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Development and Characterization of Volume Expansion Vacuum Standard

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Abstract: A multi-stage primary vacuum standard system for the generation of calibration pressures, in the range 10^0-10^{-4} mbar has been developed. It is based on volume expansion method, whereby the range is extended to lower pressures by multiple expansions. The standard system consists of four chambers that help to achieve a pressure reduction by a factor of about 10^{-5} in the main calibration chamber after a four-step expansion. The entire system is built using ultrahigh vacuum techniques. For performance characterization, results have been compared with that of a secondary standard i.e. Capacitance Diaphragm Gauge (CDG). The average value of correction factor is 0.995 while linear correlation coefficient of primary–secondary standard is found to be 1.00. Various uncertainties of the generated pressures by this method have been calculated. The combined uncertainties lie between 1.73×10^{-3} mbar and 9.18×10^{-6} mbar. The values of relative uncertainty are found to be in the range 10^{-2} - 10^{-3}. The standard system is cost effective and simple in construction, compact and user friendly with high precision.

Key words: Vacuum chambers, Expansion Ratios, Series Expansions, Uncertainties.

INTRODUCTION

In the vacuum range 10^0 - 10^{-4} mbar, mercury manometers are not suitable for the gauge calibration. In this range, the widespread and accurate method to generate pressures for such range calibration is the 'series expansion method', which was first used by Knudsen [1]. Since then, the method is referred with different names, such as pressure expansion [2, 3], volume expansion [4] and static expansion method [5-12]. In this paper, we present the novel development of a 'four-stage volume expansion vacuum standard' having a small reference volume and four vacuum chambers, with many salient features.

Methods of Pressure Generation: In order to achieve an accurate vacuum in this range, two approaches are commonly employed namely, single-stage expansion and multiple-stage expansion. In a single-stage gas expansion (Fig. 1), a small reference volume V_r at temperature T_1, filled with a suitable calibration gas at a measured (relatively high) reference pressure P_r, is expanded into a pre-evacuated large volume V at temperature T_2. It generates a final pressure P, which is calculated from the known ratio R of the system's volume, temperature and reference pressure. According to Boyle's law, this pressure is given by [13]:

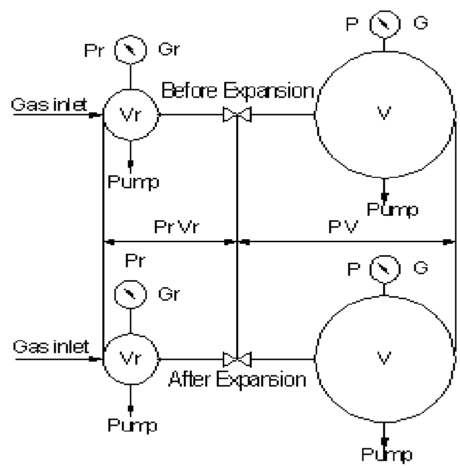

Fig. 1: Principle of Single-Stage Gas Expansion

$$P = P_r\left[\left(\frac{V_r}{V_r + V}\right)\right]\left(\frac{T_2}{T_1}\right) = P_r\left(\frac{T_2}{T_1}\right)R \quad (1)$$

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R is known as the expansion ratio $\{R=V_r / (V_r+V)\}$ of the system. The term (T_2/T_1) takes account of the temperature difference between the chambers considered. In single-stage expansion [14], for fixed volumes different values of P_r will generate appropriate pressures.

Multiple expansions are used for generating lower and larger range pressures. This is achieved with a series of large and small volumes, usually referred as stages [15, 16]. Various standard expansion systems consist of different stages [15-20].

In developing our simple and novel assembly, we have relied on multiple extension method. The developed 4-stage system has a reference volume V_r with temperature T_r and four other chambers of volumes V_1, V_2, V_3 and V_4 (Calibration chamber) with temperatures T_1, T_2, T_3 and T_4, respectively. It has been designed to have a greater flexibility to generate a continuous range of required pressures. For our system, under isothermal conditions, equation (1) takes the form [21]:

$$P = \left[\left(\frac{V_r}{V_r + V_1}\right)\left(\frac{V_1}{V_1 + V_2}\right)\left(\frac{V_2}{V_2 + V_3}\right)\left(\frac{V_3}{V_3 + V_4}\right)\right]P_r \qquad (2)$$

In this particular case the overall expansion ratio R is given by

$$R = \left[\left(\frac{V_r}{V_r + V_1}\right)\left(\frac{V_1}{V_1 + V_2}\right)\left(\frac{V_2}{V_2 + V_3}\right)\left(\frac{V_3}{V_3 + V_4}\right)\right] \qquad (3)$$

DESIGN PHILOSOPHY

The design of our standard system (Fig. 2) is based on ultrahigh vacuum (UHV) technique, as the expansions and calibrations are carried out in static conditions. In order to avoid degassing of the system, special consideration was given to the choice of material used and other components along with their surface finish. Consequently, reference volume, different chambers and other vacuum accessories are all made of Stainless Steel (SS), with properly finished internal surfaces. For further reduction of 'degassing', vacuum chambers are made of cylindrical shape to obtain inner wall surface area to volume ratio minimum. The precisely developed system consequently consists of two parts, as shown in Fig. 2a and 2b.

The first part (Fig. 2a) has a small reference volume that is the volume of connecting tubes of two valves and a reference gauge. This is further linked to a rotary vacuum pump (4 l/s) and a gas buffer by installing essential valves. This part is used to generate reference pressure.

While the second part consists of four chambers mutually connected with each other by fine quality electro-pneumatic valves. The design of these valves is so chosen that the volume change introduced by their operation is minimal. These chambers are installed with essential calibrated gauges for precise and ultimate pressure measurements. The calibration chamber (Fourth chamber) has three additional ports. One for venting purpose and other two to connect the gauge heads undergoing calibration. All the valves and gauges are

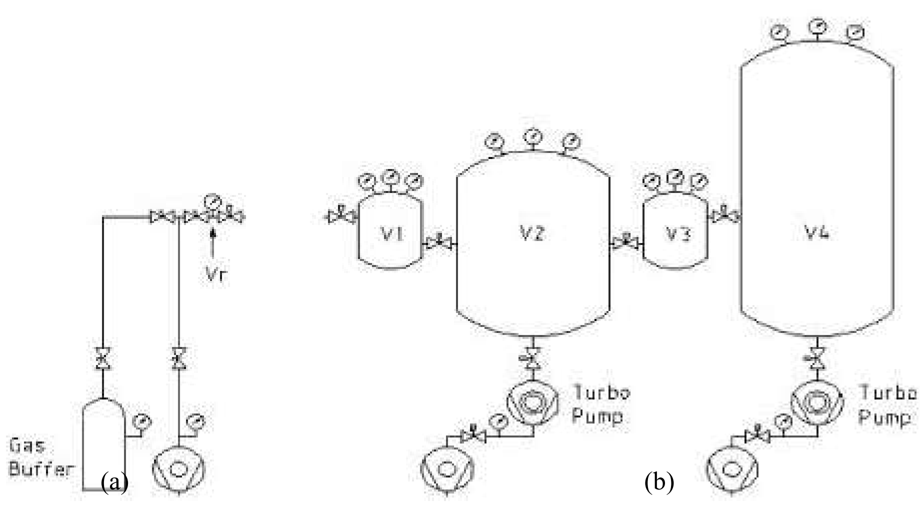

Fig. 2: (a) Reference Pressure Generation System, (b) The 4-stage Volume Expansion System

fixed using metallic seals. First and third stage chambers (comparatively small in size) are evacuated through the second stage medium chamber by a pumping unit consisting of a turbo-molecular pump (151 l/s) backed by a rotary pump (4 l/s). A separate pumping unit of the same rating, as shown in Fig. 2b, evacuates the fourth stage calibration chamber. To ensure minimal vibrations by pumping system, all the chambers as well as reference volume are fixed on a massive mild steel table. Different valves and pumps are electronically controlled through a control panel. Vacuum and temperature display units are also fixed in this panel.

THEORY OF DESIGN PARAMETERS

Expansion Ratio Determination: The most obvious technique for determining the expansion ratio of an expansion stage is to measure the volume of each chamber individually, details of which are available in literature [22]. This is done by using three methods:

***Dimensional Metrology*:** This practice is accurate for volume determination but not suitable for precise measurement of vacuum vessels and other tubing of irregular geometry.

***Gravimetric Method*:** This method is an established one, in which the volume to be determined is required to fill up with liquid (usually water) of exactly measured volume, in a constant temperature environment.

***Expansion Method*:** For this method, a known pressure in a calculated volume is expanded into an unknown volume, which has to be determined. Then by making use of Boyle s' law, the required volume is computed.

For the determination of volume ratios in our case, required volumes are calculated with second method i.e. by gravimetric method. For the accuracy confirmation, these volumes are also determined by the third method [13].

Gravimetric Method: For the determination of volumes of different chambers by this method, each chamber is fitted with concerning valves in actual position while gauge ports are kept blank. The said chambers and distilled water used for measurement are kept at a constant temperature for 24 hours before starting the volume determination. Each chamber is then carefully filled with precisely measured volume of water, using a fine graduated flask and a burette. Reference volume, volume of gauges and that of other tubes are measured similarly but separately. In our case the chamber volumes were calculated accurately and are given in Table 1.

Table 1: Volumes of various chambers measured by two different methods

	Volume (liter)		
Chamber volume	Gravimetric method	Expansion method	Percentage error
V_r	0.0198	0.0198	-
V_1	04.665	04.668	±0.06
V_2	32.599	32.587	±0.04
V_3	04.667	04.670	±0.06
V_4	59.078	59.061	±0.03

Expansion Method: After proper cleaning of all the four chambers, valves, reference volume and other vacuum accessories, the whole system is assembled as shown in Fig. 3. All the chambers along with fittings are thoroughly evacuated, made leak tight at the range 10^{-10} mar l/s and then properly baked. The system is re-evacuated properly, such that the base pressure is lower (1×10^{-7} mbar) than the lower calibration limit (1×10^{-4}mbar). The entire system is then isolated from the vacuum pumps. Reference pressure P_r (prior to expansion) is generated by filling the reference volume V_r with calibration gas (Ar) at a certain pressure. This pressure can conveniently be measured by the system reference gauge, which is 1300 mbar Capacitance Diaphragm Gauge (CDG), calibrated against standard mercury manometer already developed in the laboratory [23]. Finally, the gas of this known pressure contained in the reference volume V_r is allowed to expand in the first calibration chamber having volume V_1. In admitting or expanding gas, a certain time is given to allow the gas mass to reach temperature equilibrium with the container before the pressure is measured. Equilibrium conditions hold well when the readings of each gauge used in the pressure measurement remain constant. Following Elliott and Clapham [24], allow 1 min elapsed time between successive observations. After the said time, overall pressure of the combined volume (V_r+V_1) is noted and expansion ratio R of V_r and V_1 is determined. By this step, volume V_1 is determined, consequently by using this calculated value of V_1 as reference and repeating the same process under similar conditions, V_r is then determined by subject expansion method.

Similarly the remaining expansions are then performed in sequence, in a manner described above for calculating different expansion ratios and hence the volumes. The volumes of all the chambers thus

Fig. 3: Actual Photograph of Volume Expansion Vacuum Standard

determined are given in Table 1, along with that measured by water gravimetric method. Expansion ratios and hence volumes of the chambers have been calculated by two methods of entirely different techniques, to guarantee the reliability of the results. In each method, the practice is repeated five times and then average is taken. It was found that the volumes are almost consistent. After the accuracy confirmation of the system volumes, their overall expansion ratio R is found to be 3.39×10^{-5}.

Reference Pressure Measurement: Reference pressure is measured with a primary or secondary standard that meets the requirements regarding the pressure range and accuracy. The reference pressure P_r in a volume expansion system typically ranges from a few mbar to about 1 bar [25]. It may be measured by a reliable secondary standard. In present case, it is a 1300 mbar Capacitance Diaphragm Gauge, finely calibrated with Standard Mercury Manometer (as already mentioned) developed in the same laboratory [23].

EXPERIMENTAL PROCEDURE

For this purpose, the system (already baked and leak tested) is evacuated thoroughly to its base pressure (1×10^{-7} mbar). All the stages are isolated from each other as well as from the pumping units. Argon gas is filled in the reference volume (V_r) at some suitable pressure P_r. This pressure of reference volume V_r is expanded to volume V_1 (stage S_1). After about one minute, accumulated pressure of V_1 is expanded to volume V_2 (stage S_2), then similarly to V_3 and finally to V_4 (calibration chamber). After the stabilization time, pressure in the calibration chamber is noted with secondary standard (CDG: 1.0 to 10^{-4} mbar). The CDG is used as a secondary standard because of its correctness, consistency and reliability [26]. For every next expansion, the system is re-evacuated each time to its base pressure and above procedure is repeated for different values of reference pressures. To generate high pressure, planned stage reduction provision of the system is availed i.e. instead of V_r, V_1 is used as the reference volume with the same CDG reference gauge. Consequently, different reference and expansion pressures are generated and measured with CDGs.

For routine calibration, the calibration chamber is vented and the test gauge undergoing calibration is well installed with its test port. Precisely the gauge volume is added to that of the calibration chamber. The reference pressure generation and successive expansion procedure described earlier is repeated for critical values of reference pressures. Pressures so generated are computed and compared with the measured ones by the test gauge and thus corresponding correction factors are found.

RESULTS AND DISCUSSION

The generated pressures for different reference pressures are computed and compared with the measured values by CDG. Comparison of these three pressures and corresponding correction factors is presented in Table 2. The average correction factor and linear correlation coefficient is found to be 0.995 and 1.00, respectively.

The lower limit of calibration pressure, which can be generated in this static expansion system, is determined

Table 2: Generated and measured pressures for different reference pressures

S.No	Ref. Pressure (mbar)	Generated Pressure (mbar)	Measured Pressure (mbar)	Correction Factor (c.f)
Using V_r as reference volume				
1	5	1.69×10^{-4}	1.70×10^{-4}	1.006
2	15	5.09×10^{-4}	5.00×10^{-4}	0.982
3	25	8.48×10^{-4}	8.50×10^{-4}	1.002
4	30	1.02×10^{-3}	1.00×10^{-3}	0.980
5	150	5.09×10^{-3}	5.00×10^{-3}	0.982
6	200	6.78×10^{-3}	6.70×10^{-3}	0.988
7	500	1.69×10^{-2}	1.70×10^{-2}	1.006
8	1000	3.39×10^{-2}	3.35×10^{-2}	0.988
9	1279	4.34×10^{-2}	4.30×10^{-2}	0.991
Using V_1 as reference volume				
10	50	4.01×10^{-1}	4.05×10^{-1}	1.009
11	77	6.17×10^{-1}	6.20×10^{-1}	1.004
12	124	9.94×10^{-1}	1.00×10^{0}	1.006

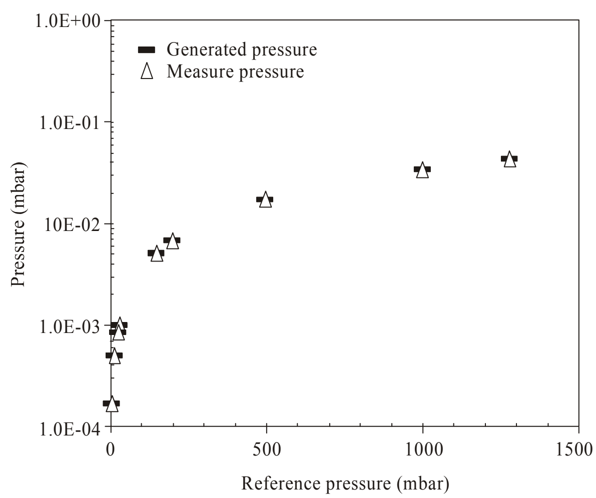

Fig. 4: Measured and generated pressures versus reference pressures

by the lowest residual pressures in the chambers and the out gassing rate of the inner walls of the system, which becomes significant below 10^{-6} mbar. In static expansion method for gauge calibration, the base pressure should be about two orders of magnitudes lower than the lowest limit of the calibration range. In our case the defined lower limit of calibration pressure of the subject device is 10^{-4} mbar, which therefore, satisfy the required condition.

Figure 4 shows results of the generated and measured pressures versus corresponding reference pressures. It is apparent that generated and measured pressures are in agreement with each other as well as with all the reference pressures in the entire calibration range. This ensures the accuracy of the subject standard system.

Pressure Transfer Gauges: Although a primary standard is defined [27] as the standard system that is designated or widely recognized as having the highest metrological quantities and whose value is accepted without any reference to other standards of the same quantity. However, in order to verify the accuracy and study the performance of the standard system, comparison is being made with another instrument of similar accuracy and sensitivity [28]. In the present case, a reference gauge i.e. calibrated CDG of measuring range 10^{0}–10^{-4} mbar, is used as a transfer gauge for calibration pressures measurements. Similarly, a 1300 mbar CDG is employed to measure reference pressure.

Temperature Measurements: Equation-2 is basically obtained assuming that the generation of pressure points develops under isothermal conditions [13]. Therefore, all possible efforts are made to keep the ambient temperature and that of the standard system constant. The four chambers of the calibration apparatus, however, showed some minor temperature differences. For accurate measurement of such minor changes, a temperature measuring sensor is attached with every chamber. For each value of the reference pressure and the generated one, temperatures have been measured accordingly [29]. Consequently the true generated pressure is obtained by multiplying the pressure given via equation-2 by correction factor (T_4/T_r), which for this 4-stage expansion system is given by:

$$\frac{T_4}{T_r} = \left(\frac{T_1}{T_r}\right)\left(\frac{T_2}{T_1}\right)\left(\frac{T_3}{T_2}\right)\left(\frac{T_4}{T_3}\right) \quad (4)$$

Table 3: Uncertainty calculations for the standard volume expansion system

Sr.No.	Generated pressure (mbar)	Uncertainties (mbar) Type-A (u_a)	Type-B (u_b)	Combined (u_c)	Relative (u_r)
1	1.69×10^{-4}	2.83×10^{-7}	9.17×10^{-6}	9.18×10^{-6}	5.41×10^{-2}
2	5.09×10^{-4}	6.22×10^{-6}	2.55×10^{-5}	2.63×10^{-5}	5.17×10^{-2}
3	8.48×10^{-4}	1.41×10^{-6}	4.26×10^{-5}	4.26×10^{-5}	5.03×10^{-2}
4	1.02×10^{-3}	1.27×10^{-5}	5.11×10^{-5}	5.27×10^{-5}	5.18×10^{-2}
5	5.09×10^{-3}	6.22×10^{-5}	2.55×10^{-4}	2.63×10^{-4}	5.17×10^{-2}
6	6.78×10^{-3}	5.94×10^{-5}	3.40×10^{-4}	3.46×10^{-4}	5.10×10^{-2}
7	1.69×10^{-2}	2.83×10^{-5}	8.52×10^{-4}	8.53×10^{-4}	5.03×10^{-2}
8	3.39×10^{-2}	2.97×10^{-4}	1.70×10^{-3}	1.73×10^{-3}	5.10×10^{-2}
9	4.34×10^{-2}	2.69×10^{-4}	2.17×10^{-3}	2.20×10^{-3}	5.06×10^{-2}
10	4.01×10^{-1}	2.90×10^{-3}	8.52×10^{-5}	2.91×10^{-3}	7.25×10^{-3}
11	6.17×10^{-1}	1.84×10^{-3}	1.31×10^{-4}	1.84×10^{-3}	2.98×10^{-3}
12	9.94×10^{-1}	4.10×10^{-3}	2.11×10^{-4}	4.11×10^{-3}	4.13×10^{-3}

As regards temperature effects caused by gas expansion, the following considerations were made. Firstly, walls of the expansion chambers are relatively thick, minor change in ambient temperature does not affect the generated pressures considerably. Secondly, when gas expands into vacuum, it does not deliver any net mechanical work. Temperature change still occurs by the intrinsic properties of a real gas (Joule-Thomson effect). But these changes are rather small, i.e. below 1ºC for an expansion from atmosphere to vacuum [30]. Thirdly, for large chambers, the extent of the temperature effects is small as disturbances in calibrations due to these effects are negligible [31]. However, by considering the small temperature deviations during the expansion ratio determination and the performance of pressure calibration, it has been observed that a relative uncertainty of the order 1×10^{-4} is arising from temperature effects.

Volumes Determinations: The involved volumes have been determined accurately by two independent methods of entirely different techniques i.e. gravimetric method (distilled water was used)] and gas expansion method. The two set of volume values obtained by these methods are equivalent within the limit. As the internal surfaces of the chambers are finely finished, outgassing starts below the range of 10^{-6} mbar vacuum. Relative uncertainty of volume ratio determination and due to outgassing is found to be 10^{-4} and 10^{-6}, respectively.

Uncertainties: In order to confirm accuracy, a due consideration needs to be given to the uncertainties. For this purpose, a method employed by various authors [32-34] has been used. There are different types of measurement uncertainties: Type-A uncertainties (u_a) are those evaluated by statistical analysis of series of observations while Type-B uncertainties (u_a) are calculated from sensitivity coefficients $u(q_i)$ and standard uncertainty $u(q_i)$, as given by the relation [32]:

$$u_b(P_i) = \sqrt{\sum C^2(q_i)u^2(q_i)} \quad (5)$$

Where $C(q_i) = \dfrac{\partial P_i}{\partial q_i}$ and q_i are parameters like reference pressure, volumes.

Combined uncertainty (U_C) may be estimated from the root-sum-squares of two components i.e. Type-A and Type-B uncertainties [33, 34].

$$u_c = \sqrt{(u_a)^2 + (u_b)^2} \quad (6)$$

The relative uncertainties (U_r) are determined from the generated pressures and corresponding uncertainties in the ratio [35].

$$u_r = \frac{u_c}{P_s} \quad (7)$$

Table 3 shows different types of uncertainties corresponding to standard (generated) pressures. The combined uncertainties are found in the range from 4.11×10^{-3} mbar to 9.18 x10^{-6} mbar. All the values of relative uncertainties range 10^{-2} - 10^{-3} which are consistent with those obtained by Jitschin [36].

The correction factor versus generated pressure is plotted in Fig. 5. The error bars represent relative uncertainties. Figure 6 shows the percent deviation of the

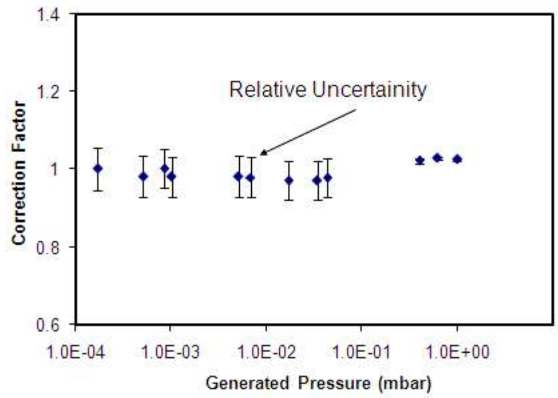

Fig. 5: Correction Factor versus Generated Pressure

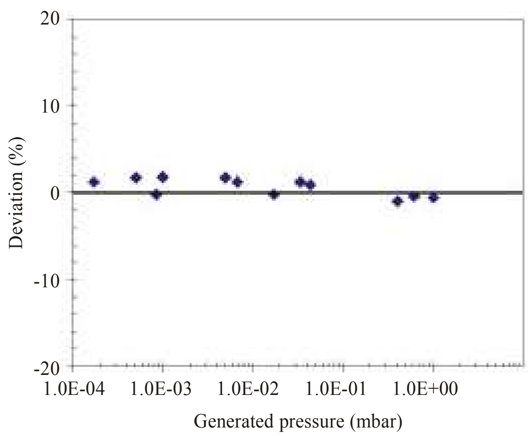

Fig. 6: Percent deviation from generated pressure

generated pressures from the measured ones. Although the deviation ranges from minimum to maximum value, however all points are close to the reference line and average deviation is 0.57%.

CONCLUSIONS

The volume expansion method has been employed in the subject primary vacuum standard because it is a fundamental and powerful method of generating calibration pressures in the defined vacuum range. In the simple and specific design of this standard system, due thought has been given to make use of the minimum number of vacuum pumps, gauges, electronic devices and other vacuum accessories without compromising on the accuracy of measurements. Each and every item along with the finally finished vacuum chambers is installed compactly on a massive metallic table. All this makes the standard system user friendly with easy operation, vibration less and specifically free from out gassing problems in the defined range.

The pressures generated by this standard system and corresponding CDG pressures are computed, compared in different ways and found to be in good agreement in the entire calibration range. The average Correction Factor is 0.995 while Linear Correlation Coefficient found is to be 1.00. The combined uncertainties lie between 4.11×10^{-3} mbar and 9.18×10^{-6} mbar. The relative uncertainties are of the order of 10^{-2}-10^{-3}. All this shows that the developed system is an improvement in the existing pool of knowledge of volume expansion systems, with essential accuracy and insignificant uncertainties. Furthermore, the subject standard system is a step further to the existing ones due to its many salient features: simple, compact, accurate, user friendly and cost effective.

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