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Techniques Used for Vacuum Standardization

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Abstract: Numerous primary vacuum standards namely Mercury Manometer Standard, Volume Expansion Standard and Orifice Flow Standard of different ranges and designs have been developed by various international vacuum metrology laboratories, for the calibration of vacuum gauges. This paper briefly describes a variety of scientific techniques that had either been used or are employed in different primary vacuum standards of these laboratories. It also describes various approaches by means of which particular scientific techniques employed for vacuum standardization, are theoretically and technically improved. This improvement is essential, to guarantee the accuracy and to make a unique development in vacuum standardization.

Key words: Vacuum gauges . calibration . vacuum standards . techniques used

INTRODUCTION

The modern, complex and refined manufacturing processes and research activities require accurate vacuum measurements. The accuracy of these measurements can have major effects on the validity of results, product quality, energy efficiency and in many cases the safe operation of different processes [1]. Therefore, measurement accuracy in every vacuum application is need of the age and of prime interest. This accuracy is achieved with the help of well calibrated vacuum gauges and this is possible only, when there exist, proper vacuum standards of required range and accuracy for calibration of these gauges. Mainly three primary vacuum standards employed for this purpose are mercury manometer, volume expansion and orifice flow standard.

PRIMARY VACUUM STANDARDS

Primary vacuum standards have the highest metrological qualities in which the pressure is deduced directly from the involved physical quantities (mass, length, time etc) uniquely with the proper accuracy, precision and fine resolution [2]. The calibration of such standard is considered according to the laws of physics along with the knowledge of its significant dimensions. A gauge which is calibrated on the primary standard and with which other gauges can be compared for calibration is known as the reference gauge or secondary standard. Such reference gauges would normally be expected to show superior qualities of accuracy, reproducibility and stability compared with a test gauge [3]. The Capacitance Diaphragm Gauges (CDGs) and Spinning Rotor Gauges (SRGs) are internationally recognized as secondary standards for medium and high vacuum respectively [4]. Commonly used primary vacuum standards are 'Mercury Manometer Vacuum Standards, 'Volume Expansion Vacuum Standards' and 'Orifice Flow Vacuum Standards'.

Mercury manometer standards: Mercury column manometers are broadly used as primary standards for the calibration of low vacuum gauges [5]. These devices employ U-tube type arrangement and are capable of most accurate measurements in this range. As given in Fig.-1 [6], any pressure applied to the left side mercury surface of U-tube displaces the mercury, thus generating a pressure $\boldsymbol{P}$ determined by the mercury density ρ, the displaced mercury height $\Delta \boldsymbol{h}$ and the local acceleration due to gravity $\boldsymbol{g}$. When the applied pressure and the displaced mercury are in equilibrium, with some reference pressure $\boldsymbol{P_{ref}}$ on right side column, the generated pressure $\boldsymbol{P}$ is given by[6]:

$$\mathbf{P = \rho g \Delta h + P_{ref}} \tag{1}$$

A number of manometers have been designed at various laboratories of the world, differing from each other with the variations in the column length determination techniques. The high range manometers established at national standards laboratories have been reviewed by Guildner and Terrien [7] while low range ones have been reviewed by Ruthberg [8] and Peggs [9]. Recently established manometers are the

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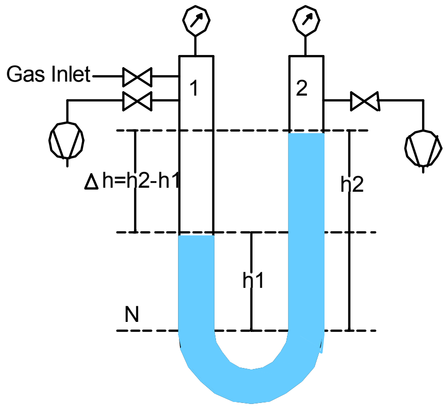

Fig.-1: U-Model manometer [6]

primary pressure standards in many advanced countries. Hong *et al*. [10] from KRISS (Korea Research Institute of Standards and Science, Republic of Korea) and Heydemann *et al*. [11] of the NIST (National Institute of Standards and Technology, USA), described an accurate mercury manometer employing ultrasonic interferometer for the determination of mercury column heights. Ooiwa *et al.* [12] from national research laboratory of Japan reported a mercury manometer using a white light Michelson interferometer. Ueki and Ooiwa [13] also developed an oil manometer interferometer for use at lower pressures. Legras and Breton [14] of the LMN (Laboratoire National de Metrologie D'Essais, France), reported a manometer using liquid gallium. In the following sections, some special techniques are presented used for liquid column determination in various standard liquid column manometers with reference to some well-known assemblies, developed in different vacuum standards international laboratories.

Liquid column determination techniques: The measurement of liquid-column height is accomplished by determining the surface of the menisci. Two techniques are used for this purpose: (a) Contacting Techniques, wherein a mechanical contact is established at the surface of the menisci and the height is calculated between this contact and a reference line. (b) Non-contacting Techniques, in which either light or ultrasound or laser reflected from menisci provide column height information [15].

Contacting techniques: The accuracy attainable with a mercury manometer mainly depends upon the exactness in measuring mercury column height. In this contest, two simple techniques are employed. In the first one, micrometer-ruler arrangement is recognized for mercury-column height determination. In the second technique, mercury column height is measured with micrometer-micrometer setup, because in accordance with Gerard [16], manometer is made significantly simple by placing a micrometer in each arm of the tube. Fig.-2 & Fig.-3 schematically show two such contacting techniques for measuring the mercury column heights.

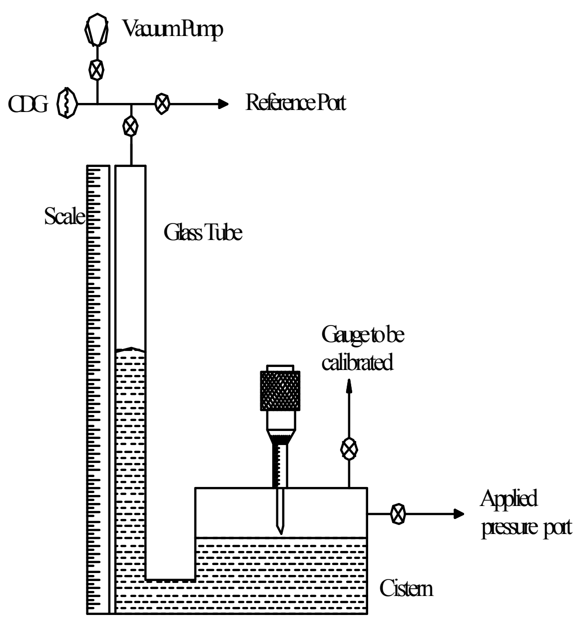

Fig.-2: Ruler-micrometer setup [17]

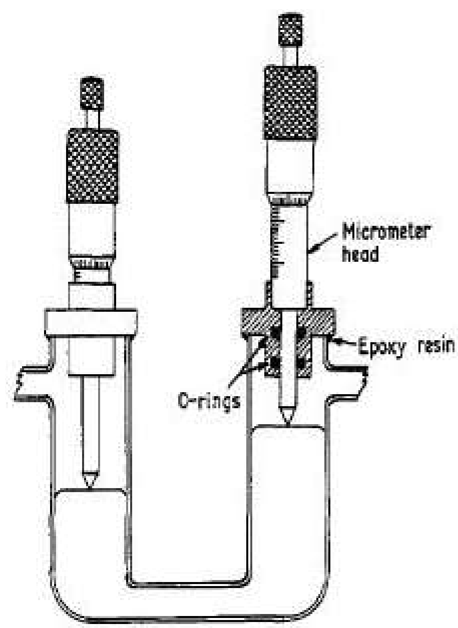

Fig.-3: Dual-micrometer setup [16]

By making the use of ruler-micrometer technique and considering all the relevant parameters, a standard mercury manometer, for the calibration of vacuum gauges from atmospheric to 1mbar, schematic of which is shown in Fig.-4, has been developed at National Institute of Vacuum Science & Technology (NINVAST), Pakistan [17]. Its proper design philosophy comply with many good points such as easiness in operation, compactness in fabrication, correctness in observation, low vibration and especially cost effective.

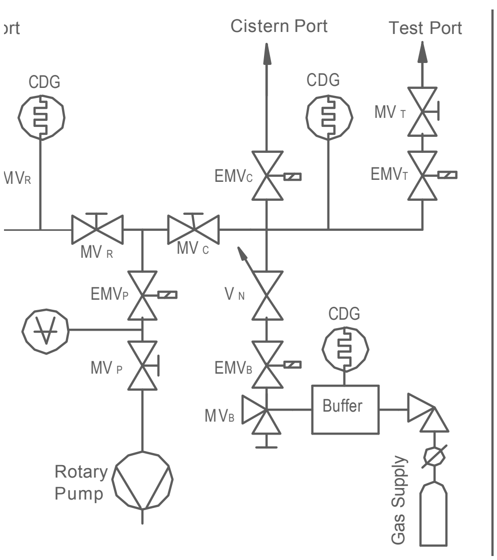

Fig.-4: [17] Schematic of vacuum system and manometer:-MV-Manual Valve, EMV-Electro-Magnetic Valve. Subscripts B, C, P, R and T stand for Buffer, Cistern Port, Pump, and Reference & Test Port respectively. CDG-capacitance diaphragm gauge

There are some disadvantages of contacting liquid-column height measuring techniques. The measurement resolution of the liquid-column heights limits the upper vacuum range. The measuring accuracy of the required height varies from operator to operator. A change in pressure during determinations requires resetting the micrometers. Due to manual operations, some determinations become time-consuming. For further improvement, the next alternative is non-contacting techniques.

Non-contacting techniques: To seek out a concrete solution of the above problems, non-contacting techniques were proposed. Most important advantages of these techniques are the remote and automatic height determinations, permitting a high degree of temperature stability, continuous tracking and measurement of the column heights with high resolution and accuracy. The displacement relative to a reference level of either the meniscus or a float resting upon the meniscus can be measured by using various techniques such as optical, ultrasonic or laser interferometry techniques. Instruments making the use of these techniques to observe minute length changes are generally known as micro manometers [15]. Three such manometers employing non-contacting techniques are discussed here, briefly.

Optical Interferometer Manometer (OIM): In such type of manometers, optical interference technique is employed, in which collimated monochromatic light is directed by adequate optics on the menisci. For zero pressure applied to the instrument, there is a reference interference fringe pattern. Any applied pressure causes the corresponding change in menisci positions which alter the fringe pattern, accordingly. The comparison of these fringe patterns gives the required information about the column height and consequently the generated pressure by the instrument. The low-vacuum U-tube manometer developed by IMGC (Istituto di Metrologia G. Colonnetti), Italy is based on this technique [18].

Ultrasonic Interferometer Manometer (UIM): In the Ultrasonic Interferometer Manometer (UIM) of KRISS, the variation in heights due to the pressure difference

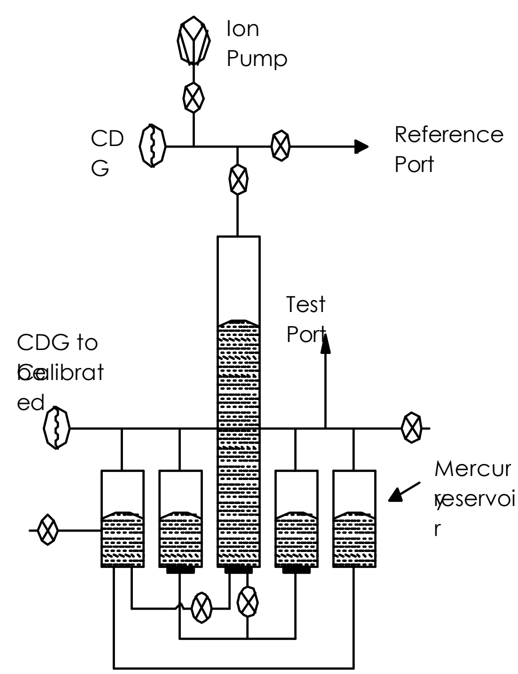

Fig.-5: UIM setup at KRISS [10]

between the mercury columns is measured with ultrasonic interferometer technique [19]. It consists of three mercury columns. One long column is in the centre with ion pump installed at its top for reference part evacuation and a gauge to measure the reference pressure. From bottom side it is connected to mercury reservoir. Other two small mercury columns on each side of long column are for test pressure application and measurement. The electronic circuits used for the excitation of the transducers are attached to the bottom of the said three mercury columns. Fig.-5 shows the entire setup of this system [10].

Laser Interferometer Manometer (LIM): In Laser Interferometer Manometer (LIM), detection of the free mercury surface is observed by using U-tube type manometer with the mercury surfaces acting as reflectors of Michelson interferometer. So the height difference of two reflectors is measured by using this interferometer. For this technique, the surfaces have to be very stable and free from vibrations. Operating principle of this manometer developed by Harrison *et al*. in Australia is easy to understand with reference to its schematic shown in Fig.-6 [20].

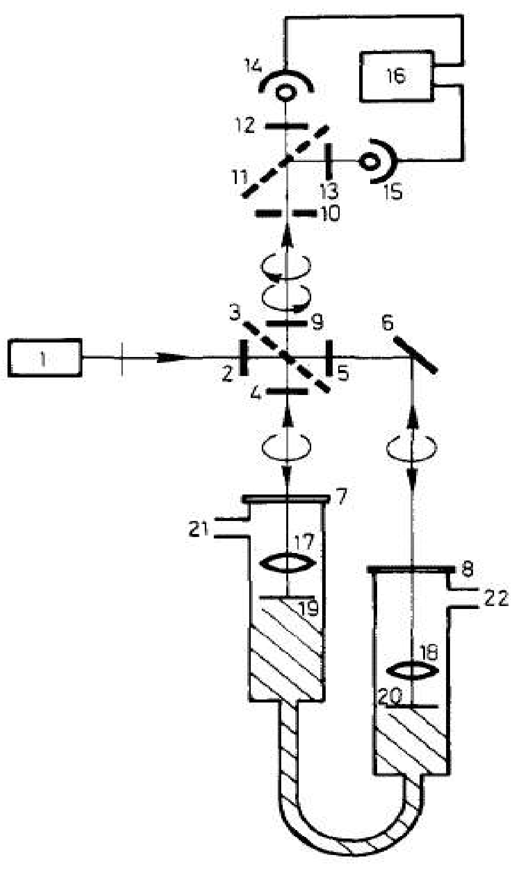

Fig.-6: LIM setup MRL [20]

In this figure, the general arrangement of the LIM various components essentially required for the operation of the manometer are labeled: 1-is He-Ne laser source; 2,4,5,9-are four quarter-wave plates; 3-is a polarization beam splitter; 6-is a beam-bending mirror; 7, 8-are two small windows; 10-is a 400 μm pinhole; 11-is another beam splitter; 12, 13-are two Polaroid analyzers; 14,15- are two photomultipliers; 16-is a reversible counter; 17, 18-are two floating cat's-eyes; 19, 20-are two free mercury surfaces; 21-a port to high vacuum pump; 22-is a port to pressure source.

The greatest disadvantage of the non-contacting technique is the mechanical instability during different determinations. Moreover, vapor pressure of mercury (2×10^{-3} mbar), may cause problems in the calibrations of medium vacuum gauges. Therefore, in the medium vacuum range when mercury manometers are not suitable, primary standards based on volume expansion of gas, are used for the generation of medium vacuum.

Volume expansion standards: In the medium vacuum range, a widespread and accurate method of generating pressure is the 'static or volume expansion method', simple model of which is shown in Fig.-7 [33]. This was first used by Knudsen [21]. Variations in the Knudsen gas expansion technique were described by a number of authors [22-32]. Discussions on this type of standard are also found in [8,33]. After a number of improvements, this method is now being used in many laboratories of the world [34], few of them are: NPL (National Physical Laboratory) UK, PTB (Physikalisch Technische Bundesanstalt,) Germany, UME (Ulusal Metroloji Enstitusu) Turkey, ETL (Electro Technical Laboratory) Japan, METAS (Federal Office of Metrology) Switzerland, CENAM (Centro Nacional de Metrolog´ia) Mexico, IMGC and KRISS, etc.

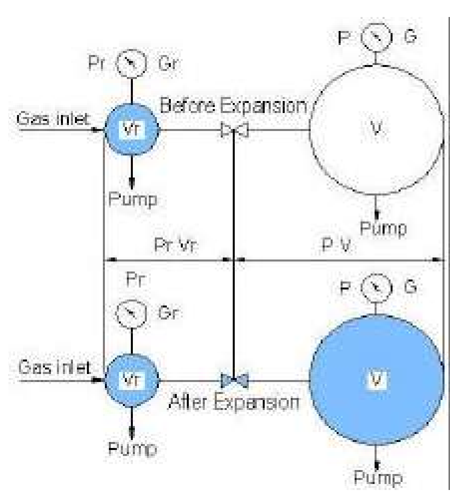

Fig.-7: Single-stage SES [33]

Volume expansion techniques: There are two types of techniques used for standard volume expansion systems [18]: single volume expansion technique and multiple volume expansion technique. Berman and Fremerey [35] used a single expansion technique. The major disadvantage of this single step expansion is that, the wide vacuum range with necessary accuracy cannot be achieved by this method. Multiple volume expansion technique is employed for generating a large number of calibration pressures than that obtained with single expansion technique. All the techniques used to develop international Static Expansion Standard (SES) systems cannot be discussed; however, here is a brief discussion of some the systems employing expansion techniques for three, four and five stages.

Three-stage SES system: The multiple volume expansion system of UME (Ulusal Metroloji Enstitusuy) in Turkey, comply three-stage expansion technique [36] as shown in Fig.-8. It is based on three large volumes (V_1, V_2, V_3) as well as three small volumes (v_1, v_2, v_3) separated from each other by different valves. Accurately found volumes are: v_1 and v_2 ˜ 0.15 liter, V_1 and V_2 ˜ 15 liter, v_3 ˜ 0.7 liter and V_3 ˜ 72 liter. The volume V_3 is the main calibration vessel and has many flanges for test gauge installation and other different purposes. The smaller vessels v_1, v_2 and v_3 are utilized as initial volu mes for the expansion processes. The entire apparatus of the system is built in accordance with requirements for Ultra High Vacuum.

Four-stage SES system: The static expansion system developed at METAS, Switzerland [37], uses four stage expansion techniques as shown in Fig.-9. It has four pairs of chambers, installed in such a way to achieve a pressure reduction ratio, up to the range of 10^{-9}. The first and second chamber has a volume ratio of about 100, whereas the third and fourth large chambers get in touch with through small chambers of 0.5 liter or 2 liter. Chamber four has a volume ratio of about 200 respective to the 0.5 liter chamber and 50 respective to the 2 liter chamber.

A 4-stage SES for medium vacuum range has also been developed at NINVAST, Pakistan, which is shown in Fig.-10 [38]. In this standard system due attention has been given to make the use of minimum number of vacuum pumps, gauges, electronic devices and other vacuum accessories without compromising on the accuracy of measurements. Moreover whole design of the subject standard system is entirely based on good mechanical engineering, surface engineering and vacuum engineering. All this made the standard system accurate, compact, vibration free, user friendly with easy operation and especially cost effective.

Five-stage SES system: A five-stage expansion technique has been used in the static expansion system of NPL in UK. It has been designed considerately and is accomplished with the special setup of five small volumes (v_1–v_5) and five large volumes (V_1–V_5), arranged in the appropriate sequence, in order to get proper output. The arrangement of various volumes along with different valves and pumping systems is shown in Fig.-11 [39].

Static expansion method is not used for the generation of high vacuum because of some inherent limitations. The ultimate vacuums achievable with this method are limited due to surface outgasing and chamber sizes. Accuracy is limited because of the uncertainties in reference pressure measurements and volume ratio determinations. Furthermore, corrections have to be made for the pumping or removal of gas by ion gauges and surface adsorption. To overcome these difficulties, the most practical method for high vacuum generation, termed as standard orifice flow method is employed. Since these devices operate with continual flow of gas, the problem due to out-gassing and the gauge interactions are significantly reduced as compared to static expansion devices

Orifice flow standards: Orifice flow method, also called "dynamic method" or "continuous expansion method" [22,40,41], is used for high & ultra high vacuum standardization. This calibration technique links with Dushman [42] and Found who applied it for the calibration of ionization gauges by setting the flow primarily along the length of a tube. Changes in this technique, using a proscribed flow of gas through calibrated orifices, were explained by various authors [43-53]. This method for generating high vacuum is used for vacuum gauge calibration in the range of molecular flow. It has also been employed at NIST (USA), PTB (Germany), LMSC (Lockheed Missiles & Space Company, California), IMGC, Italy, KRISS South Korea. Various forms that this method can take have been reviewed by Poulter [3].

Single orifice technique: A variety of forms of the orifice flow method arise either from the number of orifices techniques or reference technique or flow rate measuring techniques. Single orifice technique has known to be the most established one, schematic of which is shown in Fig.-12 [3]. This system consists of a thin orifice at the joint of two spheres.

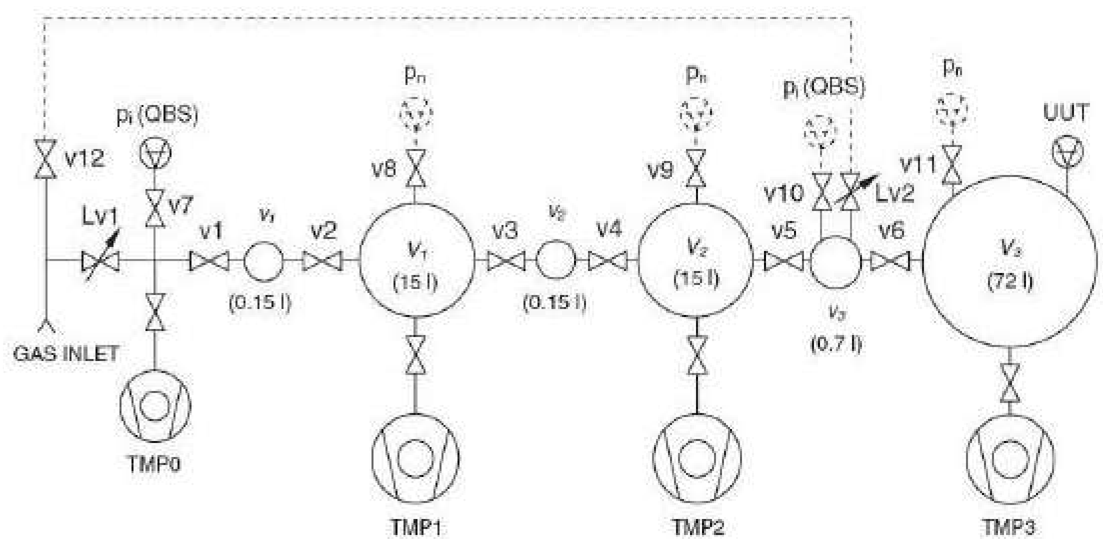

Fig.-8: Schematic of 3-stage SES in UME, Turkey [36]

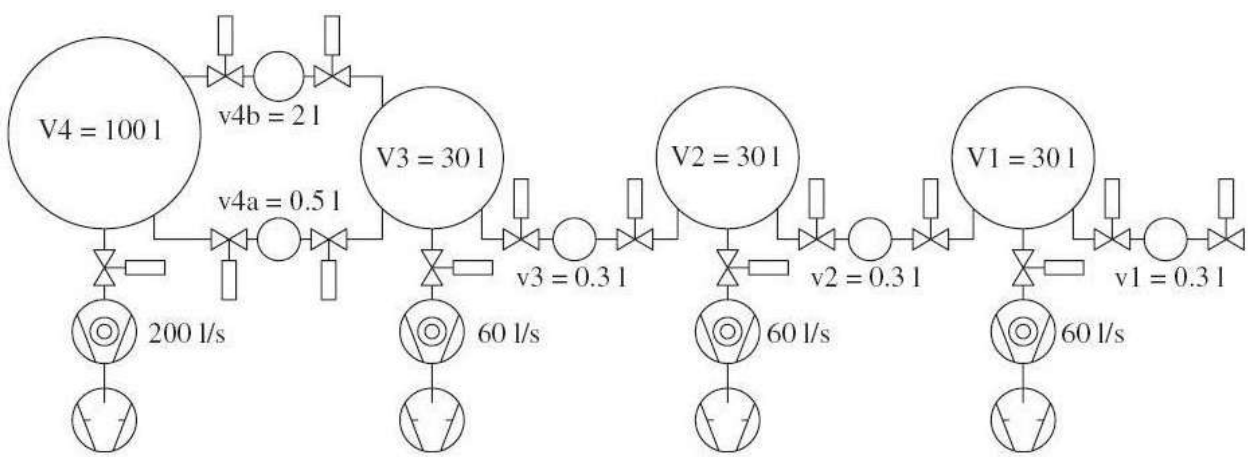

Fig.-9: Schematic of 4-stage SES in METAS, Switzerland [37]

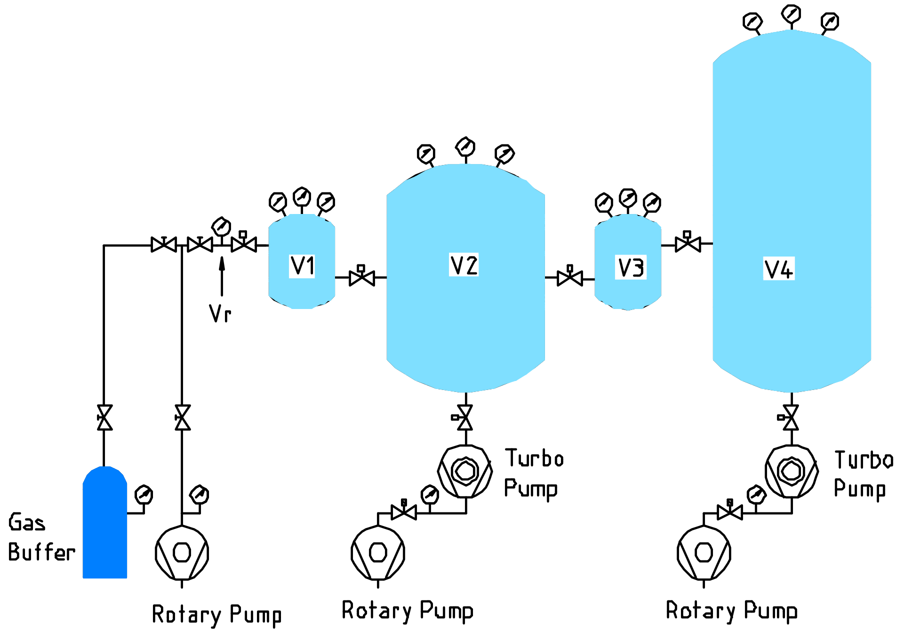

Fig.-10: Schematic of four stages SES system, Pakistan [38]

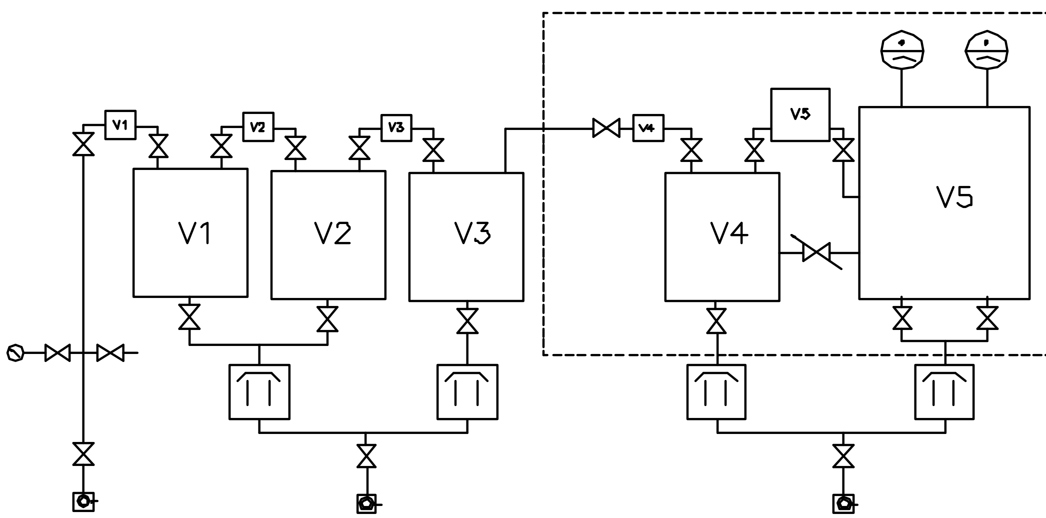

Fig.-11. Schematic of 5-Stage SES in NPL, UK [39]

An Orifice Flow Standard employing such a single orifice technique with 'constant volume-variable presssure' flowmeter, schematics of which is shown in Fig.-13 has been developed at NINVAST, Pakistan [54]. In this system the designing, fabrication, instrumentation and measuring techniques, all are on the basis of precise vacuum and surface engineering and in need of refinement to fully meet the simple formulation goals and thus matching the high vacuum design philosophy. In order to reduce the uncertainties, due thought has been given to the relative positions of the gas inlet, the gauges, the orifice and the other effects. Moreover the system is fully refined, very simple, more compact, user friendly, accurate and especially cost effective, for the calibration of high vacuum gauges.

Multiple orifice technique: A self-explanatory schematic of this technique is given in Fig.-14 [3]. For such technique, the gas flows from the first inlet

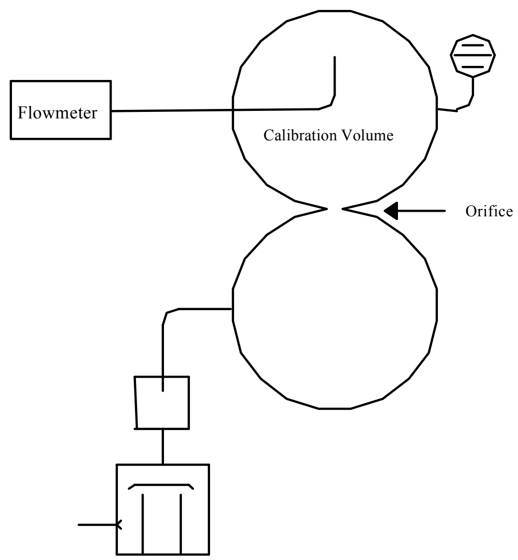

Fig.-12: Schematic of single OFS [3]

chamber from beginning to end a number of orifices, each isolated by a volume which is evacuated by further orifice. This technique is good in the sense that low pressures can be generated by making the use of higher initial gas throughputs than would be essential for the generation of same pressure in a single-orifice system.

Reference transfer technique: A variation of the orifice flow method is called Reference Transfer Method (RTM). This technique is best understood by considering the system with schematic as Fig.-15 [3]. This system has two volumes separated by an orifice of computable conductance. A secondary standard gauge is attached so that it can be connected to either upper or lower volume, making the system balanced. The test gauge is attached to the lower volume. The gas through flow meter generates the calibration pressure in the system. The indication of the secondary standard gauge when connected to the upper chamber is noted. Then the valve position is altered and the pressure in the lower chamber is watched. This time gas flow in the system is large

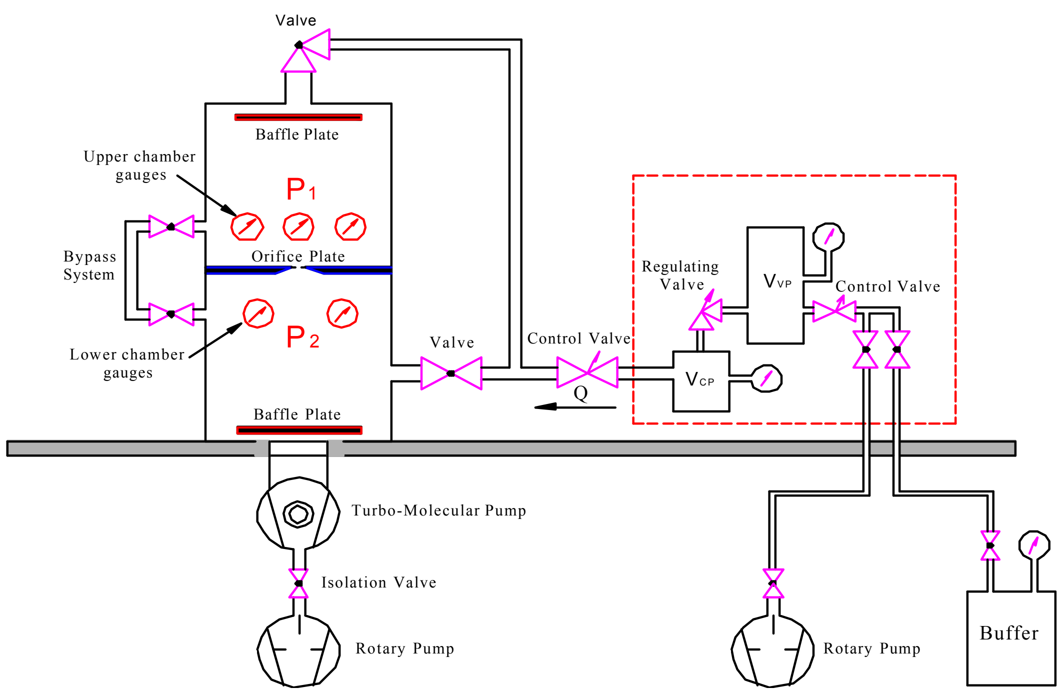

Fig.-13: Single orifice flow standard [54]

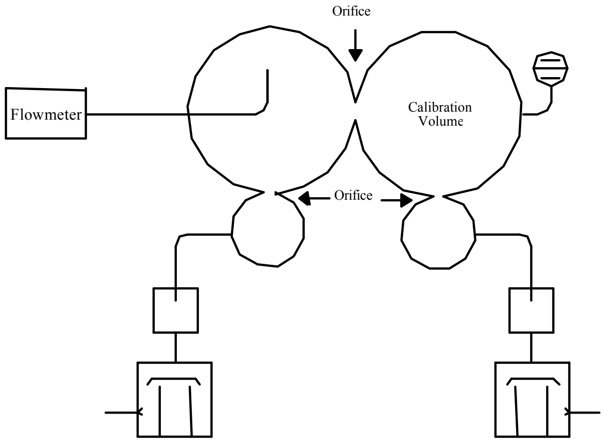

Fig.-14: Schematic of multiple OFS [3]

enough to be measured by the flow meter. At this moment the calibration pressure can be calculated from the information of the orifice dimensions and the final gas flow rate.

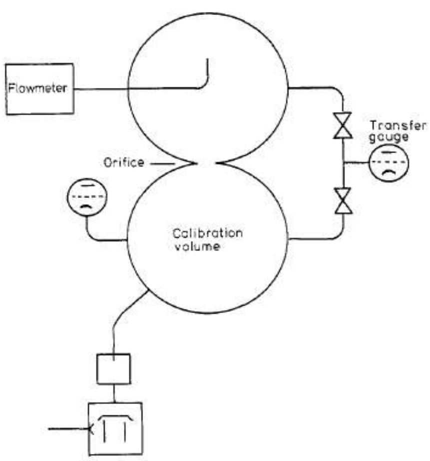

Fig.-15: Schematic 0f RTM [3]

Flow meter techniques: The orifice flow standard system mainly consists of two parts, one is high vacuum calibration chamber and other is flow measuring system as shown in Fig.-16 [3]. For the flow measuring purpose, two types of flow meters using two different techniques are being used: 'constant-pressure with variable volume' and 'constant-volume with variable pressure'. The throughput Q through the orifice of an OFS [55] is given by

$$Q = P(dV/dt)+V(dP/dt) \qquad (2)$$

where P and V are pressure and volume respectively of

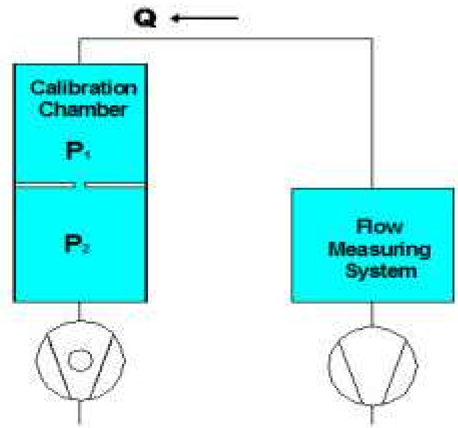

Fig.-16: Orifice flow standard [6]

the manifold of flow-meter and t is time.

- For constant-pressure flow-meters

$$Q = P\ (dV/dt) \qquad (3)$$

This type of flow-meter arrangement is used by most of the international laboratories.

- For constant-volume flow-meters

$$Q = V\ (dP/dt) \qquad (4)$$

This kind of flow-meter setup is used in USA [56] and Pakistan [54] etc.

CONCLUSIONS

After extensive and long time research in the field of vacuum metrology, it has unanimously decided that mercury manometers are the best devices for low vacuum standardization. While for medium vacuum standardization, multi-stage static expansion vacuum standards are effectively employed throughout the world. Similarly, the most accurate standards used for high and UHV standardization are the orifice flow standards. In addition, any technique used in the primary vacuum standard is an established one, with high metrological accuracy and convenience of operation. Fundamental calibration techniques have changed little over the years, because new demands for accuracy at lower and lower pressure levels, have led to interesting improvements and excellent refinements in the devices particularly developed for vacuum standardization. Consequently, each primary vacuum standard is an independent and exact apparatus for the accurate pressure generation in the defined range.

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