DEVELOPMENT OF EXPERIMENTS ON VACUUM TECHNOLOGY

A project report submitted in partial fulfillment of the requirements for the degree of

Bachelor of Technology
In
Mechanical Engineering

By

BISWARANJAN MOHANTY

NATIONAL INSTITUTE OF TECHNOLOGY
ROURKELA

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Under the guidance of
Prof. S. K. SARANGI

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CERTIFICATE

This is to certify that the Project entitled "**DEVELOPMENT OF EXPERIMENTS ON VACUUM TECHNOLOGY**" submitted by **Sri Biswaranjan Mohanty,** in partial fulfillment of the requirements for the award of ***Bachelor of Technology*** **Degree** in **Mechanical Engineering** at **National Institute of Technology, Rourkela** is an authentic work carried out by him under my supervision and guidance.

The matter embodied in the thesis has not been submitted to any of other university/institute for the award of any degree or diploma, to the best of my knowledge.

Place: NIT Rourkela
Date:

(Prof. S.K. Sarangi)
Department of Mechanical Engineering
NIT Rourkela

ABSTRACT

Improvements in vacuum science and technology have triggered its application to wide areas of knowledge and it now plays an important role in many different industrial and research environments. The increasing use of elaborate and well designed vacuum systems leads to the need for well-trained staff and engineers in this area. The education and training of researchers and technicians by actual practice in laboratories has importance and significance in a field like vacuum science and technology. This is of particular importance for undergraduate and graduate student education, given the limited time available for teaching those curricula. It is very important for students' education and training that direct quantitative measurements can be obtained during the experimental work, rather than just the simple operation of vacuum systems and qualitative analysis. Simple experiments, allowing students to perform direct measurements of the characteristics of different vacuum components and material properties, are thus important. We will describe simple experiments intended for didactic laboratory vacuum classes of undergraduate courses, where actual measurements are performed and compared with the values tabulated. These experiments are intended for five to six times 4-h laboratory classes of an introductory vacuum course for undergraduate students majoring in Physics, Physics Engineering and Material Sciences. Small high vacuum systems are used with a rough vacuum gauge at both the high-vacuum chamber and mechanical pump inlet. This allows the monitoring of the pressure in the vacuum chamber during the roughing procedure and after the high-vacuum valve is closed.

Helium leak detectors have become common in both research and industrial environments. They have changed from luxury equipment, requiring expert handling, to economic, reliable and powerful monitoring instruments, which are relatively easy to use. Therefore, it is important to include these systems in the experimental training of students. A simple experiment, using a He leak detector to measure the helium permeability of different materials, is presented.

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CHAPTER 1

INTRODUCTION

1.1. VACUUM

The Latin word *vacuum* means "empty". The vacuums achieved in "vacuum systems" used in physics and in the electronics industry are far from being absolutely empty inside, however. Even at the limits of pumping technology, there are hundreds of molecules in each cubic centimeter of volume. Still, compared to the atmospheric density of 2.5×10^{19} molecules/cm3, the relative crowding is much less in the vacuum system.

Vacuum is officially defined by the American Vacuum Society as a volume filled with gas at any pressure under atmospheric. For purposes of interesting physics, the "real" vacuum range does not begin until about 1/1000 of an atmosphere.

1.2. VACUUM TECHNOLOGY

The technology dealing with the production of such reduced-pressure environments using different scientific concepts is known as "vacuum technology".

1.3. VACUUM UNITS

The vacuum field is plagued by a super-sufficiency of units for measuring pressure, and it is worth a moment's study to familiarize oneself with the various units used in different texts and manuals. Widely used unit is the Torr or millimeter of mercury, based on the traditional mercury manometer technique for measuring pressure. One atmosphere is 1.01×10^5 Pa or 760 Torr. Other units and their inter-conversions are given in Table1

Unit	Pascal	Torr	atm	bar	psi
1 Pascal = 1 N/M^2	1	$750.06x10^{-5}$	$9.8692x10^{-6}$	10^{-5}	$1.4503x10^{-4}$
1 Torr = 1 mm Hg	133.22	1	1.3158x10-3	0.00133	0.01934
1 atm = 760 Torr	101325	760	1	1.0133	14.69
1 bar = 0.1 MPa	105	750.06	0.98692	1	14.503
psi	6895	51.717	0.068505	0.06895	1

Table 1.1 Conversion factors between various systems of pressure units.

1.4. PHYSICAL PARAMETERS AT LOW PRESSURE

The changes occurs in physical parameter in vacuum chamber, as pressure goes down is given below

- Molecular density (n)
- Mean free path (λ)
- Time to form a monolayer (τ)

Molecular density **(n)** defined as the average no. of molecule per unit volume, which is related to the gas pressure (P) and absolute temperature (T).

$$P = n \times k_B \times T$$

Where, k_B is the Boltzmann constant (1.38×10^{-23} JK^{-1})

Mean free path **(λ)** is the average distance that a molecule travels in a gas medium between two successive collisions with other molecule of the gas. According to kinetic theory of gases λ is related to particle diameter d and molecular density n.

$$\lambda = \frac{1}{\sqrt{2}\,\pi\, n\, d^2}$$

Time to form a monolayer **(τ)** is the time required for a fresh solid surface to be covered by a gas layer of one molecule thickness. τ is given by

$$Z = \frac{1}{4}\, n\bar{C}$$

$$\tau = \frac{a}{z} = \frac{4a}{n\bar{C}}$$

Where, Z is the impingement rate.

a is the number of free sites per unit surface area

$\bar{C}$ is the average speed

1.5. CLASSIFICATION OF VACUUM RANGE

The terms “high vacuum”, “ultrahigh vacuum”, “low vacuum” and the like are often used in vacuum textbooks and laboratory discussions. The borders between the regions are somewhat arbitrary, but a general guideline is given in Table 2.

Vacuum range	Pressure Range (Pa)
Low	$10^{5} - 3.3\times10^{3}$
Medium	$3.3\times10^{3} - 10^{-1}$
High	$10^{-1} - 10^{-4}$
Very high	$10^{-4} - 10^{-7}$
Ultrahigh	10^{-7} -

Table 1.2 Vacuum ranges, as given in Dictionary for Vacuum Science and Technology.

In a low vacuum range the molecules in the chamber collides more with each other than with the covering surface and hence the molecular mean free path λ is smaller compared to the characteristic dimension D, ($\lambda << D$). The flow of gases in such condition is viscous.

Gas molecules in high vacuum system are located principally on surfaces as the mean free path is larger than the characteristic dimensions ($\lambda > D$). The flow in this system is molecular without viscous drag.

In the range of ultra high vacuum, where time to form monolayer is τ is longer than the usual time for laboratory measurements ($\lambda >> D$). And the gas flow is purely molecular.

1.6. APPLICATION OF VACUUM TECHNOLOGY

Vacuum technology is an extremely important tool for many areas of physics. In condensed matter physics and materials science, vacuum systems are used in many surface processing steps. Without the vacuum, such processes as sputtering, evaporative metal deposition, ion beam implantation, and electron beam lithography would be impossible.

Vacuum cleaner, vacuum packaging of food, vacuum encapsulation of sensitive device, removing humidity from food, chemicals and removing active constituents of the atmosphere (oxygen, water vapour) are done by vacuum technology. It also accelerates filtering speed in chemical industries.

A high vacuum is required in particle accelerators, from the cyclotrons used to create radio nuclides in hospitals up to the gigantic high-energy physics colliders such as the LHC.

CHAPTER 2

PRODUCTION & MEASUREMENT OF LOW PRESSURE

2.1. PRODUCTION OF LOW PRESSURE

Since vacuum technology extends on so many ranges of pressure, no single pump has yet been developed, which is able to pump down a vessel from atmospheric pressure to the high vacuum or ultra high vacuum. The different principles are involved in the various pumps to lower the number of molecule present in the gas.

1. *Compression - expansion of the gases:-* Piston pump, rotary pump, root pump
2. *Drag by diffusion effects:-* Vapour diffusion pumps
3. *Molecular drag:-* Turbo molecular pump
4. *Ionization effects:-* ion pump
5. *Physical and chemical sorption:-* Sorption pumps, cryo-pumps

Here we had briefly discussed the operation of mechanical rotary pump and vapour diffusion pump, as these pumps are play an important role in maintaining

2.1.1. MECHANICAL ROTARY VANE PUMP

Rotary vane pumps typically have a stator and an eccentric rotor which has one to three sliding vanes that maintain close contact with the inner wall of the cylindrical stator. The stator is a steel cylinder, the ends of which are closed by suitable plates, which hold the shaft of rotor. The stator is pierced by the inlet and exhaust ports. The inlet port is connected with the vacuum system and exhaust port is provided with a valve.

The rotor consists of a steel cylinder mounted on driving shaft. Its axis is parallel to the axis of the stator, but is displaced from the axis (eccentric), such that it makes contact with the top surface of the stator. The diametrical slot is cut through the length of the rotor and caries the vane. The vanes are metal in oil sealed pumps, and carbon in dry pumps. Centripetal force acts upon the vanes in the spinning rotor so as to force them against the inner sealing surface of the stator. In some mechanical pumps springs are used to augment this action.

Rotary vane pumps may be of the single or double stage design. Single stage pumps are simpler, having only one rotor and stator, and are less expensive. The base pressure one can expect from a good single stage mechanical pump is about 20 mTorr. In a two stage design, the exhaust port of the first stage is connected to the inlet port of the second stage which exhausts to atmospheric pressure. Two stage pumps may attain a base pressure of one to two mTorr, but are more expensive than single stage pump.

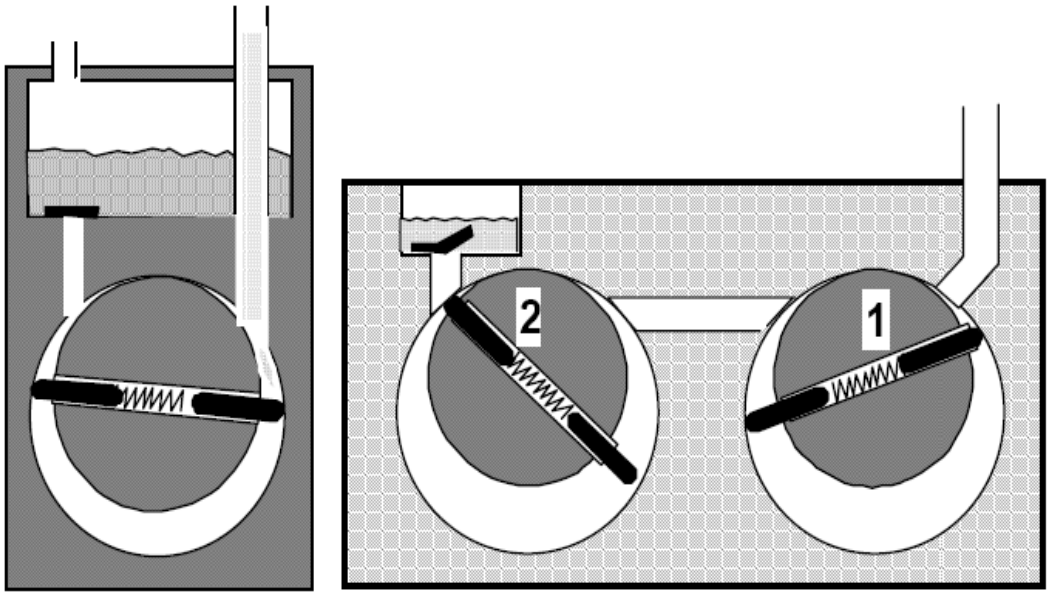


Fig 2.1 Simplified drawings, (A) single stage oil sealed rotary vane mechanical pump, (B) two stage pump of the same type.

In the compound design the high vacuum side of the pump (stage labeled 1) operates at a lower pressure due to the lack of exposure to high partial pressures of oxygen in that stage. It should be noted that supply of very little or no oil to the first stage of a compound pump in order to achieve even lower pressures can, in practice, lead to severe difficulties in the reliable operation of a compound pump.

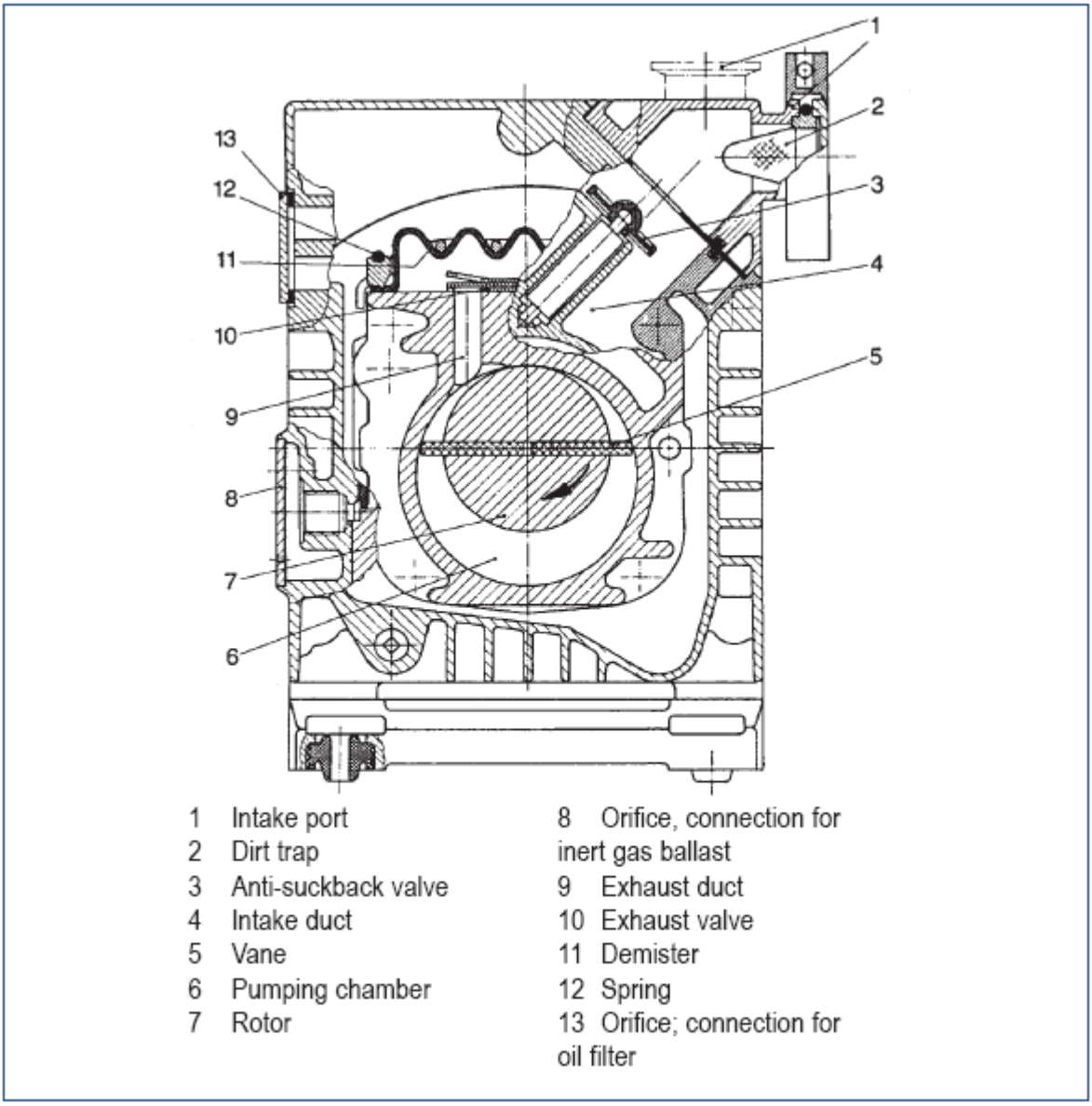


Fig 2.2 Cross sectional view of single stage rotary vane pump

The oil in an oil sealed pump serves three important functions:

- providing a vacuum seal at the pump exhaust,
- as a lubricant
- Provides cooling for the pump.

Working principle of rotary vane pump

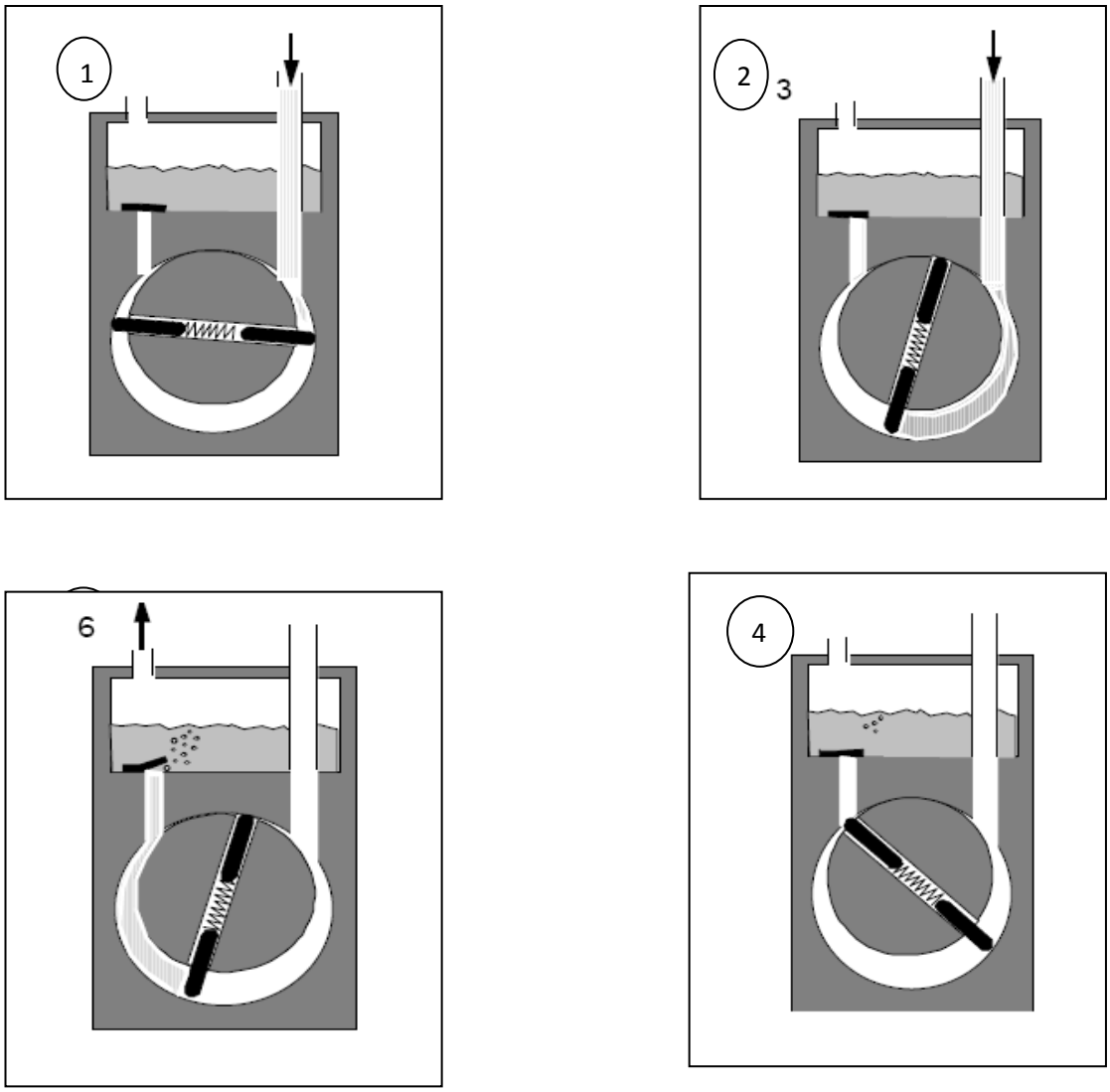


Fig 2.3 Working of single stage rotary vane pump

The gas is shown for only one cycle.

(1) Gas from the vacuum volume enters the cylinder.

(2) The rotor has turned 120º from (1) and the gas from the vacuum volume is now on the exhaust side of the cylinder.

(3) The rotor has turned an additional 180º and the gas has been forced out the exhaust.

(4) The rotor has turned an additional 120º and all gas has been forced out and the exhaust valve has been closed.

2.1.2. DIFFUSION PUMP

Diffusion pumps are vapour jet pumps or vapour ejector pumps designed for pumping rarefied gases in the high –vacuum range (<10^-2 torr) of pressures. It uses a high speed jet of vapor to direct gas molecules in the pump throat down into the bottom of the pump and out the exhaust. These are called “diffusion” pumps because of the fact that the molecules of the pumped gas penetrate the vapour jet in a manner resembling diffusion of one gas to another and carried with it to the exhaust. However, the principle of operation might be more precisely described below, since diffusion plays a role also in other high vacuum pumps.

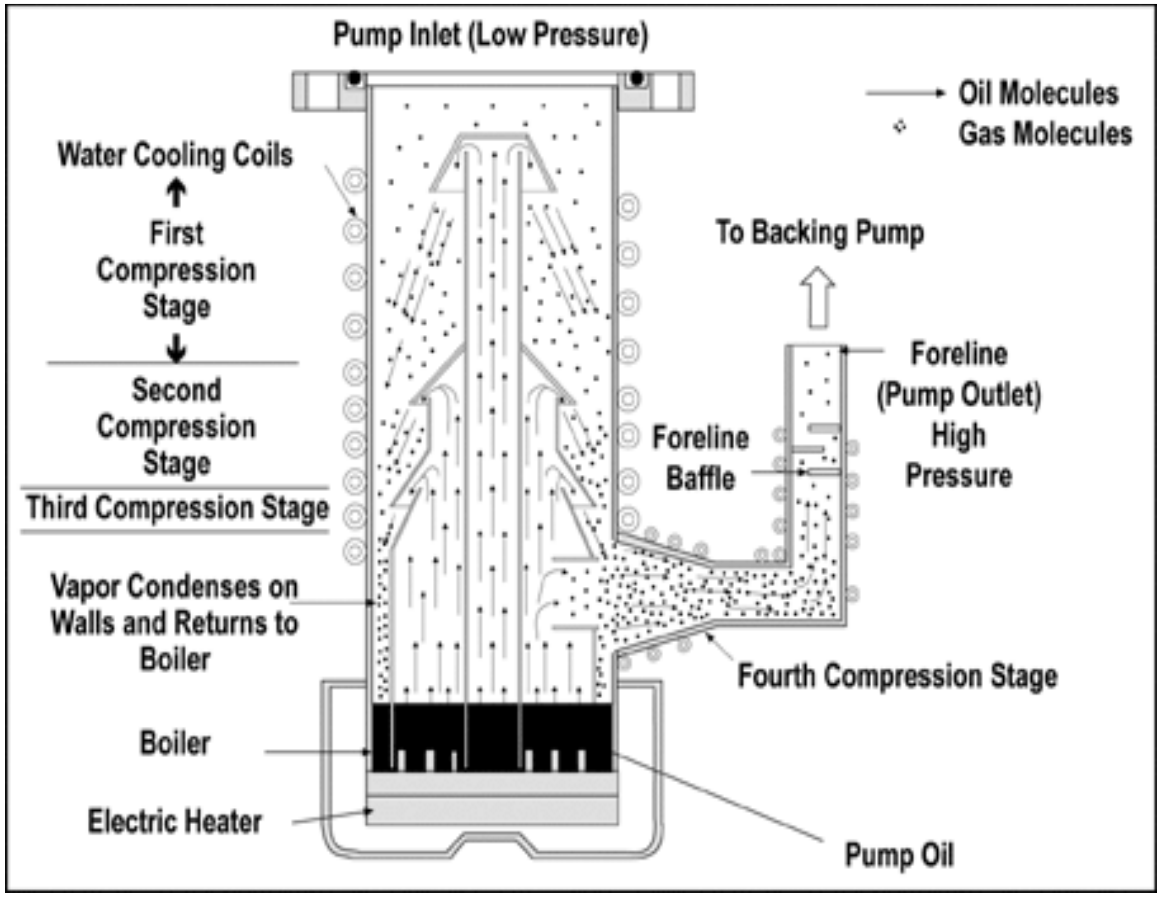


Fig 2.4 Schematic Diagram of Diffusion Pump

Working principle

A pumping fluid of low vapour pressure is boiled in the boiler by the action of heater. The oil vapour flows up through the jet chimneys, deflected at jet caps and emerges out (downwards) from the jet nozzles at supersonic velocities. The oil molecules condense on the pump walls which are water cooled and flow in the form of an oil film, back down to the boiler where the oil is re-boiled and evaporated.

Gas molecules present in the chamber above the jet assembly diffuse into the vapour stream (jet) where they are given the download momentum due to collision with heavier oil molecules. Thus the molecules are forced by the jet into the region of high pressure in the lower section of diffusion pump. The pressure here is high enough for the backing rotary pump to have a finite pumping speed so the accumulated gas molecules are drawn off through the fore – vacuum line.

The general tendency in the development of diffusion pump oil has been towards the attainment of oils of lower vapour pressure and greater resistance to oxidation. The silicone oil (DC 704) offers a higher resistance to oxidation at elevated temperature. Since, each oil type has different pressure and temperature characteristics, and since the boiler temperature affect oil decomposition, both the pump heater power and the cooling water flow have a strong effect on the performance of the pump.

The speed of diffusion pump varies pressure from different gases.

The pumping speed of diffusion pump is determined by the size of the intake clearance and Ho-factor. The area A (cm^2) of intake annulus is

$$A = \frac{\pi D^2}{4} - \frac{\pi (D - t)^2}{4}$$

Where D is the diameter of the intake port, and t/2 is the throat width.

It is impossible for a gas of molecular weight M and temperature T to pass through this area at flow rate exceeding

$$S_{max} = 3.64(\frac{T}{M})^{1/2} A$$

For air at 20^{o} C exceeding

$$S_{max} = 11.6A \text{ Lit/sec}$$

The ratio between admittance (i.e. the pumping speed S across the throat of the pump) and the maximum flow rate S_{max} is known as ho-factor or speed factor (H).this is usually H= 0.3-0.45, best modern pumps have H= 0.5.

The diffusion pump may categorized as a momentum transfer pump. The diffusion pumps uses both in industrial and research applications. Most modern diffusion pumps use silicon oil as working fluid.

These two pumps are widely used to create vacuum in lower range to higher range.

2.2. MEASUREMENT OF LOW PRESSURE

The pressure in vacuum system is defined as the force exerted by the gas per unit surface area. Instruments used to measure pressure are called **pressure gauges** or **vacuum gauges**. As the vacuum technology extends to wide range, there is no single gauge which is able to cope with such a range.

Based on different physical principles the gauges are classified as

- Mechanical gauge: Bourdon, Diaphragm, liquid Manometers and mcleod.
- Thermal conductivity gauges: Thermocouple, Pirani.
- Ionization gauges: these gauges are two types.
  - Hot – cathode gauges: Schulz and Phelps, Conventional, bayard-Alpert, Orbiton, Magnetron
  - Cold- cathode gauges: Penning, magnetron, Inverted Magnetron.

In this section we are briefly discuss about widely used gauges.

1. Bourdon gauge
2. Pirani gauge
3. Penning gauge

2.2.1. BOURDON GAUGE

A Bourdon gauge is a mechanical gauges, uses a coiled tube, which, as it expands due to pressure increase causes a rotation of an arm connected to the tube.

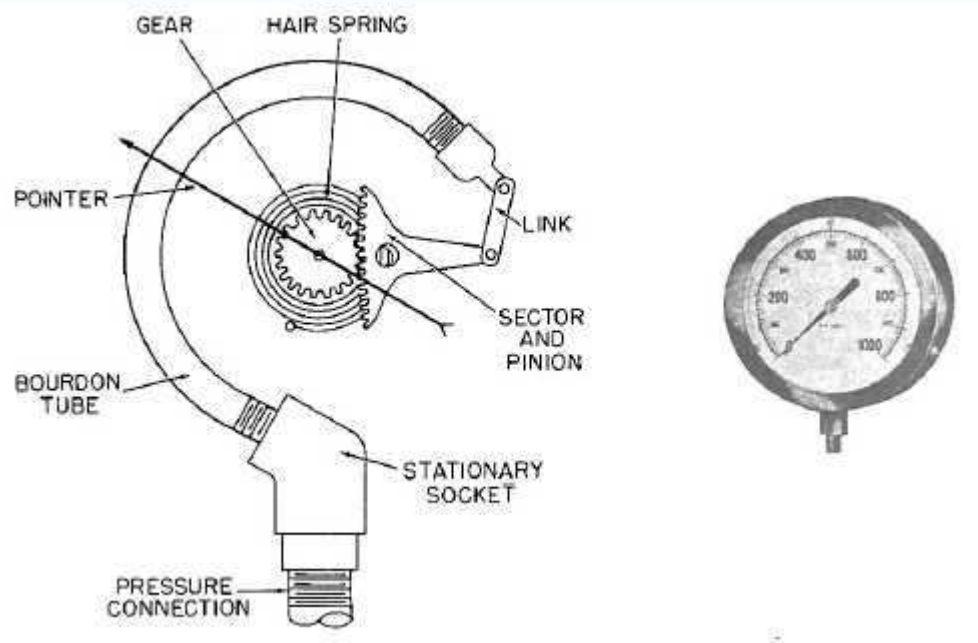


Fig 2.5 Bourdon Gauge

The pressure sensing element is a closed coiled tube sealed at one end and connected at the other to the chamber or pipe in which pressure is to be sensed. A pointer is attached by a mechanical linkage to the free sealed end of the tube and moves over a calibrated scale. As the gauge pressure increases the tube will tend to uncoil, while a reduced gauge pressure will cause the tube to coil more tightly. This motion is transferred through a linkage to a gear train connected to an indicating needle. The pressure indications are associated with particular needle deflections.

2.2.2. PIRANI GAUGE

In the Pirani gauge, one of the most widely-used gauges, it consists of a glass or metal envelope containing a heated filament of a metal with a high temperature coefficient of resistance, such as platinum or tungsten.

The measurement is carried out by heating a filament and measuring its temperature as a function of the input power. The temperature of the wire can be determined from its resistance: as the pressure in the gauge tube increases, the temperature of the filament and therefore its electrical resistance tends to decrease. The usual control for a pirani gauge is the Wheatstone bridge as shown below.

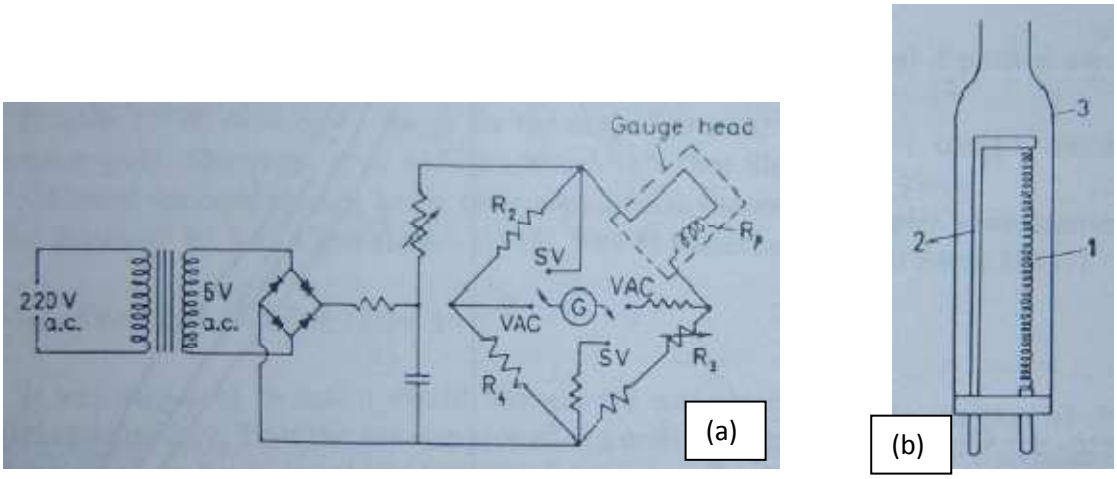


Fig 2.6 (a) Circuit for Pirani Gauge (b) Pirani Gauge Head

In which one leg of the bridge is the filament of the gauge tube R_p and the other three legs have resistance nearly equal to that of the gauge tube.

The resistance R_2 and the R_4 are fixed, while R_3 and R_p are variable. With the milliamperemeter G connected in the VAC position, the balance condition of the bridge is

$$R_p = R_2 R_3 / R_4$$

One method to measure the pressure in the gauge head R_p is to balance the bridge by varying R_3 and calculate the Resistance R_p, a previous calibration permitting to convert the values of the resistance into pressure.

Another method is to keep R_2 and R_4 constant and present R_3 and to measure the out of balance current through G. in this case it is essential to keep the voltage across the bridge constant.

The pirani gauge head includes a tungsten, nickel or platinum filament wire wound in a helix of 0.5-2 mm outside diameter, with a pitch of at least 10 weir diameters to prevent any one turn from shielding its neighbours. The filament is stretched between supports to which it spot welded. Pirani gauges can generally measure down to about 0.1 Pa.

2.2.3. PENNING GAUGE

One common type of cold-cathode gauge is the Penning or Phillips gauge. In this gauge, a much higher voltage (_2kV) is used than in hot-cathode gauges, but there is no heating to the cathode. The gauge is formed from two cathode plates with a wire Loop anode halfway between the two.

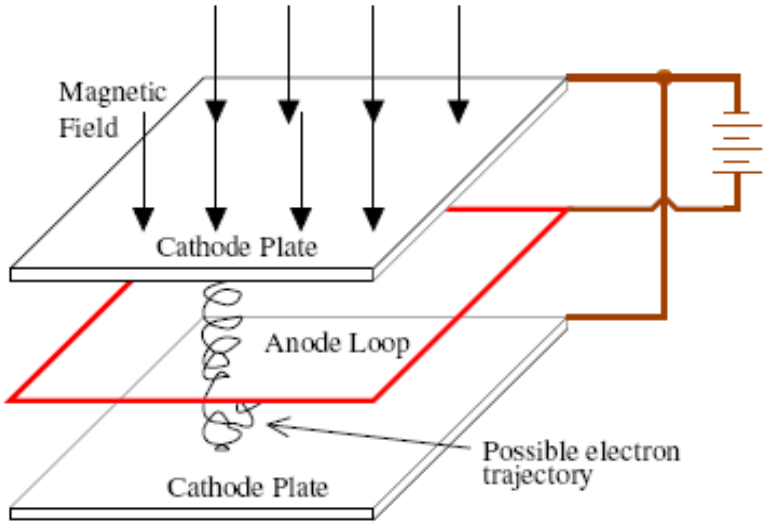


Fig. 2.7 working principle of Penning gauge

The electrons emitted from the cathode are accelerated towards the plane of the anode loop by an electric field. The entire gauge is immersed in a magnetic field (order of 500 Oerseds) perpendicular to the plane of the plates. This field makes the electrons curve in a helix. The electrons curl down through the plane of the anode loop until they are pushed back by the other cathode and they oscillate a very large

number of times in the field before encountering the ring of the anode. This long path and high kinetic energy give a very large chance of ionizing a gas molecule. The positive ions created are captured by the cathodes, producing an ion current in the external circuit. The gauge is operated from a control unit consisting rectified a.c. power supply

The only disadvantage of the gauge is that it can be hard to start the discharge at low pressures. Sometimes a small filament or beta source is used to start the discharge. Once started, the process is self-sustaining. The upper limit of this gauge's sensitivity is the pressure for glow discharge (_ 1 Pa) whiles its lower limit appears when the density becomes too low for the discharge to continue. Generally, Penning gauges are limited to 10^{-8} Pa.

CHAPTER 3

VACUUM MATERIALS

3.1. VALVES

This is a one type of assembly, used to isolate the one part from other part in vacuum system. There are different types of valve available in the market. Those are

- Butterfly valve
- Needle valve
- Solenoid cum air admittance valve

3.1.1. BUTTERFLY VALVE

Butterfly valves are useful for atmosphere to high vacuum pressures. The valve has a circular valve plates with an 'O' ring at the edge. The O ring around the valve plate makes the seal against the valve body. The valves are made of SS304 and are available both with manual and pneumatic operation. Shafts are provided with o-ring seals.

3.1.2. NEEDLE VALVE

Manually operated micrometer controlled needle valve can be used for admitting a defined, clearly reproducible flow of gas into a vacuum chamber. It essentially consists of a SS hosing, a regulating needle of SS and a valve seat made of

lead. In closed condition, the needle is pressed against the valve seat by a spring. The needle moves in axial direction and it is coupled to an actuator knob fitted with ball bearing. Fine threading of needle ensures fine control and reproducible opening for gas flow.

Features:

1. Constant gas flow
2. Reproducible gas flow
3. Leak tightness better than 10-9 std.cc/sec
4. Any installation position
6. Negligible dead volume.

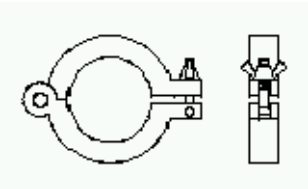
3.1.3. SOLENOID CUM AIR ADMITTANCE VALVE

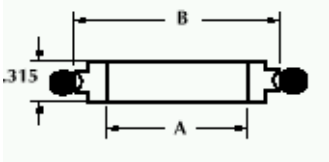
The solenoid valve is used along with rotary pump. It is fitted on to the pump inlet and is connected in parallel with the pumps electrical supply. When the electrical power is on, the valve is held closed.

In case of an interruption of power supply, the solenoid supply ceases and air vent opens and admits air into the pump. This isolates to some extent the vacuum system and prevents the pump oil from reaching the system.

3.2. CLAMPS`

These are used in joint to hold 2 parts tightly. A centre ring with the o-ring is placed between the two parts to prevent leakage of gas through the joint.





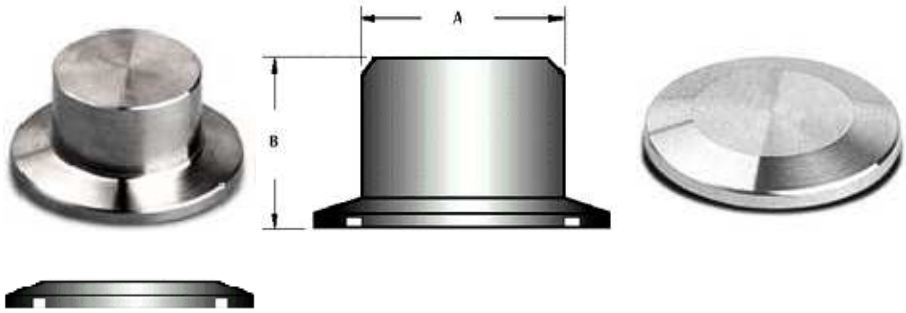
3.3. O-RINGS

These are made of either neoprene or viton. These are used for proper sealing. These are used in valves, joints of flanges, in clamps with o-ring etc.

3.4. OTHER COMPONENTS

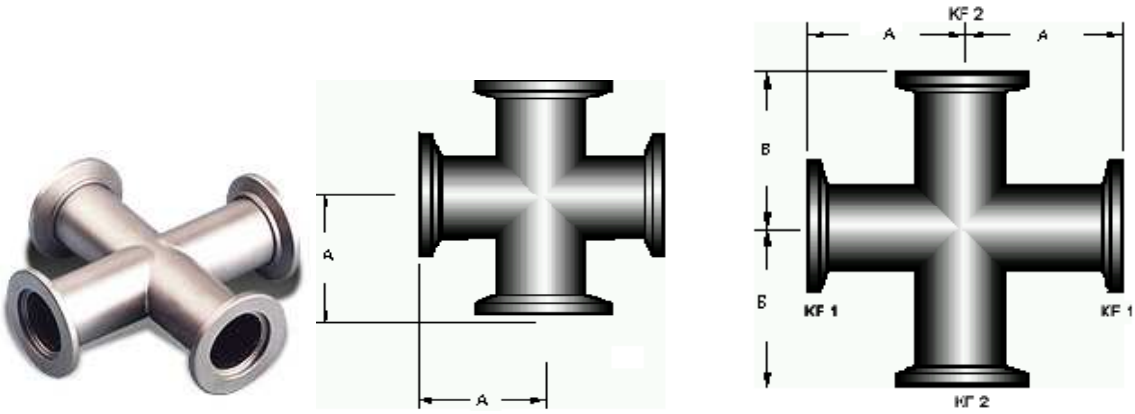
Blanks

These are used at dead end to close the opening



(KF Blank stubs) (KF Blank flanges)

4-way crosses

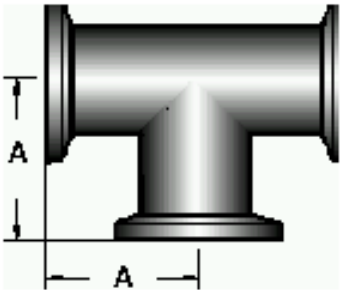


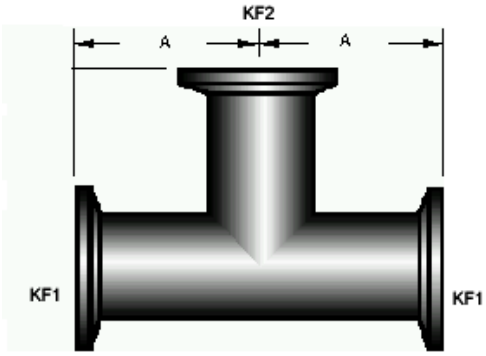
Nipple

(Half nipple)

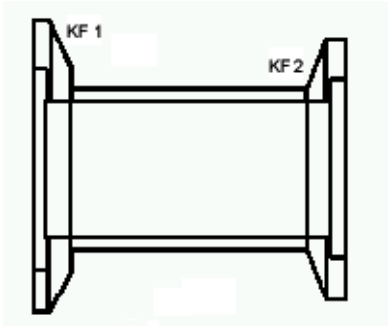
(full nipple)

Tee

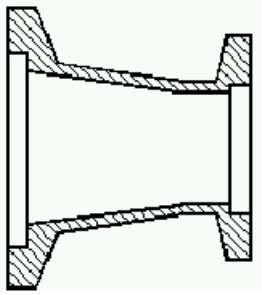




Reducers



Straight reducers



Conical Reducers

CHAPTER 4

MEASUREMENT OF PUMPING SPEED

4. PUMPING SPEED

The **pumping speed** listed by manufacturer for any given type of pump is usually the free air displacement at STP (standard temperature and pressure). As pressure decreases from atmospheric, there will be a reduction in the amount of gas pumped per unit time (the mass flow rate). The pumping speed (volumetric flow rate) will decrease only slightly until a pressure of about 1 Torr is attained. Below this pressure, the decrease in pumping speed becomes more rapid, depending upon the type of mechanical vacuum pump, and falls to zero at the ultimate pressure.

The pumps used in a vacuum system remove (evacuate) gas from the system. The rate at which the gas is removed is measured by pumping speed S_p .The pumping speed is defined as volume of the gas (which the pumping device removes from the system) per unit time. It is expressed in lit/sec, m^3/hr etc.

$$S_p = dV/dt$$

The throughput Q is defined as the product of pumping speed and inlet pressure,

$$Q = PS_p = P\ (dV/dt)$$

The throughput is also defined as the quantity of the gas, in pressure × volume units, at a specified temperature, flowing per unit time across a specified cross section. It is expressed in Torr.liter/sec or Pa.m^3/sec.

4.1. PUMPING SPEED BY CONSTANT VOLUME METHOD

4.1.1. THEORY

Determining the speed of a pump can be accomplished by measuring either pumping speed under constant volume or constant pressure conditions. The constant volume technique is generally used in the pressure range between atmospheric and one Torr. In this method, you will measure the time required to reduce the pressure

in a vessel a specified amount. The pump speed in that pressure range is then calculated using the equation.

$$S_p = 2.3\left[\left(\frac{V}{t_2 - t_1}\right) Log_{10}\left(\frac{P_1}{P_2}\right)\right]$$

Where V is the volume of enclosure, t_1 is the time at pressure p_1 and t_2 is the time at pressure p_2

MATHEMATICAL DERIVATION OF THE FORMULA

According to ideal gas equation

$$PV = \frac{m}{M} RT$$

Where P = pressure of the gas, V = volume, m = mass of the gas

M = molecular mass of the gas, R = universal gas constant, T = temperature of the gas

$$PV \propto m$$

$$\dot{PV} = Mass\,Flow\,Rate$$
$$= Throughput\,(Q)$$

$$\frac{d(PV)}{dt} = Q = S_p P$$

At constant volume

$$V\frac{dP}{dt} = S_p P$$
$$\Rightarrow V\frac{dP}{P} = S_p\, dt$$

Integrating both sides,

$$V\int_{P_2}^{P_1}\frac{dP}{P}=S_p\int_{t_1}^{t_2}dt$$

$$\Rightarrow V\ln P\Big]_{P_2}^{P_1}=S_p(t_2-t_1)$$

$$\Rightarrow S_p=\frac{V}{t_2-t_1}\ln\frac{P_1}{P_2}=2.3\frac{V}{t_2-t_1}\log\frac{P_1}{P_2}$$

Where V is the volume of enclosure,

t_1 is the time at pressure p_1 , t_2 is the time at pressure p_2

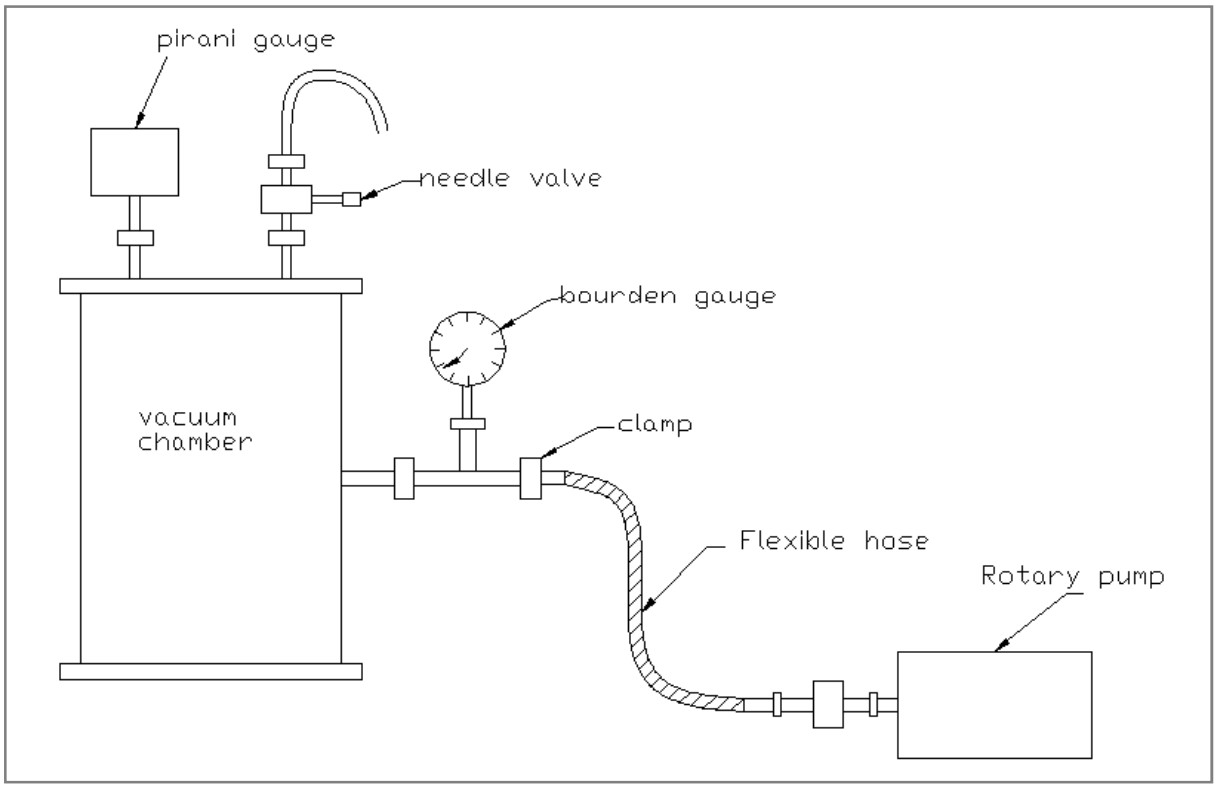


Fig. 4.1 Schematics Diagram of Experimental Setup for constant volume method

The equipments are assembled as shown above, making the connecting (flexible hose) as short as possible to minimize the line impedance (remember that any leaks in the assembly may lead to inaccuracy in the data to be collected.)

4.1.2. PROCEDURE

- Install all the setup as shown in above diagram.
- Check that all the valves are closed and the vacuum chamber is at atmospheric pressure.
- Start the mechanical pump, and after it warmed up, open the valve to vacuum vessel.
- Record the time to achieve the subsequent pressure (i.e. 1000-50,50-1,.. mbar).
- Repeat the measurement 2-3 times, and take the readings.
- Close the valve, and then turn off the rotary pump.
- Record the data in the table and calculate the pumping speed.
- Plot the log- log graph between pumping speed and average pressure.
- Plot the characteristic curve between pressure and time.

4.1.3. TABULATION

Volume of vacuum chamber (V):-_____

Sl. No.	**Initial pressur**	**Final pressur**	**Average pressure**	**Time taken to reach p2**			**Average time**	**Pumping speed**
	p_1	p_2	$p_{avg} = \frac{p_1 + p_2}{2}$	t_1	t_2	t_3	$t = \frac{t_1 + t_2 + t_3}{3}$	$S_p = 2.3\frac{V}{t}\log\frac{p_1}{p_2}$
	mbar	**mbar**	**Mbar**	**Se**	**Se**	**Se**	**Sec**	**Liter/sec**

Fig 4.2 Apparatus Set in IIT KGP for measurement of pumping speed by constant volume method

Fig 4.3 Apparatus Set in NIT RKL for measurement of pumping speed by constant volume method

4.1.4. BILL OF MATERIAL

Item	Component	Specification	Quantity
1	Rotary vane pump	VT-2006 (100 lit)	1
2	Digital pirani gauge	VT-DHP-II	1
3	Bourdon gauge		1
3	Valve	Needle valve(KF 10)	1
		Solenoid Cum Air Admittance Valve(KF	1
		Butterfly valve(KF 10)	1
4	Bellows	KF10-VT-SSB1 (50cm)	1
		KF10-VT-SSB1 (100cm)	1
5	KF Clamps (clamp+ center ring+ o ring)	KF10-VT-CS	5
		KF25-VT-CS	6
		KF40-VT-CS	2
6	KF Blank Flanges	KF10-VT-BFS	2
		KF16-VT-BFS	1
		KF25-VT-BFS	4
		KF40-VT-BFS	2
7	Conical Reducer	KF-VT-2516CR	2
		KF-VT-4025CR	2
		KF25-VT-X	5
9	Tee	KF10-VT-T	2
		KF25-VT-T	2
10	O-Rings: Neoprene	KF10-VT-ON	5
		KF25-VT-ON	5
		KF40-VT-ON	4
11	Vacuum Chamber	Capacity:-20 lit. For more detail see the attached AutoCAD drawing. (Approximate Dimensions are given).	1

SAMPLE READINGS

Sl. No.	Initial pressur	Final pressur	Average pressure	Time taken to reach p2		Average time	Pumping speed
	p_1	p_2	$p_{avg} = \frac{p_1 + p_2}{2}$	t_1	t_2	$t = \frac{t_1 + t_2}{2}$	S_p
	mbar	**mbar**	**Mbar**	**Sec**	**Sec**	**Sec**	**Liter/sec**
1	980	392	686	6.42	6.14	6.28	1999.58518
2	392	196	294	5.92	6.01	5.965	682.50288
3	196	78	137	7.7	8.17	7.935	317.8094
4	78	40	59	5.38	5.51	5.445	144.56454
5	40	5	22.5	19.48	20.31	19.895	46.98156
6	5	1	3	17.44	15.5	16.47	5.85658
7	1	0.5	0.75	9.2	7.95	8.575	1.21114
8	0.5	0.1	0.3	35.85	31.12	33.485	0.28806
9	0.1	0.07	0.085	17.47	18.94	18.205	0.03327
10	0.07	0.05	0.06	19.93	21.35	20.64	0.01954
11	0.05	0.045	0.0475	11.64	11	11.32	0.00883
12	0.045	0.04	0.0425	10.57	11	10.785	0.00927
13	0.04	0.035	0.0375	15	14	14.5	0.0069
14	0.035	0.03	0.0325	36	35	35.5	0.00282
15	0.03	0.028	0.029	25.92	22	23.96	0.00167
16	0.028	0.026	0.027	26.51	23	24.755	0.00161
17	0.026	0.024	0.025	44.52	33	38.76	0.00103
18	0.024	0.22	0.023	54.36	50	52.18	7.662E-4

Table 4.1 Readings for pumping speed by constant volume method

Sl.No.	*Pressure*	*Time*
	mbar	*sec*
	P	*T*
1	980	0
2	392	6.42
3	196	12.34
4	78	20.04
5	40	25.42
6	5	44.9
7	1	62.34
8	0.5	71.54
9	0.1	107.39
10	0.07	124.86
11	0.05	144.79
12	0.045	156.43
13	0.04	167
14	0.035	182
15	0.03	218
16	0.028	243.92
17	0.026	270.43
18	0.024	314.95
19	0.022	369.31

Sl.No.	*Pressure*	*Time*
	mbar	*sec*
	P	*T*
20	0.03	385
21	0.04	403
22	0.045	411
23	0.05	420
24	0.055	433
25	0.06	444
26	0.07	463
27	0.08	483
28	0.085	493
29	0.09	505
30	0.095	517
31	0.1	529
32	0.11	550
33	0.13	599
34	0.14	628
35	0.15	656
36	0.16	680
37	0.17	708

Table 4.2 Readings for Characteristic curve of rotary vane pump

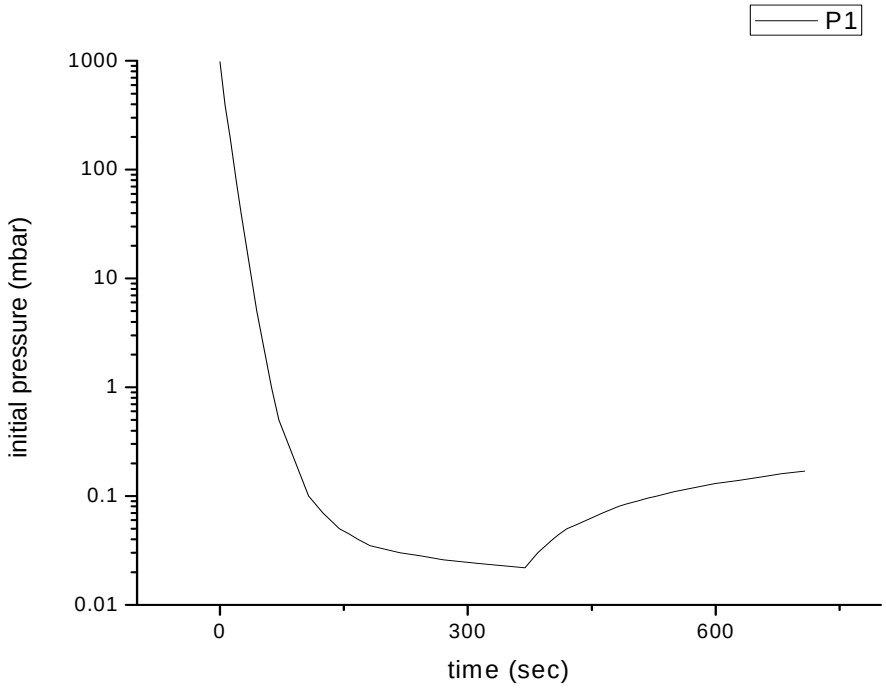


Fig. 4.4 Characteristic Curve (Pressure vs Time)

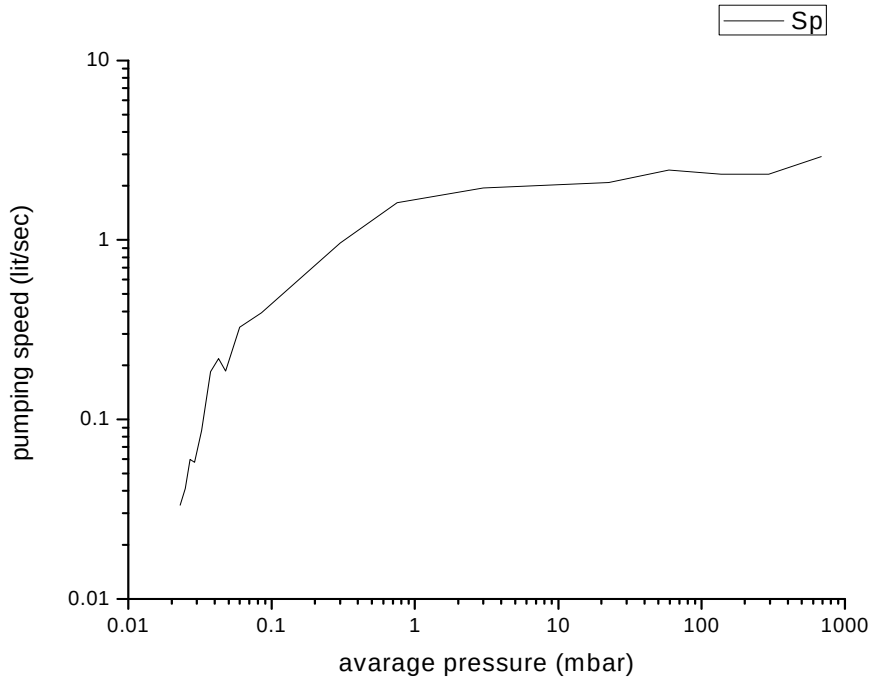


Fig. 4.5 Plot between pumping speed vs average pressure

4.1.5. DISCUSSION

From the plot in fig. 4.4, it can be seen that the pressure decreases gradually with time but when the chamber is isolated from the pump at 10^{-2} mbar range by a valve, the pressure starts increasing, which is due to gas which penetrates through leaks, that which evolves from the walls (outgassing) and that entering by permeation.

From the plot in fig. 4.5, it can be seen that the maximum pumping speed achieved by the rotary vane pump is in the region of 1000 to 10 mbar is 2.9 liter per sec which is equivalent to 180 liter per min approx., The pumping speed gradually decreases as pressure decreases. It is slow enough in the pressure range of 10^{-2} mbar. Environmental condition like presence of moisture in the air, some mechanical loss in pump etc., plays an important role in affecting pumping speed.

4.2. PUMPING SPEED BY CONSTANT PRESSURE

In contrast to the constant volume method, the measurement of pumping speed at constant pressure is typically performed in the pressure range between one Torr and the mechanical pump's ultimate pressure. To determine pumping speed by the constant pressure method, a measured *amount* of gas (Q) is admitted to the vacuum system being pumped to establish a constant pressure P.

4.2.1. THEORY

The rate at which the pump is removing air (pumping speed) can be obtained from the equation:

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

Where S_p is the pumping speed (vol. of gas per unit time)

Q is quantity of gas flowing through a pipe (Torr lit/sec)

At a constant pressure in the chamber, the quantity of gas flowing out of the chamber should be equal to quantity of gas flowing into the chamber

At P = constant, $Q_{out} = Q_{in}$

And $Q_{in} = P_{atm} \times V^{\cdot}$

Where, $V^{\cdot}$ is volume flow rate.

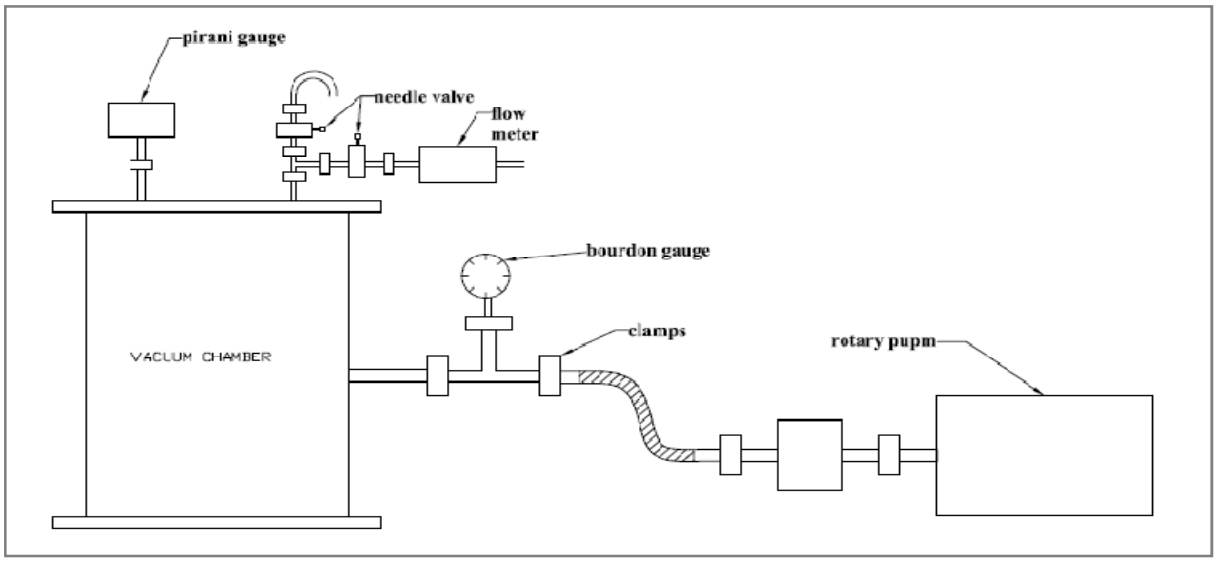


Fig. 4.6 Schematic Diagram of Experimental set up of constant pressure method

4.2.2. PROCEDURE

- Install all the setup as shown in above diagram, use the minimum amount of connecting line to reduce conductance losses
- Check that all the valves are closed and the vacuum chamber is at atmospheric pressure
- Start the mechanical pump, and after it warmed up, open the valve to vacuum vessel
- Maintain a pressure in vacuum chamber by adjusting the needle valve, and the measure the flow volume.
- Take the reading for different pressure.
- Repeat the measurement 2-3 times, and take the readings.
- Close the valves and then turn-off the rotary pump
- Record the data in the table and calculate the pumping speed
- Plot the log- log graph between pumping speed and average pressure.

4.2.3. TABULATION

Sl. No.	Pressure in vacuum chamber	Volume flow rate	Pumping speed
	P	$V^{\cdot}$	$S_P = \frac{P_{atm} \times V^{\cdot}}{P}$
	mbar	Lit/sec	Lit/sec

4.2.4. BILL OF MATERIAL

For this experimental setup, we need all materials which are used in experiment to measure pumping speed by constant volume method, given in the table 4.2. The additional things used were needle valve and a flow meter to measure the rate of volume flow.

Fig 4.7 Apparatus Set in IIT KGP for measurement of pumping speed by constant pressure method

4.2.5. DISCUSSION

This is an accurate method to calculate the pumping speed. The problem in this method is to calculate the rate of volume flow ($\dot{V}$) within a wide range of vacuum. For the measurement of volume flow rate one can use flow meter or measuring the pressure difference across a capillary tube and using Hagen Poiseuille equation. This is a difficult method but suitable and proper to calculate the pumping speed.

CHAPTER 5

PUMP DOWN CHARACTERESTICS OF DIFFUSION PUMP

5.1. PUMP DOWN CHARACTERISTICS OF DIFFUSION PUMP

We had briefly discussed about working of diffusion pump in chapter 2, as we know Diffusion pumps are used when constant high speeds for all gases are desired for long periods of time without attention. Diffusion pumps cannot discharge directly into atmosphere. A mechanical pump is required to reduce the pressure into the vacuum system to the correct operating range. The mechanical pump is then used to maintain proper discharge pressure conditions for the diffusion pump. It generally removes 99.9% of air from the vacuum.

The typical arrangement of high vacuum pumping module with valves, mechanical rotary pump, diffusion pump and piping network are shown below.

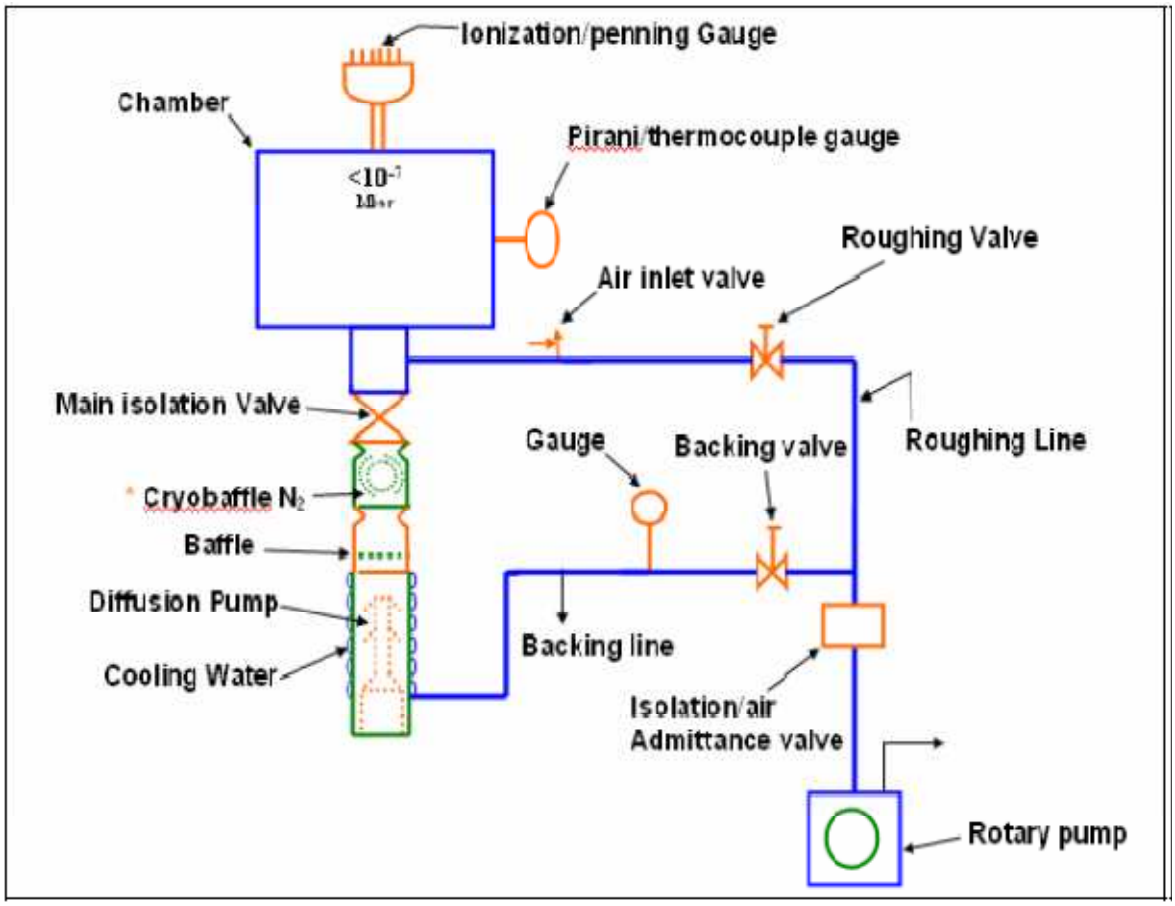


Fig. 5.1 Schematic Layout of Arrangement of valves for a diffusion pump system

The main isolation valve is used to isolate chamber from diffusion pump when required. The roughing valve is used to pump the chamber independently by a rotary pump. The backing valve is used to pump the chamber by diffusion pump along with

rotary pump. Pirani (thermal conductivity) gauge and penning (ionization) gauges are used to measure the low pressure in the chamber.

The diffusion pump fluid can be continuously contaminated by the mechanical fore-pump due to transfer of low-vapour pressure mechanical pump oil, this transfer can be can be eliminated by the use of a suitable adsorption or condensation trap in the fore line.

Backstreaming or the transfer of diffusion pump fluid (oil) beyond the pump flange can be effectively stooped by a properly designed of trap.The cryogenic trap and baffles connected above the diffusion pump improves the performance of diffusion pump in two ways.

- It acts as barrier for the flow of condensable vapour from pump to the system.
- Act as cryopump for condensable vapour emanating from the system, in which pressures far below the vapour pressure of the pump liquid may be achieved.

Precautions should be taken not to expose the hot pump oil to atmosphere to avoid oxidation. Vapour diffusion pumps, with correctly chosen accessories and appropriate pumping fluid can produce ultimate pressures down to 10^-11 torque.

5.2. PROCEDURE

- Assembled all the components such as rotary pump, diffusion pump, backing line, roughing line, water cooling circuit, baffle, pressure measuring gauges, valves etc. as shown in above diagram
- Check that all the valves are closed and the vacuum chamber is at atmospheric pressure
- Start the mechanical pump, and after it warmed up, open the backing valve to diffusion pump.
- Run the rotary pump for some times through backing line to evacuate the diffusion pump up to 10^{-2} mbar.
- Connect the chilled water line.

- Switch on the diffusion pump heater. (after 20 minutes diffusion action starts)
- After 20 minutes close the backing valve and open the roughing valve.
- Evacuate the chamber through roughing line and take the reading of pressure at different time.
- As pressure of vacuum chamber reached to 10^{-2} Torr. Close the roughing valve, and open the backing valve.
- Open the main isolation valve, to connect diffusion pump with the vacuum chamber.
- Record the pressure of vacuum chamber at different time.
- Close the main isolation valve, and then turn off the diffusion pump.
- Allow the rotary pump to run along backing line for some times after the turning off the diffusion pump
- Close the backing valve and turnoff the rotary pump.
- Plot the semi - log graph between pressure and time.

5.3. TABULATION

Sl. No.	Time (sec)	Pressure (mbar)

Fig 5.2 Apparatus Set in NIT Rourkela for pump down characteristics of Diffusion Pump

5.4. BILL OF MATERIAL

Sl. No.	Item	units
1	Diffusion pump(500 watt)	1
	Rotary pump(300 lit/sec)	1
2	Vacuum chamber	1
3	Pirani gauge	2
	Penning gauge	1
4	Main isolation valve	1
	Butterfly valve (KF 25)	2
	Air admittance valve	1
	Needle valve	1
5	Cold trap & baffle	1
6	Clamps, and other piping assembly	

Sample tabulation

sl. No.	final pressure mbar	time sec
1	1000	0
2	100	10
3	10	19
4	1	31
5	0.2	59
6	0.15	73
7	0.11	112
8	0.095	131
9	0.09	140
10	0.085	150
11	0.08	164
12	0.075	182
13	0.07	201
14	0.065	224
15	0.06	251
16	0.055	289
17	0.05	340
18	0.048	361
19	0.045	397
20	0.042	436
21	0.04	472
22	0.038	526
23	0.036	581
24	0.034	654
25	0.032	773
26	0.03	891
27	0.029	1002
28	0.028	1208
29	0.029	1310
30	0.03	1378
31	0.031	1414
32	0.032	1458
33	0.033	1497
34	0.034	1541
35	0.035	1607
36	0.036	1688
37	0.037	1732
38	0.038	1789
39	0.036	1829
40	0.034	1833
41	0.032	1839
42	0.028	1849
43	0.022	1860
44	0.015	1875
45	0.01	1879
46	0.005	1892
47	1.00E-03	1919
48	2.50E-04	1928
49	2.00E-04	1935
50	1.50E-04	1949
51	1.00E-04	1983
52	8.00E-05	2000
53	6.00E-05	2084
54	5.00E-05	2160
55	4.50E-05	2216
56	4.20E-05	2262
57	4.00E-05	2296
58	3.80E-05	2340
59	3.60E-05	2391
60	3.40E-05	2446
61	3.20E-05	2515
62	3.00E-05	2598

Table 5.1 Readings for characteristic of diffusion pump taken at IIT KGP

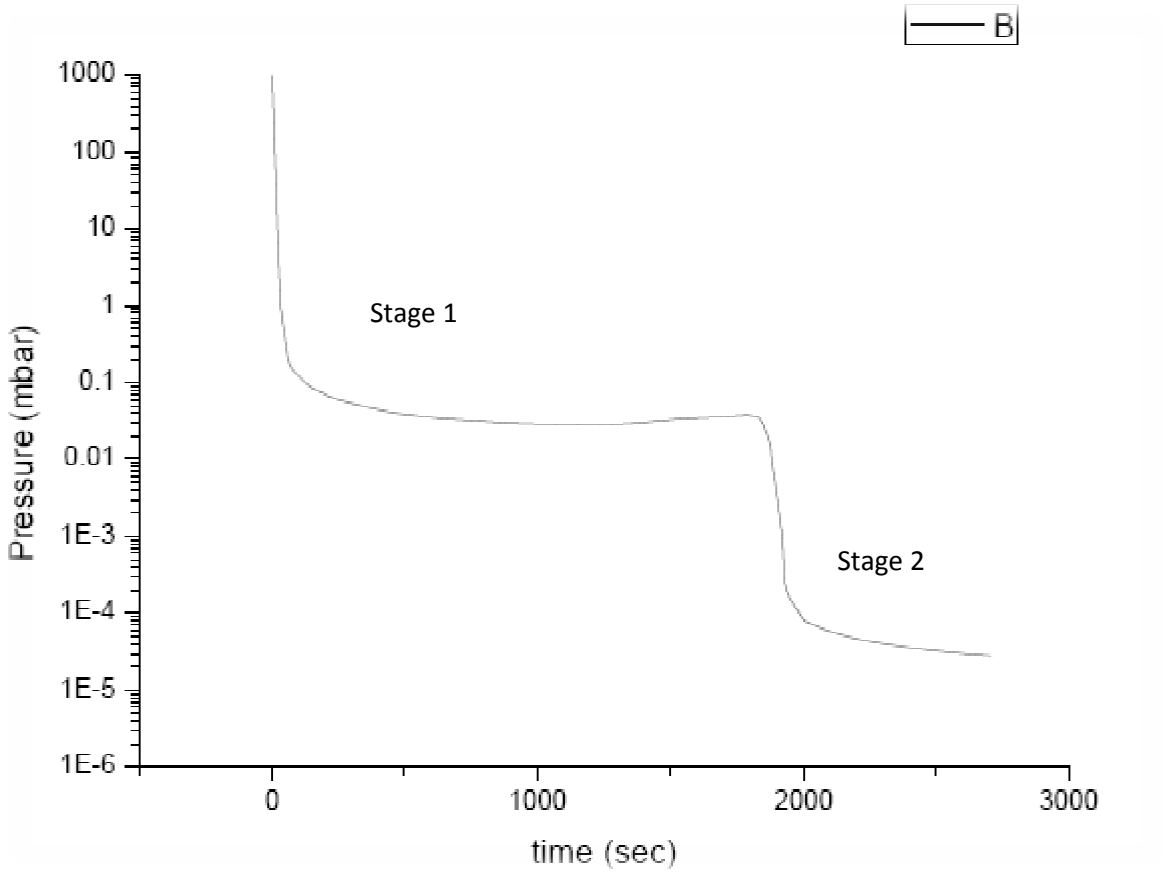


Fig 5.3 Plot between Pressure and Time for High Vacuum System (Diffusion Pump)

5.5. DISCUSSION

The stage 1 of curve is due to evacuating vacuum chamber by rotary pump only through roughing line. The ultimate pressure achieved in this way is nearly 10^{-2} mbar. Curve of stage 2 is due to evacuation of chamber by diffusion pump, along with rotary pump through backing line. The ultimate pressure achieved through the diffusion pump is nearly 10^{-5} mbar.

In the high vacuum set up, we cannot calculate the pumping speed by constant volume method, because at high vacuum the leak due to outgassing is more, so we cannot assume that the volume of air is constant. But in contrast we can measure the pumping speed by constant pressure method. By using liquid nitrogen in cold trap we can easily get the ultimate pressure of range of 10^{-8} mbar in vacuum chamber.

CHAPTER 6

MEASUREMENT OF CONDUCTANCE OF PIPE

6.1. FLOW REGIME

The gas flow can be turbulent, laminar, intermediate and molecular. The limit between the turbulent and laminar floe is defined by the value of *Reynold's number.* While those between laminar, intermediate and molecular flow are described by the value of *Knudsen number*

The Reynold's number is dimensionless quantity expressed by

$$R_e = \frac{\rho v D}{\mu}$$

In terms R_e the ranges can be

R_e > 2100 turbulent flow (viscous flow)

R_e < 1100 laminar flow (viscous flow)

The Knudsen number is the ratio between λ/D between the mean free path λ and the diameter of the pipe D. in terms of the ratio D/λ the ranges can be defined as

D/λ > 110	Viscous flow
1< D/λ < 110	Intermediate flow
D/λ<1	Molecular flow

6.2. CONDUCTANCE

The flow of gas can be interpreted as the no. of molecule N, passing per unit time through a cross section of the pipe.

Considering two subsequent cross section 1 and 2 of the same pipe, the no of molecule crossing them will be

$N_1 = A_1 v_1 n_1 = S_1 n_1$

and

$N_2 = A_2 v_2 n_2 = S_2 n_2$

Where A is the area of cross section, v is the flow velocity, n is the number of molecules per unit volume and S is the pumping speed.

In a permanent flow, the number of molecules crossing the various cross section is the same, thus $N_1=N_2=N$

$$N = S_1 n_1 = S_2 n_2$$

Since n the no. of molecules per unit volume is proportional to pressure. The drop in molecular density is proportional to the no of molecules.

$$N = C\,(n_1 - n_2)$$

The factor C is the conductance of the pipe, is given by

$$1/C = (n_1 - n_2)/N = (1/S_1) - (1/S_2)$$

Where, S_1 and S_2 is pumping speed at 1 and 2 respectively.

The unit of conductance is lit/ sec.

Through put (Q) & pressure drop (ΔP) are related by a term called Conductance "C" of the vacuum element (connecting tube)

$$C = Q/\Delta P$$

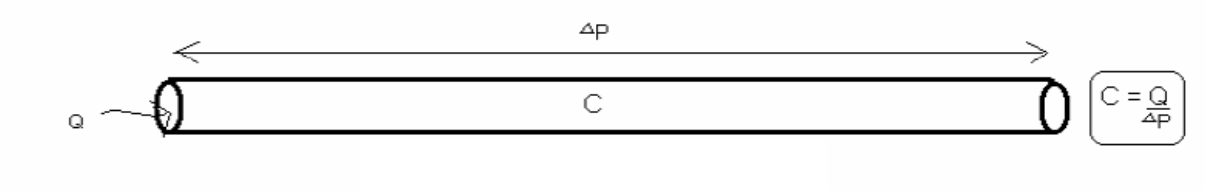


Fig 6.1 conductance of a pipe

6.2.1.1. Derivation of conductance of long tube of constant cross section in molecular flow

In this flow the molecules move in random straight lines between collisions with the wall. The no of molecules impinging on the unit surface per unit time is $\phi = nv_{av}/4$ and the number of molecule striking the wall each second is

$$q = \phi BL = BLnv_{av}/4$$

Where, B is the periphery of the cross section, and L the length of the tube.

The momentum transferred by all the molecules to the wall with drift velocity v is

$q' = qmv = BL\, n\, v_{av} mv/4$

The number N of molecule crossing the cross section A of the pipe per unit time is

$$N = Avn$$

And the pressure difference ΔP achieved corresponding to a force

$$\Delta F = A\Delta P = AkT\Delta n$$

For equilibrium condition $q' = \Delta F$, and

$$\Delta n = \frac{BL\, n\, v_{av}\, m\, v}{4\, A\, k\, T}$$

$$C = \frac{N}{\Delta n} = \left[\frac{4A^2}{BL}\right]\left[\frac{kT}{mv_{av}}\right]$$

And $V_{av} = \left(\frac{2}{\sqrt{\pi}}\right)(2kT/m)^{1/2}$

Knudsen has shown that it should be better to assume that the superimposed drift velocity of a molecule is proportional to its random velocity. On this modified assumption Knudsen found that numerical factor in above equation must be multiplied by 8/3π, so the conductance will be

$$C = \frac{8}{3\sqrt{\pi}}\left(\frac{2kT}{m}\right)^{\frac{1}{2}}\left(\frac{A^2}{BL}\right)$$

The conductance of a tube of uniform circular cross section with $A = \pi D^2/4$ and B = πD, is

$$C = 3.81\ (T/M)^{1/2}\ (D^3/L)$$

Where, all are in CGS units, for air at 20°C, $(T/M)^{1/2} = 3.81$, thus

$$C_{air} = 12.1\ D^3/L$$

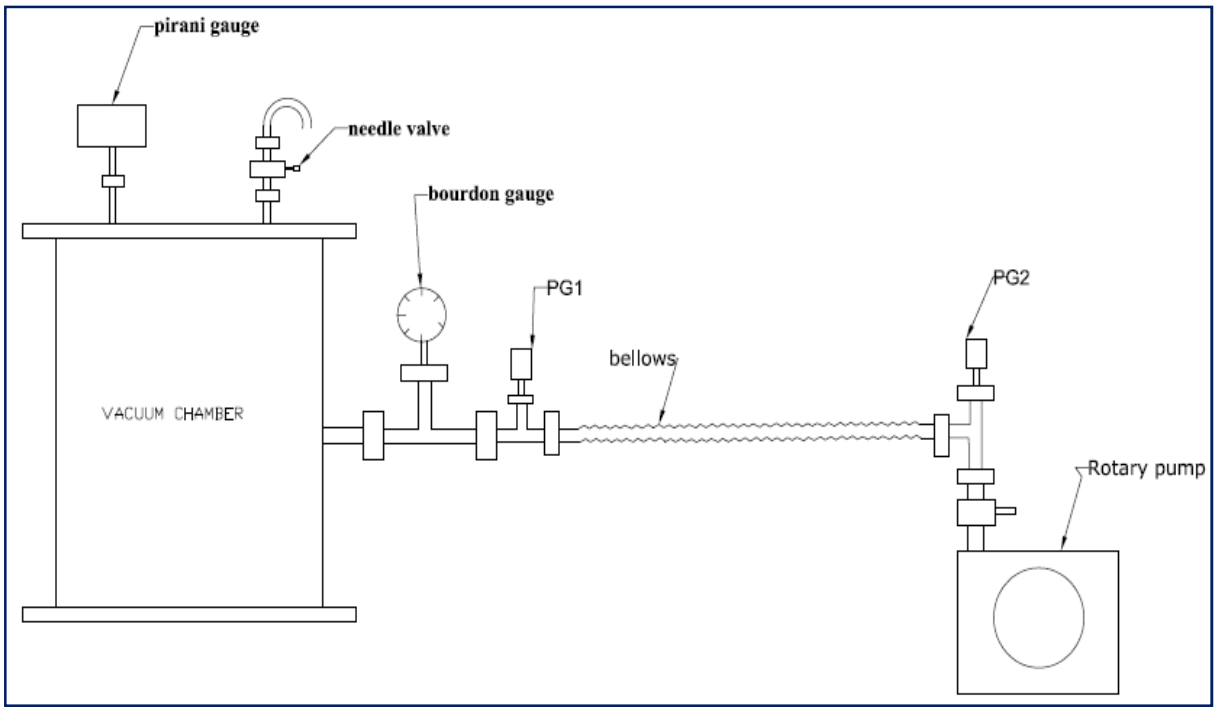


Fig. 6.2 schematic diagram for measurement of conductance of a pipe

Fig 6.3 Set up made in NIT RKL for measurement of conductance

6.2.1.2. PROCEDURE

- Install all the setup as shown in above diagram
- Check that all the valves are closed and the vacuum chamber is at atmospheric pressure
- Start the mechanical pump, and after it warmed up, open the valve to vacuum vessel
- Record the time to achieve the subsequent pressure (i.e. 1000-50,50-1,.. mbar).
- Repeat the measurement 2-3 times, and take the readings.
- Record the data and calculate the pumping speed and throughput.
- Maintain a pressure in vacuum chamber by adjusting the needle valve, and the measure the pressure difference across the bellows.
- Take the reading for different pressure.
- Close the valves and then turn-off the rotary pump
- Record the data and calculate the conductance of bellows
- Plot the graph between conductance vs average pressure.

SAMPLE READINGS

Sl. No.	Initial pressure	Final pr.	Average pressure	Time taken to reach p2 from p1		Average time	Pumping speed	Throughput
	p_1	p_2	$p_{avg} = \frac{p_1 + p_2}{2}$	t_1	t_2	$t = \frac{t_1 + t_2}{2}$	S_p	$Q = P_{avg} * S_p$
	mbar	mbar	Mbar	Sec	Sec	Sec	Liter/sec	mbar lit/sec
1	980	392	686	6	6	6	3.05087	2092.89915
2	392	196	294	6	6	6	2.3079	678.52161
3	196	98	147	7	7	7	1.9782	290.79498
4	98	60	79	5	5	5	1.96029	154.86278
5	60	40	50	4	4	4	2.02505	101.25247
6	40	5	22.5	17	17	17	2.44366	54.98224
7	5	1	3	16	17	16.5	1.94864	5.84593
8	1	0.5	0.75	10	9	9.5	1.45762	1.09321
9	0.5	0.1	0.3	41	44	42.5	0.75653	0.22696
10	0.1	0.08	0.09	17	17	17	0.26223	0.0236
11	0.08	0.06	0.07	21	24	22.5	0.25543	0.01788
12	0.06	0.05	0.055	22	24	23	0.15836	0.00871
13	0.05	0.045	0.0475	19	23	21	0.10023	0.00476
14	0.045	0.04	0.0425	21	25	23	0.10231	0.00435
15	0.04	0.035	0.0375	27	34	30.5	0.08746	0.00328
16	0.035	0.03	0.0325	83	97	90	0.03422	0.00111
17	0.03	0.025	0.0275	177	186	181.5	0.02007	5.51869E-4

Table 6.1 Readings for calculating pumping speed and throughput by constant volume method

Sl. No.	Through put	Pressure at gauge 1	Pressure at gauge 2	Difference in pressure	conductance
	Q	P1	P2	ΔP	C = Q/ΔP
	Lit mbar/sec	mbar	mbar	mbar	Lit/sec
1	1.09321	0.74	0.72	0.02	1.51835
2	0.22696	0.24	0.23	0.01	0.98678
3	0.0236	0.089	0.074	0.015	0.31892
4	0.01788	0.068	0.055	0.013	0.32509
5	0.00871	0.055	0.043	0.012	0.20256
6	0.00476	0.048	0.034	0.014	0.14003
7	0.00435	0.043	0.03	0.013	0.14493
8	0.00328	0.038	0.026	0.012	0.12615
9	0.00111	0.032	0.021	0.011	0.05296
10	5.51869E-4	0.027	0.016	0.011	0.03449

Table 6.2 Readings for calculating conductance from the throughput noted in sl. No. 8-17of above table

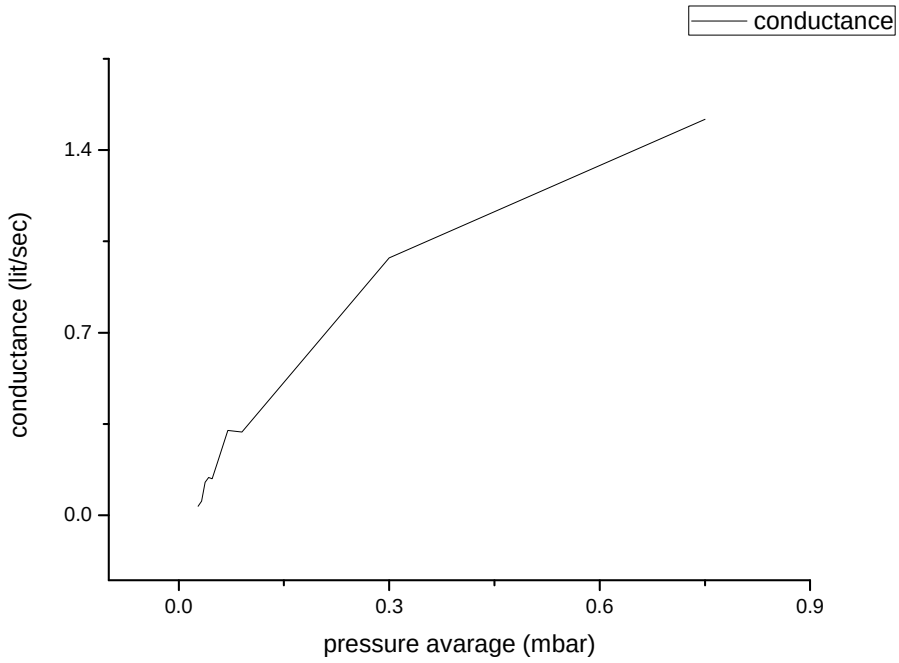


Fig. 6.4 Plot between conductance and average pressure

6.2.1.3. DISCUSSION

The conductance should be equal to theoretical value 1.9 lit/sec ($12.1D^3/L$) and remain constant through the wide range of vacuum pressure, but in the plot conductance vs pressure, it is decreasing, due to error in taking reading and calibration of gauges.

6.2.2. Conductance of pipes connected in parallel

When two pipes are connected in parallel, the number of molecule N reaching the cross section a is divided in two parts, N_1, flowing in pipe 1, and N_2 flowing in pipe 2, if the molecular density at a and b are n_a and n_b, then

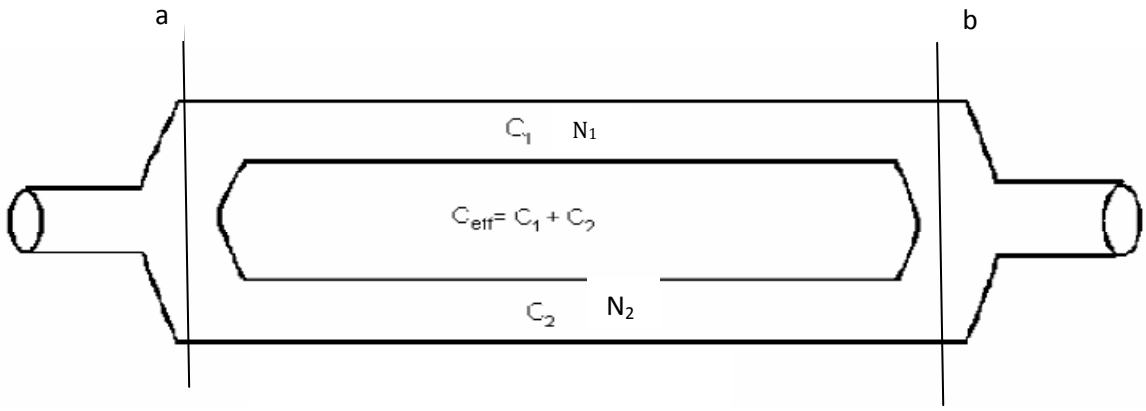


Fig. 6.5 conductance of pipe in a parallel

$$N_1 = C_1(n_a - n_b)$$

$$N_2 = C_2(n_a - n_b)$$

And since

$$N_1 + N_2 = N = C_{eff}(n_a - n_b)$$

This implies

$$C_{eff} = C_1 + C_2 + \cdots$$

Where, C_{eff} is the conductance of the system of parallel pipes.

6.2.3. Conductance of pipes connected in series

When conductance are connected in series and the molecular densities at a, b. c are n_a, n_b, and n_c, one can write

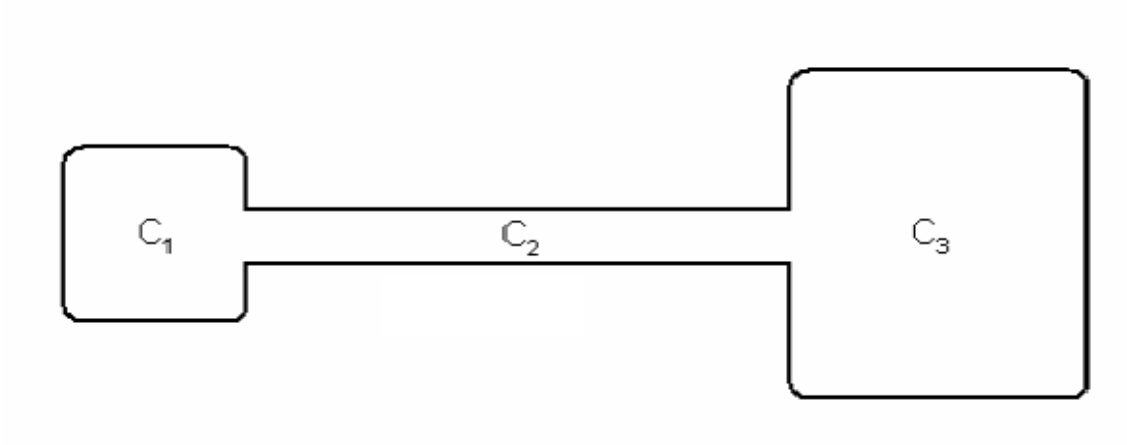


Fig. 6.6 Conductance of pipe in series

$$N = C_1(n_a - n_b) = C_2(n_b - n_c) = C_3(n_c - n_d)$$

Where, C1, C2 are the individual conductance. C_{eff} is the conductance of the system.

$$\frac{1}{C_{eff}} = \left(\frac{1}{C_1}\right) + \left(\frac{1}{C_2}\right) + \cdots \ldots \ldots \ldots \ldots$$

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