Experimental set up for vacuum laboratory

A Thesis Submitted to

National Institute of Technology, Rourkela

In Partial fulfilment of the requirement for the degree of

Master of Technology

In Mechanical Engineering

with Specialization in Cryogenics and Vacuum Technology

By

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2015

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CERTIFICATE

This is to certify that the thesis entitled, **"Experimental set up for vacuum laboratory"** submitted by **CHANDRAKANT S. SUKHDEVE** (**Roll No: 213ME5459)** in partial fulfillment of the award of **Master of Technology** degree in **Mechanical Engineering** with specialization in **Cryogenics and Vacuum Technology** during the period **2014-15** at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

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

Date:

Place: Rourkela

Prof. Sunil Kumar Sarangi,

Director, National Institute of Technology, Rourkela

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ABSTRACT

This project deals with study, procurement and installation of some vacuum apparatus for performing experiments in vacuum laboratory. This lab was proposed to be used for graduate and post graduate students. Four experimental set ups were designed as a part of this project. It includes pumping speed measurement of diffusion pump, pumping speed measurement of turbomolecular pump, measurement of conductance of different vacuum elements, and calibration of vacuum gauges. As part of project work, vacuum systems and components were studied, bills of materials were prepared, required components were purchased and installation of experimental set up was done. Pumping speed measurements of high vacuum pumps (diffusion pump and turbomolecular pump) was done by constant volume method. For getting more accuracy Vacuum chamber was tested for leak rate using a MSLD (Mass spectrometer leak detector). Conductance of vacuum elements was measured for different piping arrangements by measuring corresponding changes in effective pumping speed of pumps. Conductance was calculated in low vacuum range using rotary vane pump. Calibration of vacuum gauges was done in low vacuum range by using the method of direct comparison with standard gauge.

Keywords: High vacuum pumps, Conductance, Calibration of vacuum gauges, MSLD

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CHAPTER 1

INTRODUCTION

Vacuum is the condition of a gas in which its pressure in a chamber and hence its molecular density is less than that of the ambient surrounding atmosphere or in which the pressure of the gas is less than the atmospheric pressure. Vacuum is measured in pressure units. Commonly used units in vacuum are Torr and mbar. Depending upon the vacuum range following are the types of vacuum.

Table 1.1 Pressure ranges in vacuum technology

Pressure range	Pressure (mbar)	Molecular density ($/cm^3$)	Mean free path (m)
Atmospheric pressure	1013	2.7×10^{19}	6.8×10^{-8}
Low vacuum (LV)	1013 – 1	$10^{19} - 10^{16}$	$10^{-8} - 10^{-4}$
Medium vacuum (MV)	$1 – 10^{-3}$	$10^{16} - 10^{13}$	$10^{-4} - 10^{-1}$
High vacuum(HV)	$10^{-3} - 10^{-7}$	$10^{13} - 10^{9}$	$10^{-1} - 10^{3}$
Ultra high vacuum (UHV)	$10^{-7} – 10^{-12}$	$10^{9} - 10^{4}$	$10^{3} - 10^{8}$
Extremely high vacuum (XHV)	$< 10^{-12}$	$< 10^{4}$	$>10^{8}$

1.1 Types of Vacuum pumps:

Vacuum pumps are divided into two main categories.

1) Gas transfer vacuum pumps: These pumps in compression stages remove the gas molecules from the system by displacement transfer and eject it into the atmosphere. These pumps are of two types.

   (a) Positive displacement pumps: These pumps suck the gas particles, trap them in particular volume inside the pump chamber and then transfer it by mechanical action. Generally these pumps are rough vacuum pumps.

   e.g. Rotary vacuum pumps, roots pumps etc.

(b) Kinetic vacuum pumps: They work on the principle of drag, fluid entrapment and molecular transfer. Generally these pumps are high and ultra-high vacuum pumps.

i. Drag pumps: These pumps transfer the gas molecules from vacuum system to fore vacuum pumps by momentum transfer.

e.g. Turbomolecular pumps, turbine vacuum pumps etc.

ii. Fluid entrapment vacuum pump: In these pumps, pumping fluid vapours forces the air (or gas to be pumped) molecules from vacuum system to fore vacuum pumps.

e.g. Diffusion pumps, ejector vacuum pumps etc.

2) Entrapment vacuum pumps: These pumps remove the gasses by condensing on a solid surface, which is part of boundary of volume itself. These pumps are generally ultra-high vacuum pumps.

e.g. Adsorption pumps, getter ion pumps, cryopump, sublimation pumps etc.

1.2 Types of flow in vacuum ranges

The ratio of the mean free path to the flow channel diameter is used to describe types of flow. This ratio is called as the Knudsen number:

$$K_n = l/d \tag{1.1}$$

Where l is mean free path (m) and d is diameter of flow channel (m). Following are the types of flow according to relative changes in mean free path and dimension of the flow channel.

1) **Viscous flow** ($K_n < 0.01$): In this flow there are frequent collisions between gas molecules, but less frequently with the walls of the vessel. The mean free path of the gas molecules is significantly shorter than the dimensions of the flow channel. This flow prevails in low vacuum range.

2) **Knudsen flow** ($0.01 < K_n < 0.5$): This flow occurs in medium vacuum range. In this case the characteristic dimension of the flow channel is of the same or smaller order as that of mean free path.
3) **Molecular flow** ($K_n > 0.5$): The mean free path is significantly greater than the diameter of the flow channel. This flow occurs in high and ultra-high vacuum range. Molecular interactions will virtually no longer occur.

1.3 Applications of vacuum technology

- Electrical and electronics: semiconductor production, gas filled tubes, X ray tubes, interrupters, cathode ray tubes, waxed paper capacitors, electron tubes etc.
- Vacuum impregnation: cables, coils and windings, packaging materials casting resins, oil insulated measuring transformers and motors, asbestos objects, crayon leads etc.
- Research and development: space simulation chambers, fusion experiments, particle accelerators, cryogenic experimentations, thermal insulations, nuclear research, preparation of samples for electron microscopy etc.
- Vacuum coating: thin film technology, microcircuits, metal film resistors, photo sensitive layers, protective metal coating, hard and wear resistant layers on tools etc.
- Vacuum freeze drying: pharmaceutical products, preserving the nutritional values and tastes, proteins, yeast etc.
- Vacuum distillation: high boiling point chemicals, plasticizers, organic chemicals, fruit juices and luxury foods, preserving vitamin contents and flavours, mineral oils etc.
- Mechanical operations: railway braking systems, industrial filtering, vacuum sniffers, holding/ lifting and transporting the materials etc.
- Refrigeration and cryogenic engineering: refrigeration oils, insulation of vessels and tanks for liquid petroleum gases, cryogenic fluids etc.

- Automobile: filling of air conditioning, cooling and servo system, brake fluid systems, electrically conductive front mirror coatings for fast defrosting head lamps and rear light reflectors etc.
- Metallurgical: metal powders, production of pure metals, vacuum heat treatment of metals, vacuum alloying, vacuum casting, vacuum cleaning, degassing etc.
- Manufacturing and other Processes: electron beam machining, Brazing, sintering, etc.

CHAPTER 2
LITERATURE REVIEW

2.1 Pumping Speed measurement of high vacuum pumps

R. Y. Jou et al [1] predicted the performance of spiral-grooved turbo booster pump by computational fluid the direct simulation Monte Carlo (DSMC) method and computational fluid dynamics (CFD) method. Calculations were found accurate by CFD analysis in slip flow and continuum flow but not in the transitional flow. They found that when the Knudsen number is in the range $0.5 < Kn < 0.1$, CFD computation and DSMC simulation were not suitable for analysing the pumping speed of the pump and hence in this case, the experimental analysis was supposed to be the most suitable for analysing pumping speed.

W. Jitschin et al [2] did comparative study of measurement of pumping speed of rough vacuum pumps by constant pressure method versus constant volume method and found that both methods yields correct results if important precautions are taken during experiment. Pumping process was interrupted for achieving thermal equilibrium so that pumping speed should not be affected by change of pressure. In later method the pumping speed was calculated by evacuating the large vessel and continuously monitoring pump down curve.

F. J. Eckle et al [3] measured the pumping speed of the diaphragm pumps by intermittent pump down method and analysed the pumping speed curves for Helium, Nitrogen and Argon and ultimate pressures diaphragm pumps on the basis of flow regimes and thermal effects.

S.S. Hong et al [4] investigated the pressure distribution in the chamber of a newly developed flow control system for different gases Ar, N2, and He and found that relative distribution in pressure distribution in gas inlet and outlet were in the range of -1.3% and 1.2% respectively.

S.W. Zhang et al [5] designed automatic testing system for measuring performance of diffusion pump for use in laboratory. They proved practicability of this newly developed automated testing system.

V. D. Chaudhari and **A. D. Desai** [6] presented the theoretical procedure for calculating pump down time for vacuum pump. These theoretical calculations were compared with actual observation. Pumping curve was plotted for theoretical and actual observations.

Y. C. Liu et al [9] studied the effect of outgassing on pumping speed characteristics of vacuum pumps in ultra-high vacuum range. They attained the ultimate pressure of 4 $x10^{-12}$ mbar in a stainless steel vacuum chamber using Titanium sublimation pump, ion pump, and getter pump. They calculated the pump down time theoretically and experimentally. The effect of outgassing in high and ultra-high vacuum range was found to be predominant and found that the pumping speed was decreased with decrease in pressure. Mass analysis of gasses after baking the chamber showed that CH_4 was formed due to combination of hydrogen and carbon. After bake out, decrease in outgassing rate was found with increase in pumping speed. It was evaluated from the experimental and theoretical data.

Karl Jousten [10] described the effects of thermal outgassing in vacuum chambers and pumping speed behaviour. Also it was reviewed that there was a linear relationship between pressure (on logarithmic scale) versus time in initial stage and then asymptotic behaviour in later stage.

Phil Danielson [11] showed the effect of conductance of vacuum elements on pumping speed of pump and suggested to calculate the effective pumping speed by considering conductance of vacuum elements.

L. Peksa and **T. Gronych** [12] measured the effective pumping speed for hydrogen permeation using the method of throughput method by considering effects of permeation and

outgassing. Pressure attained during experiment was in the range of 10^{-9} Pa with the help of two diffusion pumps connected in series.They preferred diffusion pumps rather than using turbomolecular pumps because of requirement of long term continuous pumping for hydrogen and better compression ratio of diffusion pumps for hydrogen.

R J Elsey [13] examined the methods for determination of outgassing rates of some selected materials by method of known and constant pumping speed. He also suggested that known and constant pumping speed character of pump can be used for calculation of conductance of vacuum elements.

2.2 Calibration of vacuum gauges

P. J. Nash and T. J. Thompson [8] described a calibration system for vacuum gauges in the range of 10^{-4} to 10^{2} Pa with uncertainty of $\pm$10%. Capacitance manometer and triode ionization gauges with uncertainty $\pm$ 2% were used as reference gauges. A LN_2 trapped diffusion pump was used to create high vacuum. For calibration of gauges the pressure inside the vacuum vessel was balanced through a leak valve. Vacuum vessel of volume 100 litre was used so as to reduce relative amount of outgassing from gauges and to avoid changes in behaviour of apparatus with change in number of calibration items.

Anita Calcatelli [14] suggested the method of calibration for vacuum gauges in different vacuum regimes. For pressure less than 1000 Pa to 10^{-4} Pa calibration was based on static expansion or Knudsen method by single or multiple expansions. By method of multiple expansion gauges can be calibrated up to 10^{-6} Pa. For the range 10^{-1} Pa to 10^{-7} Pa, method suggested was continuous or dynamic expansion method. Constant pressure method was also suggested for this range.

William D. Davis [15] studied the problems associated with calibration of vacuum gauges at pressures below 10^{-10} mbar. A Liquid Helium pump was used to attain the pressure of 10^{-13}

mbar. McLeod gauge was used as reference standard for calibration. Calibration curves were also plotted for a mass spectrometer.

Hajime Yoshida et al [16] developed a leak element called as 'standard conductance element' (SCE) for calibrating the quadrupole mass spectrometers and ionization gauges. It was made of a stainless-steel filter with the pore size less than 1 micron. Gas flow through SCE satisfies molecular flow conditions due to very small size of pore. SCE was first calibrated by calculating measuring its conductance by evacuating a test chamber with the help of turbomolecular pump through an orifice. These calibrated SCEs were used to calibrate the gauges. First, a SCE was mounted on vacuum chamber. Chamber was evacuated and test gas was introduced into chamber with known throughput which helps to calculate pressure using formula for the SCE. Calibration was performed by comparing signal output from ionization gauge and mass spectrometer for known values of pressure.

2.3 Calculation of conductance of vacuum elements

B. Mercier [17] calculated the conductance of a conical tube in molecular flow range by Monte Carlo Simulation and presented a method for calculating conductance experimentally. Dynamic method was used to calculate the conductance experimentally. It includes introducing a known flow gas through a pipe of which conductance is to be calculated and measuring pressure difference on both sides of pipe. Surface distribution of pressure was also calculated theoretically.

James A. Fedchak and Dana R. Defibaugh [18] presented the results of measurement of conductance for an orifice having nominal diameter of 20 micron. A differential capacitance diaphragm gauge was used to monitor pressure differential and conductance was calculated by using the known flow rate of gas. They showed the difference between the conductance of Ar and N_2 gas.

Keiko Terada, Tatsuo Okano, and Yutaka Tuzi [19] described the procedure and formulation of the newly developed conductance modulation (CM) method. It was developed for measuring the pumping speeds of vacuum pumps and outgassing rates in vacuum systems. The principle of the method is based on measurement of the pressure modulation with the change in conductance between the vacuum vessel and the pump. The pumping speed was obtained from the value of the conductance and the ratio of modulated pressures without measuring the absolute pressures. A variable orifice system was inserted between test vacuum chamber and vacuum pumps (Tungsten Getter Pump and an auxiliary sputter ion pump). The ultimate pressure of $6x10^{-9}$ Pa was attained after a bake out of chamber. The orifice system was made of three components having different values of conductance.The transmission probabilities through different components of orifice system were calculated using Monte Carlo method. Pressure changes were measured using quadrupole mass spectrometer and Bayard-Alpert gauge. Pumping speed was calculated based on relative change in the chamber pressure corresponding to the change of conductance between pump and the chamber.

CHAPTER 3
EXPERIMENTAL SET UP

Proposed experimental set up consists of four experiments

1. Pumping speed measurement of diffusion pump
2. Pumping speed measurement of turbomolecular pump
3. Calibration of vacuum gauges
4. Calculation of conductance of vacuum elements

3.1 Pumping speed measurement

Pumping speed of vacuum pump is the volume of gas removed per unit time by the pump at inlet pressure. One of the important factors in the design of vacuum system is determination of pump down time (the time required to reduce the pressure of system from ambient pressure to desired operating pressure). The manufacturer's listed pumping speed for any given 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 (mass flow rate). The pumping speed (volumetric flow rate) will decrease only slightly until a certain pressure is reached. Below this pressure, the decrease in pumping speed becomes more rapid, depending upon the type of mechanical vacuum pump, and falls to zero at ultimate pressure.

Consider a general vacuum system as shown in Fig.3.1. The mass flow rate of gas from the system is given by

$$m_{out} = \rho S_s = PS_s / RT \qquad (3.1)$$

Where P = pressure within the space to be evacuated,

T = absolute temperature (assumed constant) of the gas in the system

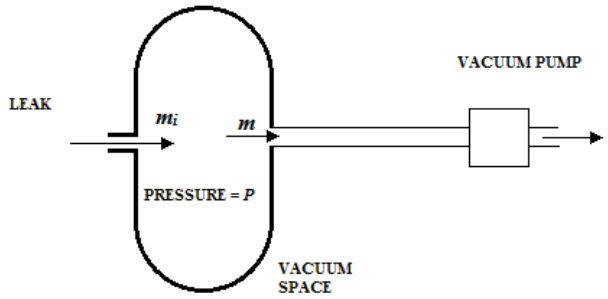


Fig 3.1 A general vacuum system

Let m_i be the mass flow rate into the system from leaks. This mass influx may be due to

(a) Actual leaks through the vessel,

(b) Virtual leaks due to gas trapped and released from pockets within the system and

(c) Outgassing of the metal wall or seals. Any material releases absorbed gasses from its surface, its interior, or both when exposed to vacuum, and this process of releasing gasses is called outgassing. For a clean, well designed and well checked vacuum system, outgassing is the major contribution to the mass inflow.

Applying the conservation of mass principle to the vacuum system and assuming that the gas obeys ideal gas equation of state,

$$m_i - m_{out} = \frac{dm}{dt} = V\frac{d\rho}{dt} = \frac{V}{RT}\frac{dP}{dt} \qquad (3.2)$$

Where V is volume of the system and t is time. If we introduce the in leak rate, defined by $Qi = m_i RT$, and the system pumping speed, $S_s = Q/P = m_{out} RT/P$, equation (3.2) becomes

$$\frac{dP}{dt} = \frac{Qi}{V} - \frac{Ss*P}{V} \qquad (3.3)$$

This is the governing equation for evaluating the pump down time for any vacuum system in general. The equation may be solved analytically or numerically if the dependence of the in

leak rate on time and/or system pressure and the dependence of the system pumping speed on system pressure are known.

After a long pumping time, the pressure of the system changes only slightly ($dP/dt = 0$) and the system pressure approaches the ultimate pressure of the system P_u. Setting LHS of the equation (3.3) equal to zero we get the relation between in leak rate and system pumping speed.

$$P_u = Q_i/S_s \tag{3.4}$$

In the operating range of most vacuum pumps, the pumping speed is constant as the pressure is varied. For constant pumping speed integrating equation (3.3) between the initial pressure P_1 at $t = 0$ to final pressure P_2at $t = t_p$ (the pump down time)

$$t_p = \left(\frac{V}{Ss}\right) ln\left(\frac{P1-Pu}{P2-Pu}\right) \tag{3.5}$$

The leak rate is often difficult to predict in design stage, so that pump selection is made in these cases by using modification of equation (3.5). A preliminary estimate of required system pumping speed can be obtained from the following expression, obtained from equation (3.5) by setting the ultimate pressure equal to zero and introducing the system allowance factor, F_s.

$$S_s = (F_sV/ t_p) ln (P_1/P_2) \tag{3.6}$$

The system allowance factor allows for outgassing within the vacuum vessel. At pressure below about $2x10^{-4}$ mbar, the outgassing rate usually controls the pump-down time, so that equation (3.6) cannot be used for this pressure region. Values of system allowance factor are given in the Table 3.1

Table 3.1 System allowance factor values

SN	Final System Pressure (mbar)	System Allowance Factor , F_s
1	1000 to 100	1.0
2	100 to 10	1.25
3	10 to 0.5	1.50
4	0.5 to 0.05	2.0
5	0.05 to 0.0002	4.0

In the high vacuum regions where outgassing becomes predominant, the pump-down relationship may be developed from the general relationship, equation (3.3), by introducing the following variation of the in leak rate and system pumping speed

$$Q_i = Q_o \, exp \, (-t/t_1) \tag{3.7}$$

$$S_s = S_o \, (1-P_u/P) \tag{3.8}$$

Where Q_o is the initial outgassing rate (at $t = 0$), t_1 is a constant characteristic of the system, S_o is the system pumping speed at pressure well above the ultimate pressure P_u of the system.

3.1.1 Calculation of pumping speed of diffusion pump

Pumping speed of vacuum pumps can be determined by either constant volume method or constant pressure method.

3.1.2 Constant volume method

In this method pressure is recorded as a function of time while the system is being evacuated and pumping speed is calculated using equation (3.9). This method is also called as pump-down method or transient state method. This method is quite easier than constant pressure method but size of vacuum chamber required in constant volume method is significantly larger than constant pressure method.

3.1.3 Experimental procedure

a) First ensure that all the connections are tight.

b) Start the rotary pump and wait till the system reaches its ultimate pressure.

c) When system reaches ultimate pressure of rotary pump (10^{-2} mbar or 10^{-3} mbar), start the diffusion pump by opening the main isolation valve (butterfly valve) and closing the roughing valve.

d) Record the values of pressure as a function of time.

e) Calculate the pumping speed by equation (3.9).

f) Plot the calculated pumping speed versus average pressure for each pressure range as given in Table 3.3.

$S_s = (V/\Delta t) \ln (P_1/P_2)$ (3.9)

Where

V= chamber volume, and

Δt =time taken to change pressure from P_1 to P_2.

Table 3.2 Observation table for constant volume method

From 1000 mbar to 100 mbar	Time (seconds)
Reading 1	
Reading 2	
Reading 3	
Reading 4	
Reading 5	
From 100 mbar to 10 mbar	

.	
.	
From 10-5 mbar to 10-6 mbar	
Reading 1	
Reading 2	
Reading 3	
Reading 4	
Reading 5	

Table 3.3 Pumping speed for different pressure range

Pressure Range (mbar)	Average Pressure (mbar)	Pumping Speed (lit/sec)
1000 to 100		
100 to 10		
.		
.		
.		
10^{-5} mbar to 10^{-6} mbar		

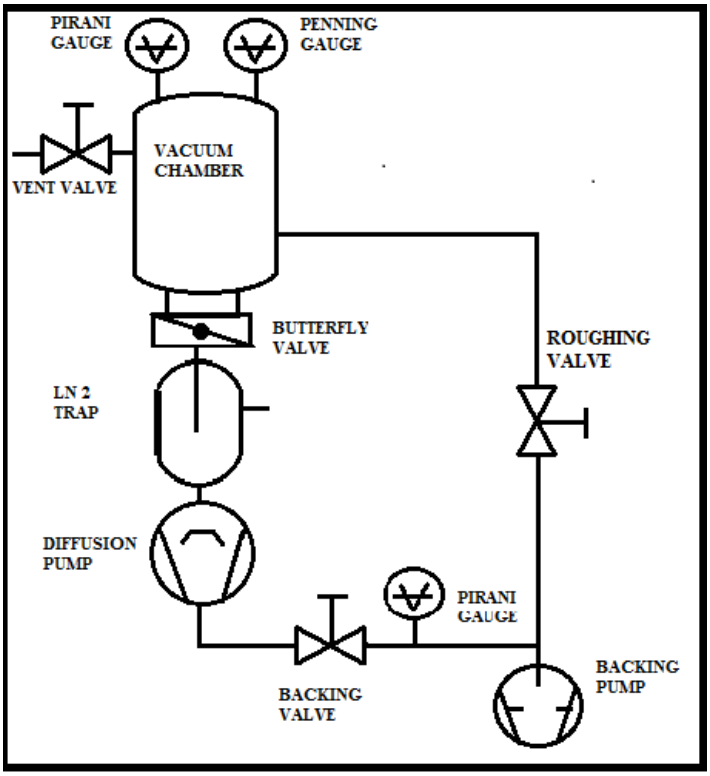


Fig. (3.2) Diagram for pumping speed measurement of diffusion pump

3.1.4 Constant pressure method

In this the pumping speed is calculated using equation (3.10). This method is more accurate and precise than constant volume method but a little difficult comparatively. As this method involves measuring pumping speed by making pressure constant, it is also called as steady state method.

3.1.5 Experimental procedure

a) First evacuate the vacuum chamber ultimate pressure of diffusion pump is achieved.

b) Allow the air to enter the chamber gradually through flowmeter via needle valve so that the vacuum chamber is still being evacuated.

c) Adjust the needle valve gradually so that we get constant reading of pressure inside the vacuum chamber.

d) Measure the flow rate for time interval □□*t* using flowmeter.

e) Calculate the pumping speed of pump using equation (3.10).

f) Repeat steps (c) to (e) for different values of pressure.

g) Plot the graph of Pumping speed versus average pressure.

3.1.6 Diffusion pump

These pumps consist basically of a pump body with a cooled wall and a three or four stage nozzle system. The oil in the boiler is vaporized by heating. The oil vapour flows through the riser tubes and emerges out with high speed from the nozzles so that the jet so formed widens like an umbrella and reaches the wall and gets condensed due to cooling of walls by circulating water. The condensed oil then flows downward along the wall and returns into the boiler and again gets heated and evaporated. Gas molecules present in the region above jet assembly diffuse into the vapour stream and are given downward momentum due to collision with heavier molecules. Hence these molecules are forced by the jet into the region of higher pressure in the lower part of the diffusion pump and then get removed by backing rotary pump. Cold traps are provided between the diffusion pump and the system to be evacuated which helps to achieve the pressure below the vapour pressure of pump fluid. Cold traps acts as barrier to the flow of oil vapours from diffusion pump to the system and also acts gives cryopumping effect for the coming from the system.

Due to spreading of the jet, the vapour density is relatively low. The diffusion of air or pumped gases into the jet is so rapid that despite its high velocity the jet becomes virtually completely saturated with the pumped medium. Therefore, over a wide pressure range diffusion pumps have a high pumping speed.

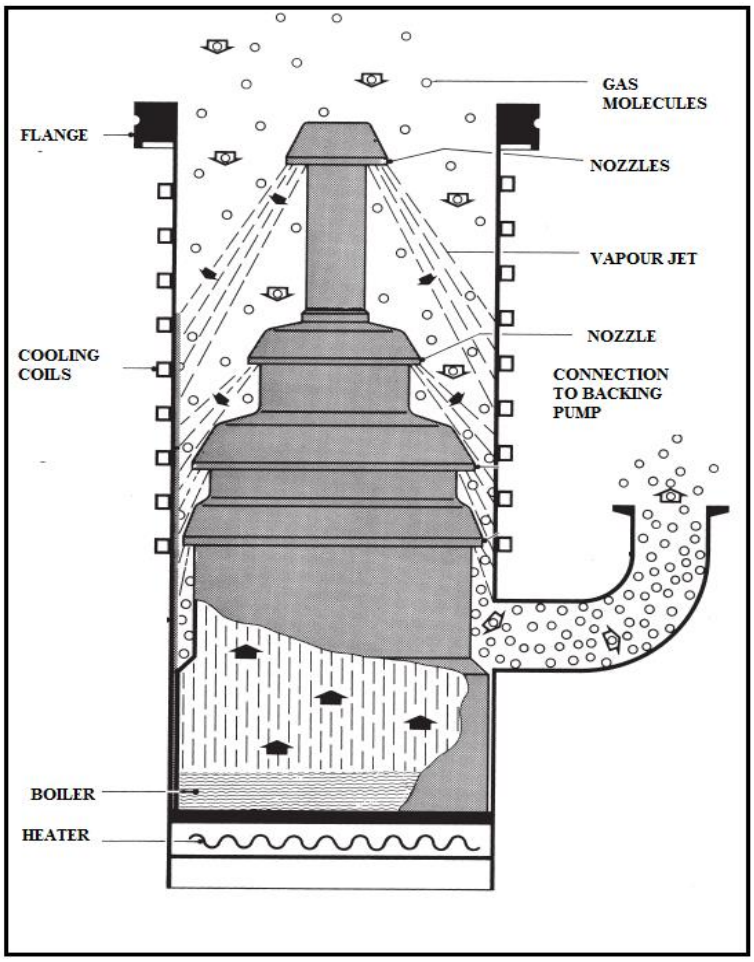


Fig. (3.3) Diffusion pump

3.2 Calculation of pumping speed of turbomolecular pump

3.2.1 Turbomolecular pump

It is a high vacuum pump having its design similar to that of turbine. Rotor rotates with very high speed of 60000 rpm and above. The working principleis based on the transfer of impulses from the rotating blades to the molecules of gas. It consists of stator blades and rotor blades. Molecules of gas colliding on the blades are adsorbed on the blades and leave the blades after certain time due to continuous rotation of rotor blades.

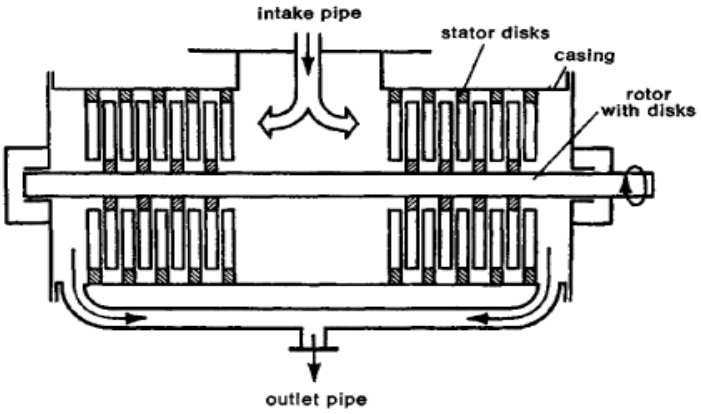


Fig. (3.4) Internal parts of turbomolecular pump

3.2.2 Applications

These pumps can be used to achieve the high and ultra-high vacuum upto the range of 10^{-9} mbar. Turbomolecular pumps are used in processes like IC manufacturing, thin film deposition and leak detectors, pumping the gasses like Argon, preparation of samples for microscopes etc. In modern days turbomolecular pumps are used instead of oil diffusion pumps in many applications due to its advantages over the oil diffusion pumps.

3.2.3 Experimental set up

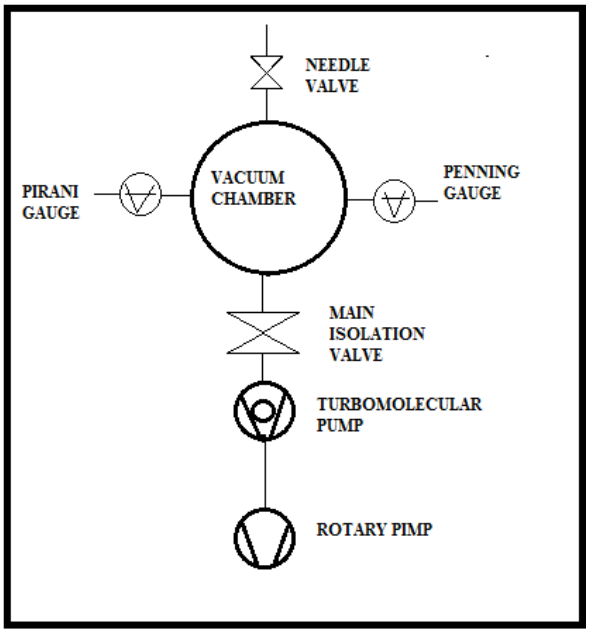


Fig. (3.5) Diagram for pumping speed measurement of turbomolecular pump

Procedure for pumping speed measurement of turbomolecular pump by constant pressure method and constant volume method is same as that of diffusion pump.

3.2.4 Experiment Procedure:

a) First open the main isolating valve of turbo-molecular pump.
b) Start the rotary backing pump and stopwatch simultaneously.
c) Note down the chamber pressure at every particular interval of time (say 10, 20, or 30 sec).
d) Continue step (c) till the pressure inside the chamber reaches the order of 10^{-3} mbar.
e) Start the turbo-molecular pump.
f) Continue step (c) till the turbomolecular pump reaches its ultimate pressure.
g) Stop the system after attaining ultimate its pressure. First turn off the turbomolecular pump but let the rotary backing pump rotate for some time (15 minutes to half an hour approximately).
h) Close the main isolation valve and turn off the rotary pump.
g) Tabulate the readings in Table 3.2.
h) Calculate the pumping speed by equation (3.9).
i) Plot the calculated pumping speed versus average pressure for each pressure range as given in Table 3.3.

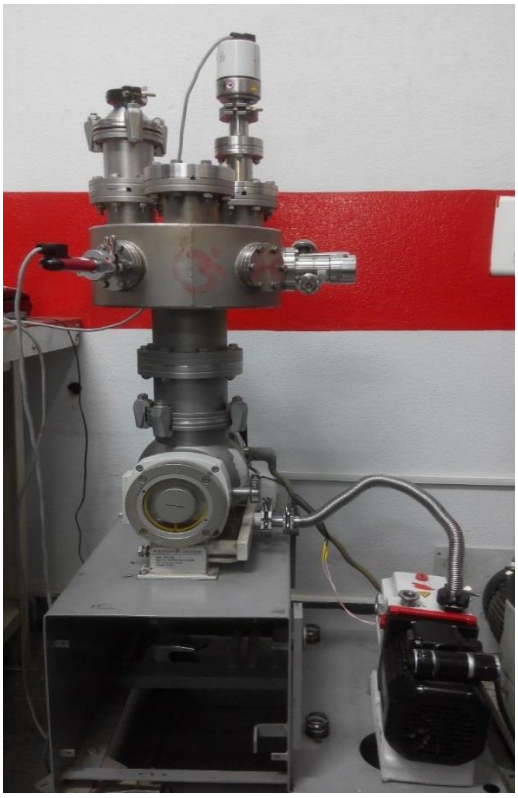


Fig. (3.6) Actual set up for pumping speed measurement of turbomolecular pump

3.3 Calibration of vacuum gauges

Vacuum gauges are the measuring instruments that are used for the measurement of pressures lower than the atmospheric pressure. The pressures measured in vacuum applications are in the range of 1013 mbar to 10^{-14} mbar. Practically it is impossible to make a single vacuum gauge which can measure the whole vacuum range hencedifferent vacuum gauges are used which have a specific measuring ranges.

3.3.1 Types of vacuum gauges

There are following two types of vacuum gauges.

a) **Primary gauges:** These gauges measure the pressure as the force which acts per unit area. This force is exerted by gas particles by their impact on the vacuum chamber walls and it depends only on the number density of gas molecules and their temperature but not on the molar mass of gas. The indicated values of pressure are

independent of type of gas in such gauges. These are the mechanical gauges and are used as reference gauges for the calibration of other gauges. These gauges are also called as direct or absolute vacuum gauges.

e.g. Diaphragm gauge, McLeod gauge etc.

b) **Secondary gauges:** In these gauges the pressure is measured as a function of pressure dependent or molecular density dependent properties of the gas. These properties include electrical conductivity, thermal conductivity, ionization probability etc. which are dependent on the pressure and the molar mass of gas. Pressure values measured by these gauges are dependent on the type of gas. These gauges are also called as indirect gauges and are calibrated with the help of primary gauges.

e.g. Pirani gauge, ionization gauge, penning gauge, etc.

3.3.2 Methods of calibration

After the gauges are manufactured they need to be calibrated. Following are the major methods of calibration of vacuum gauges

a) **Direct comparison with a reference gauge:** In this method chamber is evacuated and the gauges are calibrated against the standard gauges by for different values of pressure. The standard gauges used for calibration are precision made McLeod gauge, capacitance diaphragm gauges, spinning rotor gauges etc. by using precision made reference gauge, pressure down to limiting value of reference gauge can be measure with considerable accuracy. This method is easier as compared to other method of calibration.

b) **Generation of a known pressure (static expansion method)**: A lower pressure is reached by expanding certain quantity of a gas (with known values of pressure in working range of primary gauge, volume and temperature) in several stages. In this way, a lower pressure within the working range of ionization gauges is reached. If the gas with known

volume V_1 is expanded to a volume $(V_1 + V_2)$, and from V_2 to $(V_2 + V_3)$, and so on the after n stages of expansion:

$$P_n = P_1 . \frac{V1}{V1 + V2} . \frac{V2}{V2 + V3} \ldots\ldots \frac{Vn-1}{Vn-1 + Vn} \quad (3.10)$$

Where P_n is the calibration pressure and P_1 is the initial pressure measured directly by reference gauge.

In this method the volumes should be measured with possible accuracy and the temperature should be kept remain constant. This method also requires more cleanliness in the apparatus so that it reaches its limiting pressures to avoid errors which may occur due to desorption effects.

c) **Dynamic expansion method:** In this method, the calibration pressure P is attained by introducing a gas at constant throughput Q into the chamber while the gas is being pumped out of the chamber by a pump at a constant pumping speed S. At equilibrium,

$$P = Q/S \quad (3.10)$$

Q is measured from the amount of gas that flows into the vacuum chamber and effective pumping speed is calculated using the known values of conductance of valve. A pressure value is then fixed and the calibration is carried out for the different values of such pressures. After attaining the condition of equilibrium, sorption effects can be neglected and hence this method can be used for calibration at very low pressure where sorption effects are predominant.

3.3.3 Some important terms in gauge calibration

Calibration items: These are the gauges to be calibrated.

Working standards: These are the reference or standard gauges which are used for calibration. They can be primary gauges or already calibrated secondary gauges.

Calibratability: It is the suitable of the gauge for calibration. The calibratability is to be ascertained by external inspections and functional tests.

a) External inspections include visual inspection for damage (pointer, inscriptions, readability of indications, set-up ofmeasuring system, sealing surface), contamination and cleanliness etc., and necessary documents like technical data, operating instructions etc.
b) Functional tests includes tightness of calibration item, electrical functions, proper functions of operating elements, adjustment of elements in defined positions etc.

Adjustments of calibration item: These are the things to be adjusted for calibration**.** It includes

a) Adjustments for zero point
b) Full scale deflection
c) Measuring channel
d) Configuration of output signal, etc.

Calibration certificate: It is the document containing important details of the calibration which includes

a) measuring gas
b) adjustments on calibration item
c) mounting position of calibration item
d) auxiliary measuring equipment used
e) the calibration pressure
f) the signal (e.g. pressure indication, voltage output) of the calibration item
g) Measuring deviations (errors, uncertainties etc.)

3.3.4 Apparatus requirements for vacuum gauge calibration

(According to ISO/CD 3567):

a) Chamber volume should be at least 20 times the total volume of the connected vacuum gauge that includes associated connecting lines.

b) The size of vacuum chamber should be such that the ratio between wall surface and volume is as small as practically possible (ideally sphere). This ratio should not be exceeding the value given by a right circular cylinder with length twice its diameter.

c) The connection between vacuum chamber and the rest of the vacuum system must be such that the entering gas flow strikes neither the vacuum gauges to be calibrated nor the standards nor the orifices opening on the vacuum gauges.

d) The standards and the vacuum gauges to be calibrated must be arranged on the test chamber so that pressure and temperature differences do not lead to considerable errors (equivalent measuring connections). The conductance of the tube connections between measuring chamber and vacuum gauge should at least be some litre per second to keep the influence of adsorption and desorption effects small. The gas flow (inlet and evacuation) must not reach the active zone of the vacuum gauge directly.

e) The residual gas pressure, i.e. the pressure prevailing in the vacuum chamber without gas being admitted must not exceed 10% of the lowest calibration pressure. If a smaller uncertainty is to be reached, the residual gas pressure must be lower.

f) The vacuum gauges must not exert an influence on one another; if need be, suitable precautions have to be taken.

g) The purity of the gas should be equivalent to a maximum impurity level of 0.1% by volume.

3.3.5 Experimental procedure

a) Start the turbomolecular pump and attain the pressure of the limiting value of the gauges to be calibrated.

b) Isolate the chamber from the pumping system using main isolation valve.

c) Set a value of pressure by moving the needle valve. (say 800 mbar)

d) Set the pressure of the calibration item nearly equal to that of reference gauge using gauge controller.

e) Repeat step (c) and (d) for different values of pressure. If possible, a vacuum gauge should be calibrated for its entire working range but the minimum requirement is, it should be calibrated for at least three calibration pressures per decade (e.g. 1, 3 and 5) and at least 10 calibration pressures on the entire scale.

f) Plot the calibration curve. (Reference gauge pressure versus calibration pressure)

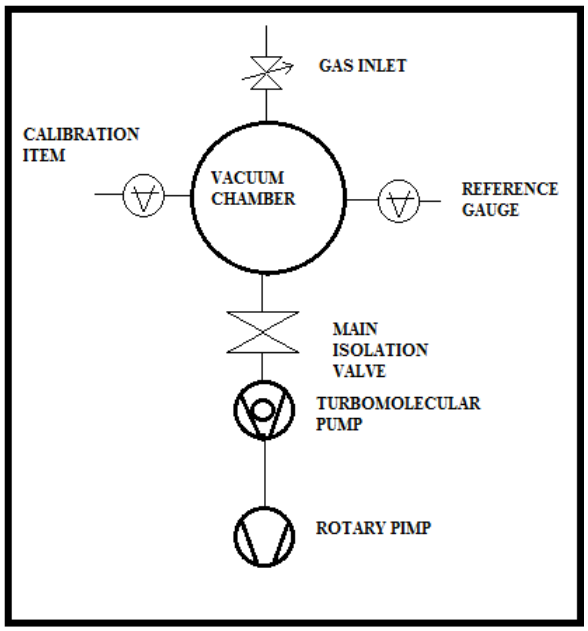


Fig. (3.7). Diagram for calibration of vacuum gauges

Fig. (3.8). Actual set up for calibration of vacuum gauges

3.4 Calculation of conductance of different vacuum elements

The effective pumping speed required to evacuate the vacuum chamber corresponds to the inlet speed of particular pump only if the pump is joined directly to the chamber but it practically such arrangement is rare. It is generally essential to use intermediate piping system consisting of valves, separators, tee, bends, cold traps, elbows etc. all such elements offer offers resistance to flowhence effective pumping speed is (S_{eff}) is always less than the pumping speed (S) of the pump.

The capacity of vacuum pumps is given in terms of pumping speed defined by

$$S = Q/P_i \qquad (3.11)$$

Where Q is the throughput of the pump and P_i is the pressure at inlet of the pump. Similarly, the effective pumping speed S_{eff} is defined by

$$S_{eff} = Q/P \qquad (3.12)$$

Where P is the pressure inside the vacuum space. The overall conductance of piping system between the vacuum space and vacuum pump is also related to the throughput by

$C_o = Q/ (P-P_i)$ (3.13)

Combining above equations, we get

$1/S_{eff} = 1/S + 1/ C_o$ (3.14)

Above equation shows that for there is no use of increasing the capacity of the pump if the conductance of vacuum elements limits the pumping speed because conductance can reduce the pumping speed by several times. e.g. for $C_o=S$, only 50 % of the pumping speed is available at vessel and for $C_o= \infty$, $Seff = S$.

The overall conductance C_o is related to the individual conductances C_i as:

$C_o = \sum C_i$ for parallel conductances, and

$1/C_o = \sum 1/C_i$ for series conductances.

In general the conductance in vacuum components is not a constant value which is independent of prevailing vacuum levels but depends strongly on the nature of flow (continuum or molecular flow). The conductance of pipes and pipe bends differs in the various flow regimes. In viscous flow they are proportional to the mean pressure and in molecular flow theyare independent of pressure. Knudsen flow represents a transition between the two types of flow, and the conductance varies with the Knudsen number.

3.4.1 Experimental procedure

a) Close the valves V2 and V3, and open the valves V1 and V4.
b) Start the rotary pump and calculate the effective pumping speed by the procedure mentioned for the vacuum pumps.
c) With the help of equation (4) calculate the conductance of the element. This conductance value will be for the pipe with valve V1 and V4 (in series).
d) Plot conductance versus pressure for this arrangement.

e) Isolate the particular element to be studied using valves V1, V2 and V3.

f) Repeat the above steps for different piping arrangements in series and in parallel connection and calculate the conductance for each arrangement.

g) Plot conductance of a pipe as a function of diameter at the same average pressure.

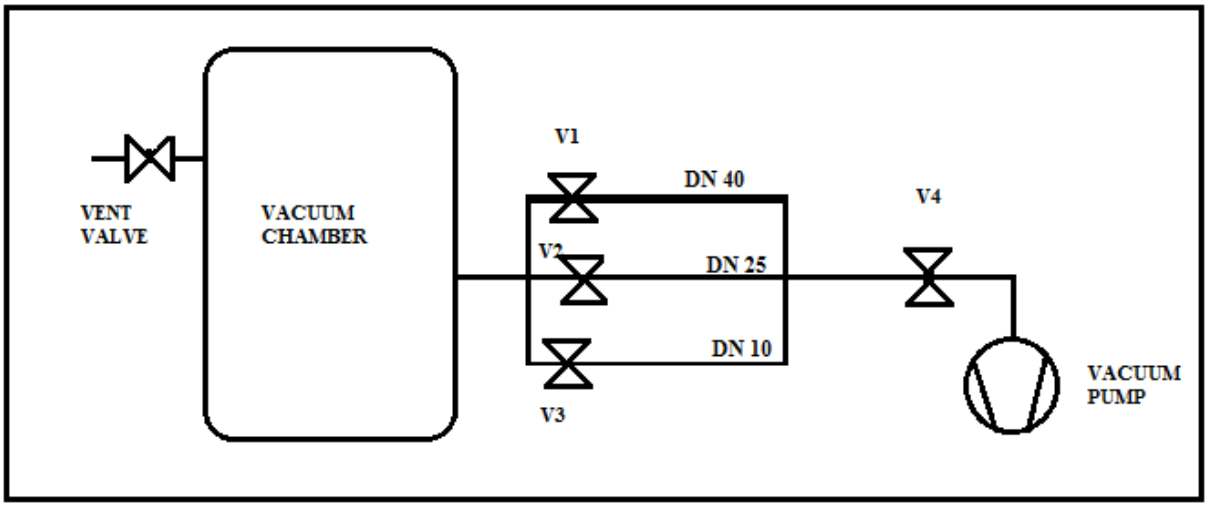


Fig. (3.9) Diagram for conductance calculation of different elements

3.4.2 Experimental procedure using calibrated leak

Other method mentioned below [Fig (3.10)] can also be used for the calculation of conductance of vacuum elements.

a) Isolate the particular pipe to be studied using valves V1, V2 and V3.

b) Allow the dry air to flow into the vacuum chamber using calibrated variable leak valve at a known mass flow rate.

c) Measure the pressure gradient across the isolated pipe with gauges.

d) The expression $Q/\Delta p$ gives the conductance at the average pressure given by $(P_1+P_2)/2$.

e) Repeat the experiment at various average pressure.

f) Plot conductance versus pressure for a particular pipe.

g) Repeat above steps for different piping arrangements (like isolated pipes, series and parallel pipes etc.).

h) Plot conductance of a pipe as a function of diameter at the same average pressure.

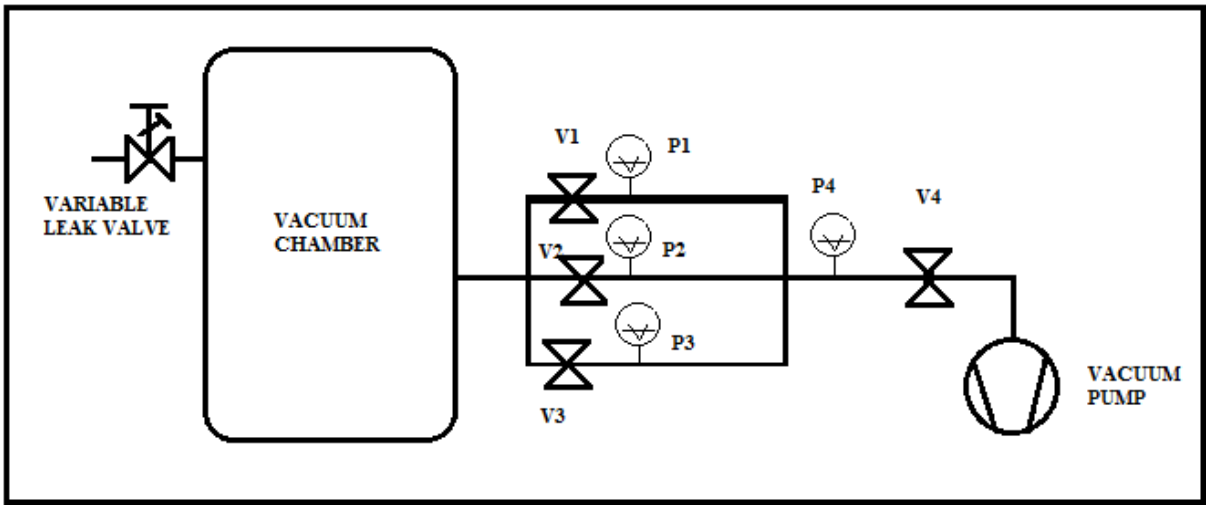


Fig. (3.10) Diagram for conductance calculation of different elements using calibrated leak

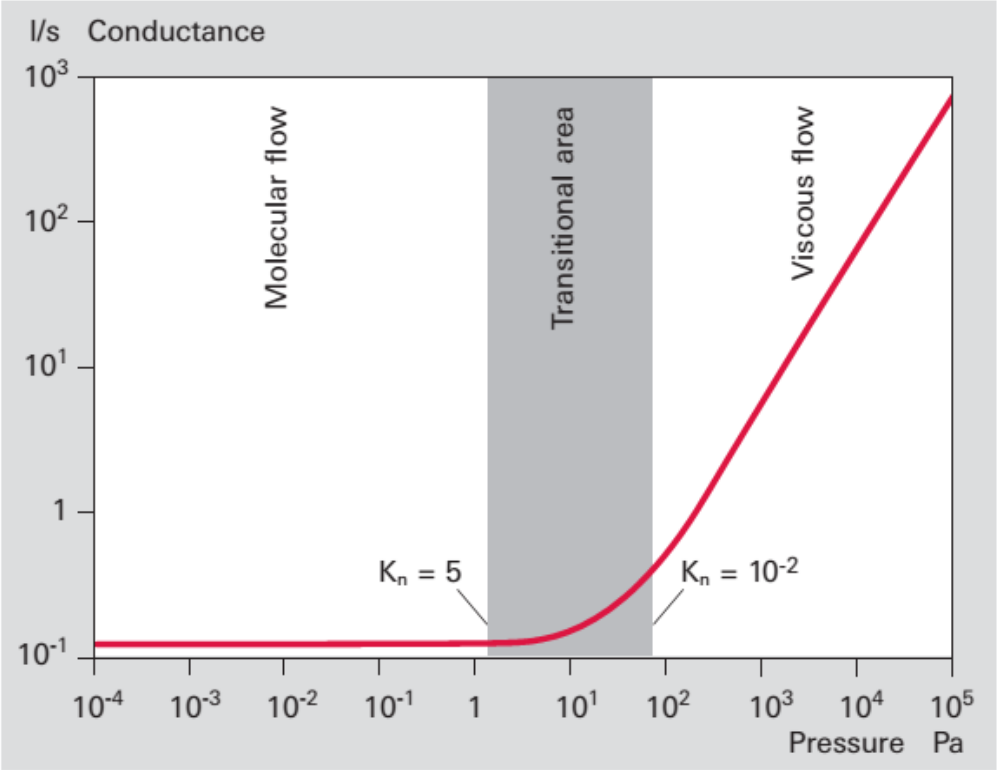


Fig. (3.11) Conductance of a smooth round pipe as a function of themean pressure in the pipe (from reference 34)

CHAPTER 4
DETAILS OF COMPONENTS

4.1 Commonly used components

Following are the commonly used vacuum components.

a) Centring O Rings: These are the vacuum seals used for mating KF flanges of same outer diameter. Its assembly consists of a centering ring (also called as carrier) and O-ring. Centering rings are made of stainless steel, brass or copper and o rings are made of neoprene, viton, silicone rubber and Teflon etc. depending upon the application, service temperature and the vacuum regime. These are specified by nominal diameter and its material.

b) Hinged Aluminium Clamps: These are used for fitting the mating flanges of vacuum elements like pipes, gauges, reducers etc. An O-ring is placed between two mating flanges and the flanges are fitted by hinged clamps and tightened with the help of wing nut provided with the clamps. These are specified by nominal diameter and its material.

c) Claw Clamps: These are used to connect mating flanges to the vacuum chamber or two mating flanges. These are used for larger flanges for which hinged clamps are not available. Single claw clamp arte used for fastening the ISO-LF flanges to tapped ISO-LFB flanges or components (valves, chambers) with a compatible sealing groove. Double claw clamps are used for fastening ISO-LF flanges.

d) Blank Flange: These are used for closing the flanges where vacuum elements like pipes, gauges etc. are not connected. These are specified by nominal diameter and its material.

e) Valve: These are used for opening and closing the flow of air or gas. Commonly used valves are ball valves, butterfly valves, needle valves etc. Needle valves are generally used for venting purpose of vacuum chamber.

f) Reducer/Adapter: These are fittings used to connect two flanges of unequal diameters. Adapters are used to change from one family of flanges to another. eg. ISO-QF 25 to CF 275. Reducer Fittings are used to reduce the size of flanges and are used within the same flange family e.g. CF600 to CF275 or ISO-LF63 to ISO-QF40.

g) Flexible Hose: These are the vacuum pipes generally made of stainless steels. They have flanges at both ends for the connection. They are specified by nominal diameter, length and material.

h) Nipples: These are the fittings with an ISO flange on both ends of a straight tube. They are used to create a weld free straight connection in vacuum systems. They also serve as straight adapters between different tube sizes and flange types. Half nipples are the fittings with a flange on one end and raw tubing on other end. They are welded to vacuum chambers. These are also made with flexible designs. These are made of stainless steel, aluminium and brass.

i) Other fitting used in vacuum systems are crosses, tees, bends, elbows etc.

j) Other necessary things are vacuum gauges, gauge displays, vacuum pump oils, vacuum greases etc.

4.2 Suppliers of vacuum components

Bills of materials were prepared and following suppliers were contacted for the materials.

Table 4.1. List of suppliers of vacuum components

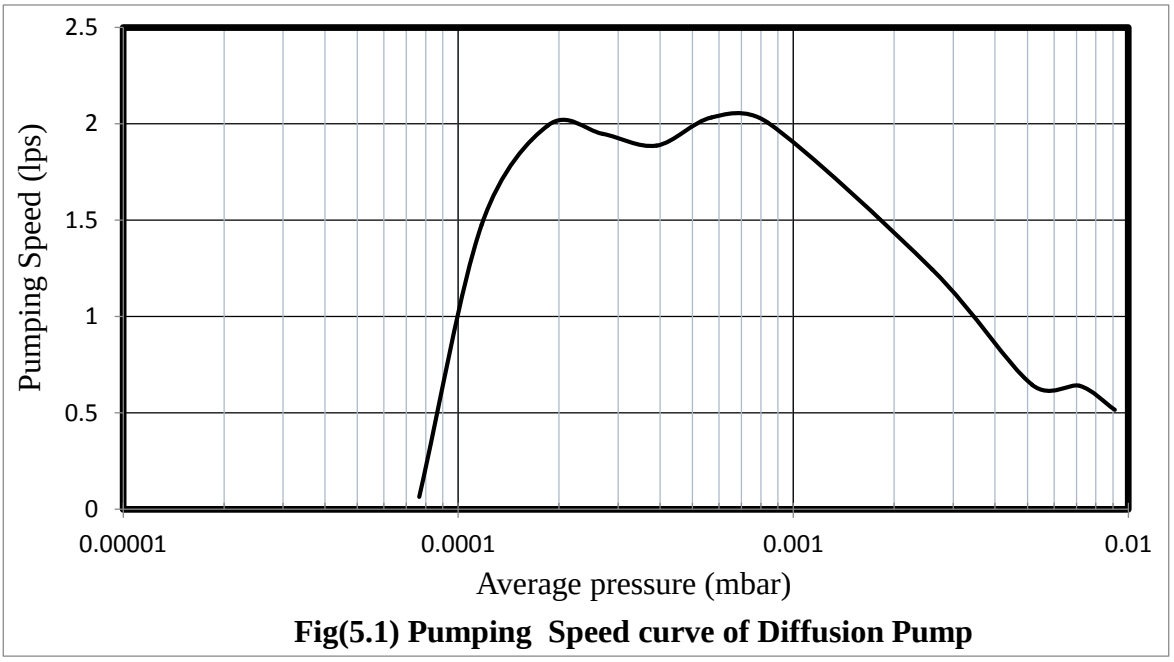
SN	Name of company	Address of company & Contacts
1	Hind High Vacuum Company Pvt. Ltd.	No. 31,34 and 37, KIABD, Industrial Area, Dabaspet, Nelamangla Park, Bangalore-562111, India Or, 34 Kabir Road, Kolkata - 700 026, Ph.: +91-9674646334, Email: shouvik@hhv.in, Web site: www.hhv.in
2	VT Vacuum Techniques Pvt. Ltd.	36A-A.G.S Layout, MSR Nagar, Bangalore-560054 Ph.: +91 9845941264 Email: vacuumtech@vsnl.net, info@vtvacuumtech.com Web site: www.vtvacuumtech.com
3	Indian High Vacuum Pumps	Indian High Vacuum Pumps, B-28, 1st cross, 1st stage, Peenya industrial Estate, Bangalore-560058, India Ph.:+91 9448076807 Email: ihvp95@yahoo.in Web site: www.indianhighvacuumpumps.net
4	Pfeiffer Vacuum (India) Pvt. Ltd.	25/5 Nicholson Road, Secunderabad 500009, Ph.: +91-40-27750014,+91 9391391544, Fax +91 40 27757774 Email: pvin@pfeiffer-vacuum.in Web site: www.pfeiffer-vacuum.net
5	Oerlikon Leybold Vacuum India	No. 82(P), 4th Phase, K.I.A.D.B. Plot Bommasandra Industrial Area Bangalore - 560 099, Mob: +91 9342548183 Email:haribabu.muniramannagari@oerlikon.com Website:www.oerlikon.com

6	Hind Vactech Scientific Pvt. Ltd	155,Ground Floor, Chirag Delhi, New Delhi-110017 Mob: +91 9958822058,+91 9711805218 Email: info@hindvactechscientific.com peeyush@hindvactechscientific.com vactechscientific@gmail.com
	Everest Blower System	435, Modern Industrial Estate, Phase I, Bahadurgarh, Haryan-124507, India Mob: +91 9582600976 Email: north.ebs@everestblowers.com Web site: www.everestblowers.com
	IVC Pumps Pvt. Ltd.	Plot No. 255, Phase-I, Near Devi masala, G.I.D.C. Estate, Naroda, Ahmedabad-382330, Gujarat, India. Tel. Ph.: +91 79 22807781/82, Mob: +91 9825708057, +91 9904707781 Email: ivc@ivcvacuumpumps.com Web: www.ivcvacuumpumps.com

CHAPTER 5

RESULTS

Pumping speed characteristics of diffusion pump calculated from experiment is as shown in Fig. (5.1). It resembles the theoretical curve but the experimental pumping speed was found less than the specified pumping speed due to conductance of the vacuum elements like pipes, valves etc.



Fig(5.1) Pumping Speed curve of Diffusion Pump

Pumping speed characteristic of turbomolecular pump is shown in Fig (5.2). It shows that pumping speed increases to a certain value and then becomes constant over a particular range. The effective pumping speed was found much less than the specified value of pumping speed at inlet. This is because of the low conductance of vacuum elements in molecular flow region. Pumping speed was found decreasing after the pressure range of 10^{-5} mbar.Pumping characteristic of backing pump for turbomolecular pump is shown in Fig. (5.3). It shows that pumping speed is initially constant over the working range of pump and then goes on decreasing as the pump reaches its ultimate pressure.

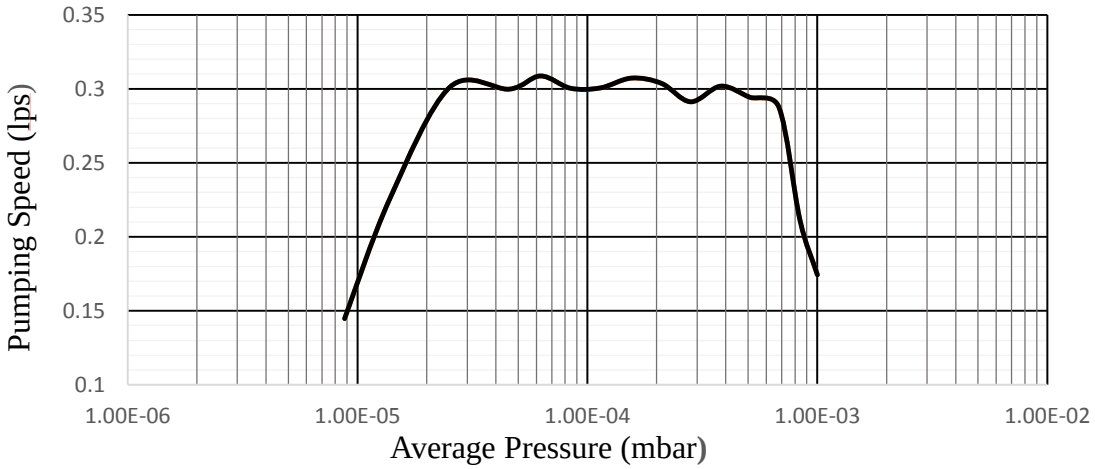


Fig (5.2)Pumping speed curve of turbomolecular pump

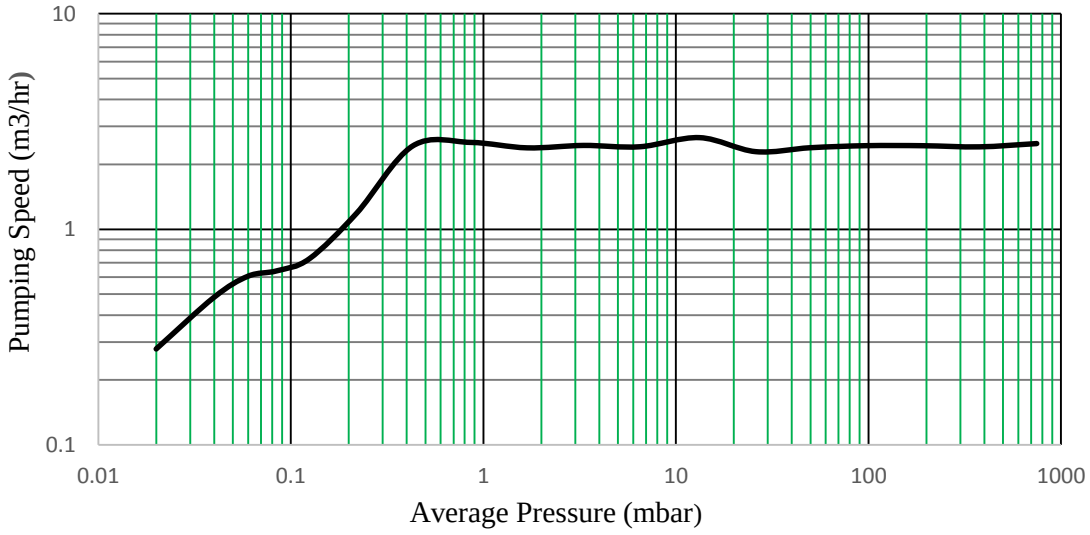
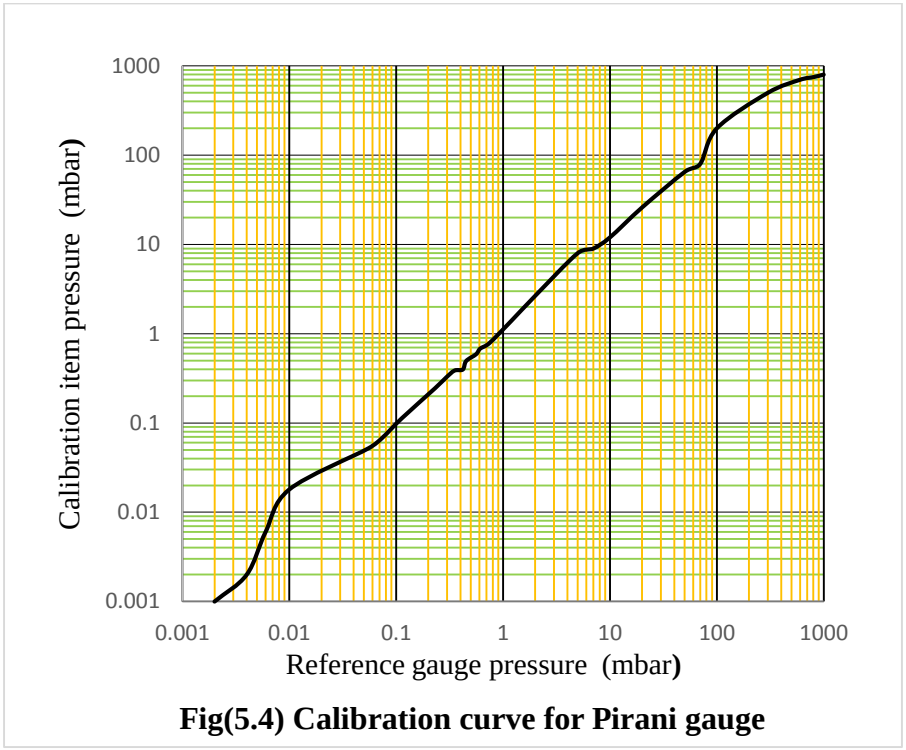


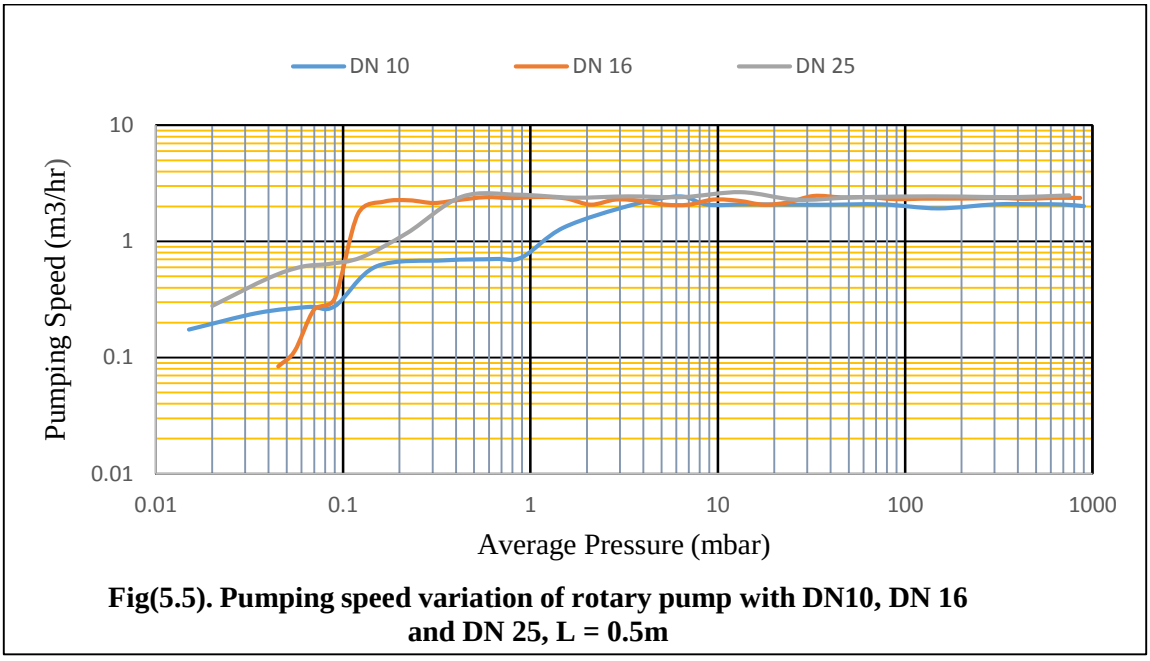
Fig. (5.3) Pumping speed characterics tic of backing pump for turbomolecular pump

Conductance of different pipes was calculated by calculating effective pumping speed of rotary pump with different hoses (DN10, DN16 and DN25). Fig (5.5) shows the variation of effective pumping speed for rotary pump with different pipes DN 10, DN16 and DN25. It was found that conductance is more for large diameter pipes. Conductance values calculated for the different hoses were found to be similar to the theoretical values and it was found that conductance in also more important for attaining ultimate pressure as it affects the pumping speed of pump to a great extent. Fig (5.4) shows the calibration curve for calibration item

(Pirani gauge). The calibration curve is almost linear from 0.01 mbar to 100 mbar and deviation are more in the range of 100 mbar to 1000 mbar and 0.001 mbar to 0.01 mbar. However the uncertainty was found in the acceptable range.



Fig(5.4) Calibration curve for Pirani gauge



Fig(5.5). Pumping speed variation of rotary pump with DN10, DN 16 and DN 25, L = 0.5m

CHAPTER 6
CONCLUSION AND FUTURE SCOPE

The above mentioned experimental set up were successfully arranged and experiments were performed. More similar experiments can be done using these procedures. It includes pumping speed measurement of sorption pumps, roots pumps etc. also pumping speed can be calculated for different sizes of vacuum chambers and the variations can be studied. Calibration of other high vacuum gauges can also be done using Precision made reference gauges by same method as mentioned in this experiment. Conductance of different vacuum elements like pipes, reducers, adapters, valves with different sizes can also be calculated in viscous flow and molecular flow using similar procedure. This laboratory work will be useful for the students dealing with vacuum technology so that can know vacuum systems with practical approach. Above experimental set up can also be used for the research work at NIT Rourkela.

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