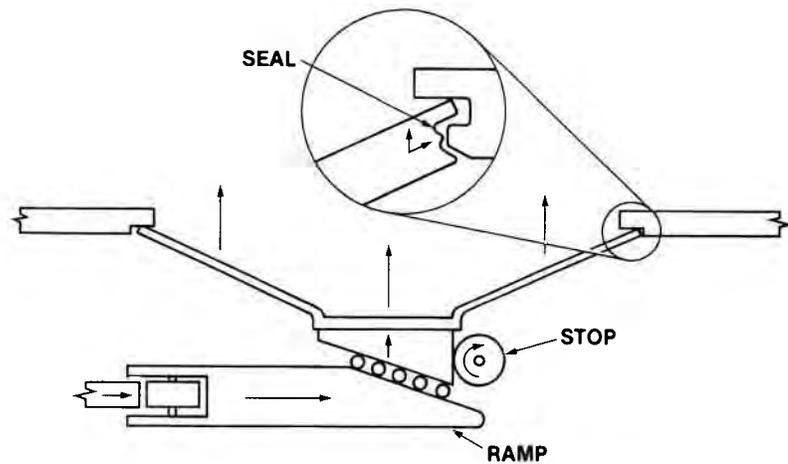
RAMP-AND-ROLLER MECHANISM

Still another bellows design incorporates a ramp-and-roller mechanism. This design offers simplicity of operation. It does not have the possible catastrophic consequences of a ruptured pressurized bellows. It has a disadvantage in that it brings a fair amount of complexity inside the vacuum system, and the possibility of generation of particulate matter. It is easier to service in the field by the user than the previous two designs.



THE VARIAN SOLUTION

The Varian solution to the sealing force requirement is seen here. The basic design resembles the Varian elastomer-sealed gate design. The seal plate in this valve is also conical, thin and lightweight. These characteristics are used to good advantage.



**SEAL PLATE
MOVING INTO SEALING
POSITION**

When moving into the sealing position, the seal plate drives against a stop built into the valve body. When this occurs, further upward force spreads the plate outward. This flexing of the seal plate actually multiplies the driving force. It also reduces the load on the drive mechanism. A gold-plated ridge machined into the seal-plate edge makes a tight leak-free seal when the plate flattens out against the seat. The other two ridges protect the center or sealing ridge against mechanical damage. This valve is bakeable in the open or closed position. Perhaps the greatest advantage of this metal-seal design is that it isn't at all sensitive to position repeatability. That is, there is no possibility of indenting eccentric circles into the seat and causing leaks as in the case of other metal seals.

Maintenance

Maintenance should be carried out according to the manufacturer's instructions. The exact dimensional tolerances needed in reassembly require specialized tools and proper training whenever possible.

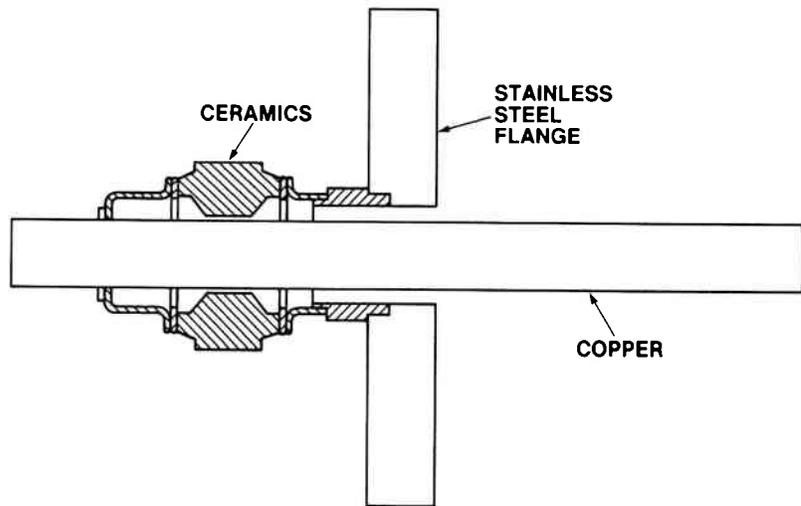
Cleaning of the valve body and actuator assembly is usually the same as discussed earlier in this chapter. Again, UHV usage demands that good vacuum practice be strictly followed. Lint, grease and cleaning residue can cause excessively long pump-down time. This is related to the outgassing load produced by contaminants in the system.

Feedthroughs

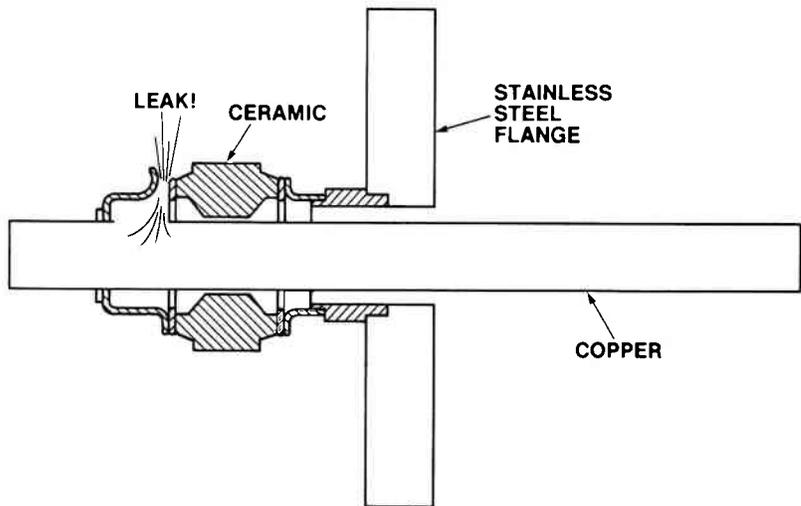
Next, let's consider feedthroughs. Some feedthroughs move components inside the chamber from the outside. Others provide operating power to coating sources and to heaters. Still others pass the signals that monitor the work being done.

What's in a feedthrough? Actually, there is plenty, but let's concentrate on just a few important features of some feedthroughs used in vacuum work.

Electrical Feedthroughs

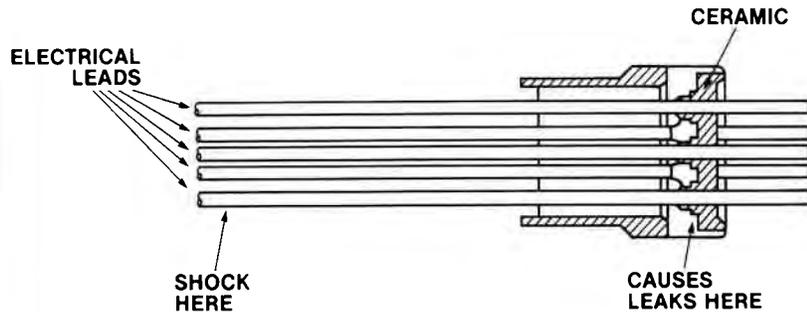


This feedthrough has a large-diameter copper rod that makes it good for high current loads. In fact, the hefty 5/8 in. copper rod conductor makes it seem pretty sturdy. But it is really not strong at all. This is because the entire weight of the copper rod is supported at a single point that also happens to be a vacuum seal.



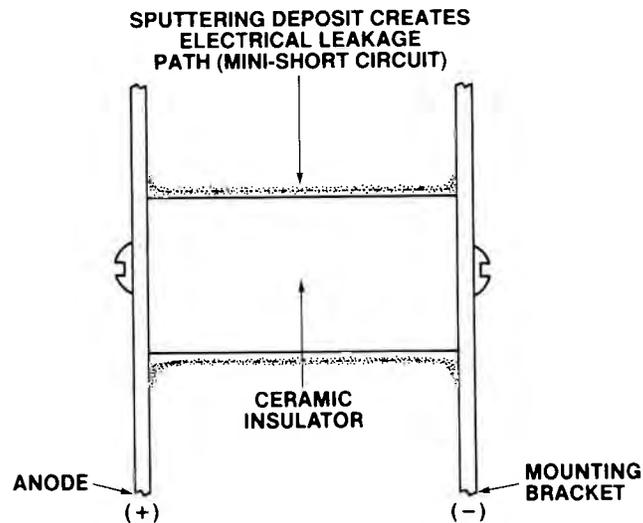
This means that the feedthrough is not intended to support any weight or to withstand any torque forces or shocks. The feedthrough is designed for electrical conduction only, and not for mechanical support.

Feedthroughs may differ, but they are sealed in pretty much the same way, and they're all easily damaged. Therefore, don't put mechanical pressures, stresses or shocks on any seal.



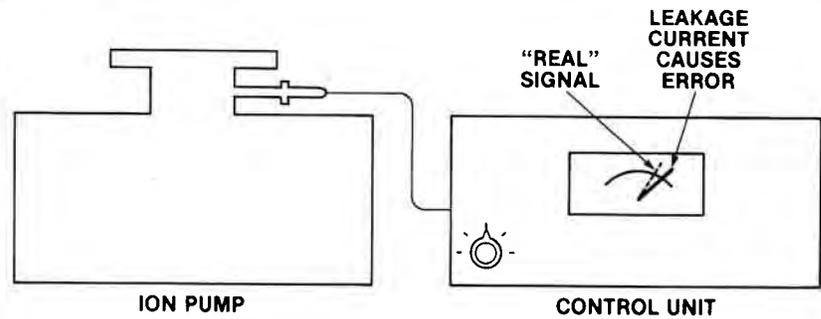
The more electrical leads on a feedthrough, the greater the chances for leaks to develop.

Cutting leads with cutting pliers can produce leaks. This is because the cutting shock transmitted to the seal area can cause leaks.

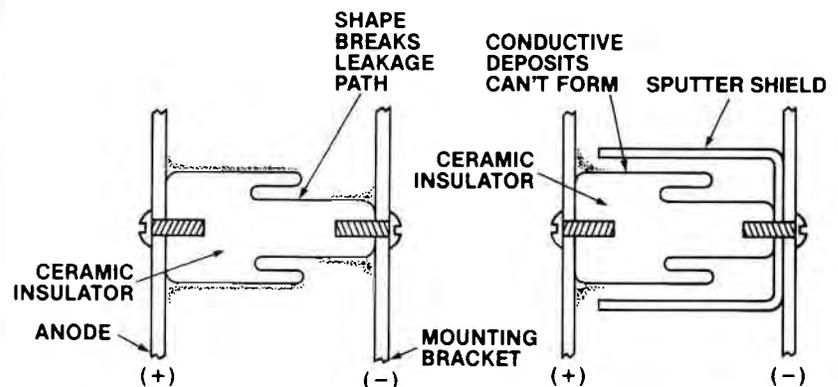


Now let's look at another possible problem, and see how it is handled. We saw in the pump section how gases are ionized by applying a high voltage across a cathode and an anode.

Recall that ionization causes sputtering. Material comes off one surface and deposits onto another. This deposition can coat an insulator and create electrical leak paths.



The leak paths may not be dead shorts, but they can cause error signals. An error signal may add to the true signal, and incorrectly indicate high pressure. This can lead to lost time searching for vacuum leaks that don't exist.



VARIAN ION PUMPS
HAVE LONG RE-ENTRANT
PATH INSULATORS

Varian ion pumps have long re-entrant path insulators. The shape of the insulators helps keep electrical leak paths from forming in the first place.

Then, the sputter shield placed over the insulator further reduces electrical leakage problems.

Ionization can also occur between the lead and the flange in high-voltage feedthroughs. Again, electrical leakage can result.

A ceramic insert placed in the high-voltage feedthrough reduces the distance between the conductor and surrounding surfaces. This minimizes ionization, sputtering and the development of electrical leak paths.

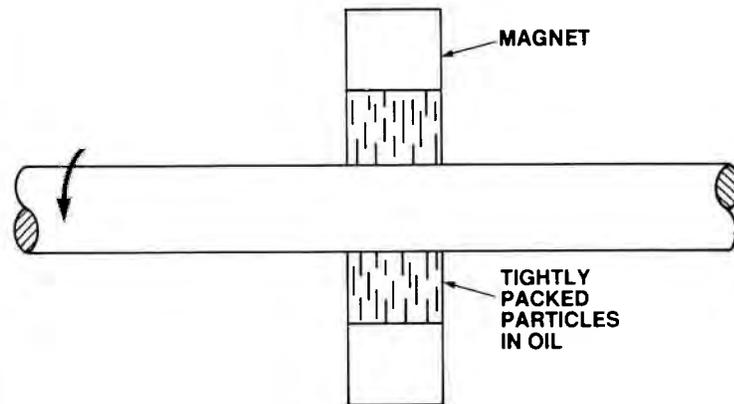
Motion Feedthroughs

There are many different types of motion feedthroughs, but they all provide rotary or linear motion, or a combination of the two. They operate valves, open and close shutters, or rotate planetary fixtures, to name a few examples of the work they do. Feedthroughs may be sealed with elastomers or may be metal-sealed.

Or, they may be “Ferrofluidically” sealed. Let’s take a look at Ferrofluidics™.

Ferrofluids can make vacuum-tight seals. A Ferrofluidic seal is a colloidal suspension of submicroscopic magnetic particles in a low vapor pressure fluid.

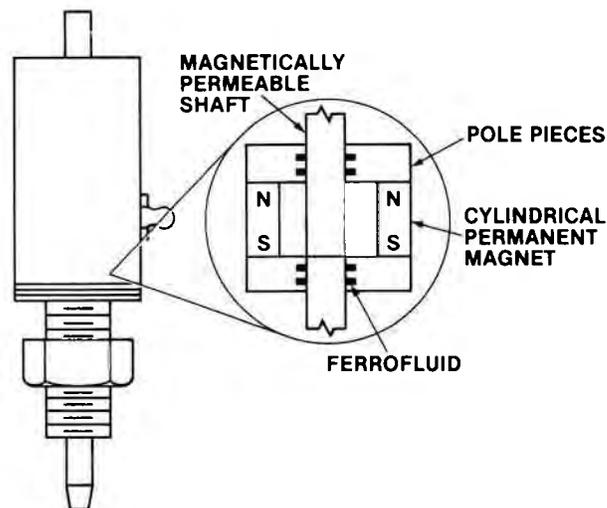
A colloid is a mixture, but unlike oil and vinegar, colloids cannot separate. In Ferrofluidic colloids, the magnetic particles cannot settle to the bottom or float to the top, but instead are permanently suspended uniformly throughout the fluid.



FERROFLUIDIC FEEDTHROUGH

In a Ferrofluidic feedthrough, a magnetic array surrounds the colloidal suspension, lining up the closely packed particles so tightly that a leak-free seal is made.

Caution: Do not apply solvents to clean or leak-check a Ferrofluidic feedthrough. This may dissolve the fluid, making it useless. Also, do not heat a Ferrofluidic feedthrough above 80°C.



ROTARY FEEDTHROUGH

In actual practice, a Ferrofluidic feedthrough has several sealing pockets.

Ferrofluids are excellent in rotary feedthroughs that drive heavy planetary wafer holders where a vacuum seal is kept up under high torque conditions. This type of seal is superior to double O-ring shaft seals, because it does not wear out or become distorted the way O-rings do.

Let's look at another aspect of motion feedthroughs. They must remain leak-free during operation as well as while static. Here are a few tips on checking whether the motion feedthrough is leak-free when leak checking a system by looking for pressure changes or while applying a tracer gas.

1. Periodically manipulate a motion feedthrough.
2. Manually rotate the crucibles in multiple pocket electron beam guns.
3. Manipulate shutters, whether manually or electropneumatically actuated.

Liquid Feedthroughs

Liquid feedthroughs carry cooling water, liquid nitrogen, or other refrigerants to components in the work chamber. Liquid nitrogen feedthroughs must withstand wide temperature changes while remaining leak-free.

Optical Feedthroughs

Pyrex and sapphire viewports allow visual and optical observation. Although they can withstand wide temperature changes, rapid changes may cause failure or leakage.

Summary

This completes our discussion on vacuum hardware components. You've seen the important roles that the materials design, fabrication, and techniques play in the manufacture of vacuum hardware components. You've also seen that the proper handling and use of components make it possible to achieve high-purity vacuum conditions. And, you've learned a number of techniques that will be valuable when troubleshooting an ailing system.

7

Systems

When you have completed this chapter, you will be able to:

1. Evaluate the major types of vacuum systems.
2. Describe how to build the major types of operating systems.
3. Perform the proper steps to operate a vacuum system.
4. Recognize normal and abnormal operation of a vacuum system.

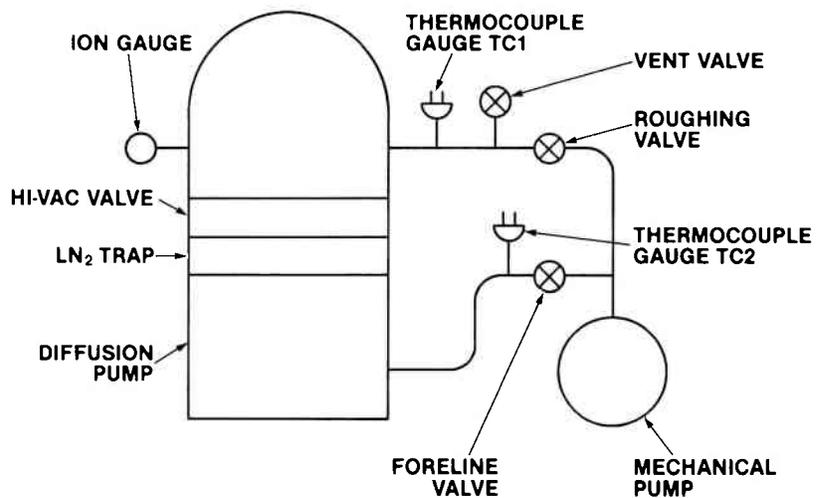
Introduction

Well, it's time to put it all together. We want to combine the hardware and instruments into a useful system. That means that the system has to produce and maintain the proper pressure in a reasonable time. It must also be made of the right materials and of the right size to do the intended work. In addition, the system must be rugged and reliable; there has to be as little downtime as possible. To accomplish this, proper construction, maintenance and operation procedures need to be followed.

Let's take a look at some vacuum systems and note their specific advantages and disadvantages. We will start with a diffusion pump system.

Evaluating Systems

Diffusion Pump Systems



DIFFUSION PUMP SYSTEM

Advantages

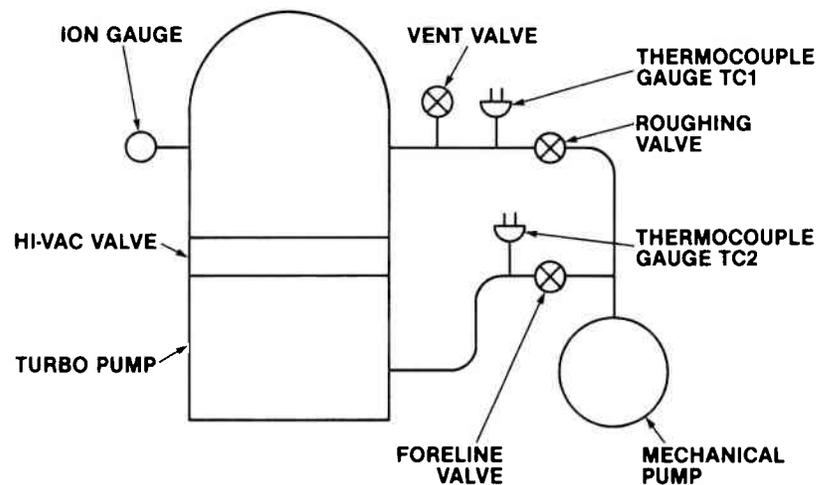
- Simple/Reliable
- High Speed
- Constant Speed
- High Throughput
- Continuous Operation
- Low Cost
- Withstands Contamination
- No Moving Parts
- Small to Large Size

Disadvantages

- Backstreaming Possible
- Requires Mechanical Pump
- Usually Requires LN₂ for Cryotrap
- High Operating Cost
- Accident-Prone
- Must Be Installed Vertically
- Proper Operation Important
- 1×10^{-3} torr Upper Limit

Turbomolecular Pump Systems

Here's the turbo pump system:



TURBO PUMP SYSTEM

Advantages

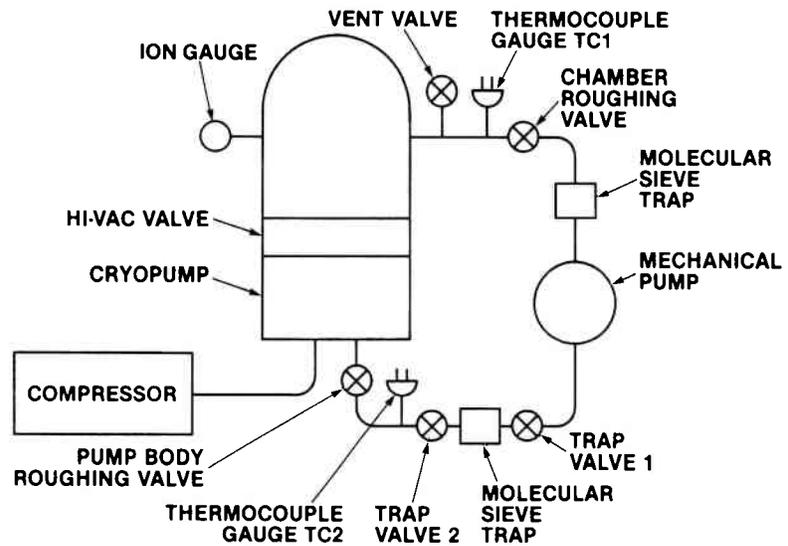
- Fast Recovery from "Dumping"
- Fast Start-up
- Constant Speed
- Clean (No Oil)
- Low Operating Cost
- High Throughput
- Small to Large Size

Disadvantages

- Sensitive to Particulates
- Requires Mechanical Pump
- High rpm (Noise/Vibration)
- Low Speed for Hydrogen and Helium
- High Maintenance Cost
- 1×10^{-3} torr Upper Limit
- High Initial Cost

Cryopump Systems

Now let's look at a cryopump system.



CRYOPUMP SYSTEM

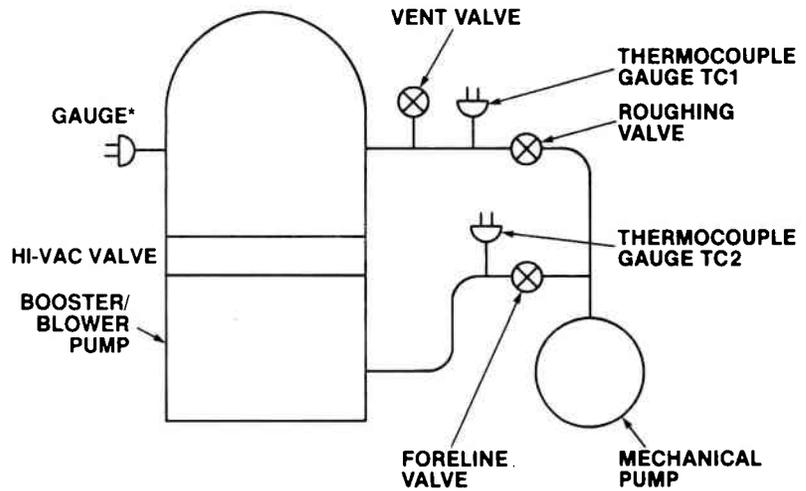
Advantages

- High Speed
- Very High Throughput
- Clean
- Low Operating Costs
- Continuous Mechanical Pumping Not Required
- Small to Large Size

Disadvantages

- Requires Regeneration
- Requires Periodic Maintenance
- Requires Ultrapure Helium
- Hydrogen Can Be Explosive
- Low Frequency Vibration
- Some Application Limits

Booster/Blower Systems



BOOSTER/BLOWER SYSTEM

Advantages

- High Pump Speed
- High Throughput
- Upper Limit 10 torr
- Low Operating Costs
- Moderate Initial Cost
- Small to Very Large Size
- No Oil Sealing
- Tolerates Particulates, Oxygen, Etchants

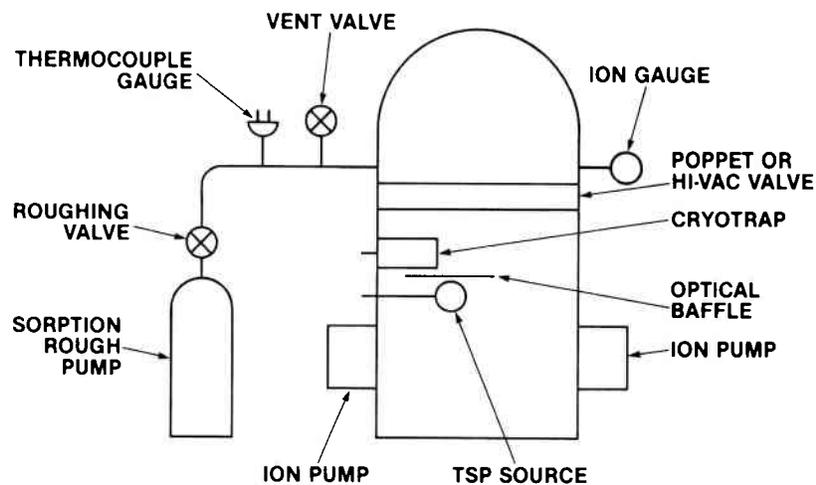
Disadvantages

- Requires Mechanical Pump
- Critical Mechanical Tolerance
- Bearings Require Cooling and Lubrication
- Lower Limit 10^{-5} torr
- Noisy

*Gauge type depends on pressure range expected.

Ion Pump Systems

Next, an ion/TSP pump system.



ION PUMP SYSTEM

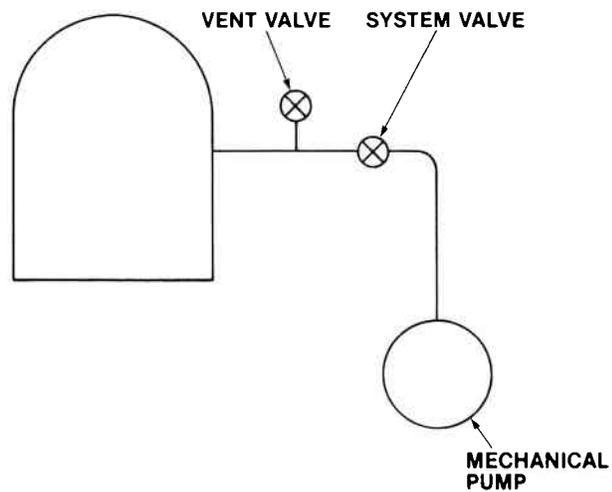
Advantages

- Ultraclean (UHV)
- Low Operating Costs
- Fail-Safe
- Forepump Not Required
- Low Maintenance
- Low Pressure Limit
 1×10^{-12} torr with TSP
- Long-Term Operation
- Self-Regulating
- Tiny to Medium Size
- No Noise/Vibration

Disadvantages

- Low Speed, Throughput
- High Initial Cost
- Speed Varies with Pressure
- Speed Varies with Gas Species
- May Generate Chamber Plasma
- Can "Regurgitate" Methane
- High Pressure Limit
 5×10^{-5} torr (steady state)
- Large/Heavy Relative to Speed
- Magnets

Mechanical Pump Systems



MECHANICAL PUMP SYSTEM

Advantages

- Can Pump from 760 torr
- Rugged Construction
- Low Maintenance
- Low Operating Cost
- Low Initial Cost
- Long Life
- Small to Very Large Size

Disadvantages

- Backstream Oil
- Oil Vapor in Discharge Gases
- Noise
- Speed Decreases Below 10^{-1} torr
- Sensitive to Particulates
- Low Frequency Vibration

Building an Operating System

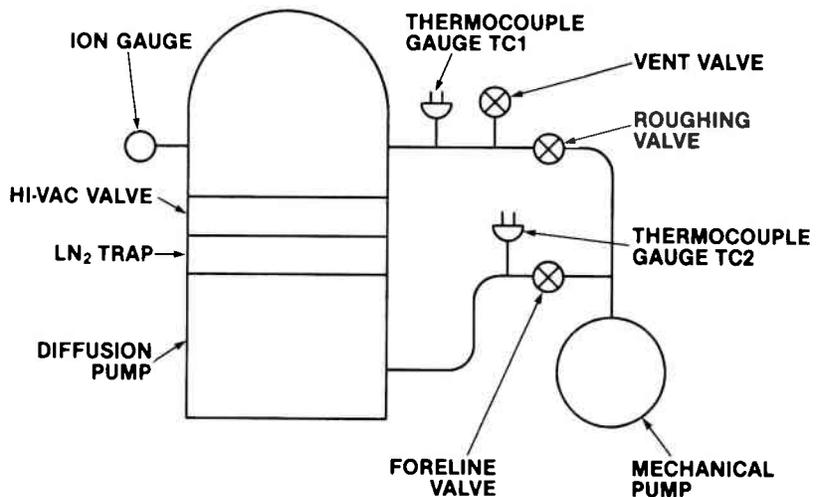
Now, let's assume that we need an operating system. Please recognize that what we "build" here probably won't look like the system that you use at all. Your system configuration will vary considerably, depending upon its application. What we are going to do is to construct on paper a "typical" system that has the minimum number of valves, gauges and pumps to perform properly.

Proper performance will be defined here as the ability to reach the desired vacuum level. We will not try to say anything about what is to go on inside the work chamber, that is, your process. Most of the systems we will look at will be high vacuum systems. Let's start with the one which most people are familiar with: the diffusion pump system.

A Diffusion Pump System

Putting the System Together

First of all, note the placement of valves and gauges on this drawing of a "typical" diffusion pump system.



DIFFUSION PUMP SYSTEM

It is equipped with the minimum number of valves and gauges to work.

Assume that the system is started and *running properly*: all services are properly connected and are ON. Follow this procedure to vent the work chamber.

Bringing the Work Chamber to Air

To bring the work chamber to air (preferably, dry nitrogen):

1. Turn off the ion gauge.
2. Close the hi-vac valve (also called a gate valve or main valve).
3. Open the vent valve (also called a back-to-air or BTA valve).

Bringing the Work Chamber to High Vacuum

To bring the work chamber to high vacuum (or to “pump it down”):

1. Close the vent valve.
2. Close the foreline valve. (Watch TC2. It should not exceed maximum tolerable foreline pressure.)
3. Open the roughing valve.
4. Wait for the chamber pressure to reach the crossover pressure. (Watch TC1 and TC2. Pump may require foreline pumpdown.)
5. Close the roughing valve.
6. Open the foreline valve.
7. Open the hi-vac valve.
8. Turn on the ion gauge.

These directions should allow us to safely and properly operate our “typical” diffusion pump system. They may need some modification to work for your diffusion pump system because of particular design or process requirements.

Start-up Procedure

Now, how do we start our system safely and properly? Let’s look at a typical start-up procedure. Assume the system is properly installed and connected to all services. All system valves are closed. All system switches are OFF. The system does not have large leaks. Check to see that the pumps have oil and that all services are in a safe condition and will not result in harm if turned on as we suggest here.

1. Turn on the main power to the system.
2. Turn on the mechanical pump.
3. Open the roughing valve and pump the chamber to about 100 mtorr pressure. (Watch TC1.)
4. Close the roughing valve.
5. Open the foreline valve. Pump the diffusion pump and trap to about 50 mtorr pressure. (Watch TC2.)
6. Turn on the diffusion pump (electrical power and cooling water).

7. Fill the LN₂ trap (may be done before step 6 if oil vapor is of concern).
8. Allow 30 minutes to 1 hour for the diffusion pump to warm up. Monitor TC2—it should remain well below maximum tolerable foreline pressure.
9. Check the chamber pressure. Rerough if necessary. (Close foreline valve, open roughing valve, pump to crossover pressure, close roughing valve, open foreline valve.)
10. Open the hi-vac valve slowly.
11. Turn on the ion gauge. Monitor the chamber pressure.
12. You are on your way! Cycle the system per directions above.

Remember that improper operation of the diffusion pump system can result in mechanical pump oil and/or diffusion pump oil in the work chamber. Also, “dumping” the diffusion pump to air by improper valving procedures will tend to decompose (perhaps even burn) the pump oil. Proper operation, on the other hand, will give many months of successful operation with minimum maintenance needs.

A Turbomolecular Pump System

Putting the System Together

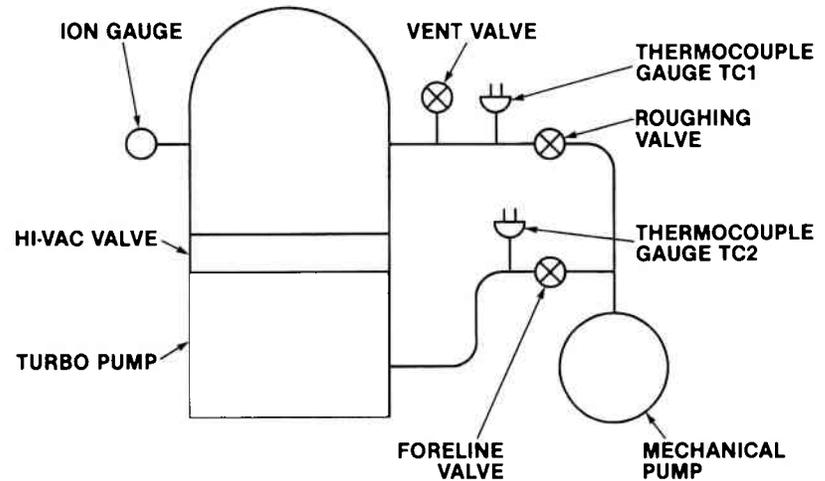
This system is much like a conventional diffusion pump system. Turbomolecular pump (TMP) systems are often assembled with a separate roughing line and foreline. The entire roughing and foreline section is the same as that in a diffusion pump system. The requirements for sizing the forepump are two-fold. It must be able to accept the maximum gas load from the turbo pump exhaust. Also, the forepump should provide an acceptable chamber roughing time.

It is desirable to use a high vacuum isolation valve to isolate the turbo pump if roughing the chamber through a separate roughing line, similar to the way diffusion pump systems are manifolded.

This is especially true on fast cycling applications such as load locks, where you want to cycle the load lock at a rate approaching the start-up time of the turbo pump. To reach the fastest cycle time, the system must transfer to the turbo pump (which is

rotating at full speed) after initial roughing of the chamber through a roughing line. As a general "rule of thumb," it is advisable to use a valved system if the chamber is going to be cycled from atmosphere to vacuum to atmosphere repetitively, with a total cycle time of less than 10 minutes.

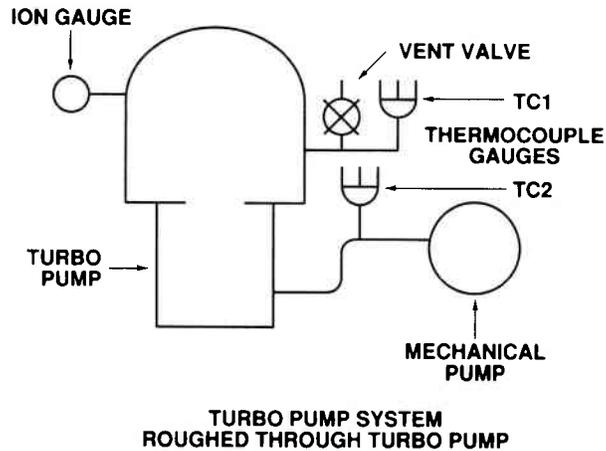
A valved system as shown here should be used when evacuating a large vacuum chamber. Roughing the chamber through the turbo pump will be slower, since the turbo pump will have rather large conductance losses due to the small exhaust port on the turbo pump.



TURBO PUMP SYSTEM

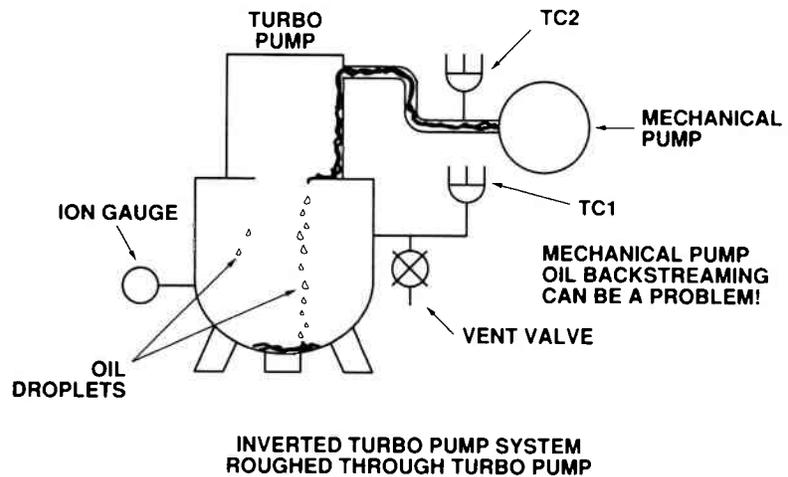
Turbo pumps allow great flexibility in the choice of vacuum manifolding. For moderately sized chambers, the turbo pump can be mounted directly to the chamber without a high vacuum isolation valve. In this case, the turbo pump is cycled from atmosphere to high vacuum with the chamber, and the chamber is roughed through the turbo. Usually, the turbo pump and mechanical pump are started and stopped simultaneously. Most modern turbo controllers can switch the mechanical pump contactor on and off with single switch control of both mechanical pump and turbo pump from the front panel.

Alternatively, if the chamber is larger and cannot be roughed through the turbo pump to several hundred millitorr within the turbo pump start-up time, you can use a delayed start with a thermocouple gauge set point to delay starting the turbo pump until the pressure falls into the high millitorr range. The advantage of roughing through the turbo is that a simple system with minimal valving can be built. A disadvantage is the increased time needed to reach high vacuum pressure levels.



Mounting Orientation

The oil-lubricated pump must be oriented within 10° of vertical. Grease-lubricated turbo pumps may be oriented in any position. Condensation of mechanical pump oil in the TMP forevacuum area is normal, since the mechanical pump operates near its base pressure. When the pump is inverted, oil droplets may contaminate the system, so we recommend using a foreline trap to prevent mechanical pump oil from entering the pump and the system, contaminating both.



If the pump is to be mounted horizontally, orient the foreline port downward and use a foreline trap between the mechanical pump and the turbo pump.

When you mount the pump vertically, no trap is required.

There will be the normal accumulation of mechanical pump oil condensate in the forevacuum of the TMP (mechanical pump backstreaming near its base pressure). A trap in the foreline would help protect the system from improper shutdown or accidental dumping of the pump.

Please remember that foreline traps require regular maintenance or they become a source of oil vapor.

Pump Cooling

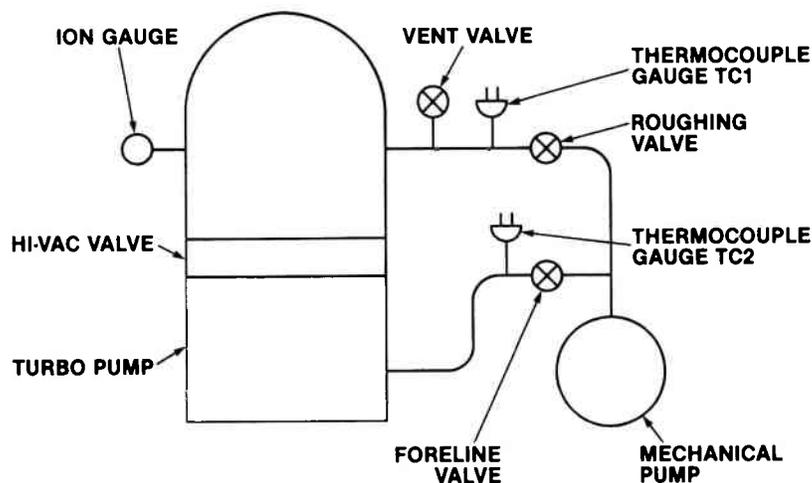
Continuous operation of the turbo pump at high pressure usually requires water cooling. Consult the manufacturer for recommended cooling method for your application.

If the pump is overheated, the thermistor on the motor stator will sense the excess heat and safely shut the pump down (at 80°C).

Overcooling your turbo pump can cause problems. If the pump is overcooled, stiffening of bearing O-rings will occur, the lubricating oil will become more viscous, the pump vibration level will increase and this, of course, may shorten bearing life.

No liquid nitrogen trap is needed on a turbomolecular pump to stop bearing or mechanical pump fluid backstreaming when the pump is operated properly. The compression ratios for all gases but hydrogen and helium, the lightest gases, are high enough so that little will flow from the foreline side to the high vacuum side, provided that the pump is rotating at the rated speed. The liquid nitrogen trap is often used with the turbo pump to increase pumping speed for water vapor.

System Operation



TURBO PUMP SYSTEM

Let's look at operating this system. The operation of the turbomolecular pump system shown above is much like that of a diffusion pump system.

Bringing the Work Chamber to Air

1. Turn off the ion gauge.
2. Close the hi-vac valve.
3. Open the vent valve.

Bringing the Work Chamber to Vacuum

1. Close the vent valve.
2. Close the foreline valve.
3. Open the roughing valve.
4. Wait until the chamber pressure has reached the crossover pressure (usually 80–100 mtorr).
5. Close the roughing valve.
6. Open the foreline valve.
7. Open the hi-vac valve.
8. Turn on the ion gauge.
9. Begin operation.

Start-up Procedure

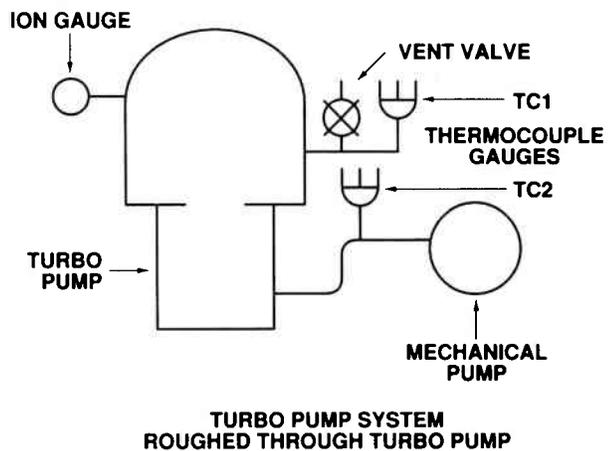
Now let's look at a start-up procedure for the turbomolecular pump. We are assuming that the system is in place, all services properly connected and plumbed. All system valves are closed. There are no large leaks.

1. Turn on the electrical power.
2. Turn on the mechanical pump.
3. Open the foreline valve.
4. Rough the pump and foreline to about 100 mtorr.
5. Turn on the turbo pump. (May need water as well as electrical power.)
6. Allow 5–10 minutes for the pump to get up to speed (longer for large pumps).
7. Close the foreline valve.

8. Open the roughing valve.
9. Rough the chamber to about 100 mtorr.
10. Close the roughing valve.
11. Open the foreline valve.
12. Open the hi-vac valve.
13. Turn on the ion gauge. You are in business!

Now, let's look at operating the other system we have discussed.

Operation of Turbo Pump System Roughed Through Turbopump



This is a much simpler system to operate. Let's look at its operation.

Bringing the Work Chamber to Air

1. Turn off the ion gauge, the turbo pump, and the mechanical pump.
2. Immediately (within one quarter of the coast-down time) open the vent valve. (Vent slowly.)

Bringing the Work Chamber to Vacuum

1. Close the vent valve.
2. Turn on the pumps. (May need a delay time on start of turbo pump.)
3. When the pressure level is satisfactory in the work chamber, begin process.

Other System Considerations

Venting a Turbo Pump

Use of a vent control will afford control over a “delay time” before air-releasing a turbo pump, once the turbo is shut down or in case of power failure. A “delay time” will prevent undesired venting in case of a power failure of short duration.

Pumps must be vented before they stop rotating to prevent contamination by mechanical pump oil and possible bearing damage upon subsequent air release (“hammer effect”).

Each model of turbo pump has a “coast-down time,” which is the time it takes for a pump to stop spinning after power is removed if it is left under vacuum. As a rule of thumb, the maximum time delay before venting a pump after switching off power should be no more than one quarter of the coast-down time.

Also available on most vent controls is an adjustable “venting time” setting, to help conserve bottled venting gas.

The minimum venting time is the time needed to reach a pressure of about 375 torr inside the pump. This pressure is high enough to avoid mechanical pump oil backstreaming and contamination of the turbo pump.

If there is no high vacuum valve isolating the turbo from the system, the required venting times will be extended, based upon chamber size. If reaching pressures less than 10^{-6} torr in a minimum time is important, the pump should be vented to a dry gas.

Particulates

Turbo pumps should not be used in applications that generate a large quantity of particulates. Particulates will make their way to the bearings and cause bearing seizure or erode pump blades. The inlet screen provided with turbo pumps collects all large particles and prevents them from entering the pump but it is ineffective against a continuous stream of fine particulates. Screen cleaning must be performed on a regular basis.

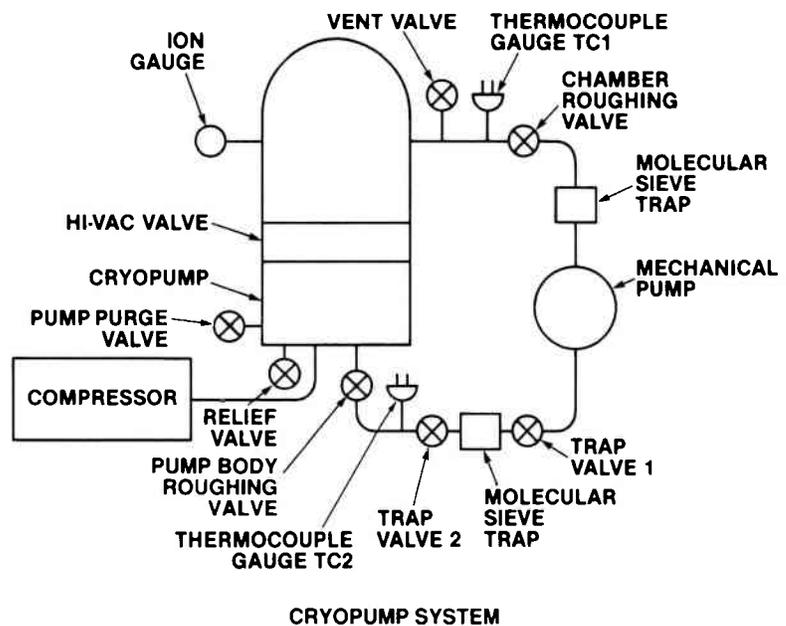
A Cryopump System

Putting the System Together

A diagram of how a cryopump fits into a vacuum system is shown on the next page. In this discussion, we refer to the vacuum system as having three major portions:

1. The *vacuum chamber*, which consists of the chamber(s), load locks, etc., where the work is being processed; the high vacuum valve; and the associated hardware and instrumentation (ion gauge, etc.).
2. The *cryopump*, which consists of the pump module, the compressor module, and the interconnecting flexible hoses.
3. The *roughing manifold*, consisting of the roughing pump, sieve trap(s), roughing valves, lines to the vacuum chamber, and roughing (TC) gauges.

Look at the cryopump system drawing. Note that the valves and plumbing have an arrangement suitable for a capture pump or storage pump. No forepump is required.



Here is a set of specific instructions for various operations on this system.

Bringing the Work Chamber to Air

1. Turn off the ion gauge.
2. Close the hi-vac valve.
3. Open the vent valve.

Bringing the Work Chamber to High Vacuum

1. Close the vent valve.
2. Open the chamber roughing valve.

3. Pump the chamber to crossover pressure.
4. Close the chamber roughing valve.
5. Open the hi-vac valve.
6. Turn on the ion gauge.

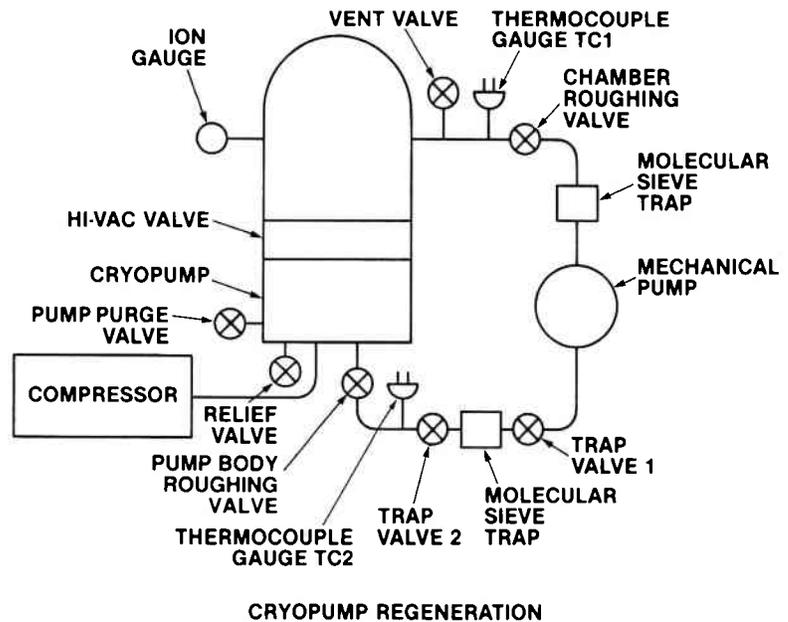
Start-up Procedure

Here's a start-up procedure for a "typical" cryopump system. We are assuming the system is properly installed and connected to all services. All system valves are closed. There are no large leaks.

1. Turn on the mechanical pump.
2. If the condition of the molecular sieve traps is not known, open trap valve 1 and bake both traps for a minimum of 1 hour.
3. When the traps have cooled, open trap valve 2 and verify a pressure of 10 mtorr or less on TC2.
4. Check the pump pressure by closing trap valve 2 and opening the pump body roughing valve. If TC2 is above 50 mtorr, rough pump the body until it is below 50 mtorr, and then close the pump body roughing valve and trap valves 1 and 2.
5. Turn on the compressor. (Wait for chardown, i.e., 20°K.)
6. Rough the chamber (to 150 mtorr). Close the roughing valve.
7. Open the hi-vac valve.
8. Turn on the ion gauge. Monitor the pressure.
9. Start process.

Regeneration Procedure

Because the cryopump is a storage pump, we must empty it when it is getting full. Here is a procedure for regeneration.



1. Close the hi-vac valve and turn off the ion gauge.
2. Turn off the compressor.
3. Purge the pump body with nitrogen (regulated to 15 or 20 psig) for the time specified by the manufacturer or until pump reaches 295°K (room temperature). If heated nitrogen is used, the temperature at the pump body must not exceed 120°C. Isolate the molecular sieve trap(s) from the cryopump. Bake the trap(s) while pumping on the trap with the mechanical pump. Nitrogen purge of the trap during bakeout is advisable, and gas ballasting the mechanical pumps may be necessary.
4. Rough the pump body to about 10 mtorr.
5. Turn on the compressor. (Continue roughing for 10 to 20 minutes, then close the roughing valve.)
6. Periodically monitor the pressure inside the pump—it should be about 50 mtorr or less.
7. After the pump has chilled to 20°K or less, return to normal operation.

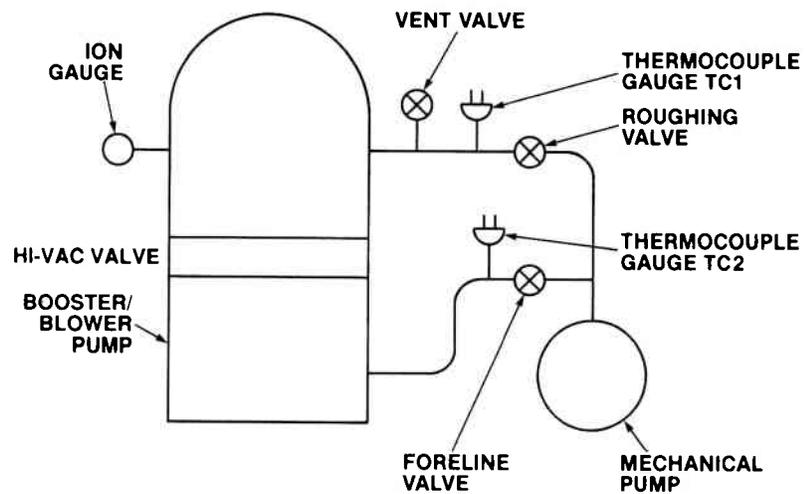
Cryopump systems differ in their operation in that they are storage pumps. Also, they do not need the assistance of a fore-pump while they are on-line and pumping. However, most people leave the rough pump running because they need to rough the chamber frequently during use. We strongly urge the use of

molecular sieve traps between the rough pump and the cryo-pump. Also, use them between the rough pump and the chamber to prevent mechanical oil from backstreaming or migrating into the vacuum system.

A way to avoid the traps, etc., is to use a dry roughing pump, which contains no oil, or use sorption roughing pumps.

A Booster/Blower System

A system using a blower as its main pump should be considered a medium-range vacuum system.



BOOSTER/BLOWER SYSTEM

Wide variations of pumpdown sequence exist, depending to a great extent on chamber volume and the volume-to-pump speed ratio. The blower is not made to pump continuously at pressures above 10 torr. The base pressure will usually be in the range of 10^{-4} to 10^{-5} torr. Its operation parallels that of the diffusion pump, or turbo pump, but valving actions may occur safely at higher pressures.

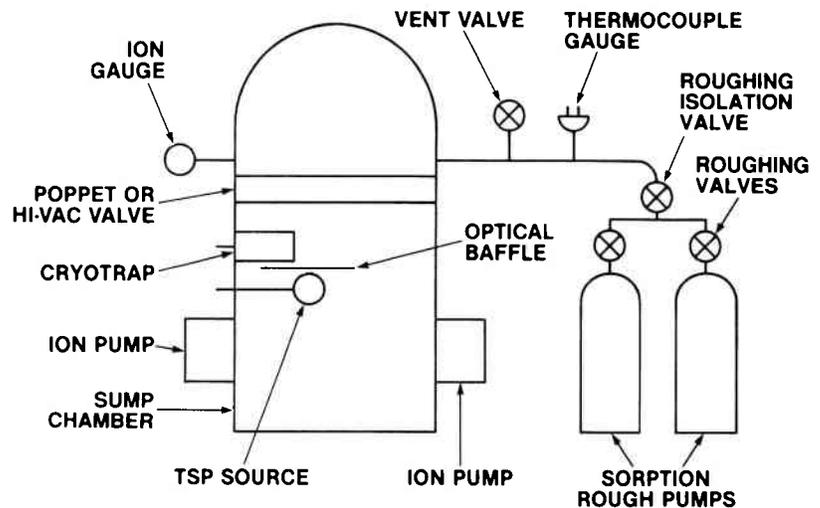
The operating concerns are similar to those of the turbomolecular pump system. Both are mechanical, high rotational speed pumps. In general, the blower is more rugged, but cannot work at

low pressures like the turbo pump does. This type of system is often roughed directly through the blower. A bypass valve is also often used to direct gases around the blower at high pressures. The bypass valve is closed as the crossover pressure is reached. These techniques eliminate the need for a separate roughing line.

An Ion Pump System

Putting the System Together

Ion pump systems may operate much as the systems we have discussed. They also may be pumped down to ultrahigh vacuum and kept there. A load-lock entry may be used for getting materials in and out of the vacuum chamber without having to vent and rough the whole work chamber. We will not cover these operations here.

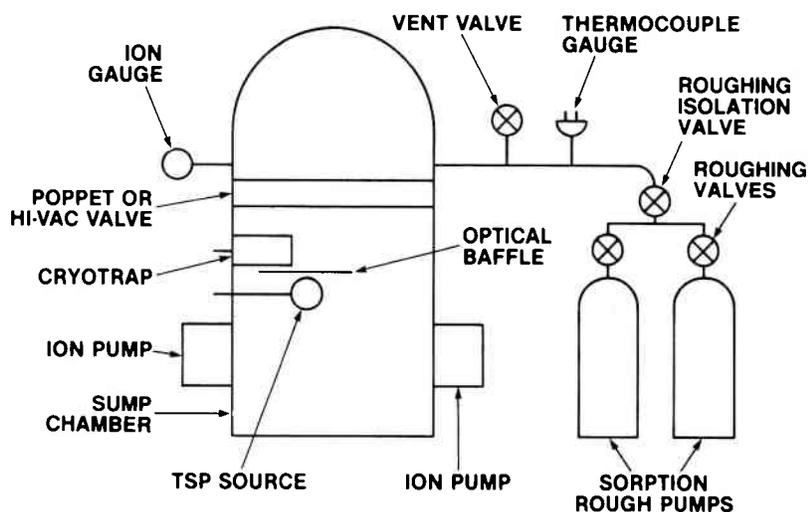


ION PUMP SYSTEM (MANUAL ONLY)

Start-up Procedure

Here's a start-up procedure for a manually operated ion/TSP pump system.

Let's assume that the system is at atmospheric pressure, the isolation and roughing valves are closed, hi-vac valve open, gauges off, vent valve closed, LN₂ trap dry. Remember that we are using sorption pumps for roughing. Of course, it is properly connected to all services. There are no large leaks.



ION PUMP SYSTEM

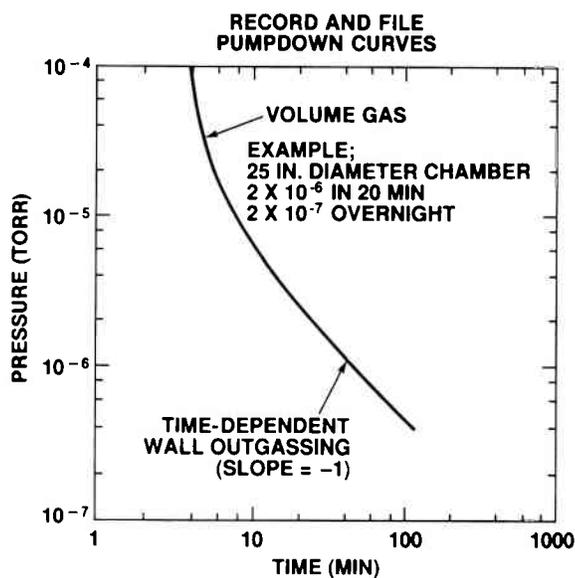
1. Pre-chill the sorption pump(s) with liquid nitrogen.
2. Turn on the thermocouple gauge.
3. Rough the complete system using the sorption pumps in sequence. Continue roughing with the second pump.
4. Fill the LN₂ trap (optional).
5. Outgas the TSP (100% duty cycle) starting it at 30 mtorr.
6. When sump pressure is less than 10 mtorr, switch the ion pump to "start mode." Turn on the power.
7. Close the main roughing isolation valve and the last sorption roughing valve.
8. Turn on the ion gauge.
9. Switch the TSP to "standby."
10. Bake out 12 hours (250°C); then cool down (8 hours).
11. Outgas the TSP (again).
12. Refill the LN₂ trap (optional).
13. Continue TSP cycling in accordance with system instructions.
14. At very low pressure, the TSP need only be used for about 5 minutes once or twice a day.

Characterizing Your System

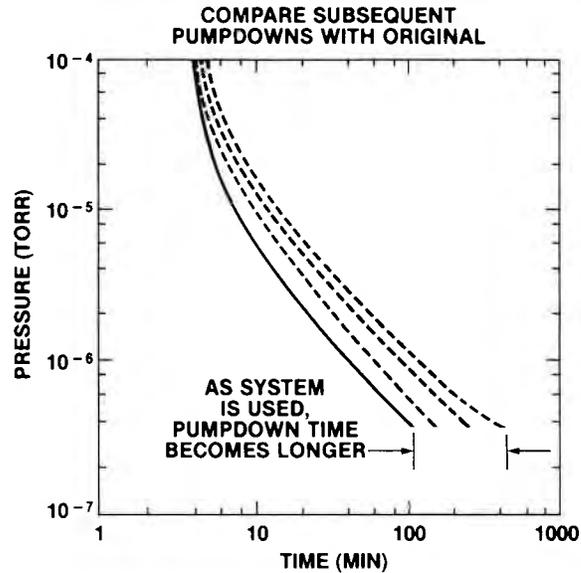
We have seen a variety of systems, but by no means have we seen them all – only the more common ones. Having compared and operated the various systems, let's next turn our attention to characterizing a system. It is necessary to determine how a system operates and how it behaves under various conditions. Recording this information in a log book will prove to be of great help when trying to determine problems with the system.

Rate of Pumpdown

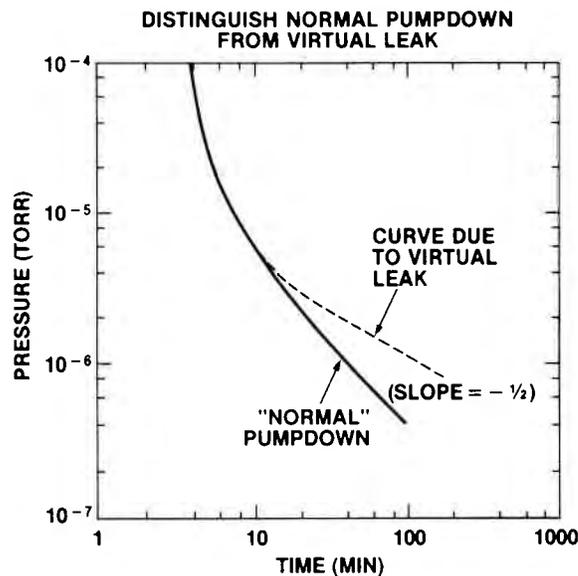
First, consider pumping time. This is necessary to establish the behavior pattern of the system.



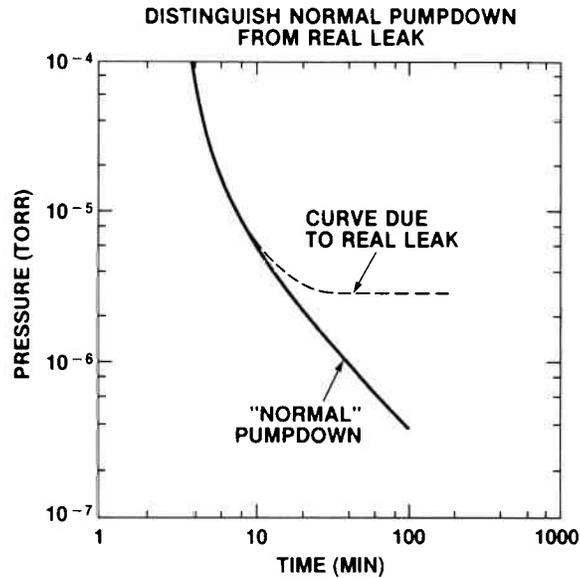
A pumpdown curve should periodically be recorded and filed for the system. The curve shown here is a plot of the inlet or system pressure on the vertical axis against time. This shows how fast the system pumps down to a base pressure. The above curve is for a typical 25-inch diameter chamber system. The system should pump to 2×10^{-6} torr in 20 minutes, and 2×10^{-7} torr in 12 hours. Once the pumpdown characteristics of a system are determined, a basis for comparison will have been established.



Having recorded and filed the pumpdown curve for a system, it is possible to compare subsequent pumpdowns with the original. Note that as the system is used, pumping performance will naturally degrade as the various process materials build up on the chamber surfaces. This will show up as a shift to the right on the pumpdown curves. The natural shift to the right is very helpful in scheduling downtime for cleaning purposes. Notice, however, that the shape of the curve remains basically the same as the original, even though pumpdown time takes longer with each subsequent use.



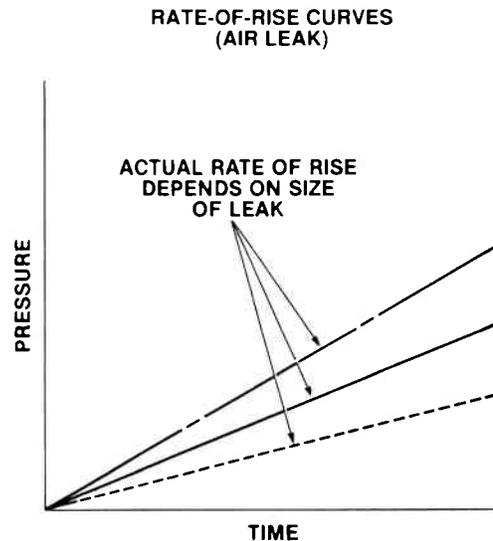
The pumpdown curves are also very useful for distinguishing normal pumpdowns from problem pumpdowns; in this case, from a problem due to a virtual leak. Notice how the normal pumpdown curve is compared against a virtual leak pumpdown. Notice how the virtual leak pumpdown curve shifts rather dramatically,



Pumpdown curves also help to distinguish normal pumpdowns from those under a real leak situation. The reason that the pumpdown curve flattens out, as shown by the dotted curve, is that at some point in the pumpdown, the incoming air through the leak exactly matches the pumping ability of the system. Therefore, the gas being expelled is exactly matched by the amount of gas being introduced through the leak. Another way to look at it is that when the pumping speed of the pumping mechanism is equal to the gas load of the system (Q), it can't reduce the system pressure any further.

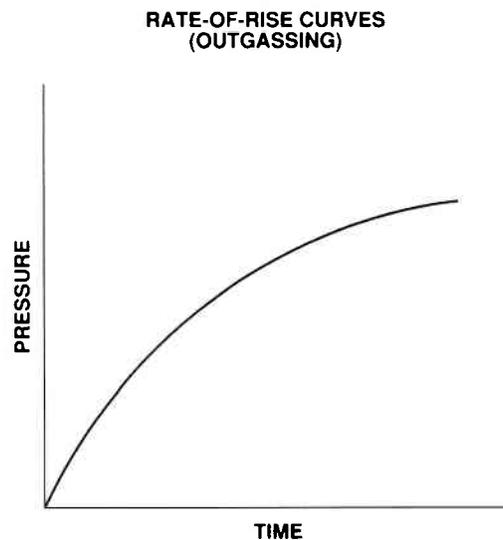
Rate of Pressure Rise

Another useful set of curves is the rate-of-rise curves. These curves are developed by pumping the chamber down, then valving off the chamber so that no further pumping occurs. At that point, the pressure in the chamber will naturally rise with time. The rate of rise, and the changing rate of rise, can be useful in determining the cause of the changes. This pressure increase is from two major factors: the normal time and temperature-dependent wall outgassing, which the pumps are expected to remove; and the gas load entering the system through real leaks from outside the system.

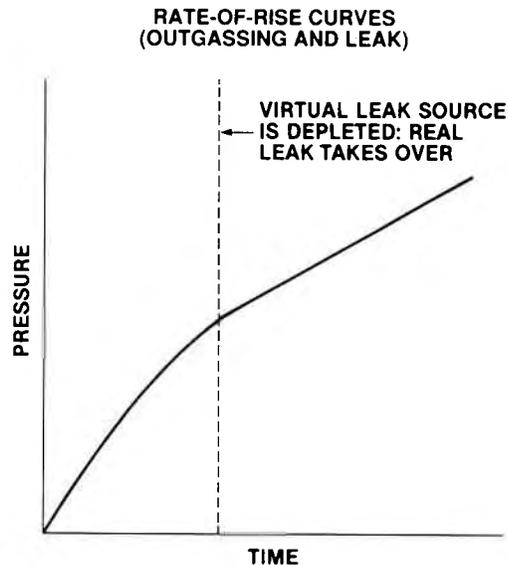


Plot the pressure on a vertical axis, and the time on the horizontal axis. To be most useful, it is best that both axes be on log scales. After pumping the chamber down to some low pressure, it is valved off or isolated from the pumping mechanism. Then the pressure is monitored as it naturally rises with time. As with the pumpdown curves, it is a good idea to establish a set of rate-of-rise curves with a system that is generally considered to be vacuum-tight in order to set up a basis of comparison.

The rate of rise of pressure, of course, will vary, depending on a number of factors. The rate of rise can be affected by the chamber volume and the amount of contamination that exists in the chamber. It can also be affected by leaks and the size of leaks. The set of rate-of-rise curves shown here indicates that the rate of rise definitely will depend on the size of the leak. Obviously, the larger the leak, the faster the pressure will rise in the chamber after the chamber is valved off from the pumping mechanism. We can distinguish between a real leak and a virtual leak.



The rate-of-rise curve shown here is due to outgassing and/or trapped volumes. Again, the pressure rise is plotted against time on log-log paper. With an air leak, we saw that the rate of rise for pressure was linear with time. That is, it rose at a steady rate. When severe outgassing or trapped volumes exist, the curve is distinctly different. In this case, the pressure rises quickly, depending on the amount of outgassing due to contamination, or due to a virtual leak. Then, as the source of gas causing the rise in pressure reaches equilibrium, the rate of that rise slows down. That is, although the pressure may continue to rise, it no longer will rise at the same rate, as seen in the plot.



The rate of rise in pressure due to a virtual leak plus a real air leak shows up as a combination of the last two plots. That is, the rate of rise due to outgassing from contamination or through a source of trapped gas will vary with time. Then, as the source of outgassing reaches equilibrium, the rate of rise due to a real leak will become linear. This is because the amount of air entering the system through a real leak does not vary with time, but instead is constant. Therefore, the rate at which the pressure rises with time from that point is also constant.

Summary

We've looked at the advantages and disadvantages of the major types of vacuum systems and learned about putting together an operating system. We've also learned how to characterize a system through the use of pumpdown and rate-of-rise curves. Now let's take a look at how to troubleshoot vacuum systems.

8

Troubleshooting

When you have completed this chapter, you will be able to:

1. Identify the most common faults in vacuum system components.
2. Check out problems in common vacuum pumps.
3. Troubleshoot common vacuum system problems.

Introduction

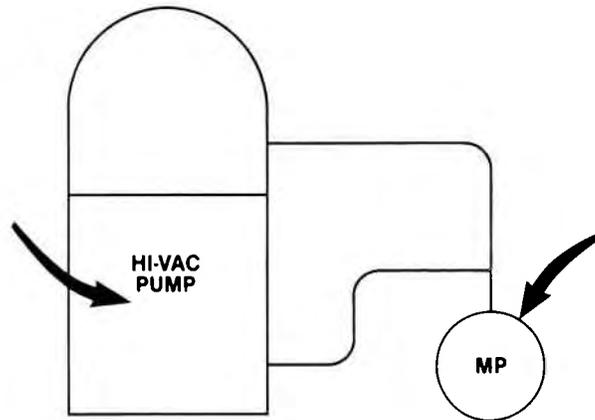
Next, let's look at troubleshooting a faulty vacuum system. Of course, we recognize that any equipment will, from time to time, be plagued by one problem or another. We also recognize that the solutions to many vacuum-related problems can be rather hard to pin down.

Vacuum malfunctions have a variety of symptoms. Some problems have overlapping symptoms, which increase our difficulties. We'll try in this chapter, therefore, to provide a guide for a systematic approach to determine the cause of vacuum system problems.

Categories of Faults

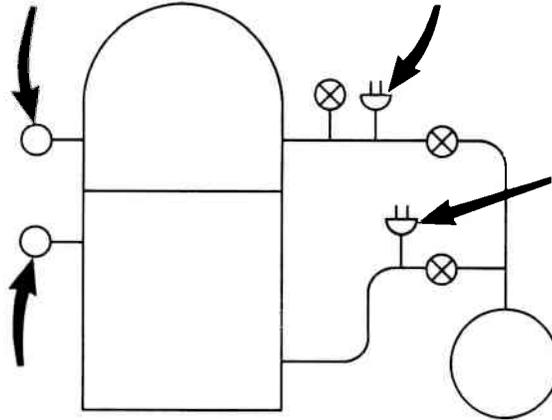
When a problem occurs in a vacuum system, it is often best to approach the situation in a systematic manner. This is better than trying to resolve the problem by any means that may occur to us at random.

For purposes of our discussion here, therefore, let's start by identifying the types, or categories, of faults that occur in vacuum systems. After we have identified the major categories, we will consider the various elements in greater depth.



Vacuum Pumps

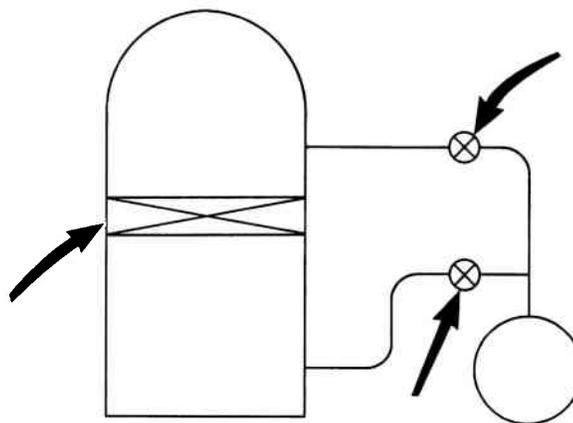
We need to consider the vacuum pumps. These mechanisms are the devices needed to create the vacuum environment. Therefore, if they are malfunctioning, our environment may be less than ideal, if not completely unusable.



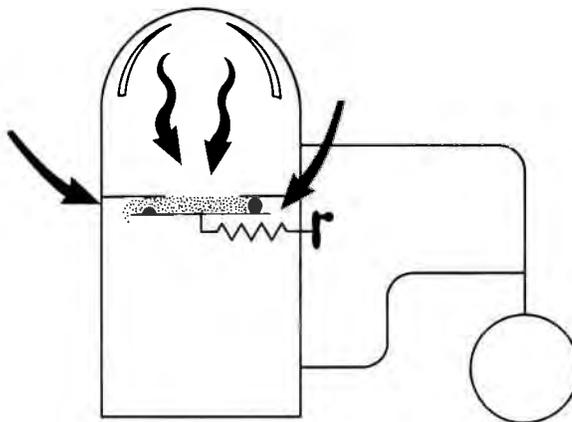
The first indication of a system malfunction is provided by the pressure gauges. However, we must also recognize that the gauges themselves can malfunction, either failing completely, or giving us false information as to the system status. Failure to recognize this fact can lead to the wrong conclusion.

Hardware

Faulty hardware can be the source of many vacuum system problems. Hardware can malfunction in a variety of ways, or can be affected by the condition of the system.

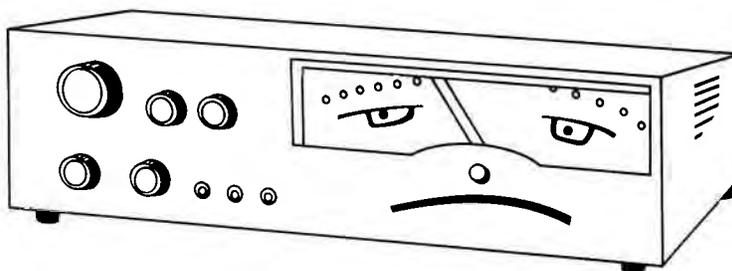


Valves can malfunction. Wear, caused by high friction forces, can prevent proper sealing. The seals wear in time. The actuating air pressure on pneumatic valves may be too low to provide proper sealing. Solenoids may be faulty.



Debris, particularly at the valve seals, is quite often the cause of system failure. Bellows or other feedthrough devices can leak. Double O-ring shaft seals can be the sources of virtual leaks.

Control Units, Gauge Tubes and Connections

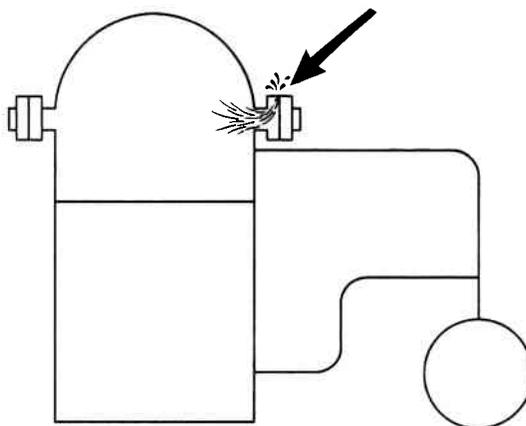


Control units, like gauges, can malfunction, or go out of calibration, giving erroneous data. Cable terminations and other interconnections can become loose or intermittent. Automated equipment may have faulty hardware or software.

Gauge tubes can become contaminated by pump oils and other materials. This may produce false pressure readings.

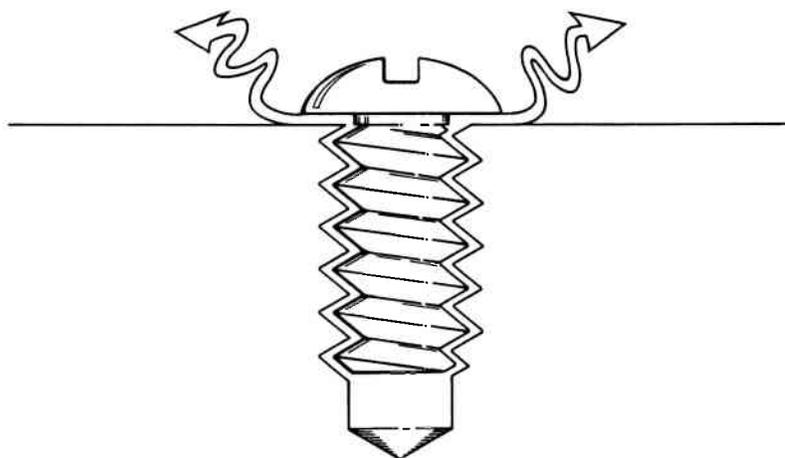
Leaks and Outgassing

Air Leaks

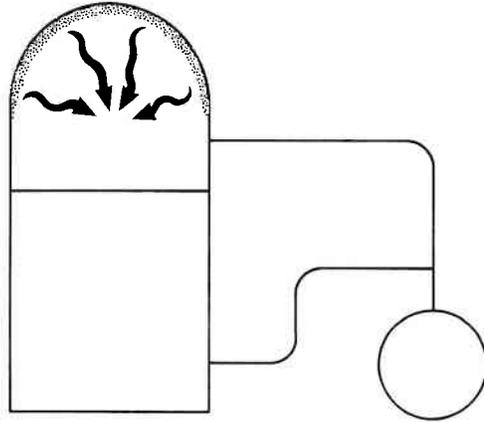


Leaks certainly are a major reason for system malfunctions. Air leaks are not uncommon. If small enough, leaks of this type can be ignored in many processes. (We'll consider sizes of leaks, or leak rates, in the Leak Detection chapter.)

Virtual Leaks

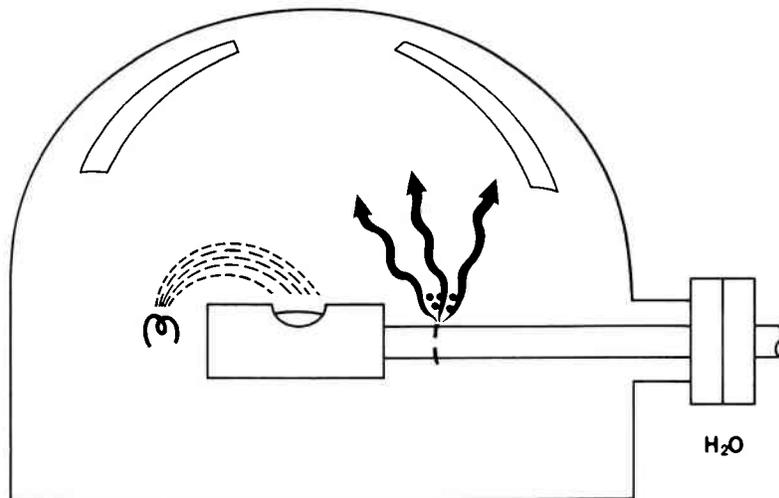


Virtual leaks, which we have already discussed, can be the cause of excessively long pumpdown times, if not complete system failures.



Outgassing, which may be considered to be a virtual leak, can also cause long pumpdown times. In production systems, long pumpdown times reduce the amount of work that can be produced by the system. Outgassing can also seriously affect the quality (yield) of the work. Simple outgassing can be often corrected by systematic, scheduled cleaning procedures.

Water Leaks



Water leaks inside the vacuum system, even very small ones, are always serious. And they often are hard to find. If small enough, their damage can be insidious, resulting in yield problems that might not be discovered until later in the manufacturing process. Additional expensive effort must then be expended on work pieces that were previously made useless by the water leak. The probability that leaks of this type will occur is increased by the number of water-cooled devices in the work chamber. Remember that water can exist as a gas, so you might not “see” a water leak! Silicone gaskets and O-rings are permeable to water vapor and can be a source of water in the vacuum system.

Utilities

The plant utilities such as air, water, electricity, process gases and liquid nitrogen can cause problems because of pressure variations, voltage, purity and temperature.

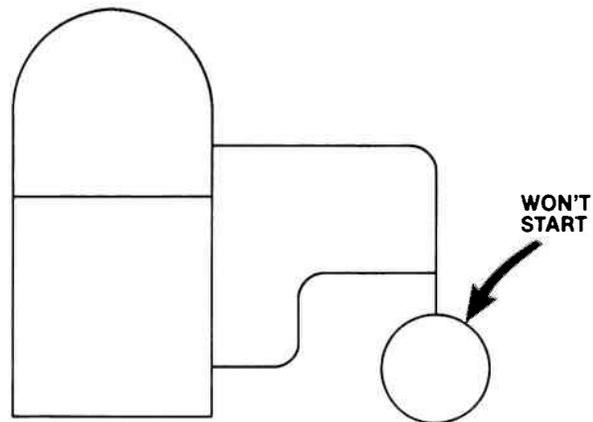
Vacuum Pumps

We have made a broad listing of several possible problem areas. Now, let's consider the various categories in somewhat greater detail, starting with the vacuum pumps.

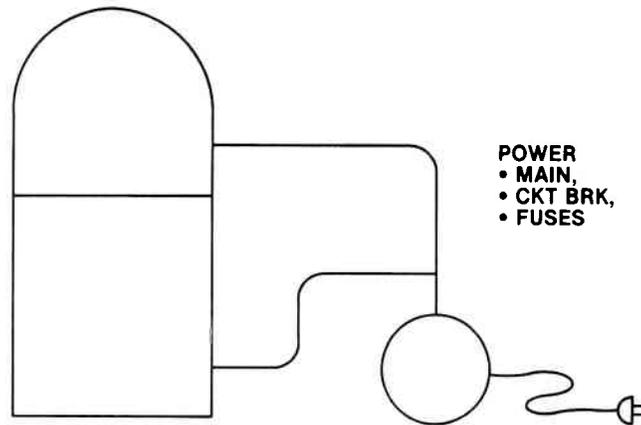
Mechanical Pump

We'll start with the mechanical roughing pump. These pumps are simple, but can be plagued by a number of problems which, as we have already said, can show up in a variety of ways. Let's look at a few of the ways.

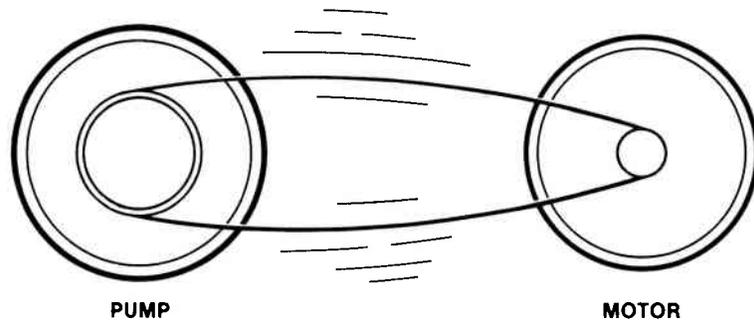
No Start-up



Start with the simplest things first. Consider a gross problem: the mechanical pump simply doesn't start when "turned on."



Check the easy things first. Is power arriving at the unit? If not, possibly a circuit breaker has opened. If the system is equipped with fuses, they may be burned out. Also, consider loose or broken connections. A pump or drive motor might be frozen, or seized. This condition might show up as a characteristic whining or buzzing sound as the unit struggles to turn, as overheating, as odor, or as all three of these symptoms.

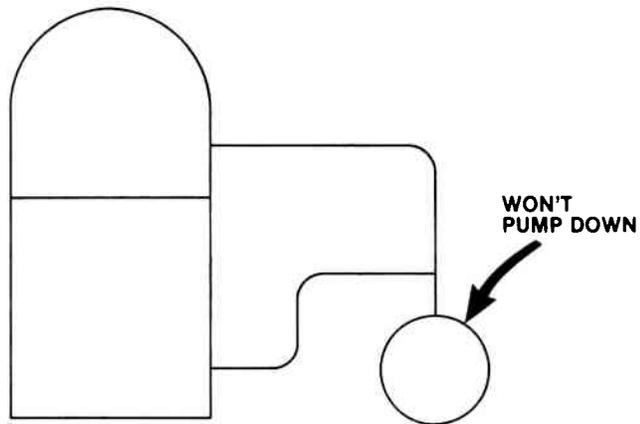


A very loose or broken drive belt can prevent a mechanical pump from operating at all. A loose drive belt might be heard slapping against the belt guard. On a single-belt drive, a broken belt will bring the pump operation to a halt; a broken belt in a multiple-belt drive unit may not be so obvious. A loose belt may not be turning the pump module at the rated speed, thus causing longer pumpdown times.

When the mechanical pump is connected to the foreline of a diffusion pump, a complete shutdown of the mechanical pump will very quickly lead to a catastrophic failure, unless the foreline pressure is monitored and action taken, or automatic shutdown is started by an interlock system.

Poor Operating Pressure

A less obvious mechanical pump problem occurs when it's impossible to reach the appropriate operating pressure normally reached by a given pump.



Contaminated Oil

Contaminated oil can result in poor operating pressure. Contamination can be caused by condensed water vapor, solvents or gases introduced into the system during the process. Or, it could also be due to failure to change the oil at the appropriate intervals.

Visually inspect the oil through the sight glass. Does it look cloudy, milky or streaky? If so, these conditions help to confirm that contamination exists. Remember to use the gas ballast if water is a problem.

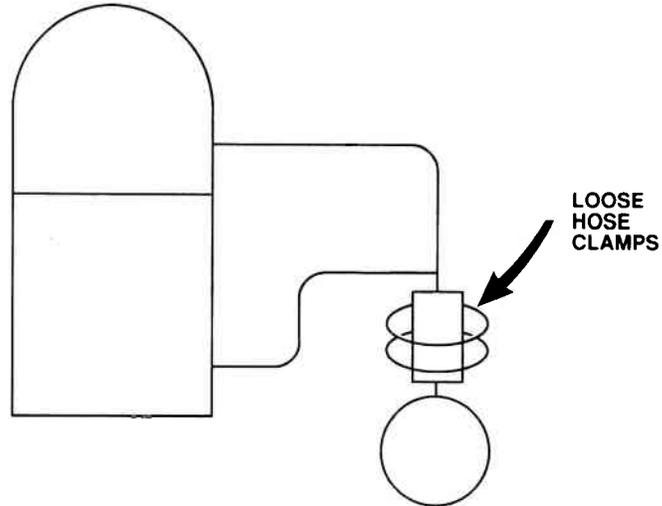
The statement has been made that 95% of mechanical pump problems can be solved by changing the pump oil.

Loose Drive Belt

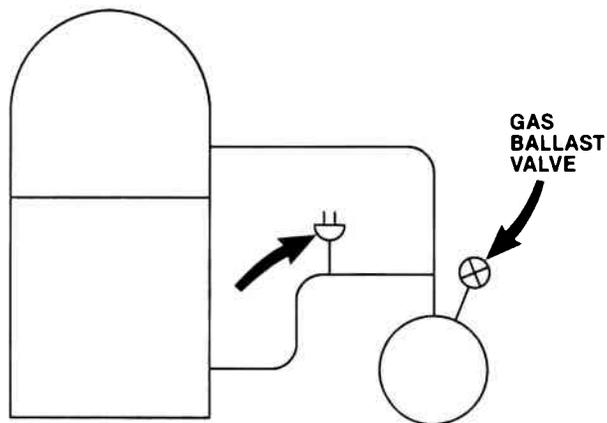
Poor operating pressure can result from a loose drive belt. Belt tension can, of course, be adjusted. Frayed belts should be immediately replaced. When replacement is indicated in multiple-belt drives, all belts should be replaced together, regardless of the condition of any one of them. Preferably, the replacement belts should be a matched set. In direct-drive pumps, coupling problems between the motor and the pump module seldom occur.

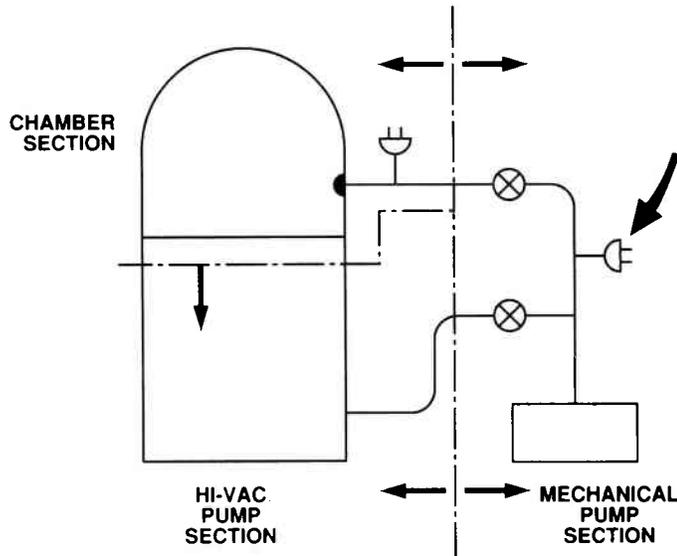
Hoses

Hose clamps may have worked loose due to pump vibration. (In small work areas, bumping against the hose connections may loosen them.) Hoses may deteriorate, particularly in hot environments. Annual replacement of hoses is good preventive maintenance.

**Open Gas Ballast Valve**

An open gas ballast valve makes it impossible to achieve normal base pressure. The gas ballast valve may have been opened to prevent water vapor condensation, or to clear the pump of water contamination, then left open unintentionally. This can often happen in a multiple-shift operation: someone on one shift may open the gas ballast valve, and the person on the next shift may be unaware that it was left open.



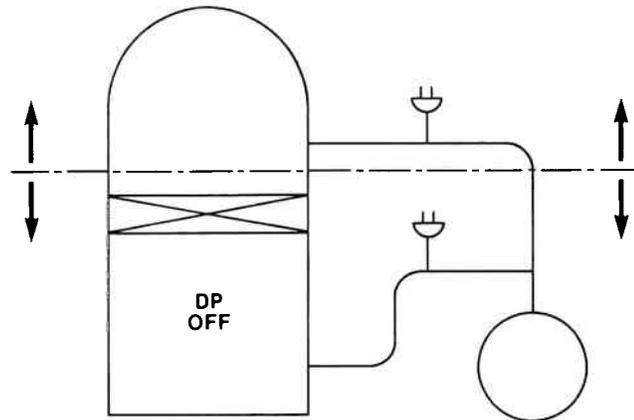


Use of valve isolation and pressure readings to identify cause of poor base pressures.

Measuring Base Pressure

A quick way to establish the base pressure of the mechanical pump is to isolate it from the rest of the system. This can be done by closing the roughing and foreline valves, and reading the pressure with a manifold TC (roughing) gauge. If a manifold TC gauge is not built into the system, the roughing valve can be left open, and the chamber roughing port can be plugged. Then the pressure can be monitored with the regular TC (roughing) gauge, if no leaks exist in the valve or manifold.

Isolation into sections by this technique also helps to establish whether the problem is in the high vacuum portion of the system, or in the roughing side. Keep in mind that manifold or valve leaks can produce abnormally high pressures.



Hi-vac valve isolation technique.

An isolation check shown in the previous drawing that is more time-consuming is one that involves turning off the diffusion pump (allow it to cool). By appropriate valving, check the mechanical pump roughing capability in both the diffusion pump and chamber sections.

If the pressure is significantly higher on one side, that section may have a leak. (A contaminated TC gauge, particularly in the fore-line, may be the cause of a substantially higher reading in that section!)

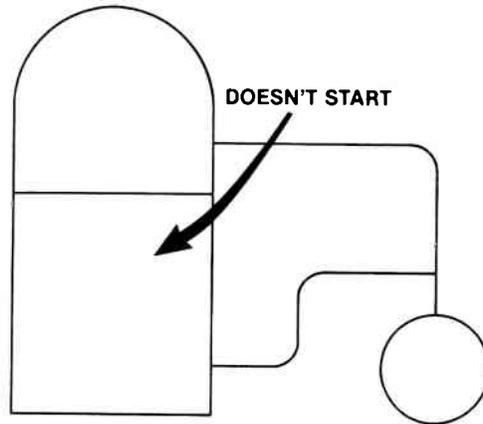
If both sections are high, the oil may be contaminated, so visually inspect again. A new change of oil may be indicated. Also, more extensive mechanical pump maintenance may be required.

Diffusion Pump

Next, let's consider the diffusion pump.

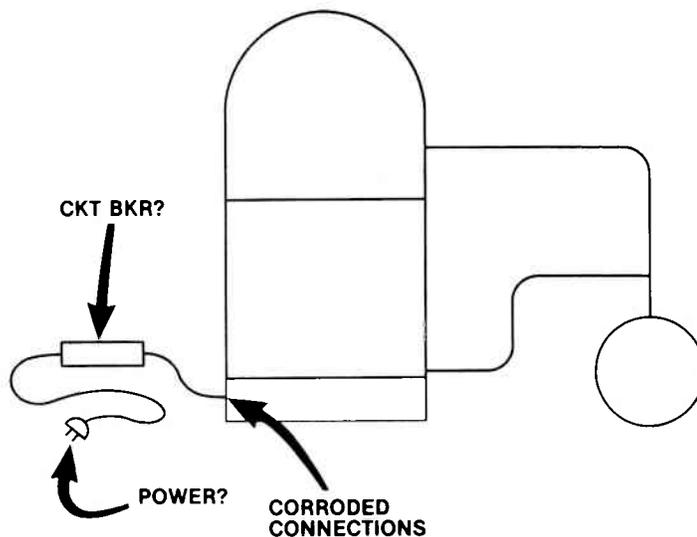
No Start-up

Again, let's start with the most obvious problems. For example, the pump doesn't start.

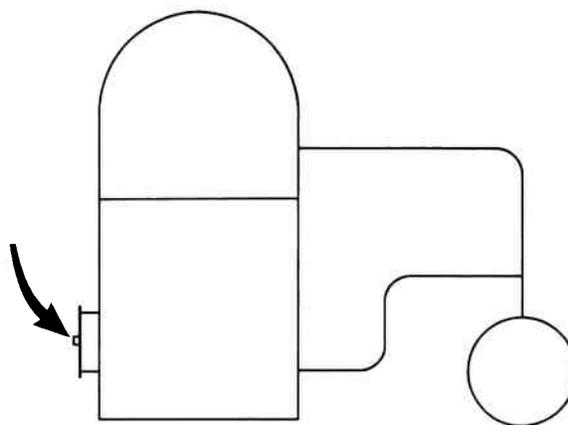


Main Input Power

Is the pump attached to a power source? How about open circuit breakers? Broken or loose connections are often sources of power failures.

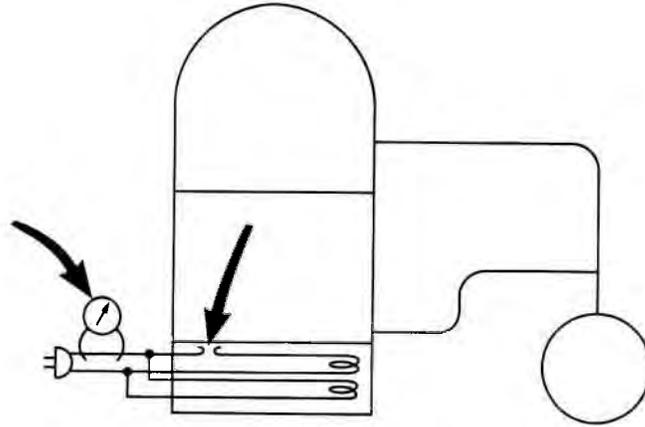
**Thermal Protect Switch**

The thermal protect switch may be open. Keep in mind that the protect switch may have opened for a very good reason. Therefore, before resetting it (providing the pump is thoroughly cold), check the fluid condition and level. If not thoroughly cool, it may even be a good idea to wait for cool-down to make these checks. Many pumps have a quick-cool water coil wrapped around the boiler to speed up the time required to cool the diffusion pump. Be sure to drain the quick-cool coil before starting the pump. Do not install a valve on the outlet of the quick-cool line. It should always have access to an open drain.



Heater

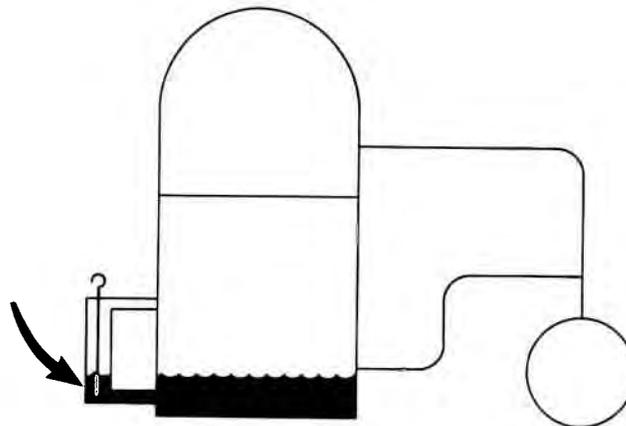
A burned-out heater in a single-element heater design will prevent pump warm-up. However, an element that burns out in a multiple-heater pump may not be so obvious. Reduced performance may cause one to look elsewhere for the source of the problem.



A clamp-on ammeter will establish whether the correct current level is being delivered to the pump. Although an ohmmeter resistance check might seem easier, particularly on single-element heaters, an ohmmeter check can overlook power-robbing, corroded terminals. Resistance is a poor indication of heater condition except for an open heater circuit.

Poor Base Pressure

Next, let's look at some of the problems that can result in poor base pressure in a diffusion pump system.

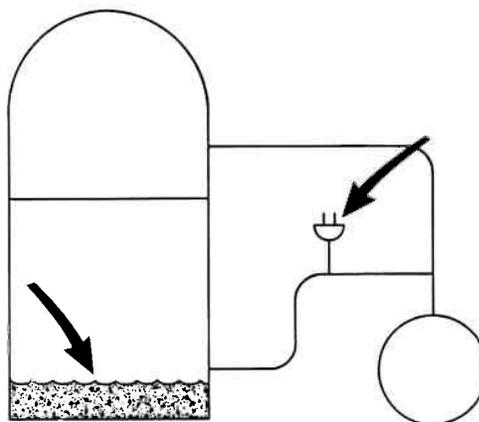


Fluid Level

The fluid level may be low. *Reminder:* Do not check the level while the pump is hot. Also, notice the color of the fluid (wipe dip stick on clean lint-free paper). If safe to do so (not on ion implanter systems or any vacuum system using toxic gases), smell the fluid. A burned smell or very dark-colored fluid indicates a need to clean the diffusion pump and replace the fluid. Always replace the fluid fill O-ring with a new one. It is located in a hot zone, and will have taken a set that will almost surely cause a leak if re-used. Use only the recommended O-ring material that can withstand the high temperatures at this location.

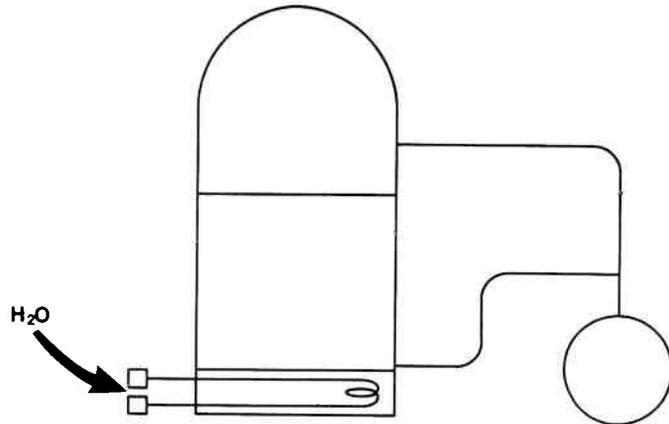
Contaminated Fluid

How about contaminated diffusion pump fluid? Besides affecting the ultimate pressure capability of the pump, this also sometimes shows up as a rapid pressure rise in the foreline when the foreline valve is closed, and before the vent valve is opened, particularly when severe contamination exists.

**Water in the Quick-Cool Coil**

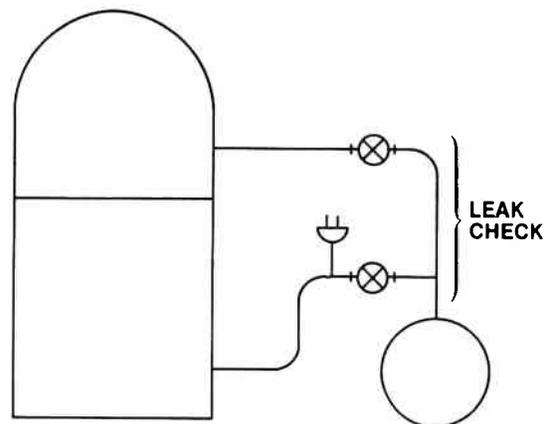
Water in the quick-cool coil? This problem might follow pump maintenance. If during maintenance, water was not drained from this coil, it could cause slow pumpdown until the water boiled away. (If there is no way for the resulting steam to escape, a

dangerous condition exists.) If water is left running, the pump will never warm up sufficiently. To minimize this problem, you could use compressed air or nitrogen to cool, rather than water. *Remember:* No valves on the outlet of the quick-cool coil. The line should always be open.



High Forepressure

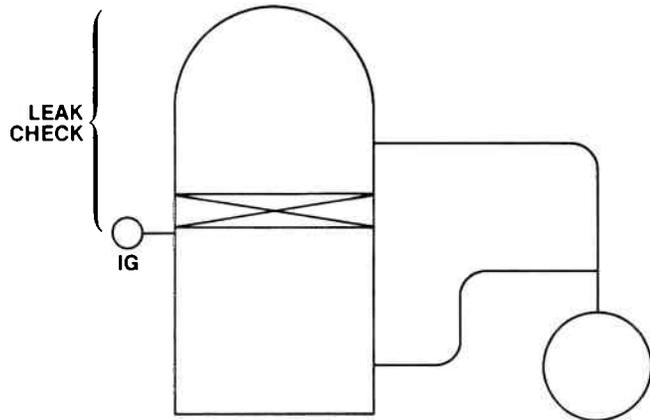
Excessively high forepressure can be caused by a problem in the mechanical pump. Also, it is a good idea to check the foreline area for leaks, as well as the entire roughing manifold. Don't forget that dirty mechanical pump oil can also cause high foreline pressure.



High Inlet Pressure

This is, of course, a familiar enough problem, and it can be caused by a number of troubles. Again, the best approach is to

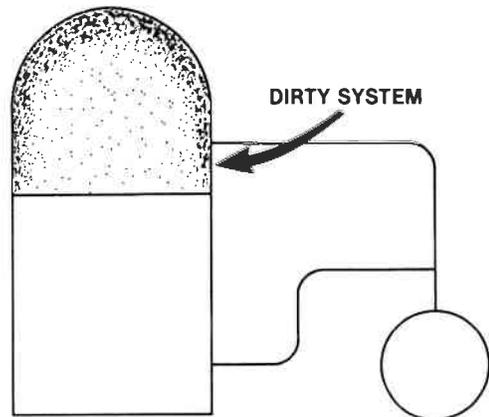
isolate the problem area from the rest of the system as much as possible. From that point, the obvious next approach is to leak-check both halves of the system.



However, the actual problem may not be so simple to solve. Leak checking is one approach to the problem; there are others. A good pressure reading below the closed high vacuum valve might eliminate that part of the system as a problem area.

Contaminated System

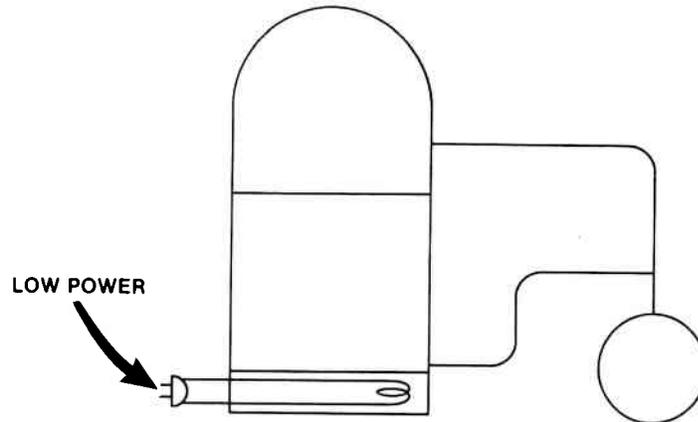
A number of things could be the cause of the problem above the high vacuum valve. For example, a symptom of a dirty system could be an inability to reach proper pressure. We have already discussed pumpdown curves and rate-of-rise curves. This information can be used to help guide us in our approach to the problem.



This doesn't mean that we always need to take exhaustive, time-consuming tests before we decide which approach to take. For example, a systems rate of rise need not be more than a simple "eye-ball check" taking no more than a few minutes. This information, together with a record of the cleaning schedule, can be a great help in determining the cause of a problem, particularly when it appears to be isolated to the upper half of the system.

Low Power

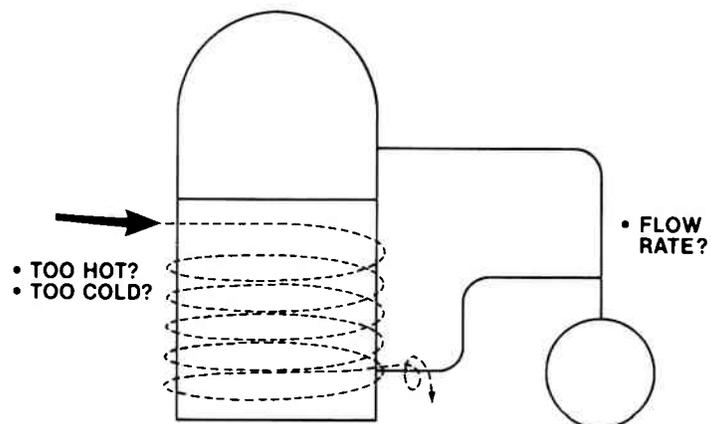
Poor base pressure performance isolated to the bottom half of the system can also have a number of causes. For example, low input power. A voltage check might quickly pinpoint the problem. A clamp-on ammeter check might give even better information. A single burned-out element in a multiple-element heater can be found with an ammeter. Also, inadequate heating due to corroded terminals can cause poor pump performance.



When replacing heating elements, be sure to tighten the elements, the clamps, and the crush plate to provide good intimate contact and good heat transfer. Also, make sure that the proper heating element is used. Check the voltage rating on the replacement element before installing it. If a crush plate is used, replace it with a new one when you replace the element. If wires or wire lugs must be replaced, they must be the nickel wire and lugs provided by the pump supplier.

Water

Diffusion pump cooling-water temperature and flow rate are important. If the pump cooling-water discharge temperature is too hot (above 130°F, 55°C), it is not performing its cooling function properly. On the other hand, when the water is too cold, the fluid may become very viscous and not return to the boiler fast enough.



Most modern industrial high-production systems are equipped with water flow-rate meters. These flow-rate meters are adequate for most situations. However, they do not answer all questions. For example, most diffusion pumps have water temperature range specifications. And, they have a flow-rate specification.

The best water check, however, is made at the output on the foreline. The output water temperature should range between 110°F and 130°F (45°C and 55°C). If it is not within the proper temperature range at the output, it is not doing a proper job.

Random Pressure Spikes

Poor pumping performance can be caused by a low fluid level. This may show up as slow pumpdown and poor base pressure. Look for random pressure spikes as a further clue to this condition; there may not be enough oil in the reservoir to supply a steady stream of oil out of the jets. The low fluid level may cause poor heat transfer, which in turn may cause overheating and uneven boiling. Random pressure spikes can also be caused by leaky O-rings in the fill-drain tube.

Slow Pumpdown Following Cleaning

Slow pumpdown following cleaning is to be expected. This is due to a number of factors. Residues of the solvents used for cleaning the equipment will outgas for a period of time. Even with the best of vacuum practice, human oils will contribute to the vacuum environment; even lint-free gloves cannot completely eliminate body oil contamination. Finger cots only protect against the oils produced by the finger tips (not the rest of the hands).

Excessively long pumpdown periods also might be due to incomplete cleaning, incomplete rinsing, and incomplete drying. Pay special attention to grooves such as O-ring grooves and leak-check grooves. Even the mechanical pump and diffusion pump fluids which are used to produce the vacuum require a period of time to outgas.

Let's go on now to look at vacuum system problems. Note that by changing the words from diffusion pump to cryopump or turbo pump, we find that many of the comments here also apply quite well to other types of vacuum systems.

The Vacuum System

The next category of problems we will consider relates to the entire system. We will also suggest some ways to help with troubleshooting.

Record and Monitor Performance

A record of normal system performance will establish a basis for comparison against abnormal performance. Abrupt changes from normal are immediately apparent; gradual changes indicate a need for routine maintenance such as cleaning.

As indicated earlier, pumpdown and rate-of-rise curves can be very valuable. When a problem occurs, compare the curves with those previously taken when the system was working normally. We have already discussed how these curves can be used to distinguish between the types of problems.

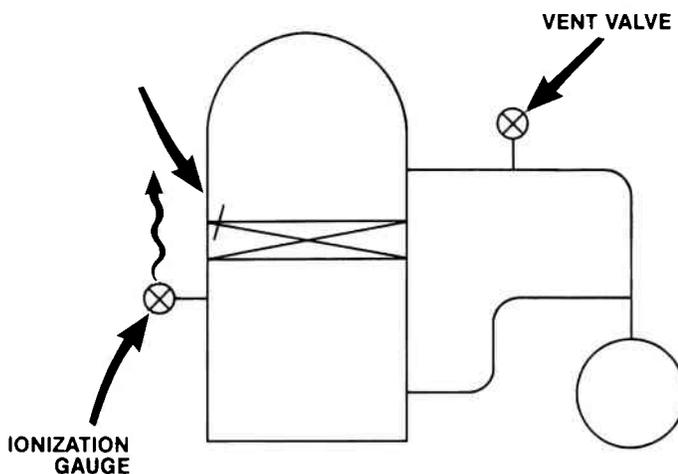
Here's another use for these performance curves. When a problem is definitely indicated, try pumping down twice. Pump down to some low point and record the remainder of the pumpdown from that point. Call this portion of the pumpdown T1. Valve off the suspected portion of the system, allowing the pressure to rise naturally to the point where the period T1 started. Then valve in that portion of the system again and pump down again.

How long did the second pumpdown take? If time T2 is approximately equal to time T1, this would indicate that the problem might be due to an air leak. This is because the source of gas is constant, indicating an unvarying physical leak.

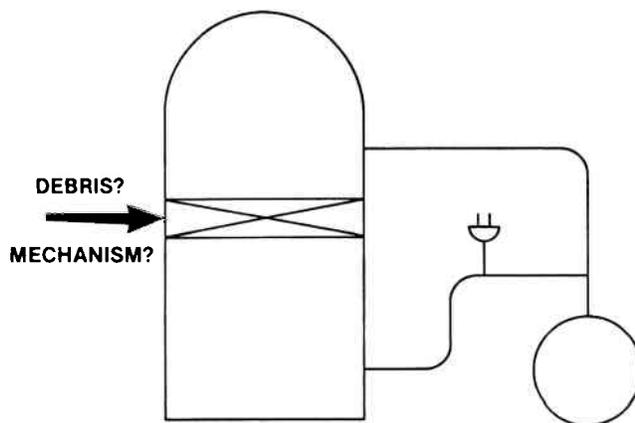
If the test resulted in time T2 being about one-third to one-fifth the pumpdown period of time T1, then this is probably indicative of a virtual leak or an outgassing problem. The reason for this is that during the first pumpdown period, much of the gas load, including the virtual leak and outgassing, was removed. Then, during the second pumpdown, the pump didn't have as large a load to handle, and therefore was able to pumpdown considerably faster.

A similar test using “rate-of-pressure rise” can be done in conjunction with the pumpdown test. If the timed rise in pressure after a volume has been isolated is always the same, an air leak probably exists. If the time for the pressure to rise the same amount becomes longer for each subsequent test, outgassing or a virtual leak is indicated.

High Vacuum Valve Sealing Problems



If the pressure goes up (indicated on the ion gauge below the high vacuum valve, if you have one) when the system is vented, this is an indication of a high vacuum valve sealing problem. The pressure should not rise even slightly. There could be dirt, debris, hair on the seal, improper adjustment of the high vacuum valve, or insufficient sealing pressure. The foreline TC gauge can also be used for making this check; however, it is not as sensitive as an ion gauge below the high vacuum valve.



After checking for seal problems, check for sealing mechanism problems. Pump the chamber to around 1,000 mtorr. (The high vacuum valve, of course, is still closed.) Now, if the pressure in the gauge below the high vacuum valve or the foreline TC gauge pressure goes down, a sealing mechanism problem is indicated. The reason is that the reduced pressure above the high vacuum valve now allows the sealing mechanism to affect a proper seal. Caution! This check, like many others, is not necessarily 100% perfect. However, it is included here as an aid in solving system problems.

Problems After Cleaning

After a major system disassembly for cleaning or other maintenance work, do not attempt to pump the system to high vacuum immediately. Instead, use the roughing pump to pump the entire system with the diffusion pump off. Monitor the pressure to check for outgassing and/or leaks. Remember that long-term roughing may allow oil vapors to enter your newly cleaned system!

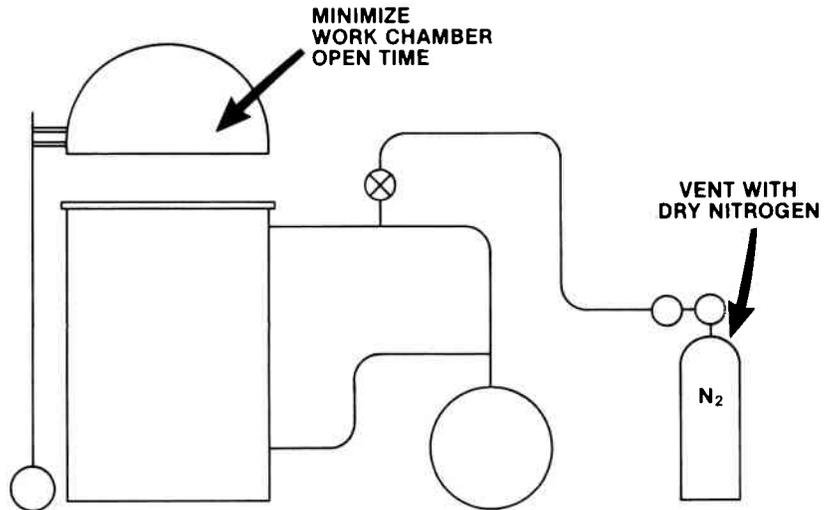
During system reassembly, an O-ring could have worked loose or twisted in its groove, causing a gross leak. If the diffusion pump were to be turned on under this condition, the entire purpose of cleaning the system could easily be defeated. What's more, the diffusion pump fluid could be ruined.

Instead, if the roughing pump cannot evacuate the system, the problem is indicated without risk to the diffusion pump. However, keep in mind that there should be an allowance for outgassing. Residual solvents, fingerprints, and the pump oils themselves must be given a reasonable outgassing period.

General Tips

Now let's consider a few ideas that will help to minimize problems before they begin.

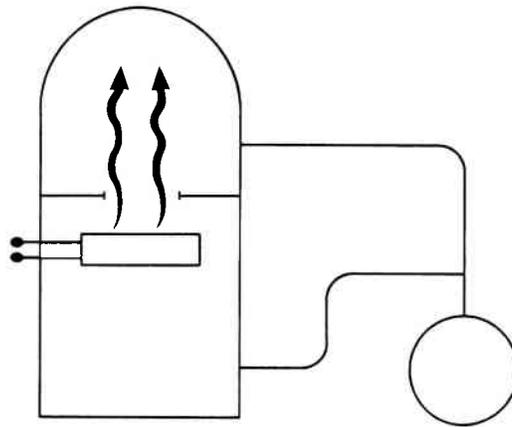
Minimize Work Chamber Open Time



The time that the work chamber is left open and up to atmosphere should be reduced as much as possible. Five minutes is not unreasonable. When many work pieces are processed per cycle, they generally are mounted on fixtures of some kind. By having spare work-piece holders on hand, it is possible to greatly reduce the chamber open time.

It is also becoming common practice in high-production vacuum processing systems to vent the work chamber to a dry gas, usually nitrogen, instead of exposing the work chamber to atmospheric air (which, of course, contains water vapor). When possible, also store loaded fixtures in a dry nitrogen atmosphere.

Keep Cryotrap Filled



The liquid nitrogen trap should never be allowed to go dry while the high vacuum valve is open. This is sometimes mistakenly practiced on weekends when the system is on standby. However, as the LN₂ trap warms up, it releases all the trapped gases, particularly water vapor, that it had captured during the work week. A significant amount of water vapor and other contaminants are then free to go back into the work chamber.

Rough Below Normal

When a subtle, difficult-to-define problem exists, it may pay to try roughing to well below the normal crossover point. This is because if a small leak exists, it is possible for the combined action of the diffusion pump and the mechanical pump to disguise the situation. Now if the mechanical pump is unable to pump the chamber down, the problem area might be somewhat more clarified.

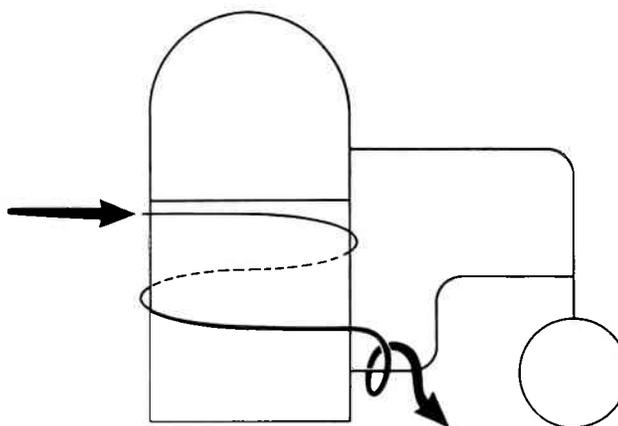
However, it should be kept in mind that a number of factors might cloud the issue. The mechanical pump itself may not have the capacity to reduce the chamber pressure very far below the normal crossover point. Its oil may be slightly contaminated. The roughing lines might be conductance-limited. The chamber walls might be severely contaminated. (Make a visual inspection.) When roughing below normal, be sure to trap the mechanical pump!

Check Plant Air Pressure

What is the air pressure in the line for the pneumatically-actuated valves? A common enough problem occurs when the plant facility air pressure is not high enough to do the job. Watch out for dirt and water in the house or shop air lines. Use and maintain air line filters and traps.

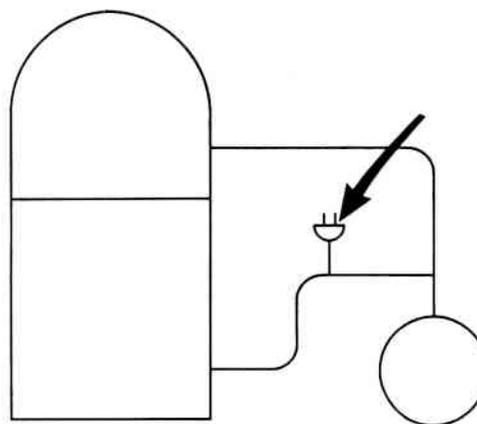
Note also that a given utility, although adequate as originally designed, may not be able to keep up with the demand of increased work loads.

Check for Hard Water



How is the water in your facility? Hard water is full of minerals which, when deposited over a long period of time, can plug (or partially plug) the water lines. This defeats their cooling function. Thirty-five percent muriatic acid and water flushed through the water lines will help to unplug them. Do not use deionized water! Most facilities today use a treated, recirculated water supply. Water filtration and treatment may be a good investment if your area has water problems.

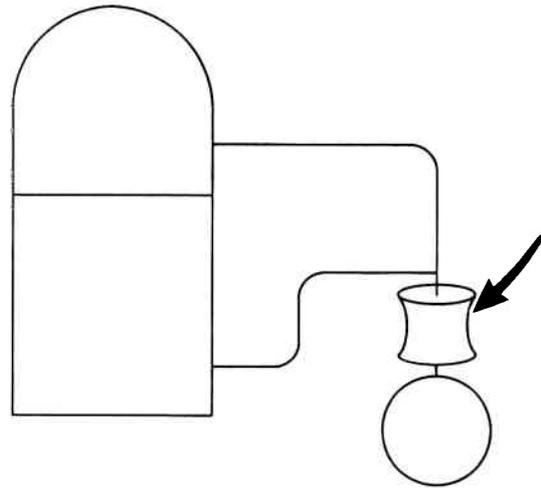
Check Foreline TC Gauge



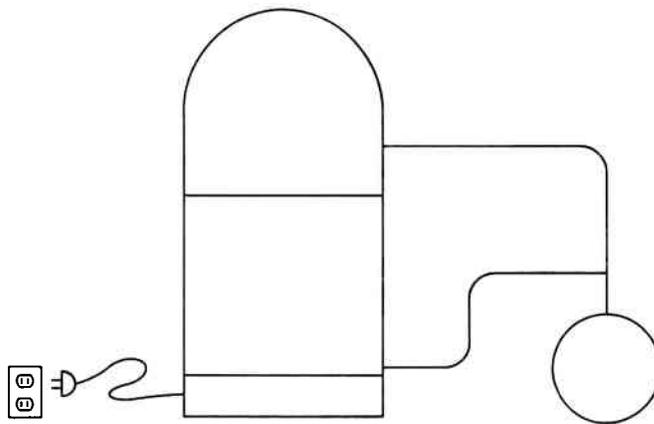
It should be recognized that a foreline TC gauge is especially subject to oil contamination. Therefore, it is more apt to fail or give incorrect readings sooner than TC gauges located in other parts of the system.

If necessary, the TC gauge can be cleaned with acetone or Freon TF, rinsed with alcohol, then dried with dry air. Keep in mind that the TC gauge may have to be readjusted. This will serve as a temporary remedy if a replacement is not immediately available.

Check Hoses



Hoses should be used as convenient couplings, not as long tubulations. In any case, the hose walls should be thick enough so that they will not collapse under a 15 psig pressure differential. When kept in inventory, hoses should be capped or kept in plastic bags so they will not soak up water vapor during their shelf life. Tygon is generally recommended. Also, overly tight hose clamps can cause leaks.



Check Diffusion Pump on Weekend Standby Operation

Another problem that often begins on a weekend is based on the mistaken belief that the system can be set on standby by turning off the diffusion pump and keeping the system evacuated with the roughing pump in the interest of economy, or energy saving. Unfortunately, most vacuum processes cannot tolerate the oil backstreaming from the roughing pump that occurs from this type of weekend standby. If you want to leave the mechanical pump running, close the valve between the pump and the system.

SOLVENTS FOR DIFFUSION PUMP FLUIDS

Remember that some solvents will be definite hazards to your health if you use them improperly. Always wear the proper protective clothing and use proper ventilation. Dispose of used solvents properly.

For hydrocarbon fluids such as Apiezon A, B, C, Litton Oil or Convoil, use acetone, followed by alcohol.

For silicone-based fluids such as DC-702, 703, 704 or 705, use Freon TF or hot 1,1,1-trichloroethane, followed by acetone, then alcohol.

For polyphenyl ether fluids such as Santovac 5 or Convalex 10, use Freon TF or methanol, followed by acetone, then alcohol.

For fatty ester fluids such as Octoil, Octoil-S, butyl phthalate, Amoil, Amoil-S or Invoil, use acetone, followed by alcohol.

Diffusion Pump System Troubleshooting

We've covered diffusion pump troubleshooting in the earlier part of this chapter. Refer to pages 228-35.

Ion Pump System Troubleshooting

If your ion pumps are slow to start (more than 30 minutes), look for the following:

- a. Air leaks
- b. Foreign materials such as molecular sieve pellets, glass beads and other materials

- c. Buildup of titanium compounds
- d. Pump let up to air while hot
- e. Pump approaching end of life

If your ion pumps are slow starting when using sorption pumps:

- a. Regenerate the sorption pumps. (Bakeout!) Check for poor conductance to the sorption pumps.
- b. Check for improper rough pump valving, roughing pump seal leaks, excessive exposure of Viton, or the system, to air.
- c. Check for pressure relief or vent valve leakage.
- d. (Large sorption pumps) A possible cause might be the re-evolution of condensed vapors into the system. Use a heat gun during roughing, or install an extra valve on-line near the system to isolate the roughing manifold.
- e. Check molecular sieve material for dark, glazed surfaces. Replace the sieve material.

In the case of short circuits, check for shorted anode support insulator and flakes. Replace parts; clean or replace the insulators.

If the pump power control unit goes off, check for air leaks. Look for cable or feedthrough arcing.

For pressure bursts, look for flakes and clean the pump.

A possible cause of high leakage currents is field emission caused by "whiskers." Hi-pot (10–20 kV AC at 20–100 mA) the pump.

TSP

Troubleshooting

If there is no increase in pumping speed when the sublimation rate is increased, a possible cause is an abundance of unsaturated titanium already available. Cycle on-off times for greatest efficiency.

In the case of pressure spikes during pumpdown, check for an expended filament or source. Switch filaments or replace source.

Cryopump System Troubleshooting

Here are some troubleshooting procedures for the cryopump. For detailed instructions, refer to your manufacturer's instruction manual.

If the compressor does not turn on:

- a. Is the switch on? If not, turn it on.
- b. Is the power connected? If not, connect it.
- c. Is the remote switch installed and on? If not, turn it on.
- d. Is the compressor charge low? If so, recharge the compressor.

If the compressor turns on, then off:

- a. Check the water flow to be sure it is not too low. If it is, correct it.
- b. Is the line voltage correct? If not, correct it.

If the compressor is on, but not the expander:

- a. Check the expander cord. Is it connected? If not, connect it.
- b. Is the expander motor wiring defective? Check for continuity.
- c. Check for a compressor power supply malfunction. If so, call the pump manufacturer's service department.

If the cryopump does not cool down:

- a. Poor vacuum in the pump? Check for leaks in the vacuum system. If you find a leak, fix it. Check for leaks in the cryopump, and if you find a leak, fix it. Was the pump rough pumped adequately? Rough for proper time.
- b. Are all hoses connected completely? If not, connect/tighten the hoses.
- c. Is the expander performance degraded? Call the pump manufacturer's service department.

- d. Check for low compressor pressure. Recharge the compressor.
- e. Check for a system thermal overload. Remove the thermal load.
- f. Faulty temperature sensor? Replace the sensor.

If the cryopump is slow to pump down the chamber or there is a high operating pressure in the system, you should:

- a. Verify that gauge tube is not dirty. Replace if questionable.
- b. Determine if the pump is near capacity. If so, regenerate.
- c. Check the vacuum system for contamination (RGA analysis). If needed, clean the system.
- d. Check for leaks in the vacuum system or the pump, especially the relief valve. Fix if present.
- e. Check for contamination of the second-stage charcoal by removing the expander from the pump body and checking for oil odor on the charcoal. If pumping toxic gases, do not attempt to check for contamination by smelling.
- f. Check for degraded expander performance. Call the pump manufacturer's service department.
- g. If the compressor is noisy, call the pump manufacturer's service department.

Should you have an excessively noisy expander:

- a. Check to be sure the phasing capacitor is properly connected. If not, reconnect the capacitor.
- b. Check for compressor capacitor failure. Call the pump manufacturer's service department.
- c. Check to determine if the compressor adsorber (final helium filter) is overdue for replacement. This may have contaminated both the expander and the compressor. If the helium in the refrigerator has been contaminated, call the pump manufacturer's service department.

Summary

You have looked at the various categories of faults that can cause vacuum system difficulties. You have seen in some depth the major problem areas that can beset a variety of pumps.

You have also learned to troubleshoot common vacuum system problems.

Let's now move on to our last topic, detection of leaks that cause problems in vacuum system operation.

9

Leak Detection

When you have completed this chapter, you will be able to:

1. Tell outgassing and virtual leak problems from real leak problems.
2. Be familiar with the sizes of leaks that affect vacuum systems and product life.
3. Identify the principal methods of leak detection.
4. Identify the major components of two of the major types of helium leak detectors.
5. Understand how helium mass spectrometer leak detectors work.
6. Be familiar with the type of calibrated leak commonly used to tune and calibrate a helium leak detector.
7. Identify some welding practices that can make leak detection difficult.

Introduction

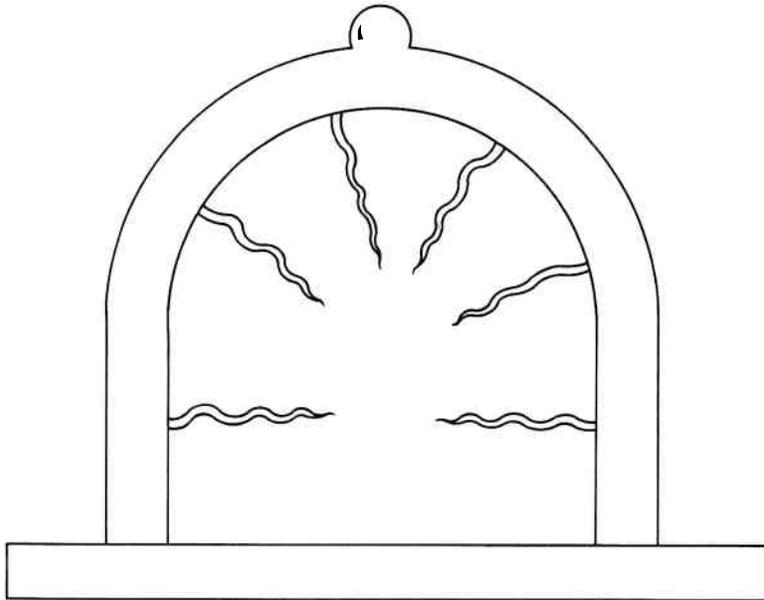
In this section, we will consider types of leaks, leak rates, detector principles, and leak detection techniques. Many instruments can be used for leak detection, including simple pressure gauges. Here, however, we will concentrate on the use of the helium mass spectrometer leak detector and its techniques.



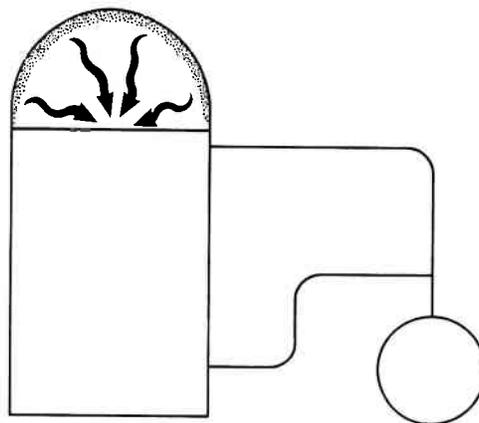
To begin with, everything leaks. It's simply a matter of how much. In some cases, the leaks are so small that they do not hurt the vacuum system process. In other cases, problems occur in vacuum systems that appear to be caused by leaks, but are not. Let's first try to separate real leaks from other types of problems (which themselves are very real).

Problems That Appear to Be Leaks

Outgassing



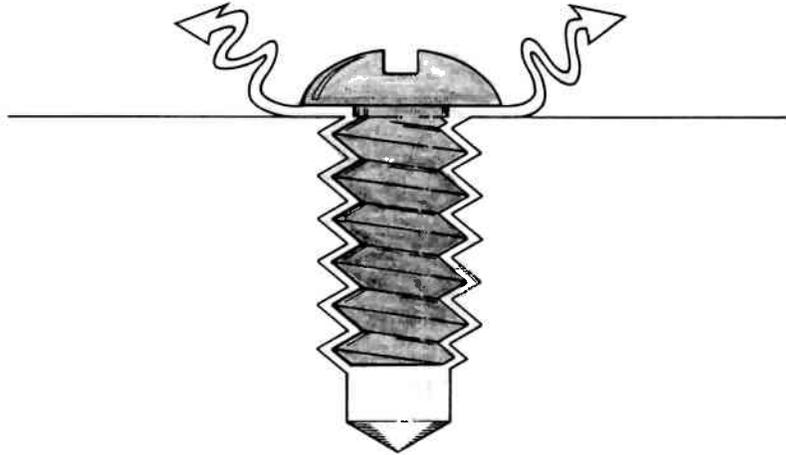
Outgassing can keep a system from reaching satisfactory pressure. Outgassing can be caused by techniques or materials that never should be used with vacuum. These can be improper handling, such as using bare hands in the vacuum system and excessive use of greases. They also include using too many materials which in small amounts might actually be all right to use with vacuum. Your process may cause deposits to form on the walls of the chamber. These deposits may contribute to the outgassing load.



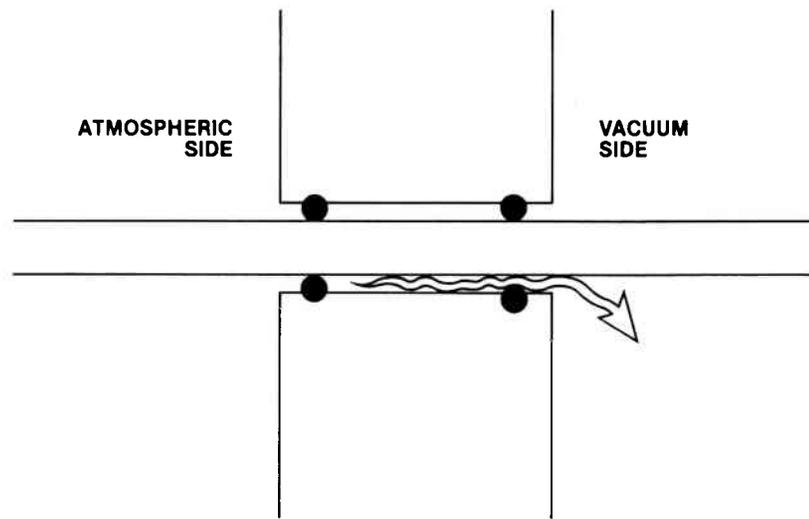
virtual leaks

Contaminated systems can outgas to the point that they make it impossible to reach satisfactory pressure. Such a problem can appear to be the result of real leaks. We often call these *virtual leaks*. Virtual leaks are any source of gas that is already inside the vacuum system. Two examples are outgassing and trapped volumes.

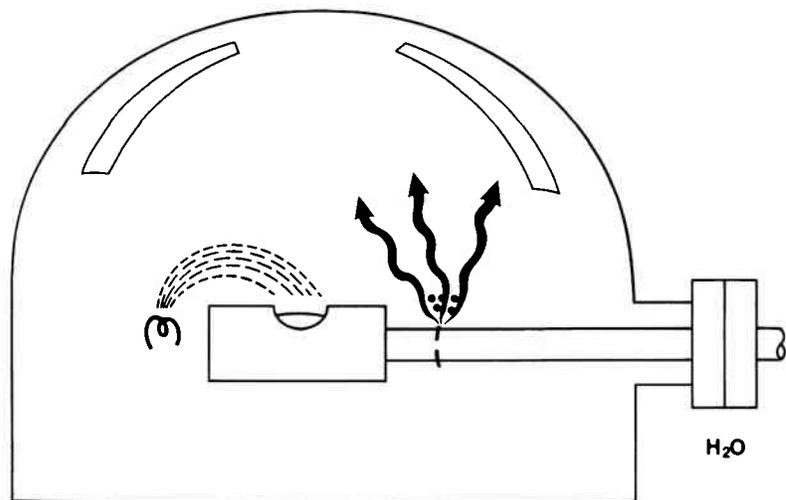
Trapped Volumes



Trapped volumes, also called virtual leaks, are sources of gases that are inside the vacuum system but which are released very slowly into the system. For example, water vapor on the walls, solvent residues, and trapped volumes can release gases into the system over a long period of time. This can make it impossible to reach satisfactory pressures until all of the gas is completely released.



Virtual leaks can also result from double O-ring-sealed shafts.

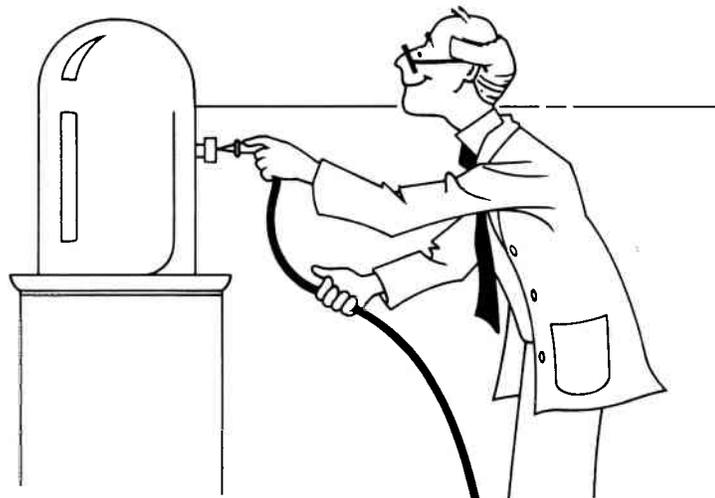


There are real leaks which act like virtual leaks. An example is a cooling line leak which “looks” like a virtual leak because it is not directly connected to the outside.

Permeation Leaks

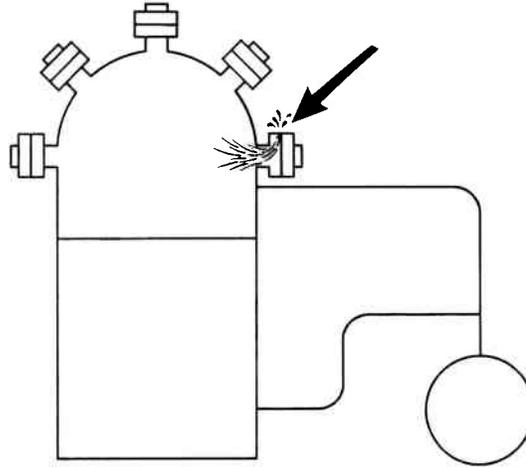
Gases may penetrate through some “solid” materials such as elastomers (O-rings). We call these permeation leaks. These are different than real leaks because the only way to prevent or stop them is to change to a less permeable material.

Real Leaks



Real leaks (cracks and holes) are the types of problems that can be relatively easy to solve by leak detection techniques. These are the types we will concentrate on in this section. We often refer to real leaks as *air leaks*.

Sizes of Leaks



In modern industrial vacuum systems containing many feed-throughs, there are many opportunities for leaks to occur. You can see an example of a leaky flange in the illustration above.

Vacuum-related leaks are small. The types of leaks that we will consider are not gaping holes. Instead they are tiny fissures that can develop at joints, welds, seals, or even in the materials that make up the system.

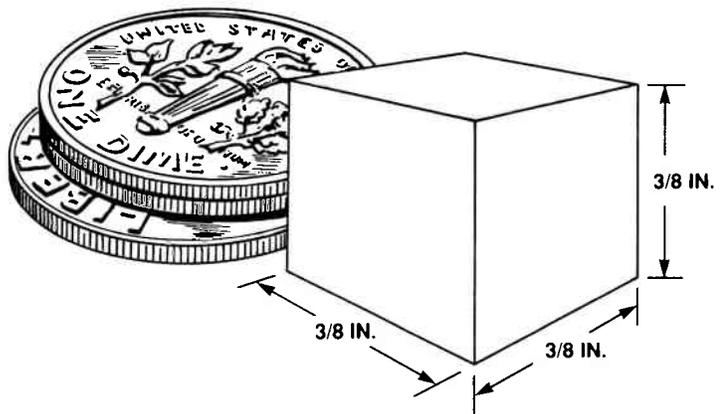


Even tiny leaks can have a negative effect on the productivity of a system or cause it to become useless.

Sizes of Leaks That Affect Vacuum Systems

leak rates

So, let's next consider *leak rates*, or the sizes of leaks that affect a vacuum system.



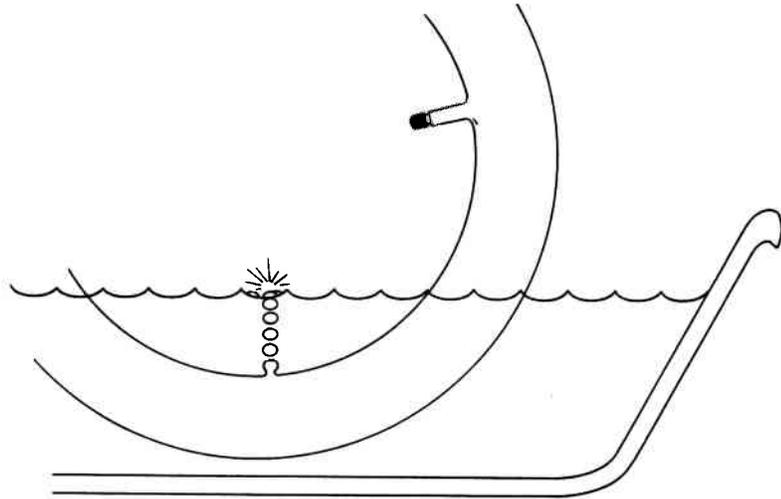
standard cubic centimeter

A one *standard cubic centimeter* per second (std cc/sec) leak rate is equivalent to 1 cc of gas at the standard pressure of 760 torr and temperature of 0°C passing through a leak at a pressure differential of 1 standard atmosphere. Recall that the volume of

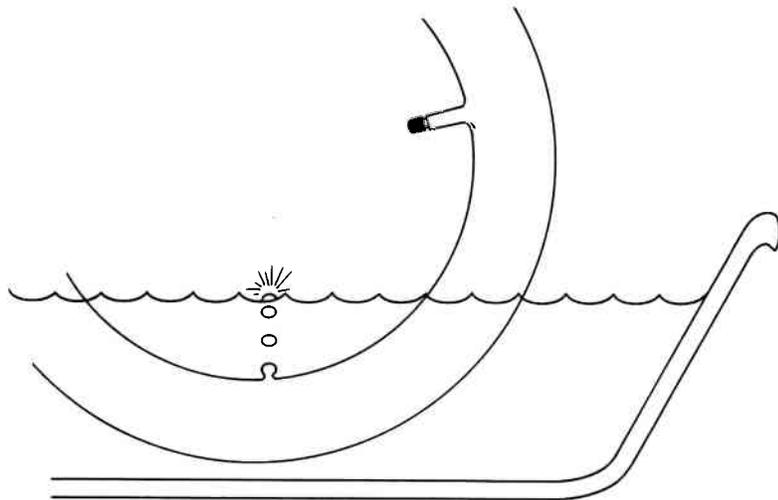
1 cc is equal to a cube approximately three-eighths of an inch on each side, or the volume of three dimes. One standard cc per second can also be expressed as 0.76 torr-ℓ/sec. Let's next look at these leak rates in more familiar terms.

$$1 \text{ std cc/sec} = 0.76 \text{ torr-ℓ/sec}$$

This flow can occur at any pressure differential. Remember that we are talking about a flow rate here, *not* a pressure reading.



The bubble test is a commonly used method of locating a leak in a bicycle tube. A bicycle inner tube leak that releases a steady stream of very small bubbles releases about 1 std cc of air in 10 seconds. In leak detector terms, this is about equivalent to a 10^{-1} std cc/sec leak rate.



A bicycle tube leak that releases one bubble per second will release a total of 3 cc of air per hour. In leak detector terms, this is about a leak rate of 10^{-3} std cc/sec. A leak rate of 10^{-4} std cc/sec

releases 1 cc every 3 hours, or one bubble in 10 seconds. Smaller leaks than this cannot be detected by the bubble technique.

Although the bubble test is used in many industries, we will not discuss this technique further in this section. Looking at leak rates in terms of bubbles helps to get a feel for the leak sizes that we will consider.

LEAK RATES

10^{-1} std cc/sec	— 1cc/10 sec
10^{-3} std cc/sec	— 3cc/hour
10^{-5} std cc/sec	— 1cc/day
10^{-6} std cc/sec	— 1cc/2 weeks
10^{-7} std cc/sec	— 3cc/year
10^{-9} std cc/sec	— 1cc/30 years

A leak rate of 10^{-5} std cc/sec releases a volume of gas equal to 1 std cc every 24 hours. A leak rate of 10^{-6} std cc/sec releases 1 std cc in a 2-week period.

For a vacuum system, the gas for the most part is not released from the system, but instead is admitted into the system from the atmosphere. A 10^{-7} std cc/sec leak rate admits 3 std cc of air into the system per year, and a 10^{-9} std cc/sec leak rate admits 1 std cc of air into the system in 30 years!

This last example, of course, doesn't sound like a very serious leak rate because it is so tiny. Indeed, in many processes, a leak rate this small can be ignored. However, in UHV systems this might be a serious leak.

Sizes of Leaks That Affect Product Life

This leads us to consider the sizes of leaks that affect product life. Of course, the maximum acceptable leak rate for a given product depends on what the product is. In general, static sealed systems require stricter specifications than other systems. In any case, if a tiny leak does not negatively affect a product, there is no need to worry about it or test for it. To do so would be very uneconomical, both with time and money.

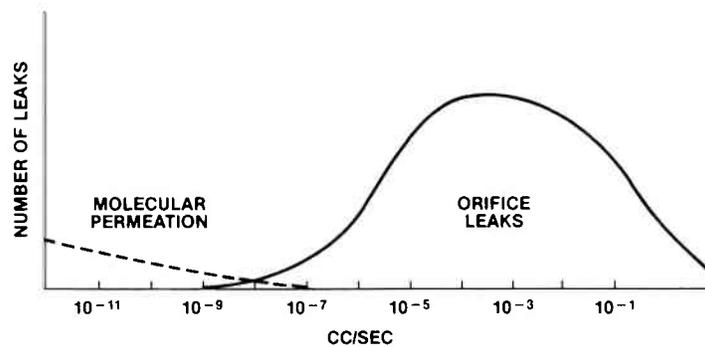
Product or System	Leak Rate Specification
Chemical Process Equipment	10^{-1} to 1 atm cc/sec
Torque Converter	10^{-3} to 10^{-4} atm cc/sec
Beverage Can End	10^{-5} to 10^{-6} atm cc/sec
Vacuum Process System	10^{-6} to 10^{-7} atm cc/sec
IC Package	10^{-7} to 10^{-8} atm cc/sec
Pacemaker	10^{-9} atm cc/sec

This table lists the suggested leak rate specifications for different types of products. It can be seen that the range is quite wide. For example, a leak in a torque converter greater than that specified as acceptable shows up as a fluid leak.

The leaky beverage can pull-tab lid shows up as a can of very flat beverage if it leaks at a greater rate than the specified limit. Another way to look at an acceptable leak rate in a beverage pull-tab lid is to consider that the beverage should retain its carbonation over a three-month shelf life.

Helium mass spectrometer leak detectors are commonly used for testing can lids, damaged vacuum process systems, IC packages, and pacemakers, as well as many other industrial and consumer products.

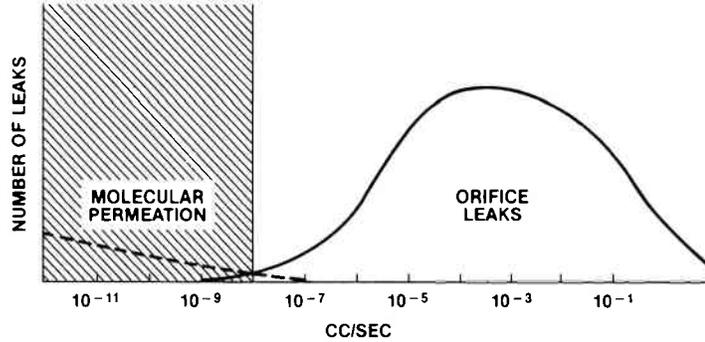
Distribution of Leaks by Size



DISTRIBUTION OF LEAKS BY SIZE

Next, let's look at the distribution of leaks by size. Long experience has shown us that leaks that affect vacuum equipment occur in different sizes. We have also learned that some sizes of leaks occur more often than others. For example, there are relatively few very large leaks above 10^{-1} std cc/sec or very small leaks below 10^{-7} std cc/sec. Instead, the greatest number of leaks occur at a rate that ranges from 10^{-2} std cc/sec on the high side, to 10^{-5} std cc/sec on the low side.

This curve shows that medium-size leaks occur most often. Both very large leaks and very small leaks occur much less often. Note also that most of the leaks represented by the large hump in the curve are orifice leaks. That is, they are holes or cracks or splits between joints such as welds, brazes, and seals. We also commonly call these *air leaks*.



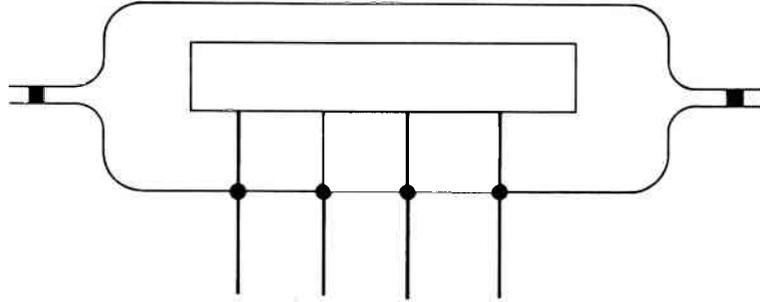
DISTRIBUTION OF LEAKS BY SIZE

Leaks below 10^{-8} std cc/sec may occur through molecular permeation. That is, they occur because gases actually penetrate through the material. This illustrates the need to select materials that are correct for vacuum work, and materials which have been properly made. (We saw earlier how voids can be elongated to create leak paths.) Products having leak rates in the 10^{-8} to the 10^{-9} range generally will have a five-year "leak-free" life.

It may not be economically feasible to 100% leak-check large production systems unless a problem is indicated. Such a problem might be that it is impossible to reach satisfactory vacuum levels. On the other hand, some of the individual components used in the system may routinely be 100% tested. These may include the system components such as the feedthroughs.



Some leaks keep happening over and over and occur in definite patterns. For example, special high-speed presses are used to manufacture pull tab lids for beverage cans. The pull tabs may have leaks as a result of worn or misaligned dies in the high-speed presses. Once the machine produces leaky pull tabs, it will continue to do so. It has been found that sample testing after a definite number of pull tabs have been produced detects almost all leakers.



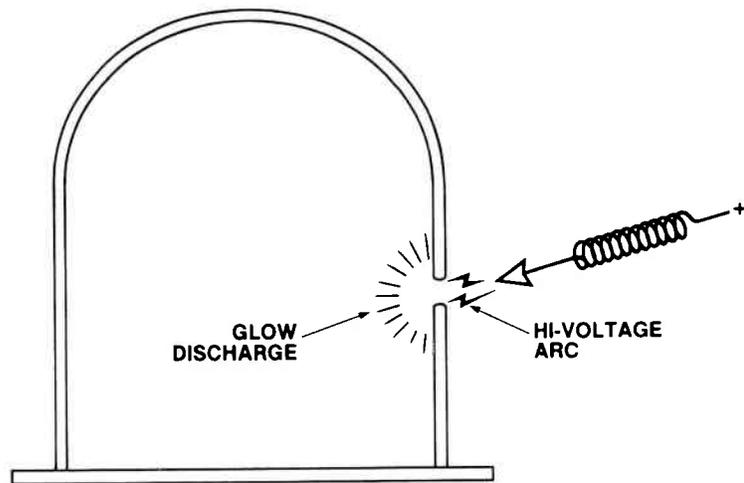
IC PACKAGES

An example of leaks that don't occur in definite patterns can be found in the semiconductor industry. Typically, the average frequency of leakers in production IC packages is 1% to 2% with no observable pattern of size or incidence of leaks.

Methods of Leak Detection

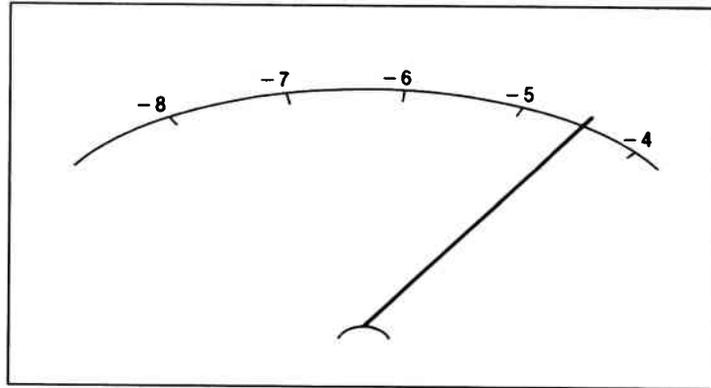
Next, let's look at a few leak detector techniques. Of course, we will concentrate on the helium mass spectrometer leak detector techniques. However, to give us a frame of reference, we will compare this technique with several other leak-check methods.

Spark Coil Technique



The spark coil technique uses a high-voltage or tesla coil to create an arc. This causes the ionization of air at the point where it enters the system. Ionization, of course, creates a glow discharge which is visible in glass systems. This is a simple technique; however, it has a number of drawbacks. First, it can only be used in glass systems. Further, this technique is potentially dangerous. (The high-intensity arc could cause an implosion.)

Pressure Change Method



Another simple technique uses the pressure gauges which are ordinarily used to monitor the system performance. Suspected leak sites can be squirted with a solvent while watching the gauge for a pressure rise that occurs when the solvent enters the leak. This method also has its shortcomings. Solvent entering a leak can actually freeze and plug up the leak. This can result in an inability to pinpoint the leak site. The problem is that the plugging is not permanent and instead will reopen at some later, unpredictable time. This technique also has limited sensitivity. Solvents may also attack vacuum grease and cause O-ring degeneration. Use of solvents for leak checking may require that O-rings be replaced.

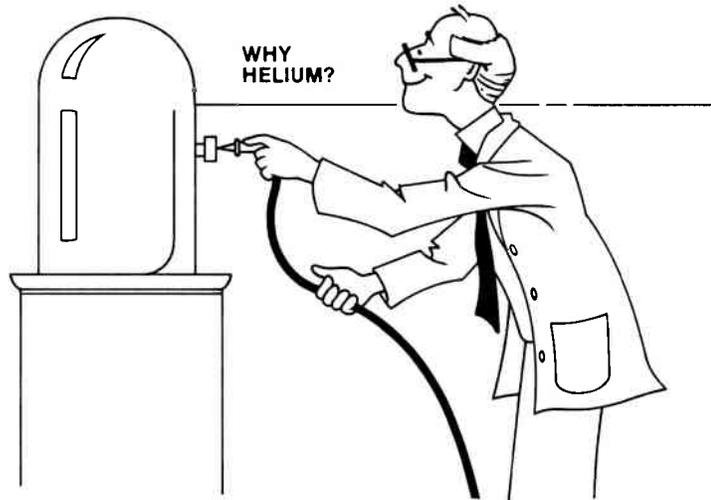
We have also mentioned the residual nitrogen analyzer and the residual gas analyzer in the chapter on gauges. These are also used to detect leaks when the pressure in your vacuum system is between 10^{-4} torr and 10^{-8} torr.

Helium Mass Spectrometer Leak Detector

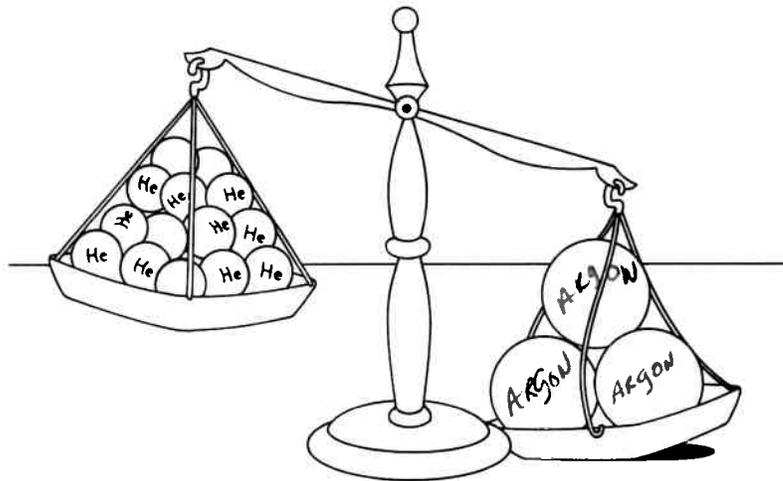
Now, let's concentrate on the helium mass spectrometer leak detector technique. This is one of the most commonly used techniques. It is the most sensitive, and it is very easy to use.

The helium mass spectrometer leak detection technique depends on the ionization, separation, and detection of helium ions. The leak-rate meter displays a value proportional to the helium ion concentration present in the spectrometer tube. Thus, the spectrometer tube is the most important part of the leak detector.

Why Helium Is Used



Why is helium used? Helium has a number of useful characteristics.



Helium Is Very Light

Helium is very light; in fact, it is the lightest and smallest of the inert gas molecules. Therefore, it flows through very small leaks easily. This makes it well suited to high-sensitivity leak detection. Even very small amounts can be readily separated and identified by a relatively simple mass spectrometer.

COMPOSITION OF DRY AIR
AT 760 TORR (SEA LEVEL)

Gas	Percent by Volume	Partial Pressure (Torr)
Nitrogen	78.08	593.40
Oxygen	20.95	159.20
Argon	0.93	7.10
Carbon Dioxide	0.03	0.25
Neon	0.0018	1.38×10^{-2}
Helium	0.0005	4.00×10^{-3}
Krypton	0.0001	8.66×10^{-4}
Hydrogen	0.00005	3.80×10^{-4}
Xenon	0.0000087	6.60×10^{-5}

Another aspect of helium that recommends it for use in mass spectrometer leak detection is that it constitutes only 5 parts per million in the atmosphere. Under most conditions, this makes it easy to distinguish the helium that is deliberately introduced into a leak with a helium probe, from the helium that enters with the atmospheric air. The instrument can be easily calibrated to show a zero response to the natural helium background.

Helium Permits Dynamic Testing

Helium also permits easy testing for leaks while a system is operating (dynamic testing). The system need not be shut down to perform a leak check. Therefore, valuable operating time can be saved. Also, the testing can proceed under the system's normal operating conditions rather than under unrealistic, static conditions.

Helium Permits Nondestructive Testing

The use of the helium probe also permits nondestructive testing. Many systems or products that are shown to have leaks by this technique can be repaired. Those that are rejected are rendered useless by the leak, not by the leak test.



Helium Is Safe

Another useful feature of this technique is that it is nontoxic, inert, and nonreactive. Helium released into the test area presents no personnel safety hazard. Even if a breath is deliberately inhaled, the worst result is that a person will sound rather silly for a few seconds. Helium is also nonhazardous. It is not flammable, nor explosive. We should caution you that breathing a pure helium atmosphere for a period of minutes would cause suffocation.

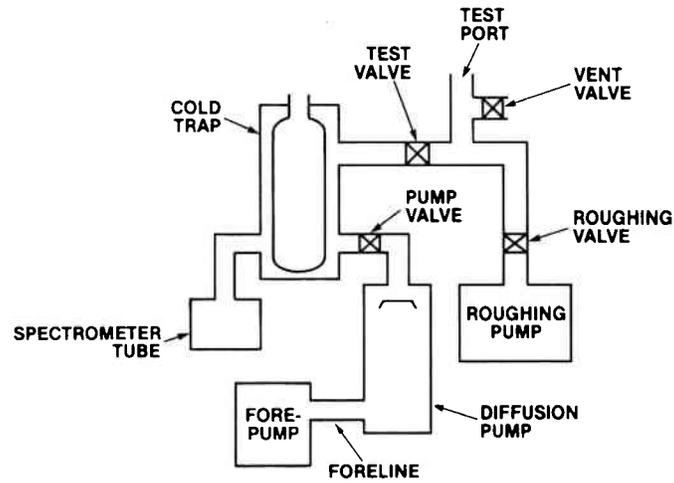
The Helium Leak Detector System

Helium leak detection systems work as follows: Helium is introduced to a test part that is connected to the leak detector. The helium from the test part leak travels into the leak detector, its partial pressure is measured, and results are displayed on a leak-rate measurement meter as flow rate.

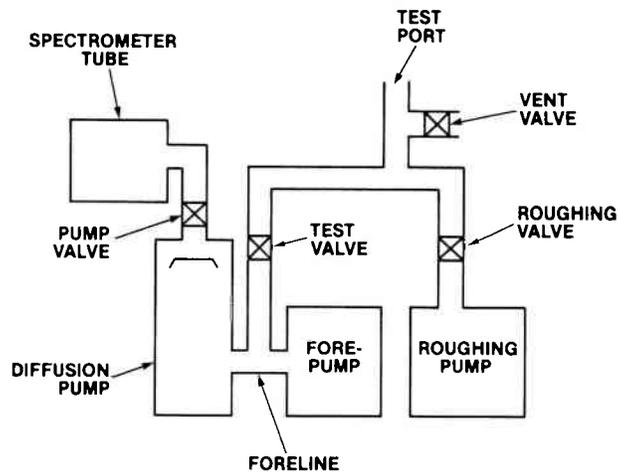
There are two general types of leak detectors—the conventional design and the Contra-Flow™ design.

Standard System Components

A conventional leak detector is arranged as shown here. It is an ordinary diffusion pump system. Also shown is the Contra-Flow leak detector.



CONVENTIONAL LEAK DETECTOR



CONTRA-FLOW™ LEAK DETECTOR

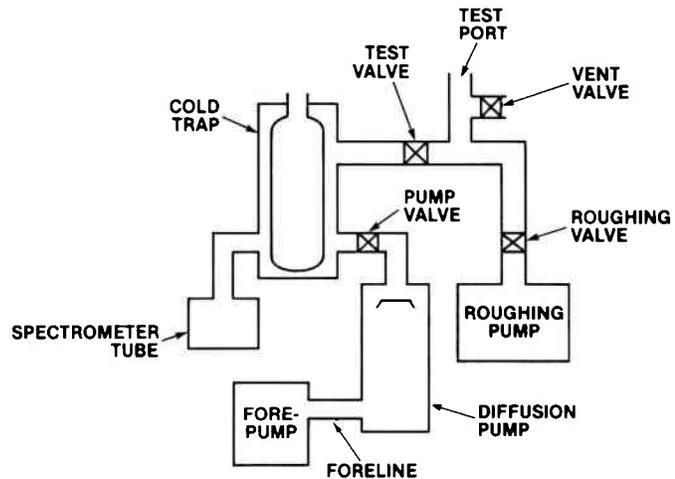
The leak detectors have two mechanical pumps. The first one is used for roughing purposes. This pump rough pumps the test piece, which is connected to the test port of the leak detector. The second mechanical pump is used as a forepump, or backing pump, for the diffusion pump. The conventional leak detector, like many diffusion pump systems, includes a cold trap. And finally, both versions include a spectrometer tube, which is used to sense the presence of helium.

Contra-Flow System Components

The Contra-Flow system differs from the conventional system in that the test port is connected directly to the foreline of the diffusion pump. Also, there is no cold trap.

How the Conventional System Works

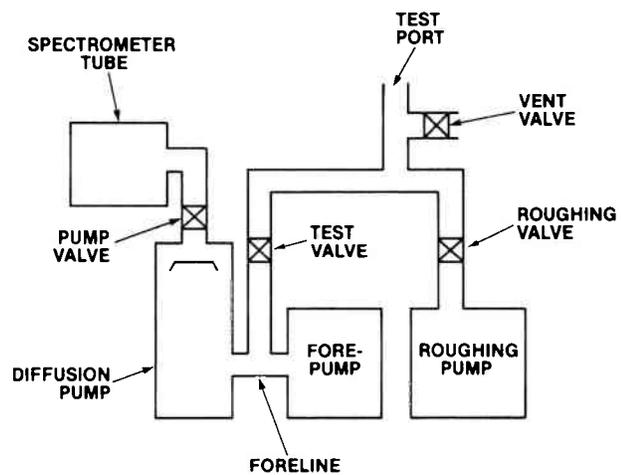
In the conventional system, vacuum is maintained in the spectrometer tube by use of a diffusion pump in combination with a mechanical pump. A cold trap pumps condensable vapors such as oil and water. A roughing pump is provided to evacuate the test port to a pressure level that will not disrupt diffusion pump operation.



CONVENTIONAL LEAK DETECTOR

The part to be tested is connected to the test port. It is evacuated to a suitable pressure (usually less than 10 mtorr) and then valved off from the rough vacuum system and onto the spectrometer tube vacuum system. Helium is sprayed on the surface of the part using a helium probe.

When leak testing is complete, the test port is valved off from the leak detector and vented to air.



CONTRA-FLOW™ LEAK DETECTOR

How the Contra-Flow System Works

In the Contra-Flow system, helium (and other inlet gases, such as those resulting from a leak in the test piece) are introduced into the diffusion pump foreline rather than into the "normal" pump inlet, as in conventional leak detectors. Helium diffuses backward through the diffusion pump to reach the spectrometer tube, where it is detected in the normal manner. The helium can diffuse backward because the diffusion pump has a much lower compression ratio for helium than other gases contained in air.

The diffusion pump acts as a filter that prevents the other gases and contamination from reaching the spectrometer tube. This filtering action of the diffusion pump eliminates the need for any cryogenic trapping.

The part to be tested is coupled to the test port. It is evacuated to a suitable pressure (usually 100 mtorr or less) and then connected to the foreline of the diffusion pump. Helium is sprayed on the surface of the part using a helium probe.

When leak testing is complete, the test port is valved off from the leak detector and vented to air.

The Two Systems Compared

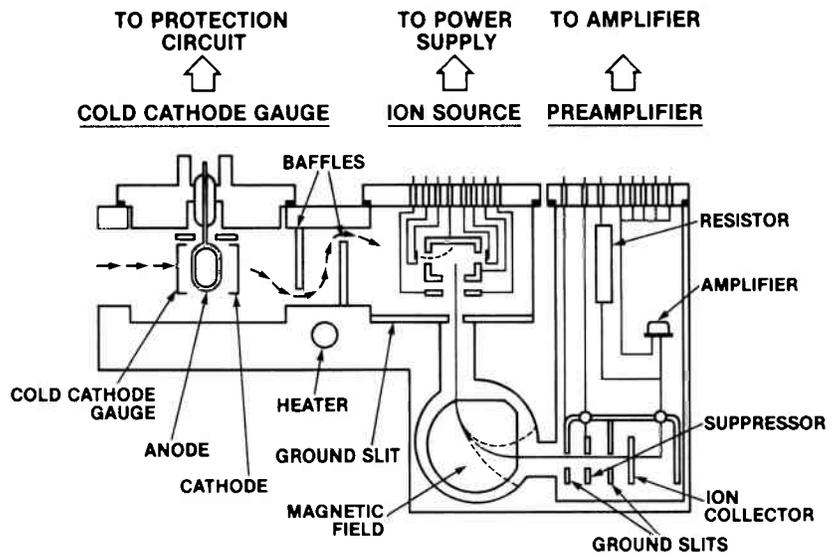
The test port in a Contra-Flow design is connected to the foreline of the diffusion pump, which is a relatively high-pressure region. Therefore, it is possible to check for much larger leaks than can be tolerated by the conventional leak detector design. Also, it is not necessary to wait for the system to get into the high vacuum range before fine leak testing can begin.

The advantage of the conventional leak detector design is that it is more sensitive. That is, all gases, including helium, pass from the leak in the test piece through the spectrometer tube and diffusion pump and finally to the mechanical pump and to the atmosphere in a more or less single direction. This contrasts with the Contra-Flow principle where most of the gas doesn't even make it to the spectrometer tube, only the helium.

Both types of leak detectors share a problem. They will back-stream mechanical pump oil if improperly used. If you are leak checking a system which is sensitive to oil vapor, you should have an external trap on your leak detector. Remember that there is a mechanical pump in the leak detector that will be directly attached to your system. Trap it.

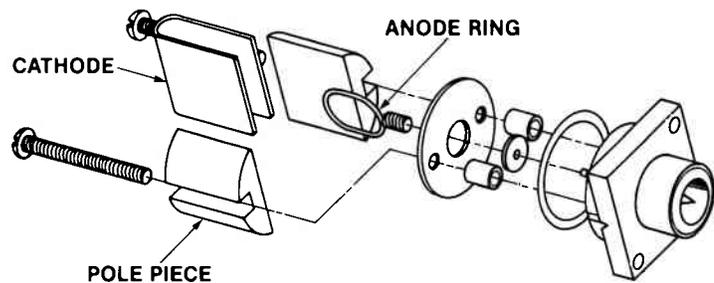
Now, let's talk about the heart of the leak detector, the spectrometer tube.

The Spectrometer Tube



SPECTROMETER TUBE

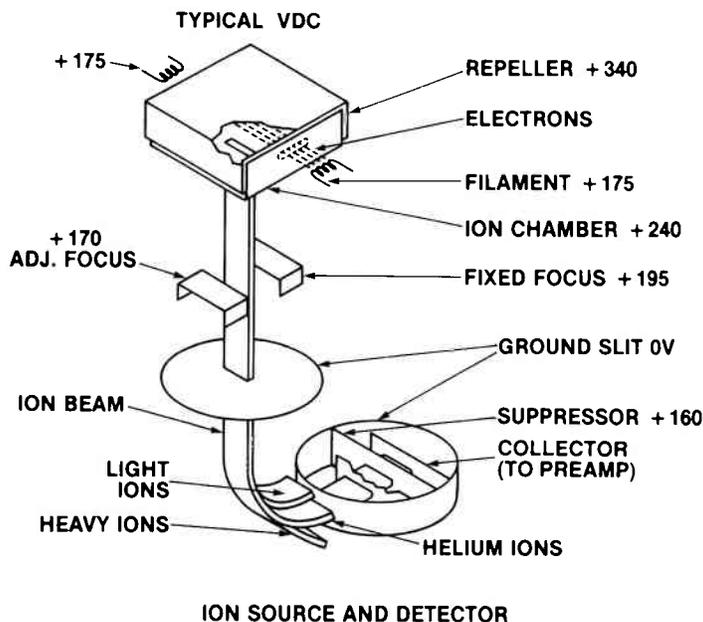
The spectrometer tube is the device around which the leak detector is built. The spectrometer tube is a simple, rugged component. It includes a cold cathode or ion gauge for pressure measurement, an ion source, and an ion collector and preamplifier assembly. The spectrometer tube housing also includes a magnet.



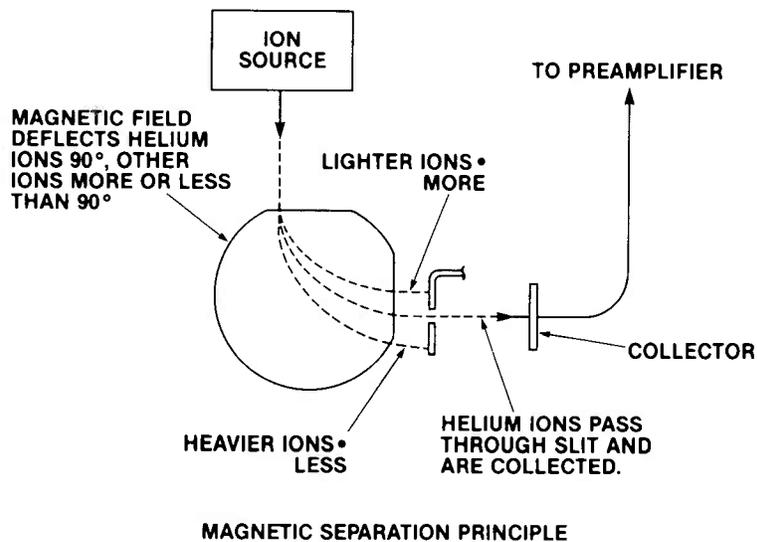
COLD CATHODE GAUGE

The cold cathode gauge is a device that we have already discussed. In this spectrometer tube it consists of a cathode and an anode structure, surrounded by a magnet array. It measures the pressure by ionizing the gases and measuring the resulting ion current.

The cold cathode gauge circuitry also includes an overpressure protection circuit. This circuit opens the ion source filament circuit to protect it if the pressure rises above a predetermined level. It may also close the test valve in an automatically valved unit.



The ion source consists of two filaments and an ion chamber. In the top half of the ion chamber, the repeller plate is at a more positive voltage than the bottom half of the ion source. Therefore, an ion beam is formed and directed out of the slit at the bottom of the ion chamber. It is directed between the focus plates, through the ground slit, and into the analyzing magnet section of the spectrometer tube.



The function of the magnetic field is to bend and separate the ion beam. In the bending process, ions that are heavier than helium are bent less, and those that are lighter (hydrogen) are bent more. Both of these ions are caused to crash into the walls. When the ion source voltages are properly adjusted, the magnet array causes helium ions (mass 4 amu) to be bent at the proper angle, causing them to pass through the ground slit and suppressor of the preamplifier assembly. They finally pass to the ion collector

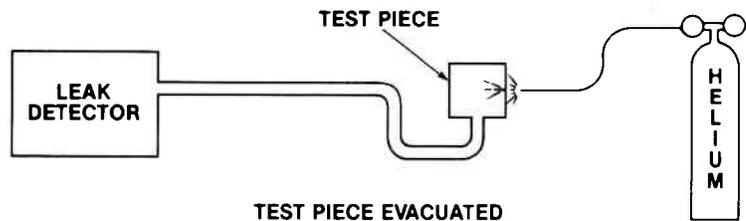
where they are detected to show—what else—a leak! The ability of a spectrometer tube to pick up and display small concentrations of a particular ion species is called its sensitivity. The helium mass spectrometer leak detector is widely used because of its extremely high sensitivity. That is, its sensitivity is as low as about 10^{-11} std cc/sec.

Methods of Helium Leak Detection

Let's now turn to leak detector methods. Most leak detector methods depend on the use of a *tracer gas* passing through the leak and being detected on the other side. There are basically four different methods, or techniques, for finding leaks—two "Outside-In" methods and two "Inside-Out" methods.

tracer gas

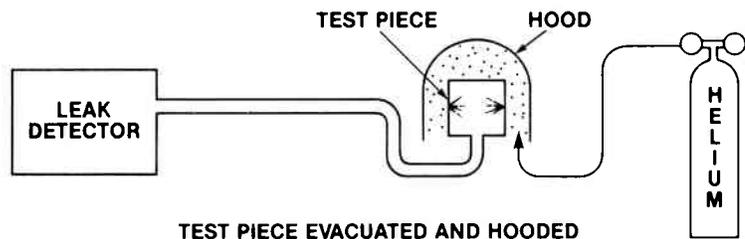
First Outside-In Technique: Exposed Test Piece



outside-in leak detection technique

In the most commonly used *outside-in leak detection technique*, the object to be tested is evacuated. With the leak detector connected to the evacuated test object, the surface of the test object is "probed" with a small jet of helium to locate individual leak sites. Helium entering the test piece enters the leak detector and the spectrometer tube (whether through the conventional flow or the Contra-Flow design) where it is detected as a leak. You can define or "pinpoint" the leak location as well as measure its size.

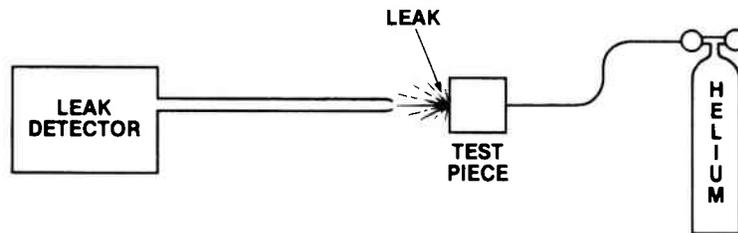
Second Outside-In Technique: Hooded Test Piece



The second outside-in technique consists of hooding the test piece with something like a plastic sheet or dome and flooding the hood with helium as shown. This technique has two obvious advantages. First, it helps to quickly establish whether or not a test piece leaks. Secondly, it establishes the total leak rate. That is, the problem may be the result of a number of very small leaks. Each of the leaks may not individually constitute a problem. But

their cumulative effect may render the system or product unusable or inoperable. This technique is most useful on production lines where a part may be accepted or rejected on the basis of the total leak rate.

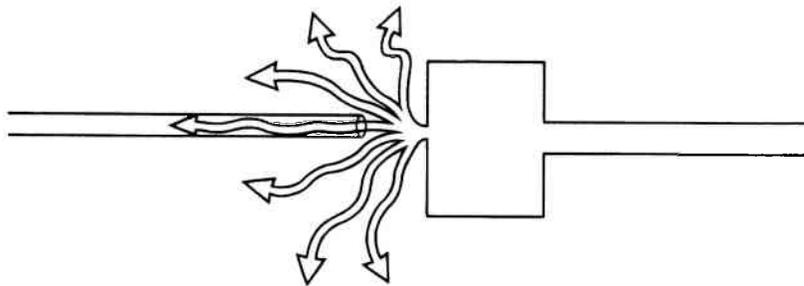
First Inside-Out Technique: Detector Probe



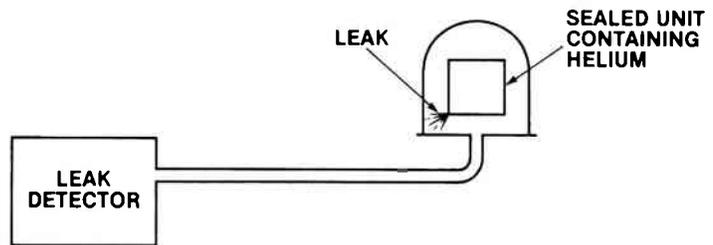
inside-out leak detection technique

The test configuration can be reversed. That is, instead of being evacuated, the test piece can be pressurized with helium as shown. This is an *inside-out leak detection technique*. In this case, the leak detector is equipped with what is called a detector probe that admits air and helium into a small hole at the end of a flexible hose. The test piece is probed with the detector probe around suspected leak sites. *Caution:* Many components used with vacuum systems have not been designed to hold positive pressures. Overpressure may cause rupture or even explosion-like forces to develop.

MOST HELIUM ESCAPES

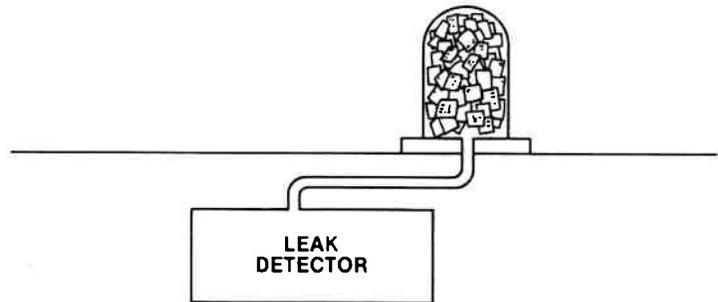


A limitation of the detector probe technique is that the total leak rate is virtually impossible to establish by this technique. This is because only a small percentage of the helium coming from the leak actually enters the detector probe. Most of the helium escapes into the atmosphere. It is very useful to establish leak location. It is not as sensitive as the other methods because the helium in the air is constantly being admitted into the detector. The smallest practical leak detectable with a detector probe is about 1×10^{-6} atm cc/sec.

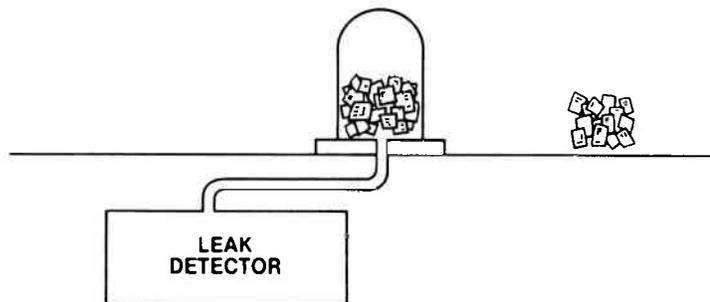
Second Inside-Out Technique: Bombing

bombing technique

In the test arrangement shown here, the test piece is pressurized. This technique allows batch testing; that is, a large number of pieces can be tested simultaneously. It is often called the *bombing technique* because the parts are placed in a pressurized helium vessel which allows the helium to leak into the parts which leak. This method is much more sensitive than the sniffer probe technique because the helium does not escape to the atmosphere before entering the leak detector.



This technique, of course, does not pinpoint the site of the leak; however, it quickly establishes whether a leak (or leaks) exist(s).



IF LEAK IS INDICATED, REMOVE HALF THE TEST PIECES, AND RETEST.

If the bombing technique above indicates that a leak exists, the piece can be probed to pinpoint the location. If the test was a batch test, half of the test pieces can be removed from the bell jar and the test repeated. If the second test shows that there are no

leaks, half of the pieces are automatically eliminated as possible leakers. Next, half of the remaining test pieces are placed in the bell jar, and the test repeated. It can be seen that this technique is well suited to batch (fast) testing.

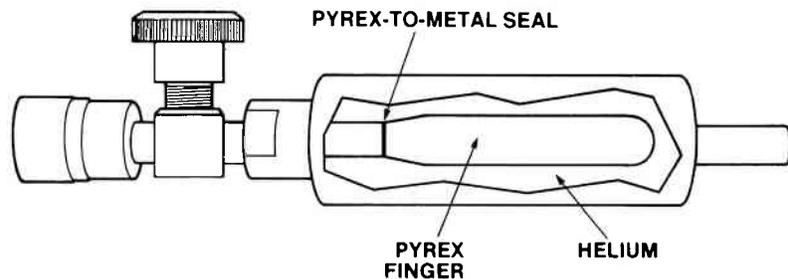
Each technique has advantages and limitations.

The leak test techniques indicated above are the main methods for establishing whether leaks exist, and for locating where they are. Of course, each of the techniques we have discussed has its own advantages and shortcomings. Each also has its own individual set of variations. You will learn to recognize the advantages, shortcomings, and variations of each as you gain experience with them.

Calibrated Leaks and Measurement Accuracy

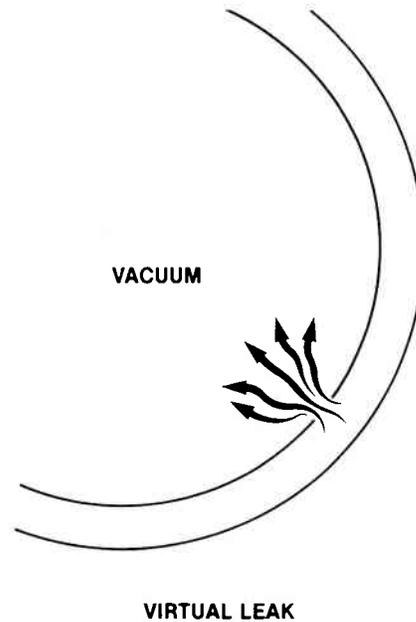
calibrated leak

You must use some sort of standard to set the scale properly on your leak detector. This standard is usually a *calibrated leak*. Several companies manufacture this type of leak calibration device. It consists of a metal container with a pyrex glass tube inside. The helium is placed inside the metal container so that it must permeate through the pyrex glass in order to escape. The valve on the container allows you to set the zero on your leak-rate meter for no helium "leaking" into the leak detector. Opening the valve then allows you to set your meter to read the value of the leak stamped on the container. You use the zero control knob to set the zero; the calibrate control knob to adjust the leak detector to read the value of the calibrated leak. Your calibrated leak should be stored with the valve left open. This permits the helium to leak freely and does not saturate the inside of the calibrated leak with helium.

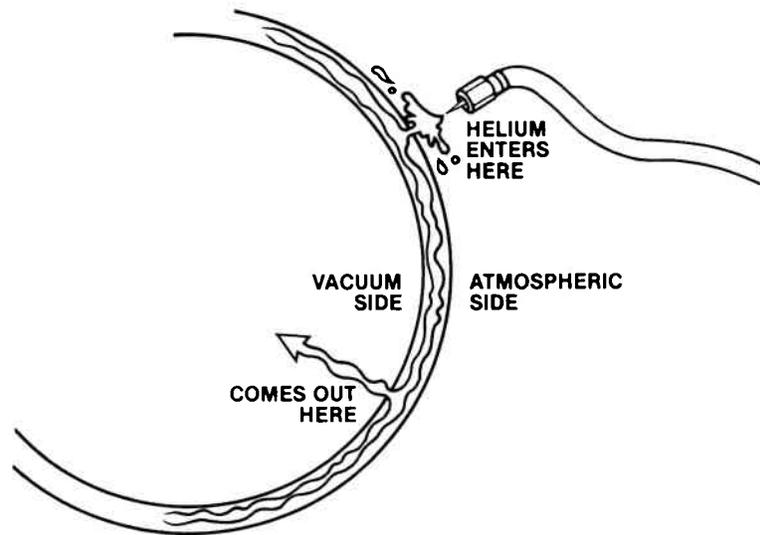


PERMEATION LEAK WITH RESERVOIR AND VALVE

Welding Techniques and Leaks

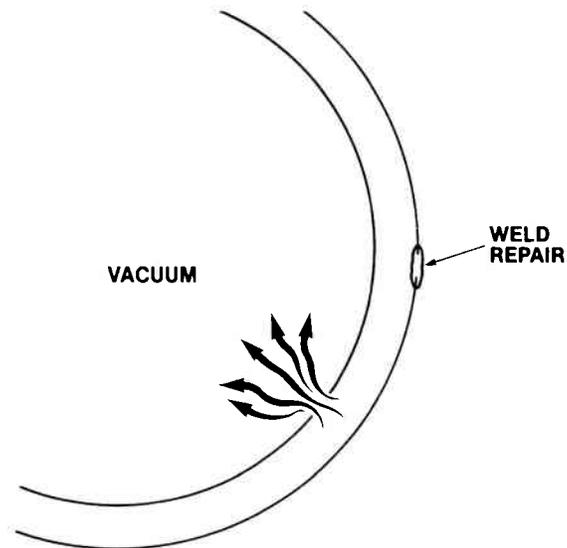


A virtual leak can result from bad welding techniques. If the joint is welded both on the outside and inside (or atmosphere and vacuum side) and if a tiny hairline crack develops on the vacuum side, every time the system is vented, the space between the welds is immediately loaded with atmospheric gas. This is, of course, a double problem. The partially trapped gas will contribute to the pressure over a long period, and may appear to be a real air leak. But, what is worse, it is impossible to successfully use the leak detector helium probe technique to detect this problem.



REAL LEAK

Now, if both sides of a weld leak, there is no guarantee that they will leak in the same location. This means that the helium used in the leak detection helium probe technique can enter the outside leak at one point, and emerge from the inside leak at another location.



VIRTUAL LEAK

We can repair the outside weld, but this doesn't solve our problem. Since the inside leak was not located and repaired, it now is a virtual leak. (The solution to this problem is to use full welds on the inside, or vacuum, side, and tack welds on the outside, or atmospheric side.)

Summary

You have seen how virtual leaks such as outgassing and trapped volumes can masquerade as real leak problems. By now, you are familiar with the sizes of leaks that affect both vacuum systems and product life. You have learned what sizes of leaks occur most often.

We have described the principal means of leak detection and the major components of two of the major types of helium leak detectors. We have also looked at how each type operates.

Helium leak detection is a very useful method of quality control in a wide variety of systems and products. It is enjoying increasingly extensive use in many areas of science and industry.

10

Summary

You have learned what vacuum is, how it is created, and how it is measured. You have seen what the basic vacuum concepts are and how materials and contamination can affect achieving a desired pressure.

We have described the commonly used vacuum pumps and how they work, as well as some general maintenance information for the pumps. We have also described other major vacuum components—gauges, which tell you what is happening inside the vacuum system; materials, which are important to minimize gas load; seals, which keep unwanted gases out of the vacuum system; and valves and feedthroughs.

Next, we combined the hardware and instruments into typical vacuum systems. The advantages and disadvantages of these systems were indicated. You also learned the basics of troubleshooting vacuum systems.

Finally, we discussed leaks, which prevent proper vacuum system operation, and how to detect those leaks.

Now it's up to you. Put your knowledge to work when working with your vacuum system. We have given you a start.

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Glossary

For a more detailed discussion of a specific term, turn to the page number listed next to it in the left margin.

- 16 *absolute pressure* See *pressure, absolute*.
- 10 *absolute temperature* The temperature scale which starts at "true" or absolute zero. It is often called the Kelvin scale.
- absorption* The binding of a gas in the interior of a solid or liquid.
- adsorption* The condensing of a gas on the surface of a solid.
- 11 *atmosphere, standard* See *standard atmosphere*.
- 17 *atom* The smallest identifiable part of an element. An atom has a nucleus with particles called protons and neutrons. Under normal conditions, it is surrounded by a number of electrons equal to the number of protons. Neutrons are neutral, protons are positively charged, and electrons are negatively charged.
- 17 *atomic mass unit* A way of classifying atoms according to their weight, or mass. Atoms of the different elements have different weights, or masses.
- 20 *Avogadro's Law* The gas law that states that one mole of any gas has 6.023×10^{23} particles and under standard conditions occupies 22.4 liters.
- 47 *backing pump* See *forepump*.
- 48, 68 *backstreaming* The small amount of pump fluid vapor that moves in the wrong direction, i.e., toward the work chamber.
- 108 *bakeout* The degassing of a vacuum system by heating during the pumping process.
- 12 *bar* Unit of pressure measurement. There are 1.010 bar in one standard atmosphere. One bar equals 1×10^6 dynes per square centimeter.
- 152 *base pressure* That pressure which is typically reached with your system when it is clean, empty, and dry.
- bell jar* A container open at the bottom and closed at the top which is used as a vacuum chamber or test vessel. Also called a work chamber.
- 169, 174 *bellows-sealed valve* A valve type in which the stem seal is accomplished by means of a flexible bellows, one end of which is attached to the sealing disk, the other end to either the bonnet or the body.

- 61 *blower pump* A type of vacuum pump which functions from 10 torr to 0.0001 torr. Also called a booster or Roots pump.
- body* That part of a valve which contains the external openings for entrance and exit of the controlled fluid.
- 273 *bomb test* A form of leak test in which enclosures are immersed in a fluid. The fluid is then pressurized to drive it through possible leak passages and thus into the internal cavities. The enclosures are then placed in a leak detector to detect the escaping fluid.
- 169 *bonnet* In general, that part of the valve through which the stem enters the valve, and which is rigidly attached to the valve body.
- 125 *bourdon gauge* A roughing gauge that responds to the physical forces that a gas exerts on a surface.
- 20 *Boyle's Law* The gas law that states $P_1V_1 = P_2V_2$, or original pressure times original volume equals new pressure times new volume. This equation predicts new pressure or new volume whenever the other is changed by any amount (providing that the temperature is unchanged).
- 274 *calibrated leak* An external reference standard that permits calibration of a helium leak detector.
- 126 *capacitance manometer* A vacuum gauge which senses pressure by the change in capacitance between a diaphragm and an electrode.
- 22 *Charles' Law* The gas law that describes what happens to the volume of gas as the temperature is changed. As a gas is cooled, its volume gets smaller. As a gas is heated, its volume increases (at constant pressure).
- chemisorption* The binding of a gas on or in a solid by chemical action. See *gettering*.
- 93 *closed-loop refrigeration system* A refrigeration system in which the coolant is recycled continuously.
- 65 *cold cap* A component mounted on top of the jet assembly in a diffusion pump. This cap helps to keep pump fluid vapor out of the work chamber.
- 118 *cold cathode discharge* A visible glow caused by the recombination of electrons and ions. The color is characteristic of the gas species present.

- 137 *cold cathode gauge* See *ionization gauge*.
- 78 *cold trap* See *cryotrap*
- 14 *condensation* The process of a gas turning back into a liquid.
- 27 *conductance* A term used to indicate the speed with which atoms and molecules can flow through a particular region such as an orifice or pipe.
- 30 *conductance limited* The inability to make use of the rated speed of a pump due to the use of an opening or pipe smaller than the inlet diameter of the pump.
- 128 *conduction* The transfer of energy (heat, light, etc.) by direct contact. In the case of gaseous conduction, the transfer of energy by molecules directly contacting surfaces and other molecules.
- 128 *convection* The transfer of heat from one place to another by the circulation of currents of heated gas or other fluid.
- 72 *critical forepressure* See *maximum tolerable foreline pressure*.
- 100 *crossover* The pressure at which a vacuum chamber is changed from being pumped by a roughing pump to being pumped by a high vacuum pump.
- 55, 96 *cryocondensation* The pumping of gases that are condensed at cold temperatures. For example, water vapor on a liquid nitrogen trap at -196°C .
- 55, 97 *cryosorption* The pumping of gases that are not readily condensed (or pumped) at cold temperatures, by the process of sticking onto a cold surface.
- 78 *cryotrap* A device usually placed before the inlet of a high vacuum pump to "trap" or freeze out gases such as pump oil vapor and water vapor. Cryotrap commonly use liquid nitrogen as the coolant. Also called cold trap or liquid nitrogen trap.
- degassing* The removal of gas from a material, usually by application of heat under high vacuum. See *bakeout*.
- 17 *desorption* See *outgassing*.
- diffusion* (1) The flow of one substance through another by random molecular motion. (2) The process by which molecules intermingle as a result of their thermal motion.

- 65 *diffusion pump* A vapor pump having boiler pressures of a few torr and capable of pumping gas continuously at intake pressures not exceeding about 2 mtorr and discharge pressures (forepressures) not exceeding about 500 mtorr. The term diffusion should be applied only to pumps in which the pumping action of each vapor jet occurs as follows: The gas molecules diffuse through the low-density scattered vapor into the denser, forward-moving core of a freely expanding vapor jet. Most of the gas molecules are then driven at an acute angle toward the wall and on into the fore vacuum.
- 169 *dynamic seal* A seal that moves. (See *static seal*.)
- 17 *electron* A negatively charged particle. (See *atom*.)
- 14 *evaporation* The process that happens when a liquid or solid becomes a gas.
- 182 *feedthrough* A device used to allow some sort of utility service to go from the outside world to the inside of a vacuum system while maintaining the integrity of the vacuum; for example, an electrical feedthrough.
- 66 *foreline* The section of a pump through which the gases leave. The exhaust line of a pump.
- 76 *foreline valve* A vacuum valve placed in the foreline to permit isolation of the pump from its forepump.
- 47 *forepump* The pump which is used to exhaust another pump which is incapable of discharging gases at atmospheric pressure. Also called the backing pump.
- 67 *fractionation* A process that helps to purify the condensed fluid in a diffusion pump. This process distills out contaminants produced by decomposition of pump fluid.
- 10 *gas* A state of matter where the individual particles are free to move in any direction and tend to expand uniformly to fill the confines of a container.
- 47 *gas ballast* A method used with any oil-sealed rotary pump which allows a quantity of air to be admitted during the compression cycle to prevent condensation of water vapor. The amount of air admitted is regulated by the gas ballast valve. The use of a gas ballast raises the ultimate pressure of the pump.
- 26 *gas density* The number of molecules per unit of volume.

- 32, 34 *gas load* The amount of gas being removed from a vacuum chamber by the vacuum pumps. Typically measured in torr-liters per second, cubic feet per minute, or cubic meters per hour.
- gauge pressure* See *pressure, gauge*.
- 23 *Gay-Lussac's Law* The gas law that states that if the temperature of a volume of gas at 0°C is changed by 1°C, the volume will change (plus or minus, as appropriate) by 1/273 of its original value.
- 24 *general gas law* The gas law that covers pressure, volume, and temperature in one single equation, or $P_1V_1T_2 = P_2V_2T_1$.
- 109 *gettering* A method of pumping gases through chemical reaction of a material with gas molecules. The material usually used is an active element such as titanium. See *chemisorption*.
- 262 *helium mass spectrometer leak detector (HMSLD)* See *mass spectrometer leak detector*.
- 17 *high vacuum* Pressure which ranges from about 10^{-4} torr (0.0001 torr) to approximately 10^{-8} torr (0.00000001 torr).
- 41, 63 *high vacuum pump* A vacuum pump which will function in the high vacuum range. Common examples are the diffusion pump and the mechanical cryopump.
- high vacuum valve* A large diameter valve usually placed between the vacuum chamber and the vacuum pumps. It is used to isolate the vacuum chamber from the pumps when it is necessary to work on something in the chamber. Also called hi-vac valve, gate valve, or trap valve.
- 17 *implosion* In vacuum work, the inward collapse of the walls of a vacuum system, caused by external pressure.
- 272 *inside-out leak detection technique* A method of leak detection whereby the tracer gas is placed under pressure inside the container to be leak-checked. A detector probe attached to a leak detector is used to locate leaks.
- 19, 115 *ion* A charged particle consisting of an atom or molecule which has an excess of positive or negative charge. Typically produced by knocking an electron(s) out of an atom or molecule to produce a net positive charge.
- 115 *ionization* The process of creating ions. See *ion*.

- 133 *ionization gauge* A vacuum gauge that has a means of ionizing the gas molecules, electrodes to enable the collection of the ions formed, and a means of indicating the amount of the collected ion current. Various types of ionization gauges are identified according to the method of producing the ionization. The common types are:
- 134 1. *hot cathode ionization gauge* The ions are produced by collisions of gas molecules with electrons emitted from a hot filament (or cathode) and accelerated by an electric field. Also called hot-filament ionization gauge, or simply ion gauge.
- 137, 269 2. *cold cathode ionization gauge* The ions are produced by a cold cathode discharge, usually in the presence of a magnetic field which lengthens the path of the electrons.
- 116 *ion pump* An electrical device for pumping gas. The ion pump includes a means for ionizing the gas with a system of electrodes at suitable potentials, and also a magnetic field. The ions formed move toward a cathode or a surface on which they are reflected, buried, or cause sputtering of cathode material.
- 65 *jet assembly* A nozzle assembly that directs oil vapors in a diffusion pump.
- 152 *leak* Leaks may be of three different types: (1) a real leak, which is a crack or hole allowing gases to pass through; (2) a virtual leak, which is caused by outgassing of some volatile material inside a vacuum system or trapped volume; and (3) a permeation leak, which consists of atomic-scale holes throughout the material of construction: for example, O-rings are quite permeable.
- leak detector* A device for detecting, locating, and/or measuring leakage.
- 255 *leak rate* Mass flow through an orifice per unit time. Vacuum system leakage rates are typically measured in atm cc per second or torr-liters per second.
- 78 *liquid nitrogen trap* See *cryotrap*
- 17 *mass* A fundamental characteristic of matter which is most closely related to the unit of weight.

- 262 *mass spectrometer (MS)* An instrument that is capable of separating ionized molecules of different mass/charge ratio and measuring the respective ion currents. The mass spectrometer may be used as a vacuum gauge that measures the partial pressure of a specified gas, as a leak detector sensitive to a particular tracer gas, or as an analytical instrument to determine the percentage composition of a gas mixture.
- 262 *mass spectrometer leak detector* A mass spectrometer adjusted to respond only to the tracer gas. Helium is commonly used as the tracer gas, and thus the instrument is normally referred to as a helium leak detector.
- 72 *maximum tolerable foreline pressure* A measure of the ability of the diffusion pump to pump gases against a certain discharge pressure. Also called critical forepressure.
- 26 *mean free path* The average distance between molecular collisions. Of importance for vacuum systems where one is interested in getting some particular type of particle from a source to a surface. For example, ion implanters, coaters, or television tubes.
- 12 *micron* Pressure unit equivalent to 1 mtorr.
- 12 *millibar* Unit of pressure measurement, equal to 1/1000 bar.
- 12 *millimeter of mercury* See *torr*.
- millitorr* Unit of pressure measurement, equal to 1/1000 torr.
- 20 *mole* The number of particles in equal volumes of gases under the same conditions of temperature and pressure. One mole of any gas has 6.023×10^{23} particles.
- 26 *molecular density* The number of molecules in a unit of volume such as a cubic centimeter. There are approximately 3×10^{19} molecules per cc at one standard atmosphere.
- 25 *molecular flow* The type of flow which occurs when gas molecules are spread far apart. There are few collisions so that the molecules tend to act independently of any other molecules which may be present. The molecular directions are completely random.
- 54 *molecular sieve* A very porous material used to contain the pumped gases in sorption pumps. May also be used in a foreline trap to contain oil molecules.

- 48 *molecular sieve trap* A device used to collect oil vapors backstreaming from oil-sealed mechanical pumps.
- 19 *molecular weight* A way of classifying molecules according to their weight, or mass. Molecular weight or mass is the sum of the individual atomic weights that make up the molecule.
- 19 *molecule* One atom, or two or more atoms joined together and having definite chemical and physical characteristics.
- 17 *neutron* A particle located in the nucleus of an atom which has no electrical charge but does have mass. (See *atom*.)
- 17 *nucleus* The dense center portion of an atom containing protons and neutrons. (See *atom*.)
- 78 *open-loop refrigeration system* A refrigeration system in which the coolant vents to atmosphere.
- 17, 152, 251 *outgassing* The process in which a gas particle leaves a surface and moves into the volume of a vacuum chamber. This, of course, adds to the gas load and may or may not be desirable. In extreme cases, it prevents "pumping down" a vacuum system to the specified pressure. The system is then said to be "hung up," or outgassing. Also called desorption or virtual leak.
- 271 *outside-in leak detection technique* A leak detection technique where the leak detector senses a tracer gas which passes from the outside of the container to the inside of the container. May be used to determine the size and/or the location of a leak.
- 13 *partial pressure* See *pressure, partial*.
- 12 *pascal* Unit of pressure measurement. There are 101,325 pascals in one standard atmosphere. A pascal equals one newton per square meter.
- 253 *permeation leak* Molecular-scale holes through a material of construction. See *leak*.
- 131 *Pirani gauge* A vacuum gauge used to measure pressure in the rough vacuum range.
- 6 *powers of ten* A convenient way of describing very large and very small numbers. A number is written as some value from 1 and up to 10 (but not including 10). Then, it is multiplied by either a positive or negative power of ten. Also called exponential notation or scientific notation.

- 3, 11 *pressure* Force per unit area. The force is created when atoms, molecules, or "particles" strike the walls of their container. Common pressure units for vacuum work are torr, pounds per square inch relative (psig), inches of mercury, millimeters of mercury, bar, millibar, and pascal.
- 16 *pressure, absolute* Pressure above zero pressure (corresponding to totally empty space) as distinguished from "gauge" pressure. In vacuum technology, pressure is always measured from zero pressure, not atmospheric pressure, and therefore the term absolute pressure is not required.
- pressure, gauge* The difference between absolute pressure and atmospheric pressure. The most common unit is probably psig.
- 3 *pressure measurement* A measurement of the pressure (the number and intensity of particle impacts) on a given unit of area. There are several different scales for pressure measurement: for example, torr, millitorr, bar, millibar, and pascal. These scales may be used as absolute or relative scales.
- 13, 139, 141 *pressure, partial* A measurement of the pressure of one particular gas in a mixture of gases. For example, the partial pressure of oxygen in air is about 160 torr.
- pressure, relative* See *pressure, gauge*.
- 13 *pressure, total* The sum of all of the partial pressures of every gaseous species. The force exerted by all the gas molecules in any mixture of gases. We commonly assume that a pressure gauge reads total pressure.
- 14 *pressure, vapor* The pressure exerted by molecules after they have escaped from a liquid or solid and formed a vapor (gas). One tries, in general, to put substances of low vapor pressure into a vacuum system so as to decrease the gas load on the vacuum pumps.
- probe* A tube having a fine opening at one end, used for directing or collecting a stream of tracer gas.
- probe test* A leak test in which the tracer gas is applied by means of a probe so that the area covered by the tracer gas allows tracer gas to enter and locate the leak.
- 17 *proton* A positively charged particle. (See *atom*.)
- 12 *psia* Pounds per square inch absolute, a unit of pressure measurement. There are 14.69 psia in one standard atmosphere.

- 12 *psig* Pounds per square inch gauge, a unit of pressure measurement. Gauge pressure is the difference between absolute pressure and atmospheric pressure. One standard atmosphere equals 0 psig.
- 211 *pump-down curve* A graphic plot of pressure versus time as a vacuum system is being pumped. Usually plotted on log-log graph paper. Can be used to distinguish real leaks from virtual leaks.
- 31 *pumping speed* A measure of the ability of a vacuum pump to remove gases. It is typically measured in liters per second, cubic feet per minute, or cubic meters per hour.
- 129 *radiation* Heat transfer by energy from infrared light. Radiated heat is the only way to transfer heat inside of a vacuum system at high vacuum.
- 213 *rate of rise* The rate of pressure increase versus time when a vacuum system is suddenly isolated from the pump by a valve. The volume and temperature of the system are held constant during the rate-of-rise measurement.
- 213, 262 *rate-of-rise test* A method of determining whether a leak is present in a system, or of obtaining an estimate of the magnitude of a leak, by observing the rate of rise of pressure in the evacuated system when the system is isolated from the pump. This method also can determine if leakage is real or virtual.
- 152, 253 *real leak* A crack or hole that allows gases to pass through in both directions. See *leak*.
- 58, 101, 114 *regeneration* Some vacuum pumps and traps fill up from usage (containment pumps) and must be emptied periodically. The process of emptying the pump is called regeneration.
- 141 *residual gas analyzer* A gauge that measures partial pressure.
- Roots blower* See *blower pump*.
- roughing* The initial evacuation of a vacuum system.
- 41, 42 *rough pump* A vacuum pump which will function in the rough vacuum range. A roughing pump is often used to "rough" a vacuum chamber. Typical examples of rough pumps are the mechanical pump and the sorption pump.
- 17 *rough vacuum* Pressure which ranges from just below atmospheric pressure to about 10^{-3} torr (0.001 torr).

- sniffer probe* See *probe*. (More correctly called a detector probe.)
- 116 *sputtering* The release of one or more molecules from a cathode surface when that surface is struck by a high-energy ion.
- 11 *standard atmosphere* At 45° N latitude, at sea level, and 0°C, the average pressure exerted on the earth's surface. This average pressure is 14.69 pounds per square inch (absolute), or 14.69 psia.
- 20, 255 *standard cubic centimeter* The quantity of gas in a volume of 1 cc at standard temperature and pressure (0°C, 760 torr).
- 170 *static seal* A seal that does not move. (See *dynamic seal*.)
- 14, 110 *sublimation* The process in which a substance can go directly from the solid state to the vapor state, without passing through a liquid state.
- 110 *sublimes* Changes directly from a solid to a vapor state.
- TC gauge* See *thermocouple gauge*.
- 10 *temperature* A qualitative measurement of energy. The hotter something is, the more energy it contains, thus its temperature is higher.
- 153 *thermal expansion rate* Materials change in size as their temperature changes. This size-to-temperature relationship of the material is called its thermal expansion rate.
- 129 *thermocouple gauge* A vacuum gauge used to measure pressure in the rough vacuum range.
- 31 *throughput* Pumping speed times the pressure. It is a term used to measure the quantity of gas per unit of time flowing through a vacuum system or through a component of that system, such as a pump. Typical units are torr-liters per second. It is a unit of power:
$$5.70 \text{ torr-liters/sec} = 1 \text{ watt}$$
- 12 *torr* Unit of pressure measurement, equal to the force per unit area exerted by a column of mercury one millimeter high. There are 760 torr in one standard atmosphere.
- 271 *tracer gas* A gas which, passing through a leak, can be detected by a specific leak detector and thus reveal the presence of a leak.
- transfer pressure* See *crossover pressure*.

- 28 *transition range* A range of pressure that cannot be correctly defined as either a viscous flow condition or molecular flow condition.
- 48, 78 *trap* A device which will hold selected molecules and not let them pass. Two common types are the molecular sieve trap and the liquid nitrogen trap.
- tubulation* A pipe or hose used in a vacuum system.
- ultimate pressure* The lowest pressure a vacuum pump or vacuum system can reach when clean and empty. Is dependent upon the particular gas species being pumped.
- 17 *ultrahigh vacuum* Pressure which ranges from about 10^{-8} torr (0.00000001 torr) to less than 10^{-14} torr.
- 41, 107 *ultrahigh vacuum pump* A vacuum pump which will function in the ultrahigh vacuum range. Typical examples are the ion pump and the TSP (titanium sublimation pump).
- 40 *useful operating range* The pressure range of a vacuum pump between the higher pressure limit where it will begin pumping and the base (or ultimate) pressure, which is the pump's lower operating limit.
- 2 *vacuum* Any pressure lower than atmospheric pressure.
- 4 *vacuum pump* A type of pump which is capable of removing the gases in an enclosed volume such as a vacuum chamber. Vacuum pumps are typically divided into three broad categories: (1) roughing pumps, (2) high vacuum pumps, and (3) ultrahigh vacuum pumps.
- 14 *vapor* The gas produced as a result of evaporation.
- 14 *vapor pressure* See *pressure, vapor*.
- vent valve* A valve used for letting atmospheric air or other gas into a vacuum system. Also called a BTA or back-to-air valve.
- 152, 252 *virtual leak* An apparent leak that is caused by release of gas from a trapped volume or outgassing of some volatile material or trapped gas inside a vacuum system. See *leak*.
- 25 *viscous flow* The type of flow which occurs when gas molecules are packed closely together and collide with each other quite frequently.
- 2 *work chamber* A contained volume from which some of the air and other gases have been removed. The work chamber separates the vacuum from the outside world. The portion of a vacuum system where the process is performed. See *bell jar*.

Appendix

VACUUM DIMENSIONS AND FORMULAS

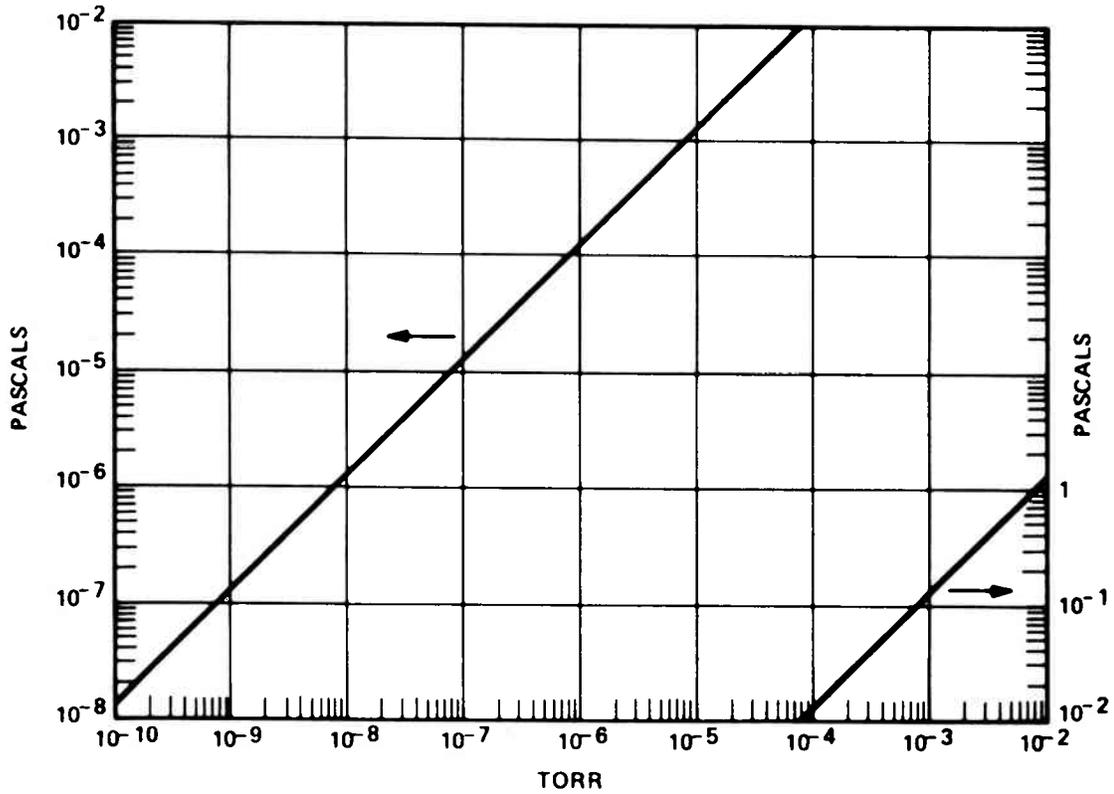
<p>I. Units of Length</p> <p>1 Meter = 100 Centimeters (cm) 1 cm = 10 Millimeters (mm) 1 mm = 1000 Microns (μ) 1 μ = 10^{-3} mm 1 μ = 10^{-6} Meter 1 Liter = 10^3 cm³ 1 Inch = 2.54 cm 1 Ft³ = 1728 in³ = 28316.8 cm³ 1 Ft³ = 28.3 Liters 1 Mile = 160,934 cm = 63,360 in = 5,280 ft</p> <p>II. Units of Mass</p> <p>1 Pound = 453.5 Grams (g) Density of Mercury = 13.546 g cm⁻³ Density of Water = 1 g cm⁻³</p> <p>III. Units of Force</p> <p>1 Dyne = Force Required to Accelerate 1 g, 1 cm sec⁻² 1 Gram = 980.6 Dynes (Gravity, Sea Level, 45° Lat) 1 Newton = Force Required to Accelerate 1 Kg, 1 Meter Sec⁻² 1 Newton = 1×10^5 Dynes 1 Pound = 4.45×10^5 Dynes 1 Pound = 4.45 Newtons</p>	<p>IV. Units of Pressure (Force/Area)</p> <p>1 Atmosphere = 1.013×10^6 Dynes cm⁻² 1 Atmosphere = 14.69 lbs. in⁻² 1 Atmosphere = 1030 Grams cm⁻² 1 Atmosphere = Column of Hg. 76 cm High 1 Atmosphere = Column of Hg. 29.9 Inches High 1 Torr = 1/760 Atmosphere 1 Torr = Column of Hg. 1 mm High 1 Torr = 1.33×10^3 Dynes cm⁻² 1 Micron = 10^{-3} Torr = 1.33 Dynes cm⁻² 1 Bar = 10^6 Dynes cm⁻² (English) 1 Bar = 750 Torr 1 Millibar = .75 Torr 1 Microbar = 7.5×10^{-4} Torr 1 Barye = 1 Microbar = .9975 Dynes cm⁻² 1 Micron = 1.01 Dynes cm⁻² 1 Pascal = 1 Newton Meter⁻² 1 Pascal = 7.5 Microns 1 Torr = 133.3 Pascals 1 lb. in⁻² = 2.036 Inches Hg = 5.17 cm Hg (0°C, 45° Lat)</p>
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VOLUME

from/to	cm ³	liter	in ³	ft ³	fl oz	pt.	qt.	gal.
cm ³	1	.001	0.06102	3.53×10^{-4}	.03381	.00211	.00106	2.64×10^{-4}
liter	1000	1	61.02	0.03532	33.81	2.113	1.057	.2642
in ³	16.39	0.01639	1	5.79×10^{-4}	.5541	.03463	0.01732	.00433
ft ³	2.83×10^4	28.32	1728	1	957.5	59.84	29.92	7.481
fl oz	29.57	0.02957	1.805	0.00104	1	.06250	.03125	.00781
pt	473.2	0.4732	28.88	0.01671	16	1	0.500	0.1250
qt	946.4	0.9463	57.75	0.03342	32	2	1	0.2500
gal (US)	3785	3.785	231	0.1337	128	8	4	1

UNITS OF PRESSURE

Prepared by
American Vacuum Society Standards Committee



	Pascal (N/m ²) (Pa)	Torr	Standard Atmosphere (atm)	Millibar (mbar)	Dyne per Square Centimeter (dyn/cm ²)
1 Newton per square meter (N/m ²) = Pascal =	1	7.5 x 10 ⁻³	9.87 x 10 ⁻⁶	10 ⁻²	10
1 Torr = 1mm Hg =	133	1	1.32 x 10 ⁻³	1.33	1,330
1 standard atmosphere (atm) =	101,000	760	1	1,010	1,010,000
1 millibar (mbar) =	100	0.75	9.87 x 10 ⁻⁴	1	1,000
1 dyne/square centimeter (dyn/cm ²) =	10 ⁻¹	7.5 x 10 ⁻⁴	9.87 x 10 ⁻⁷	10 ⁻³	1

V. Relationship of Temperature Scales

Fahrenheit	Centigrade	Kelvin	
212°	100°	373°	Water Boils
32°	0°	273°	Water Freezes
-459.72°	-273.18°	0°	= Absolute Zero

$$T_F = 1.8 (T_C) + 32 \quad ; \quad T_K = T_C + 273$$

VI. Units of Pump Speed (Vol/Time)

$$\begin{aligned} 1 \text{ Liter Sec}^{-1} &= 2.12 \text{ CFM} \\ 1 \text{ CFM} &= .47 \text{ Liter Sec}^{-1} \\ 1 \text{ Liter Min}^{-1} &= .035 \text{ CFM} \end{aligned}$$

VII. Units of Gas Mass (PV)

$$\begin{aligned} 1 \text{ Molar Volume} &= 22.41 \text{ Liters} \\ &\text{(at standard conditions-STP)} \\ 1 \text{ Mole} &= 6.023 \times 10^{23} \text{ Molecules} \\ 1 \text{ Liter-Atmos} &= 2.68 \times 10^{22} \text{ Molecules} \\ 1 \text{ Std. cc} &= 2.68 \times 10^{19} \text{ Molecules} \\ 1 \text{ Torr-Liter} &= 3.52 \times 10^{19} \text{ Molecules} \\ 1 \text{ Std. cc} &= .76 \text{ Torr-Liter} \\ 1 \text{ Std. cc} &= 1 \text{ Atmos cm}^3 \\ 1 \text{ Cubic Foot} &= 7.6 \times 10^{23} \text{ Molecules} \\ &\text{(Standard Conditions are 1 Atmosphere at 273°K)} \end{aligned}$$

VIII. Units of Throughput (Q = PV/Time)

$$\begin{aligned} (\text{PV} = \text{Work}; \text{Work/Time} = \text{Power}) \\ 1 \text{ Std. cc Sec}^{-1} &= 760 \text{ Micron-Liters Sec}^{-1} \\ &= 1.6 \text{ Torr CFM} \\ 1 \text{ Torr CFM} &= .62 \text{ Std. cc Sec}^{-1} \\ &= 472 \text{ Micron-Liters Sec}^{-1} \\ 1 \text{ Micron-Liters Sec}^{-1} &= 1.32 \times 10^{-3} \text{ Std.} \\ &\text{cc Sec}^{-1} \\ &= 2.12 \times 10^{-3} \text{ Torr CFM} \end{aligned}$$

IX. Vacuum Formulas

A. Conductance = Volume of Gas Flowing Through Orifices and Pipes Per Unit Time

1. Molecular Flow (below 10^{-3} Torr) through orifices

$$\begin{aligned} C_O &= 11.6 \text{ Area in liters per second} \\ &= 11.6 \frac{\pi D^2}{4} D \text{ in cm} \end{aligned}$$

2. Molecular Flow (below 10^{-3} Torr) through long pipes, for air

$$C_p = 11.6 \frac{D^3}{L} \text{ in liters per second}$$

D and L in cm

3. Viscous Flow (10^{-1} to 100 Torr) through pipes*

$$C_v = 180 \frac{D^4}{L} \bar{P} \text{ in liters per second (for air)}$$

D in cm
L in cm
Average P in Torr

4. Conductances in Parallel = C_{Total}

$$C_{\text{TP}} = C_1 + C_2 + C_n \dots$$

5. Conductances in Series = C_{Total} (for long units)

$$\frac{1}{C_{\text{TS}}} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_n \dots}$$

6. Resultant Pumping Speed at a Vacuum System (for compatible elements)

$$\frac{1}{R} = \frac{1}{S} + \frac{1}{C} \quad \begin{array}{l} C = \text{Conductance} \\ R = \text{Liters Per Second} \\ S = \text{Pump Speed} \\ \text{(in liters per sec)} \end{array}$$

B. Fundamental Equation for a System in Equilibrium

$$\begin{aligned} Q &= PS \\ Q &= \text{System gas load in Torr-liters sec}^{-1} \\ P &= \text{Equilibrium pressure in Torr} \\ S &= \text{Effective (resultant) pumping speed} \\ &\text{in liters sec}^{-1} \end{aligned}$$

* Above 100 torr, check for choked-flow conditions

(Q) is also called throughput and is work per unit time ($\frac{PV}{t}$).

C. Ideal Gas Equation

$$PV = nRT$$

$P = \text{Torr}$
 $V = \text{Liters}$
 $n = \text{No. of Moles}$
 $R = \text{Molar Gas Constant}$
 $T = \text{Degrees Kelvin}$

or:

$$PV = nkT$$

$P = \text{Dynes}$
 $V = \text{cc}$
 $n = \text{No. of Molecules}$
 $k = \text{Boltzmann's Constant}$
 $T = \text{Degrees Kelvin}$

D. Mean Free Path, for air

$$L = \frac{5 \times 10^{-3}}{P_{\text{Torr}}} \text{ in centimeters}$$

E. Molecular Velocity

$$a) \bar{V} = 1.5 \times 10^4 \sqrt{\frac{T}{M}} \text{ cm sec}^{-1}$$

$$T = \text{K}^\circ$$

$M = \text{Molecular Weight}$

$$b) \bar{V} = \sqrt{\frac{8KT}{\pi m}} \text{ cm sec}^{-1}$$

$k = \text{Boltzmann's Constant}$
 $T = \text{K}^\circ$
 $m = \text{Molecular Mass in Grams}$

F. Pumpdown (Roughing) Formulas, excluding outgassing

$$a) P = P_0 e^{\frac{-st}{V}}$$

P_0 in Torr (original pressure)
 P in Torr
 S in liters per second
 V in liters
 t in seconds

$$b) t = \frac{V}{S} \ln \frac{P_0}{P}$$

X. Common Values

Acceleration-Gravity g	= 980.6 cm sec ⁻² = 32.174 Ft sec ⁻²
Angstrom Unit \AA	= 10 ⁻⁸ cm
Area Constant π	= 3.1416
Atomic Mass Unit AMU	= 1.66 x 10 ⁻²⁴ grams
Avogadro's Number n	= 6.023 x 10 ²³ mols per mole
Boltzmann's Constant k	= 1.38 x 10 ⁻¹⁶ ergs-deg ⁻¹ -molecule ⁻¹
Electron Charge q	= 1.602 x 10 ⁻¹⁹ coulomb
Mechanical Equivalent of Heat J	= 4.185 x 10 ³ Joules K cal ⁻¹
Molar Gas Constant R	= .82 liter-atmos deg ⁻¹ mole ⁻¹ = 8.31 x 10 ergs deg ⁻¹ mole ⁻¹ = 62.36 Torr-Liter deg ⁻¹ mole ⁻¹
Molecular Diameter d	≈ 3 \AA
Natural Log Base e	= 2.7183
Velocity of Light c	= 3 x 10 ¹⁰ cm sec ⁻¹
Velocity of Molecule \bar{V}	= 4.9 x 10 ⁴ cm sec ⁻¹ (1100 MPH)
Velocity of Sound S	= 3.3 x 10 ⁴ cm sec ⁻¹ (750 MPH)

PHYSICAL PROPERTIES OF GASES AND VAPORS

Gas	Chemical Formula	Molecular Weight M	Molecular Diameter (10 ⁻⁸ cm)
Hydrogen	H ₂	2.016	2.74
Deuterium	D ₂	4.028	2.74
Helium	He	4.002	2.18
Methane	CH ₄	16.04	4.14
Ammonia	NH ₃	17.03	4.43
Water vapor	H ₂ O	18.02	4.60
Neon	Ne	20.18	2.59
Nitrogen	N ₂	28.01	3.75
Oxygen	O ₂	31.99	3.61
Argon	A	39.94	3.64
Carbon dioxide	CO ₂	44.01	4.59
Krypton	Kr	83.80	4.11
Xenon	Xe	131.30	4.85
Mercury	Hg	200.59	4.26

Conductance in Viscous Flow

The volume of gas per unit time (ℓ/sec) that can flow through a long pipe under laminar and nearly incompressible viscous flow conditions is given by:

$$C_v = \frac{180 \ell D^4}{\text{torr cm}^3 \text{sec L}} P_{\text{ave}}$$

where D is the diameter in centimeters,

L is the length in centimeters,

P_{ave} is the average pressure in torr.

Example:

What is the conductance of a 0.25-in. ID tube, 12 in. long, from 10 torr to 1 torr?

Changing inches to centimeters:

$$0.25 \text{ in.} \times 2.54 \text{ cm/in.} = 0.64 \text{ cm}$$

$$12 \text{ in.} \times 2.54 \text{ cm/in.} = 30.5 \text{ cm}$$

Changing atm to torr:

$$1 \text{ atm} = 760 \text{ torr}$$

Calculating the average pressure, P_{ave}

$$\frac{10 \text{ torr} + 1 \text{ torr}}{2} = 5.5 \text{ torr}$$

Substituting into our formula:

$$C_v = \frac{180 \ell}{\text{torr cm}^3 \text{sec}} \times \frac{(0.64 \text{ cm})^4}{30.5 \text{ cm}} \times 5.5 \text{ torr}$$

$$C_v = 68 \frac{\ell}{\text{sec}}$$

These equations are correct only under laminar viscous flow conditions and only for long tubes. As the pressure decreases and molecular flow develops, these equations can't be used.

(continued on next page)

Conductance in Molecular Flow

Here are some equations for molecular flow. They will help you figure the flow through some simple pipes under molecular flow conditions. The first equation is for an orifice:

$$C_o = KA$$

where
$$K_{cm} = 11.6 \frac{\ell}{\text{sec cm}^2} \quad (\text{for air only})$$

or in the English system:

$$K_{in.} = \frac{75 \ell}{\text{sec in.}^2} \quad (\text{for air})$$

A is the area in cm^2 or in in.^2 .

Let's use this formula to calculate the conductance of a 6-in. diameter hole.

Remember that the area of a circle is:

$$A = \pi r^2$$

Substituting the diameter (d) for the radius (r):

$$A = \pi \left(\frac{d}{2}\right)^2 \text{ or } \frac{\pi d^2}{4}$$

Now we can work our problem above; substituting the values into our formula ($C_o = KA$), we have:

$$\begin{aligned} C_o &= \frac{75 \ell}{\text{sec in.}^2} \times 3.14 \times \frac{(6 \text{ in.})^2}{4} \\ &= 2,100 \ell/\text{sec} \end{aligned}$$

Now, let's try the conductance of a 15.24-cm hole:

$$\begin{aligned} C_o &= \frac{11.6 \ell}{\text{sec cm}^2} \times 3.14 \times \frac{(15.24 \text{ cm})^2}{4} \\ &= 2,100 \ell/\text{sec} \quad (\text{for air}) \end{aligned}$$

(continued on next page)

Let's apply this to a length of pipe.

The equation is called the short tube formula and is to be used where the pipe length is not more than 1.5 D:

$$C_p = KAW$$

where K and A are as before and W is:

$$W = \frac{1}{1 + \frac{L}{D}}$$

L is length of pipe and D is diameter.

Let's say we need to know the conductance of a 6-in. diameter pipe, 6 in. long.

$$\begin{aligned} C_p &= \frac{75 \ell}{\text{sec in.}^2} \times 3.14 \times \frac{(6 \text{ in.})^2}{4} \times \frac{1}{1 + \frac{6}{6}} \quad (\text{for air}) \\ &= 1,060 \ell/\text{sec} \end{aligned}$$

Let's try the conductance of a 15.24-cm diameter pipe, 15.24 cm long.

$$C_p = \frac{11.6 \ell}{\text{sec cm}^2} \times \pi \times \frac{(15.24 \text{ cm})^2}{4} \times \frac{1}{1 + \frac{15.24 \text{ cm}}{15.24 \text{ cm}}} = 1060 \ell/\text{sec}$$

What is the conductance of the 0.25-in. tube 12 in. long in molecular flow?

$$\begin{aligned} C_p &= \frac{75 \ell}{\text{sec in.}^2} \times \pi \times \frac{(0.25 \text{ in.})^2}{4} \times \frac{1}{1 + \frac{12 \text{ in.}}{0.25 \text{ in.}}} \\ &= 0.074 \ell/\text{sec} \text{—Not much!!} \end{aligned}$$

In all cases, a change in diameter has a large effect on the conductance. Please remember that these equations must be used only under molecular flow conditions, if answers are to be anywhere near correct.

The long tube formula:

$$C = \frac{Kd^3}{L}$$

applies when the length is more than 10 times the diameter of the tube and in molecular flow.

**VAPOR PRESSURES OF SOME ELEMENTS
AT VARIOUS TEMPERATURES**

Element	Ref.	Vapor Pressure (mm Hg)						mp (°C)
		10 ⁻⁵ at °C	10 ⁻⁴ at °C	10 ⁻³ at °C	10 ⁻² at °C	10 ⁻¹ at °C	1 at °C	
Aluminum	1	882	972	1082	1207	1347	1547	659
Antimony	1	382	427	477	542	617	757	630
Barium	1	417	467	537	617	727	867	710
Beryllium	1	902	987	1092	1212	1367	1567	1283
Bismuth	1	450	508	578	661	762	892	271
Cadmium	1	149	182	221	267	321	392	321
Calcium	1	402	452	517	592	687	817	850
Carbon	1	1977	2107	2247	2427	2627	2867	—
Cesium	1	46	75	110	152	206	277	30
Chromium	1	1062	1162	1267	1392	1557	1737	1903
Cobalt	1	1162	1262	1377	1517	1697	1907	1495
Copper	1	942	1032	1142	1272	1427	1622	1084
Gold	1	987	1082	1197	1332	1507	1707	1063
Indium	1	670	747	837	947	1077	1242	156
Iridium	1	1797	1947	2107	2307	2527	2827	2454
Iron	1	1107	1207	1322	1467	1637	1847	1539
Lead	1	487	551	627	719	832	977	328
Lithium	1	348	399	460	534	623	737	181
Magnesium	1	287	330	382	442	517	612	650
Manganese	1	697	767	852	947	1067	1227	1244
Mercury	1	-28	-8	16	45	81	125	-39
Molybdenum	1	1987	2167	2377	2627	2927	3297	2577
Nickel	1	1142	1247	1357	1497	1667	1877	1452
Osmium	2	2101	2264	2451	2667	2920	3221	2697
Palladium	1	1157	1262	1387	1547	1727	1967	1550
Platinum	1	1602	1742	1907	2077	2317	2587	1770
Potassium	1	91	123	162	208	266	341	64
Rubidium	1	64	95	133	176	228	300	39
Silicon	1	1177	1282	1357	1547	1717	1927	1415
Silver	1	757	832	922	1032	1167	1337	961
Sodium	1	158	195	238	290	355	437	98
Strontium	1	342	394	456	531	623	742	770
Tantalum	1	2397	2587	2807	3067	3372	3737	2997
Thorium	2	1686	1831	1999	2196	2431	2715	1827
Tin	1	882	977	1092	1227	1397	1612	232
Tungsten	1	2547	2757	3007	3297	3647	—	3377
Uranium	1	1442	1582	1737	1927	2157	2447	1130
Zinc	1	208	246	290	342	405	485	420
Zirconium	1	1837	2002	2187	2397	2647	2977	1852

PROPERTIES OF SOME VACUUM GREASES AND OILS

Material	Vapor Pressure (mm Hg at t°C)	Melting Point (°C)	Remarks
Apiezon Oil J	10 ⁻³ at 200°C about 10 ⁻⁵ at 20°C	—	Used where a moderately viscous oil is needed—oil-sealed stopcocks, etc.
Apiezon Oil K	10 ⁻³ at 300°C 10 ⁻⁹ to 10 ⁻¹⁰ at 20°C	—	Used where an exceedingly viscous oil is needed.
Apiezon Grease L	10 ⁻³ at 100°C 10 ⁻⁹ to 10 ⁻¹¹ at 20°C	47	Well-fitting ground joints (not stopcocks). Safe temp. = 30°C.
Apiezon Grease M	10 ⁻³ at 200°C 10 ⁻⁷ to 10 ⁻⁸ at 20°C	44	General purpose grease. Safe temp. = 30°C.
Apiezon Grease N	10 ⁻³ at 200°C 10 ⁻⁸ to 10 ⁻⁹ at 20°C	43	Glass stopcocks. Safe temp. = 30°C.
Apiezon Grease T	about 10 ⁻⁸ at 20°C	125	Safe temp. = 110°C. Used where temperatures up to this value are encountered.
Celvacene, light	10 ⁻⁶ at 20°C	90	Vacuum seals and taps where heating is encountered.
Celvacene, medium	less than 10 ⁻⁶ at 20°C	120	General vacuum grease. Useful where heating is encountered.
Celvacene, heavy	less than 10 ⁻⁶ at 20°C	130	Rubber gaskets and metal to rubber joints where heating is encountered.
Lubriseal	less than 10 ⁻⁵ at 20°C	40	General vacuum grease.
Silicone stopcock grease	less than 10 ⁻⁵ at 20°C	215	Good at high temperatures.
Vacuseal, light	10 ⁻⁵ at 20°C	50	General vacuum grease.
Vacuseal, heavy	10 ⁻⁵ at 20°C	60	General vacuum grease.

DIFFUSION PUMP FLUIDS

Trade Name	Chemical Name	Molecular Weight	Vapor Pressure at 20°C (torr)	Flash Point (°C)	Viscosity 20°C at (centistokes)	Surface Tension dyn/cm
Octoil	Di-ethyl hexyl phthalate	391	10^{-7}	196	75	
DC-704	Tetraphenyl tetramethyl trisiloxane	484	10^{-8}	216	47	30.5
Apiezon C	Paraffinic hydrocarbon	574	4×10^{-9}	265	295	30.5
DC-705	Pentaphenyl trimethyl trisiloxane	546	5×10^{-10} (25°C)	243	170 (25°C)	>30.5
Santovac 5/ Convalex 10	Mixed 5-ring polyphenyl ether	447	1.3×10^{-9} (25°C)	288	2500 (25°C)	49.9

PROPERTIES OF VACUUM MATERIALS

	Melt Point (°C)	Thermal Conductance	Thermal Expansion	Resistance (ohms)	Magnet	Untreated Outgassing Rate (T-ℓ/sec/cm ²)
Aluminum	645	.53	23.6×10^{-6}	2.9×10^{-6}	No	6.6×10^{-9}
Brass	890	.28	20.5×10^{-6}	6.7×10^{-6}	No	4×10^{-7}
Buna-N				$>10^{14}$	No	2.2×10^{-6}
Ceramic	2050	.07	6.9×10^{-6}	$>10^{14}$	No	2×10^{-7}
Copper (OFHC)	1083	.94	16.8×10^{-6}	1.67×10^{-6}	No	1.9×10^{-8}
Glass (Pyrex)			3.5×10^{-6}	$>10^{14}$	No	7.4×10^{-9}
Gold	1063	.72	16.1×10^{-6}	2.3×10^{-6}	No	1.6×10^{-7}
Kovar	1450	.04	5.5×10^{-6}	49×10^{-6}	Yes	
Nickel	1453	.21	13.3×10^{-6}	7×10^{-6}	Slight	
Steel (Mild)	1516	.14	12.2×10^{-6}	15.9×10^{-6}	Yes	5.4×10^{-7}
Steel (304 SS)	1460	.04	15.4×10^{-6}	7.25×10^{-6}	No	1.3×10^{-8}
Tantalum	2996	.13	6.5×10^{-6}	15.5×10^{-6}	No	
Teflon		5.8×10^{-4}	16×10^{-5}	10^{19}	No	6.8×10^{-8}
Titanium	1725	.04	9×10^{-6}	61×10^{-6}	No	1.1×10^{-8}
Tungsten	3400	.40	4.5×10^{-6}	5.5×10^{-6}	No	
Viton						1.1×10^{-6}

PERMEABILITY OF POLYMERIC MATERIALS TO VARIOUS GASES

Type or Trade Name	$\frac{\text{std cc cm}}{\text{sq cm sec bar}} \times 10^8$ (Note bar = 10^4 dynes per sq cm)						
	Carbon Dioxide	Helium	Hydrogen	Nitrogen	Oxygen	Propane	Water
Buna S	92.8	17.3	30.1	4.7	12.8		
Butadiene-acrylonitrile copolymer							
Perbunan 18		12.7	18.9	1.89	6.12		
German perbunan		9.20	11.3	0.84	3.0		
Hycar — OR — 15		5.13	5.35	0.178	0.72		
Hycar — OR — 25		7.40	8.85	0.45	1.76		
Cellulose acetate							
Celanese P-912			6.6		0.81		
Cellulose acetate butyrate							
Kodapak II		10.8	15.8	1.1	2.8	2.0/31c	
Cellulose nitrate		5.1			1.48	0.0057	4740.
Ethyl cellulose							
Ethocel 610	31.6		22.4	5.5	17.9		9750.
Hydropol	36.3	11.8		3.0	8.5	40.5	
Mipolam MP	3.95		4.3	0.2	0.69		
Neoprene G	19.2	3.38	10.2	0.88	3.0		
MEM-213 Polycarbonate*	713.	90.	150.	75.	128.	900.	750.
Nylon 6	0.92			0.0063	0.023		53.
Polybutadiene	103.9		31.6	4.85	14.3		
Polychlorotrifluoroethylene							
Kel-F			0.73	0.0025	0.02		0.22/30c
Trithene		25.5/30c	0.73		0.0079/23c		
Polyethylene							
Alathon 14	9.5	3.7	5.88	0.73	1.65		
Polyethylene terephthalate							
Mylar A	0.888	0.73	0.44	0.0031	0.019		97.8
Polypropylene (0.907 g/cm ³)				0.33/30c	1.72/30c		36.8
Polystyrene							
Dow 0641	5.63/20c		67.6	5.8	18.3		
polyvinyl chloride-dioctyl phthalate							
101-EP-100	0.765/30c		2.6/30c				
Polyvinylidene chlorid							
Saran 517	0.024	0.01	0.05		0.0018	0.00027	7.4
Rubber, butyl							
Oppanol-B-200			4.8	0.22	0.89		
Rubber, hydrochloride							
Pliofilm 140-N2	0.48		1.20				
Pliofilm PM1	0.70			0.109	0.40		
Rubber, methyl	8.3	10.9	12.8	0.36	1.6		
Rubber, natural	105.	25.0	38.3	7.4	17.5	126.	2570.
Rubber, polysulfide							
Thiokol B	2.37		1.2		0.22		
Rubber, dimethyl silicone	2030.	263.	495.	210.	450.	3080.	28500.
Teflon							
FEP	7.51	30.1	9.89	1.44	3.37		
TFE		523.	17.8	2.4	7.5		
Glass							
Fused silica		0.75	0.00011				
Vycor		1.13	0.00038				
Pyrex		0.09					
Soda lime		0.00056					
X-ray shield		0.000 000 31					
Vitreosil		0.480					

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AVS STANDARD (tentative)

AVS 7.1—1966

Graphic Symbols in Vacuum Technology

INTRODUCTION

Purpose. The purpose of this standard is to establish a uniform system of graphic symbols in vacuum technology.

Definition and Application. The graphic symbols are a shorthand used to show graphically the functioning and interconnections of vacuum components in a single-line schematic or flow diagram.

A single-line diagram is one in which the graphic symbols are shown without regard to the actual physical location, size, or shape, of the components.

A symbol shall be considered as the aggregate of all its parts.

The orientation of a symbol on a drawing, including a mirror image presentation, does not alter the meaning of the symbol.

A symbol may be drawn to any scale that suits a particular drawing.

Arrows should be omitted unless necessary for clarification.

Explanation. The graphic symbols are divided into two separate sections, general and specific symbols.

Wherever possible, the general symbol illustrates the function or appearance of a component without regard to special features.

The special symbols elaborate upon the general component categories with individual symbols, which illustrate in detail the special features of the component. Wherever possible, the special symbol utilizes the general symbol outline. Parts from two or more special symbols may be combined, as shown among others by valves Item No. 6, 8, and 9 in Example No. 3 at the end of this standard.

For definitions of the terms used in the description column, see American Vacuum Society, *Glossary of Terms used in Vacuum Technology* (Pergamon Press, New York, 1958).

LIST OF SYMBOLS

I. General Symbols

Item	Description	Symbol	Remarks
1	Pump		
1.1	Mechanical		
1.2	Diffusion		
1.3	Sorption		
2	Vacuum gauge		
3	Valve		
4	Baffle		

Item	Description	Symbol	Remarks
5	Feed-through		Including rotating, sliding, and fixed
6	Vacuum chamber		
7	Lines		
7.1	Connected		Minimum diameter of dots five times line width
7.2	Not connected		

Approved by American Vacuum Society, Inc., 20 August 1965

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AVS Standard

II. Special Symbols

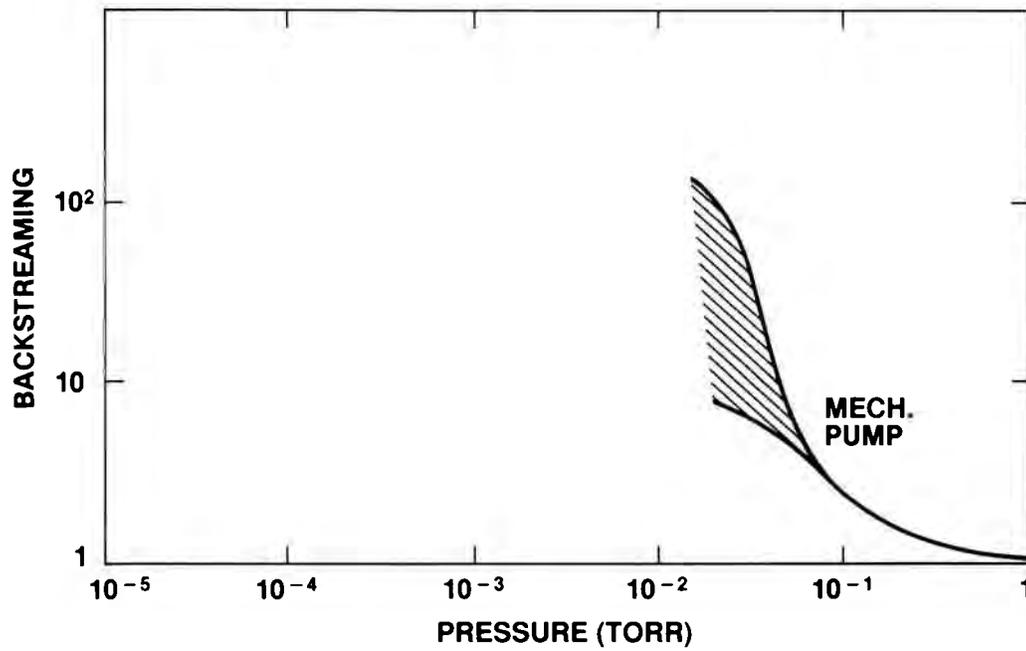
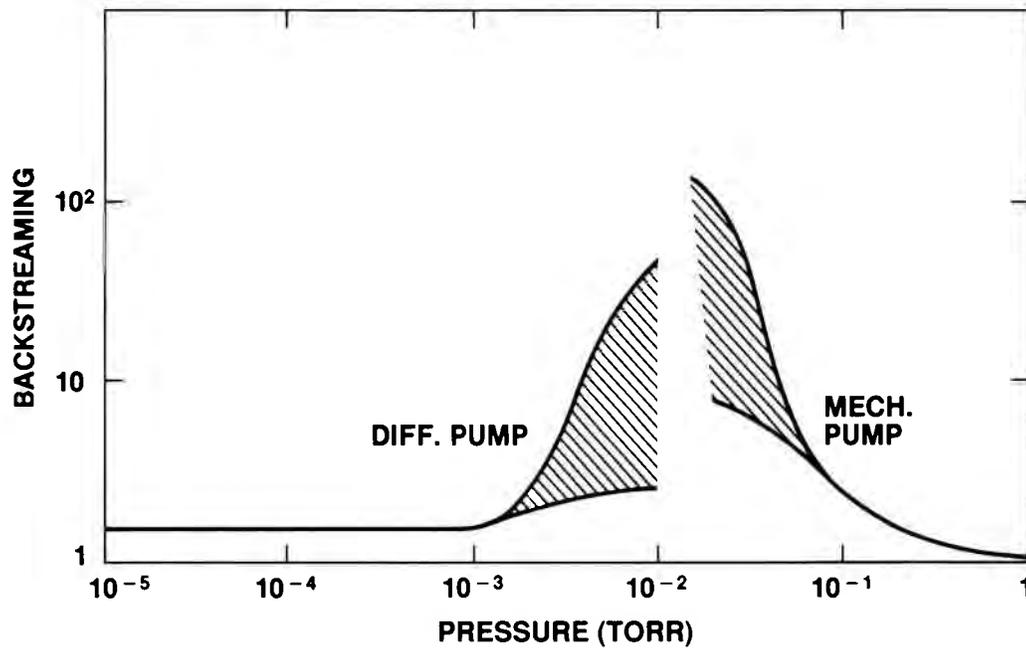
Item	Description	Symbol	Remarks
1.10	Mechanical Pumps		
1.11	Liquid-sealed, single-stage		
1.12	Liquid-sealed, compound		
1.13	Blower, lobe-type single-stage		
1.14	Blower, lobe-type compound		
1.15	Turbomolecular		
1.20	Diffusion Pumps		Optional: add chemical name of oil below symbol
1.21	Diffusion, oil		
1.22	Diffusion, mercury		
1.23	Diffusion, booster		Optional: add chemical name of oil below symbol
1.24	Diffusion-ejector		Optional: add chemical name of fluid below symbol
1.25	Ejector		
1.30	Sorption Pumps		Use element symbol for designation of getter material
1.31	Getter-evaporation		
1.32	Sputter-ion		
1.33	Cryo		Vacuum line (solid) omitted in cryo panels Cryogenic lines (dotted) optional

Item	Description	Symbol	Remarks
1.34	Cryo-sorbent		
2.0	Vacuum Gauges		
2.1	Manometer, liquid level		
2.2	Manometer, diaphragm		
2.3	McLeod		
2.4	Thermocouple		
2.5	Pirani		
2.6	Ionization, cold cathode		
2.7	Ionization, hot cathode		
2.8	Knudsen		
2.9	Residual gas analyzer		
2.10	Radioactive		
2.11	Nude		To specify type of nude gauge, add after N the proper letter or letters from above list.
3.0	Valves		
3.1	Gate or slide		With seat orientation Without seat orientation
3.2	Gate, with bypass port		

AVS Standard

Item	Description	Symbol	Remarks
3.3	Poppet or globe, in-line or angle		Diameter of dot approximately five times line width.
3.4	Ball		
3.5	Butterfly or quarter swing		
3.6	Solenoid		
3.7	Pneumatic		
3.8	Bellows—sealed		
3.9	Throttling or calibrated leak		
3.10	Air admittance		
3.11	Stopcock 2-way, 2-position		
3.12	Stopcock 3-way, 2-position		
3.13	Stopcock 3-way, 3-position		
4.0	Baffles		
4.1	Ambient		
4.2	Refrigerated		For others substitute LN with name of coolant or cooling means.
4.3	Thimble trap		

Item	Description	Symbol	Remarks
4.4	Sorbent		
5.0	Feed-throughs		
5.1	Rotating		
5.2	Sliding		
5.3	Bellows sealed		
5.4	Electrical		
6.0	Vacuum Chambers and Accessories		
6.1	Bell jar		
6.2	View port		
6.3	Blind flange port or door		
7.0	Lines and Connections		
7.1	Flexible line		
7.2	Demountable coupling		

BACKSTREAMING VS. PRESSURE CURVES**RELATIVE AMOUNT OF BACKSTREAMING OIL
FROM A MECHANICAL PUMP****PUMPING AND LUBRICATION FLUID BACKSTREAMING
IN THE TRANSITION ZONE
BETWEEN DIFFUSION AND MECHANICAL PUMPS**

Do You Know Your Solvents?

Jeremy Agnew

Vicon Instrument Company
Colorado Springs, Colorado

Even though we all know better, commonly used chemicals are often treated with such familiarity that we forget the potential hazards that are associated with their use. It is the responsibility of anyone dealing with such chemicals to be aware of all the hazards involved, as well as to conform to government regulations that specify safe handling and storage of chemical solvents. This article summarizes fire, explosion and toxicity hazards for some of the commonly used solvents in the semiconductor and electronics industry. The information is presented in a convenient table for ready reference.

THERE IS A GROWING AWARENESS among many manufacturers that solvents they use may be hazardous, and that they should be handled with care. While this is obviously only a good manufacturing practice to begin with, it is also rigidly specified and enforced by OSHA, as well as a growing number of state and local regulations.

In light of this increasing awareness of workers and management to the hazards of chemical processes, many are finding out that perhaps they don't know the properties of the solvents they use as well as they should.

To help users gain a better understanding of some of the problems involved and to act as a ready reference for the non-chemist, the accompanying Table of Solvents commonly used in the industry has been compiled.

In addition to this specific information, several properties of these solvents can be discussed in general terms. First, there is no such thing as a safe solvent, in spite of the fact that this is what some solvents are often called. For a solvent to be "safe," the conditions of use have to be carefully defined. For example, a solvent that is generally non-hazardous in normal use or in small quantities, may suddenly become very dangerous if a large spill occurs, if there is a malfunction in the ventilating system or if there is an undetected leak in a storage or piping system. All solvents are dangerous to some degree and should therefore be used with suitable caution.

Even a solvent that is considered non-toxic, in the poisonous sense, can kill. Due to improper ventilation, a slow leak or a large spill, fumes from a volatile solvent can displace all the air in an area, not leaving enough oxygen to support life and causing death by suffocation. This can easily happen in large

storage tanks where fumes have accumulated, even though the tank has been emptied.

Similarly, the danger of explosion exists in drums and tanks that have been used for solvent storage. Even though the tank is empty, extreme caution must be observed with open flames, sparks, welding, or even the static electrical discharges that can occur during handling, since enough fumes may remain in the tank to form an explosive fume-air mixture.

Solvent fumes, in general, are dangerous. Even very low concentrations of fumes can form explosive mixtures in the air. For obvious reasons, smoking and open flames must not be allowed in solvent areas and good ventilation should be maintained at all times.

Fumes also present great toxic danger. Inhaling solvent fumes is the most common way of ingesting a solvent. The effect produced will depend on the particular solvent inhaled. Problems can range from eye and respiratory tract irritation, temporary headache, nausea or unconsciousness, to more serious effects that can cause permanent damage to internal organs, such as the liver and kidneys. A more insidious problem is that some solvents can cause chronic poisoning by cumulative absorption of small amounts over a long period of time.

Another danger of fumes, sometimes unrecognized, is that while they may not be particularly hazardous by themselves, they can be changed into other toxic products. An example of this is toxic decomposition by-products formed by heat. Many of the chlorinated and fluorinated hydrocarbons, such as trichloroethylene, 1,1,1-trichloroethane or trichlorotrifluoroethane, when heated, will decompose into hydrochloric acid and phosgene (carbon oxychloride, carbonyl chloride). Both of these are extremely toxic. Hydrochloric acid vapor can cause

chemical burns to the eyes and respiratory tract, and phosgene is so toxic that it was used as a poison gas during World War I.

Though at first the words, "heating a solvent," bring to mind boiling a beaker of trichloroethylene over a bunsen burner, remember that decomposition can also be caused by solvent fumes near an open flame, being drawn through a space heater, or getting into an electric heater or furnace. Another danger, even though remote, is the use of the old-style carbon tetrachloride fire extinguishers in an enclosed area. In all these cases, toxic by-products can be formed.

To control dangers from fumes, suitable precautions must be taken. The obvious one is to eliminate all sources of fumes to begin with. Open solvent containers should be eliminated, spills and leaks stopped. Adequate fresh-air ventilation should be provided at all times. For added safety, work involving solvents should be performed under an exhaust fume hood, wherever possible.

If the work area is to be vented to the outside, be sure to check federal and local clean air regulations, which limit the amount and type of chemicals that may be discharged into the atmosphere.

Fumes are not the only toxic hazard when dealing with solvents. All body contact with solvents is to be avoided. Like inhalation, problems caused by direct contact range from the mild to the serious. All solvents, by their very nature, tend to dissolve oils and fats, and they will do so in contact with the skin by removing the natural skin oils. This defatting action can lead to a drying-out and irritation of the skin, and eventually dermatitis, which can be quite painful and slow to heal.

On the more serious side, some solvents, such as benzene, carbon tetrachloride and trichloroethylene, can be absorbed directly into the body through unbroken skin. These are extremely dangerous solvents too, as they can cause damage to the internal organs and lead to kidney and liver failure. In some cases, such as with benzene and toluene, the toxic effects are cumulative and repeated exposure to even small concentrations can cause later problems.

As with fume hazards, the obvious solution is the best one, allow no skin contact with solvents. Suitable solvent-resistant gloves and aprons can

eliminate this source of danger. To protect the face and eyes, safety glasses or, even better, a full face shield should always be worn when handling solvents. The eyes are particularly vulnerable and solvent absorption through the eyes into the system is extremely rapid. Using a glass-fronted fume hood will help minimize the danger from splashes.

It is not within the scope of this article to discuss first-aid, but provision should be made to have adequate equipment and personnel trained to provide emergency assistance. Eye fountains and showers should be available to provide emergency flushing of solvent-affected areas of the body. Workers dealing with large volumes of solvents should know the techniques of artificial respiration in case someone should be overcome with fumes. Emergency plans should be worked out in advance so that, should an accident occur, emergency procedures can be carried out quickly and efficiently.

Finally, read the labels of all chemicals, note the manufacturers' procedures and cautions and follow them. The information given in this article is believed to be accurate, but it cannot be guaranteed. It is in terms of general recommendations, given for information only. In addition this should not be interpreted as replacing any manufacturer's or user's procedures or recommendations.

For specific information, along with the latest Threshold Limit Values (TLV's) and handling procedures, contact the manufacturer. It is the obligation of each chemical user to assure himself that he has the latest information and is using correct procedures for handling and storage of all chemicals.

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CHARACTERISTICS OF COMMONLY USED SOLVENTS

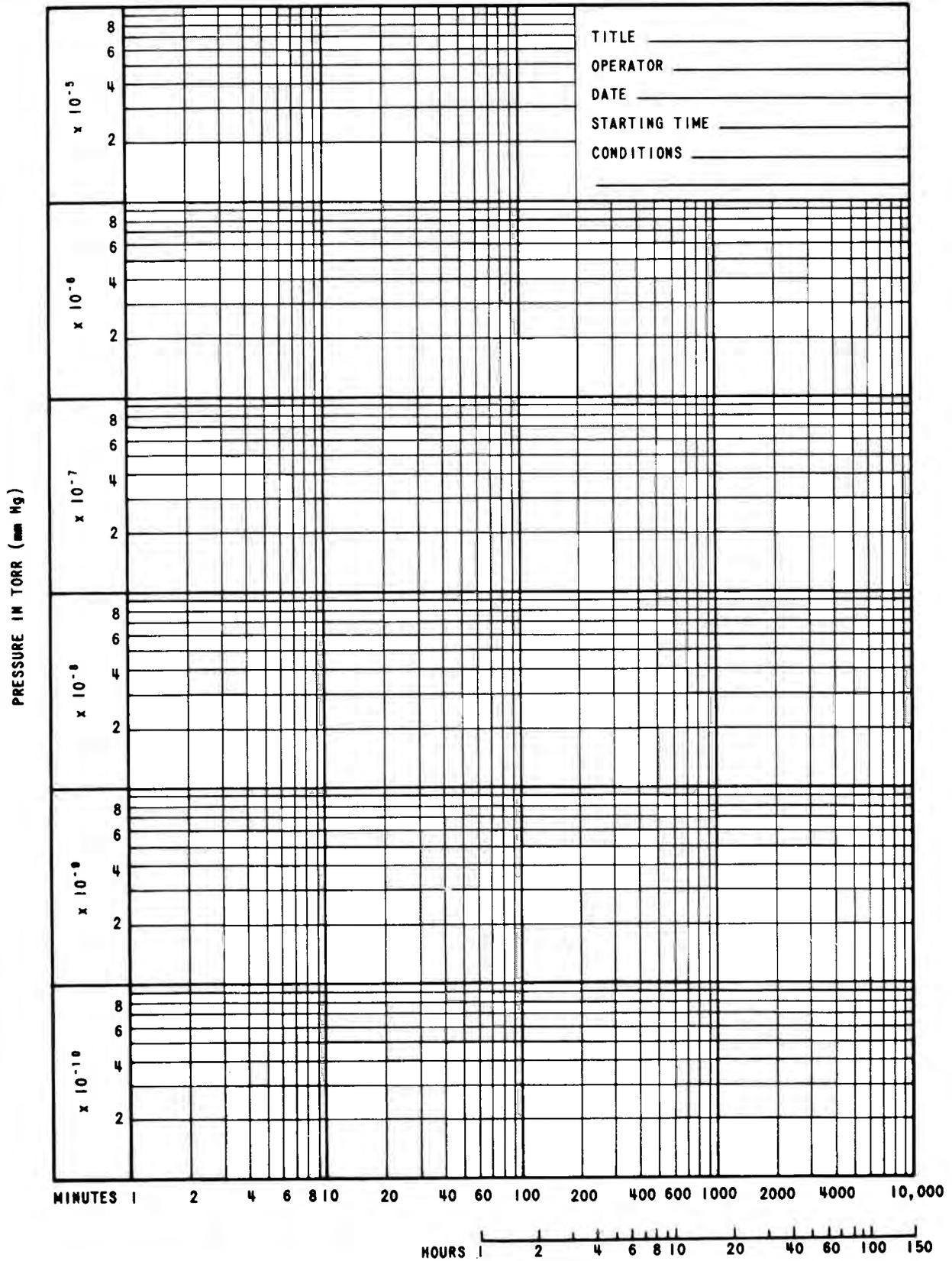
KEY TO TABLE

TLV = Threshold Limit Value. Maximum safe solvent concentration for repeated exposure, in parts per million in air. TLV numbers are periodically revised. Check with the manufacturer for the latest data. Largest assigned TLV number = 1000.

N = Not generally considered hazardous.
L = Generally considered low hazard.
M = Moderately hazardous.
E = Extremely hazardous.
** = Not defined.

Name	Other Common Names	Chemical Family	TLV	Chemical Formula	Fire Hazard	Explosion Hazard	Toxicity Hazard	Toxicity
Acetone	Dimethyl Ketone 2-Propanone	Ketone	1000	CH ₃ COCH ₃	E	E	L	Low toxicity; narcotic action; causes headaches; irritates eyes and respiratory tract.
Benzene	Coal Tar Naphtha Benzol	Aromatic Hydrocarbon	25	C ₆ H ₆	E	M	E	Extremely dangerous; absorbed directly through unbroken skin; chronic poison that can lead to internal organ damage.
n-Butyl Acetate		Ester	150	CH ₃ CO ₂ C ₄ H ₉	M	M	M	Irritates eyes and respiratory tract; narcotic action.
Carbon Tetrachloride	Tetrachloromethane	Chlorinated Hydrocarbon	10	CCl ₄	N	N	E	Extremely dangerous; use not recommended for anything; absorbed directly through unbroken skin; can cause kidney and liver damage; toxic fumes when heated.
Ethyl Alcohol	Ethanol Grain Alcohol	Alcohol	1000	C ₂ H ₅ OH	E	M	L	Low toxicity; irritates eyes and respiratory tract; causes headaches.
Ethylene Dichloride	Ethylene Chloride	Chlorinated Hydrocarbon	50	CH ₂ ClCH ₂ Cl	E	M	E	Irritates respiratory tract; can cause liver and kidney damage.
Isopropyl Alcohol	Isopropanol 2-Propanol	Alcohol	400	CH ₃ CHOHCH ₃	E	M	L	Low toxicity, but irritating to eyes and respiratory tract.
Kerosene	Fuel Oil No. 1	Aliphatic Petroleum	**	Hydrocarbon Mixture	M	M	L	Low toxicity; narcotic action.
Methyl Alcohol	Methanol Wood Alcohol	Alcohol	200	CH ₃ OH	E	M	M	Irritates eyes and respiratory tract; produces headaches and nausea; continued exposure may cause internal damage; ingestion can cause blindness.
Methylene Chloride	Dichloromethane Methyl Dichloride	Chlorinated Hydrocarbon	500	CH ₂ Cl ₂	N	N	E	Highly toxic; can damage internal organs; toxic fumes when heated.
Methyl Ethyl Ketone	MEK 2-Butanone	Ketone	200	CH ₃ COC ₂ H ₅	E	M	M	Irritates eyes and respiratory tract.
Mineral Spirits	Paint Thinner Petroleum Spirits	Aliphatic Petroleum	**	**	M	M	L	Low toxicity, but use adequate ventilation.
Perchloroethylene	Drycleaning Solvent	Chlorinated Hydrocarbon	100	C ₂ Cl ₄	N	N	M	Irritates eyes and respiratory tract; produces headaches; toxic fumes when heated.
Toluene	Toluol	Aromatic Hydrocarbon	100	C ₇ H ₈	E	M	M	Moderately toxic; absorbed through unbroken skin; chronic effect can cause cumulative poisoning.
1,1,1-Trichloroethane	Methyl Chloroform	Chlorinated Hydrocarbon	350	CH ₃ CCl ₃	N	N	M	Toxic fumes when heated.
Trichloroethylene	Ethylene Trichloride	Chlorinated Hydrocarbon	100	C ₂ HCl ₃	N	N	M	Absorbed through unbroken skin; can lead to kidney damage; toxic fumes when heated.
Trichlorotrifluoroethane	"Freon" (T.M. of E.I. du Pont de Nemours & Co., Inc.)	Fluorinated Hydrocarbon	1000	CCl ₂ FCClF ₂	N	N	L	Generally considered non-toxic, but use adequate ventilation.
Xylene	Xyloil	Aromatic Hydrocarbon	100	CH ₃ C ₆ H ₄ CH ₃	E	E	E	Highly toxic; absorbed through unbroken skin.

LOG-LOG CHART FORM FOR PRESSURE VS. TIME*



*You have our permission to copy this chart.