The KRISS Primary Vacuum Gauge Calibration Standards: A Review

Wakil KHAN*1, S. S. HONG*2,a), T. SATAR*1, M. AHMED*1, Zulfiqar A. KHAN*1 and M. Zafarullah KHAN*1

*1National Institute of Vacuum Science and Technology (NINVAST), National Center for Physics, QAU, Islamabad, Pakistan
*2Korea Research Institute of Standards and Science (KRISS), Daejeon 305–340, Rep. of Korea

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The calibration of vacuum gauges is mainly carried out in two ways; i) using the primary standards in which the readings of a test gauge are compared with the pressures generated by the standard, and ii) by comparison method in which the readings of the gauge under test are compared with the output signal of a secondary standard. The former type of standards are, usually, large and complicated systems comprising of vacuum pumps, chambers, valves, and gauges etc. while the latter type are simply vacuum gauges, with superior qualities, which are attached to a properly designed calibration system. The Vacuum Measurement Lab at Korea Research Institute of Standards and Science (KRISS), Rep. of Korea, maintains primary standards as well as systems for calibration by comparison method. The KRISS primary standards can be used for the calibration purpose in the pressure range from $\sim 10^{-2}$ Pa to 133 kPa. For bilateral as well as key comparison of these standards, KRISS has participated in the past where its standards have good degree of equivalence and hence international recognition, with other national standards like that of NIST, PTB, NPL(UK), etc. Besides, the KRISS Vacuum Measurement Lab also has comparison system which can be used for calibration by comparison method. However, here the KRISS primary vacuum gauge calibration standards are discussed briefly with the aim to provide enough information to the readers in a single paper.

1. Introduction

During the past decades, the need for thoroughly practical, consistently accurate, wide-range vacuum-gauge calibration methods has increased in accordance with the changing role played by vacuum in research and technology[1]. The purpose of a calibration is to ensure correct readings on a gauge by making the indicated quantity directly or indirectly traceable to SI units. This is important not only for physical but also economical reasons[2]. Calibration of vacuum gauges is generally achieved by one of the following methods: (i) direct comparison with an absolute gauge, (ii) expansion method, and (iii) flow method or pumpdown method[3].

The calibration instruments used in these methods are called “primary standards”. There is another method in which the reading of the gauge under calibration is compared to another calibrated vacuum gauge while attaching both to a suitably designed vacuum system. This method is usually referred to as “comparison method” and the device is called “secondary standard”[2]. The Vacuum Measurement Lab at KRISS has both types of calibration facilities; primary standards as well as systems for comparison calibrations. It is recommended that the secondary standards are first calibrated on the relevant primary standard and the former are, then used for calibration of working standards. Before a review of the KRISS primary standards, an introduction to some prevailing techniques used in the primary standards (and relevant systems) in different vacuum ranges are discussed below.

2. Primary Vacuum Gauge Calibration Standards

An absolute pressure standard, usually referred to as a primary standard, is an instrument whose calibration is calculated from the knowledge of its significant dimensions and physical constants[4]. The values of such instruments are accepted without reference to other standards of the same quantity[1]. They are mainly categorized into manometers, static (volume, series) expansion systems, and dynamic expansion (orifice, pumpdown) systems.

2.1 Manometers

In the rough vacuum range (100 Pa–100 kPa), the internationally recommended primary standards are the “manometers” which measure an unknown pressure in terms of a fluid columns’ heights. Generally, a U-shaped glass tube, and hence the name U-tube, filled with the fluid is used for this purpose. U-tube manometer was first invented by Dutch physicist Christian Huygens in 1661, which was a modification of Torricelli’s barometer for determining gas pressure differences. Although the U-tube manometer is one of the earliest pressure measuring instruments, it is still widely used because of inherent accuracy and simplicity of operation[5].

Modern manometers operate on the same basic principle that pressure $p$ can be determined if one knows the density $\rho$, the acceleration of gravity $g$, and height $h$ of a liquid column[6] [refer to Fig. 1(a)] i.e.

$$ p = \rho gh $$

(1)

When pressure is applied to both ends of a U-tube manometer, the differential pressure on both sides of the manometer is given by

$$ p_2 - p_1 = \rho g(h_1 - h_2) $$

(2)

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E-mail: sshong@kriss.re.kr

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If the reference pressure $p_1$ is reduced to zero (i.e. less than the vapor pressure of the fluid used) by evacuating the space above the liquid in the reference column [Fig. 1(b)] an absolute pressure is determined. As a general practice, in case of two columns the temperature does not remain constant and so the density of the fluid in the two sides of the manometer is different. Under such condition, equation (2) can be written as$^7$:

$$p_2 - p_1 = g(r_1 h_1 - r_2 h_2)$$

(3)

where $p_1$ and $p_2$ are the temperature dependent densities of the fluid in the two columns.

Mercury is widely used as working fluid in the liquid manometers as its reference density is well known, its thermal expansion and vapor pressure are relatively small, and its density is high enough to allow measurements in the atmospheric pressure range with reasonable column heights.

As the most demanding accuracy requirements of pressure measurements in many industrial processes and R&D activities exist in the barometric pressure region$^9$, researchers continuously explore new techniques to reduce the uncertainties in the measured values in this range of vacuum; particularly the uncertainty in the measured value of $\Delta h$. For this, different techniques are used in top-level laboratories of the world including$^9$–$^{12}$; manual, laser, optical, and ultrasonic measurements. M. Bergoglio et al$^{13}$ has summarized comprehensively these techniques along with relevant lab as given below;

**White light interferometer**
- Bureau International des Poids et Mesures (BIPM)–Sevres-France
- Institut National de Metrologie (INM)–France
- Istituto di Metrologia “G. Colonnetti” (IMGC-CNR)–Italy
- National Research Laboratory of Metrology (NRLM)–Japan

**Laser interferometer**
- Istituto di Metrologia “G. Colonnetti” (IMGC-CNR)–Italy
- National Physical Laboratory (NPL)–United Kingdom
- National Institute of Metrology (NIM)–People’s Republic of China
- State Institute for Physicotechnical and Radiotechnical Measurements (NIIFTRI)–Russia
- Commonwealth Scientific Industrial Research Organization (CSIRO)–Australia

**Capacitance interferometer**
- Institut National de Metrologie (INM)–France
- Physikalisch-Technische Bundesanstalt (PTB)–Germany

**Ultrasonic interferometer**
- National Institute of Standards and Technology (NIST)–USA
- National Physical Laboratory (NPL)–India
- Korea Research Institute of Standards and Science (KRISS)–Korea

Schematics/ photographs of some of the corresponding standard systems are shown in Fig. 2 with their employed techniques. It is worth mentioning here that not only mercury but oil$^{14}$ and water are also being used as fluids in manometers. The effect on overall error/ uncertainty of the manometer is different for different fluids$^9$.

It is worth mentioning here that the measuring techniques for fluid columns’ heights are broadly grouped into two types; (a) the contacting and (b) the non-contacting techniques. As obvious from the names, surface of the fluid is touched in the former type while in the latter the heights of columns are measured without touching the fluid surface. In contacting technique, micrometers while in non-contacting technique, the laser, the white light and ultrasonic techniques are used. As has mentioned above in$^1$), the KRISS manometer employs the ultrasonic interferometric technique, keeping in view which, this technique will be briefly explained here.

An ultrasonic interferometer manometer (UIM) has mainly three mercury columns; two smaller side columns and one central column of approximately 1 meter length. The central column is at midpoint of the line joining the centers of the outer columns$^9$. The first interferometer using the ultrasound as source for detecting the fluid columns’ heights was developed, in 1977, at NIST by P. L. Heydemann et al$^7$. This is an extremely accurate
instrument in which a short (20 μs) pulse with a carrier frequency of 10 MHz is sent to the transducer mounted under the bottom closure of the fluid (liquid, usually mercury) column. The transducer transforms the electrical signal into the mechanical analog, and a short ultrasonic wave train is launched vertically upwards into the mercury. This signal is reflected at the surface of the liquid column and travels back to the transducer. Here a very small part of the ultrasonic energy is reconverted into an electrical signal. The rest is reflected back into the column.

In a mercury column of a few centimeters length, 20 or more echoes can usually be observed, whereas in oil the number is reduced by at least a factor of four. The echoes received by the transducer are amplified and then phase sensitive detected in two detectors with a 90° phase shift between their reference signals. Each of the pulses represents one echo received from the liquid column. The transducer is pulsed every 10 ms. One must therefore sample the instantaneous value of a chosen echo once every 10 ms and then hold it until it is pulsed 10 ms later. The sampled and held signals are then smoothed with a low-pass filter. These signals are connected to an up-down counter, which will count each zero crossing. Changes in the length of the mercury column cause corresponding changes in ultrasonic transit time and phase of the received signal. Careful measurement of this phase allows measurements of the changes in the length of column with typical standard deviation of 1–2 x 10⁻⁵ mm. The working principle of mercury UIM is shown in Fig. 3 above. For high accuracy of the generated pressure, the various factors as well as errors are well explained for both manual and ultrasonic manometers in [7–9].

Some general conditions to be met by equation are given below.

- As in manometers, liquids used are generally mercury (most common), water and diffusion pump oils. Also, the density of mercury is dependent on temperature, correction must be made if the system is operated at temperature higher than 20°C as this is the standard temperature for calculating the density of mercury. The value adopted for mercury density at 20°C and 100,000 Pa is ρ = 13.545.8507 kg m⁻³ [8,11].
- The local acceleration due to gravity g must be accurately known at the location of measurement. The relative uncertainty in g contributes the same relative uncertainty to the differential pressure.
- In order to reduce the adhesive/ cohesive forces effect, the manometer tubes must have sufficiently large diameters, usually, 6 to 7.5 cm.
- Thermal as well as mechanical instabilities also contribute errors to the generated pressures and, therefore, must be minimized as much as possible.

Advantages of the ultrasonic interferometer manometers include; attainment of a length resolution of about 10⁻⁸ m, a complete measurement in 2s only, no mechanical/ thermal disturbance of the manometer/components. However, the greatest disadvantage of this technique is the mechanical instability (due to vibration, etc.) during determinations/measurement of generated (vacu-
um) pressures. Keeping in view their numerous advantages, UIMs are preferred all over the world for barometric pressures measurements irrespective of their technique for the measurement of mercury columns' heights.

### 2.1.1 The KRISS Ultrasonic Interferometer Manometer

The KRISS UIM shown in Figs. 4 & 5\(^1\)\(^2\)\(^3\) was assembled and evaluated as an international cooperation project between the National Institute of Standards and Technology (NIST) in the United States and KRISS in the beginning of 1988. The manometer has an operating range between 1 Pa and 133 kPa. Its design and operation are based on those of mercury ultrasonic manometers\(^4\)\(^5\)\(^6\). In order to reduce the error, three glass columns are used. The inner diameter is selected 68 mm so as to minimize the surface tension effects of Hg.

The principle of the KRISS UIM is somewhat different to the traditional mercury manometer. The difference in height due to the difference in pressure between the mercury columns is measured using ultrasonic interferometry. A short pulse with a carrier frequency of 10 MHz is sent to the transducer. The signal is reflected at the surface of the liquid column and travels back to the transducer. This process continues until all the ultrasonic energy is lost from the signal by absorption and diffraction. The echoes received by the transducer are amplified, and then the phase sensitivity is detected using two detectors with a 90° phase shift between their respective reference signals. The amplitude of the phase sensitive detected signals is proportional to the amplitude of the received signal and to the phase shift between the measured and reference signals. When the liquid-column length changes, the phase of each received echo also changes. The output signals from the ultrasonic interferometer are related to the column length \(L\) as:

\[
u_1 = A \cos qL \quad \text{(4)}
\]

\[
u_2 = A \sin qL \quad \text{(5)}
\]

where \(A\) is the amplitude factor, \(q = 2f/c\), \(f\) is the carrier frequency.
frequency, and \( c \) is the speed of sound in mercury. The standard pressure was calculated in the KRISS UIM by using the relation;

\[
p = \frac{p_r}{1 + \beta_1 \Delta T + \beta_2 \Delta T^2} \cdot g \cdot \left( L_2 - \frac{L_1 + L_3}{2} \right) - \rho_{N_2} \cdot g \cdot \left( R_l - \frac{L_1 + L_3}{2} \right) + p_{\text{back}}
\]

where the \( p_r \) is the density of mercury at 20°C (13.5458507 × 10^3 kg m^-3), \( \beta_1 \) and \( \beta_2 \) are volume expansion coefficients of mercury, \( \Delta T \) is the temperature difference between the reference (20°C) and operating temperature and, \( L_1 \) and \( L_3 \) are the heights of the test columns of mercury, \( L_2 \) is the height of the reference mercury column, \( \rho_{N_2} \) is the density of nitrogen gas in the test column, \( \rho_{N_2} \) is the density of nitrogen gas in the reference column, \( p_{\text{back}} \) is the base pressure and \( R_l \) is the distance between the transducer and the position of test gauge.\\

The temperatures of the Hg columns were measured using six high quality standard platinum resistance thermometer (SPRT) temperature sensors while the density of Hg was calculated using the traditional equations which incorporate for temperature corrections. Besides, the heights of Hg columns were measured using the technique of time of flight (TOF) in which the distance travelled across the mercury column is accurately measured by comparing the outputs of phase sensitive detectors.

The uncertainty contributions to the generated pressure are summarized in the last articles. It should be noted that the pressure of the reference column is not exactly zero due to the vapor pressure of the mercury. Therefore, the value of \( p_{\text{back}} \) must be measured and a correction must be made to calculate the test pressure. The expanded uncertainty in this range was estimated as 0.04 Pa to 4 Pa for the capacitance diaphragm gauge (CDG) calibration.

\subsection{Comparisons with other Standards}

\subsubsection{CCM.P–K4 (Consultative Committee on Mass and related quantities Key comparison in Pressure in the range from 10 Pa–1000 Pa)}

Initially, a key comparison of the KRISS UIM was performed successfully between the vacuum laboratories at NIST, the Istituto di Metrologia Colonnetti (IMGC, Italy), the Commonwealth Scientific and Industrial Research Organization (CSIRO, Australia), PTB in Germany, the National Physical Laboratory (NPL, India), and the National Physical Laboratory (NPL, England). The KRISS UIM range for this comparison was from 10 Pa to 1000 Pa. The results of the key comparison CCM.P–K4 are given in Fig. 6. In almost all ranges of comparison, the KRISS values are close to the mean value/zero line.

![Fig. 6 CCM.P-K4 Key comparison results where the uncertainty bar of the KRISS UIM is indicated by arrows and are close to the zero line.](image-url)
2.1.2.2 Bilateral Comparison with PTB CCM.P–K4 (below 10 Pa)

Since, the CCM.P–K4 was carried out from 10 Pa to 1000 Pa, it was necessary to compare this system with another system in the range below 10 Pa. For this, a bilateral comparison was performed with the PTB static expansion system that had a high degree of equivalence with the key comparison reference values\(^{24}\). The primary standard of PTB was a static expansion system (SES) in which pressures were generated by expanding gas of known pressure from two alternative small volumes of nominally 0.1 L and 1 L into a vessel of nominal volume of 100 L.

Comparison procedure

Two CDGs of 133 Pa and 1333 Pa full-scale (CDG1 and CDG2, respectively) were chosen as transfer gauges for this comparison. The pressure ratios, \( r \), of the CDGs were determined in each laboratory. The gauges were calibrated in the range from 3 Pa to 100 Pa. Each target pressure was established three times during a single calibration and three separate calibrations on different days were carried out at each laboratory where \( r \) was calculated from:

\[
r = \frac{p_{\text{ind}}}{p_{ij}}
\]

where \( p_{\text{ind}} \) is the CDG reading, and \( p_{ij} \) is the pressure generated by the standard \( j \). Percentage of the relative difference for CDG1 and CDG2 was calculated using the following relation:

\[
d_i = \frac{r_{\text{PTB}} - r_{\text{KRISS}}}{r_{\text{KRISS}}} \times 100
\]

where \( r_{\text{KRISS}} \) is the mean of \( r_{\text{KRISS}ij} \) and \( r_{\text{KRISS}ij} \) for the two transfer gauges \( i = 1, 2 \). Since the temperature differences at the different calibrations were not larger than 0.2 K, the differences due to the thermal transpiration effect were smaller than 0.04% and were therefore neglected. To estimate the transport instabilities, half of the relative difference of \( r_{\text{KRISS}ij} - r_{\text{KRISS}ij} \) near full-scale were considered as the standard uncertainty. In order to generate a common value for the two CDGs, a mean was calculated by using:

\[
d = \frac{d_1 + d_2}{2}
\]

The relative uncertainty over the range of the comparison was estimated to be between \( 1.6 \times 10^{-3} \) and \( 2.0 \times 10^{-3} \) with a coverage factor \( k = 2 \). Fig. 7 illustrates the results for \( d(p) \) with \( 2 \times u_d(p) \) as uncertainty bars. The degree of equivalence, \( E_r(p) \), was calculated from:

\[
E_r = \frac{d(p)}{2 \times u_d(p)}
\]

The results from Fig. 7 indicate that all the \( E_r(p) \) values between KRISS and PTB, -0.029 and 0.305, at each target pressure are in good agreement. Since the uncertainty bars overlap with the zero line, an equivalence of the two primary standards (the KRISS UIM & the PTB SES) can be assumed. This bilateral comparison validated the performance of the KRISS UIM in the range below 10 Pa that was the requirement of KRISS.

2.2 Static Expansion Systems

In the medium pressure range 0.1 Pa to 1000 Pa, mercury manometers are not suitable and the characteristics of continuous expansion systems become poorly defined, as they depend on pressure, so primary standards are based on static expansion of a gas\(^{25}\). That is why, the static expansion systems (also called volume or series expansions systems) are used as primary standards for the calibration of vacuum gauges\(^{26,27}\).

The method originated from Knudsen (1916) and the systems are in use at several standard laboratories for the calibration of vacuum gauges\(^{28}\). Static expansion systems have been used routinely for more than 35 years as a primary definition of low pressure\(^{29}\). This is the most suitable method above 10\(^{-1}\) Pa\(^{30}\), however, newly developed systems (UME of Turkey) have extended this limit from \( 9 \times 10^{-4} \) Pa to 10\(^3\) Pa\(^{26}\). In such systems, known pressures are generated by expanding a known gas amount enclosed in a small volume \( V_1 \) into a much larger evacuated volume \( V_2 \) by opening a valve in between the two volumes.

Considering the gas to be in ideal conditions (obeying the Boyle’s law and with isothermal conditions), the gas pressure is reduced by the ratio of the small volume to the sum of the small and large volume\(^{31}\):

\[
f = V_1/(V_1 + V_2)
\]

and the pressure generated is:

\[
p_2 = p_1 \times \frac{V_1}{V_1 + V_2}
\]

where \( p_1 \) and \( p_2 \) are the initial and final pressure of the vessels. The ratio \( f = V_1/(V_1 + V_2) \) is called “expansion ratio” and its inverse is the “volume ratio” \( f^{-1} = (V_1 + V_2)/V_1 \). If the initial pressure \( p_i \) and volumes are known, the generated pressure \( p_2 \) is directly calculable from the initial pressure and the expansion ratio\(^{31}\).

For highest accuracy, the initial pressure, which ranges typically from 1 kPa to 300 kPa, is measured with
a piston gauge or a suitable secondary standard. The pressure \( p_1 \) in the initial chamber \( V_1 \) (figure 8) referred to as “transfer sampler” is then expanded to the final chamber also known as the “calibration chamber”. The final pressure or calibration pressure is reduced in the calibration chamber according to the volume ratio of the chambers stated above.

In case of non-isothermal expansion, the chambers’ ratio of temperatures is taken into consideration as given below;

\[
p_2 = p_1 \frac{V_1}{V_1 + V_2} \left( \frac{T_2}{T_1} \right)
\]

where \( T_1 \) and \( T_2 \) are the temperatures of the small and large volumes, respectively.

In Fig. 8, the operation of a single-stage static expansion system is shown. However, in the medium vacuum as well as high vacuum range (UME Turkey), various low calibration pressure points are desired and, hence, they can be generated by combining a number of transfer samplers of two different sizes. Such systems are called multi-stage expansion systems and have been developed at various metrological labs including the PTB of Germany, UME of Turkey, and METAS of Switzerland (shown in Fig. 9), etc.

There are several techniques for the accurate determination of expansion ratios for such systems. The main ones are the gravimetric technique and the expansion technique. Usually the gas expansion method, already briefly discussed, is used for the calculation of volume ratios of the involved chambers.

In multi-stage expansion systems where various pressure levels are required, under ideal and isothermal conditions, the final pressure after \( n \) expansions is;

\[
p_2 = p_1 \left[ 1 - (1 - f)^n \right]
\]

where \( p_1 \) is the initial pressure and \( f \) is the expansion ratio. The number of expansions varies among laboratories for example; the NPL had performed 30 expansions, the PTB and CEM each 25, and so on.

However, if the calibration gas deviates from the ideal gas conditions, i.e. not obeying the Boyle’s law as well as in non-isothermal environment, then the final pressure in the calibration chamber can be calculated by using;

\[
p_2 = p_1 \left( \frac{V_1}{V_1 + V_2} \right) \left[ 1 - \left( \frac{B}{R_m T} \right) p_1 \right] \frac{T_2}{T_1}
\]

where the factor \( T_2/T_1 \) represents the correction for the temperature difference between the two volumes and the term \( 1 - (B/R_m T) p_1 \) is the correction for real gases with the molar gas constant \( R_m \).

The most challenging but essential aspect when employing the static expansion method is an accurate determination of the volume ratio. As stated earlier, various techniques are used to measure the absolute volume of the individual vessels; however, earlier workers used gravimetric techniques in this regard which are still useful but have the serious disadvantages that the system must be disassembled every time re-measurement is required. Similarly, to generate pressures as low as
10^{-6}$ Pa, an overall volume ratio of at least $10^7$, and preferably $10^{10}$, is desirable. To achieve such large ratios, alternative techniques are possible which include the single-stage expansion system, the multi-stage expansion system, etc.

The factors affecting the accuracy of static expansion systems include:

- Invalidation of the Boyle’s law (it remains valid when (a) the molar quantity of gas transferred through the system is maintained constant, (b) the process of pressure generation develops under isothermal conditions, and (c) the gas used for pressure generation obeys the ideal gas laws) and
- The systematic errors in the measurement of the different quantities involved in the evaluation of the pressure generated in the calibration chamber.

### 2.2.1 The KRISS Static Expansion System

The KRISS static expansion system (SES), comprising of two stages, was assembled in 2005 as shown in Fig. 10. It consists of a gas reservoir $D$ (having volume $V_d$), three vacuum chambers $A$, $B$, & $C$ of different volumes, vacuum pumps and associated vacuum gauges and thermometers. The volumes of $A$, $B$ and $C$ predicted through dimensional calculation were 73 ml, 8.4 liter and 67 liter, respectively. The initial pressure of $A$ is measured with a quartz Bourdon gauge (QBG) while an ionization gauge is used to measure the ultimate pressure of $C$.

The volume ratio of the KRISS SES was calculated using the same method as used in the NMJES procedure in Japan i.e. cumulative gas expansion method described in detail in the NPL Report. Capacitance diaphragm gauges (CDGs) of 13.3 kPa and 133 Pa full-scale ranges were used as pressure measuring sensors for this purpose. Two volume ratios $I/X_1=(A+B+C)/A$ and $I/X_2=(A+B)/A$ are involved in the KRISS SES. A summary of their calculating procedure is that; for the volume ratio $X_j$, after recording the initial pressure of $B+C$, $p_{Y_j1}$, gas is introduced into $A$ and the pressure of $A+B+C$, $p_{Y_j2}$, is noted. Then $A$ is isolated from $B+C$ and the pressure, $p_{Y_j2}$, is recorded. After evacuation of $(A)$ gas is again introduced and the process is repeated $n$ times and the pressure of $B+C$, $p_{Y_j2}$, is noted. Here, $Y_j$ is $\{\text{Final pressure of } B+C \text{ after } n \text{ expansions / Initial pressure of } B+C\}$ and $Y_j$ is $\{\text{Pressure of } (A+B+C) / \text{Pressure of } (B+C)\}$.

The volume ratios for the chambers, $I/X_1=(A+B+C)/A$ and $I/X_2=(A+B)/A$, are measured to be 1061.61 and 116.79 with standard deviation of 0.9777 and 0.0359 and their uncertainties not more than 0.1%.

The pressure in calibration chamber of the system can be calculated by using the following relation:

$$p_r = p_i \left( \frac{p_{Y_2}}{p_{Y_1}} \right)^{N} \left( 1 - \frac{p_{Y_2}}{p_{Y_1}} \right)^{1/n} \left( \frac{p_{Y_2}}{p_{Y_1}} \right)^{1/X_c} \left( \frac{T_A}{T_C} \right) \tag{15}$$

where $p_i$ (Pa) is pressure generated by SES; $p_i$ (Pa) is initial pressure of $A$; $p_{Y_2}(Pa)$ is initial pressure of $A$ to calculate $X_2$; $p_{Y_2}(Pa)$ is final pressure of $A+B$ to calculate $X_2$; $N$ is number of exhausions of $B$ to generate pressure in $C$; $p_{Y_2}(Pa)$ is initial pressure of $B+C$ to calculate $X_1$ and $Y_1$; $p_{Y_2}(Pa)$ is final pressure of $A+B+C$ measured after exhausting $A$ times to $X_1$; $Y_1$; $n$ is number of exhausions of $A$ to calculate $Y_1$; $p_{Y_2}(Pa)$ is initial pressure of $A+B+C$ with valve (a) and (b) opened to calculate $Y_2$ of $X_2$; $p_{Y_2}(Pa)$ is final pressure of $A+B+C$ with valve (a) closed and (b) opened to calculate $Y_2$ of $X_2$; $T_A$ (K) is temperature of $A$ and $T_C$ (K) is temperature of $C$.

In the standards' laboratory at KRISS, very high quality temperature controller is installed. In order to obtain the proper (stable) temperature, the controller must operate for more than a week continuously. Besides, for the KRISS SES, for deviation from the ideal gas behavior, the values of $p_{Y_2}(Pa)$ and $p_{Y_2}(Pa)$ were lower than 0.05%.

In the overlapping range of pressure from 3 Pa to 100 Pa, two CDGs (CDG1 and CDG2) were first calibrated on the KRISS UIM and then on SES, respectively, and the result was compared.

The $E_{m,pj}$ (Fig. 11) was found to vary between $-0.02151$ and $0.00331$, showing that the two standards
were consistent with each other within the expanded uncertainty limit. Particularly at pressures below 6 Pa, the uncertainty was much smaller in the SES than in the UIM. On the basis of these results, the SES contributions are great not only as a national medium vacuum standard but also to the improvement of industrial calibration services.

### 2.3 Dynamic Expansion Systems

In the high and ultrahigh vacuum ranges, the dynamic expansion method, also known as the “orifice flow method”\(^{35}\) and the “continuous expansion method”, is widely used for the calibration of vacuum gauges. Since at low pressures, about \(10^{-6}\) Pa, the generated pressure is affected by the outgassing of vacuum vessels and the adsorption of gas molecules on the inner surfaces of the vessels, the most suitable method is the dynamic expansion method\(^ {37}\). Steady, known pressures can be generated with the help of dynamic expander systems which makes them an important calibration technique in vacuum metrology\(^ {38}\) and more effort has gone into the development of this procedure than any other\(^ {39}\).

In dynamic expanders, a known flow of gas is expanded continuously from a volume at high pressure into the vacuum pump via two conductances. If there is no sinks or sources of gas between the two conductances, the equation of continuity is valid and the net flow through the two orifices must be equal under isothermal conditions\(^ {40,2}\). The two conductances are respectively in the form of a flowmeter and an orifice. The flowmeter generates precise gas flows while orifice is made, usually, in a SS plate which divides the system’s vacuum chamber into two parts; the upper part as calibration chamber while the lower one as pumping chamber. A typical system for generation of dynamic flow is shown in Fig. 12 above.

Under steady state condition, the well-known equation relating the pressure \(p\) to conductance \(C\), gas flow \(Q\) and pumping speed \(S_p\) of the pump in the molecular range and down to at least \(10^{-6}\) Pa during continuous gas flow is given by\(^ {12}\);

\[
\Delta p = \frac{Q}{C(1-C/S_p) F_T F_i}
\]

(16)

where \(F_T\) is the correction factor for temperature of the various parts of the system, \(F_i\) the correction factor for real gas behavior, gauge pumping or degassing, gas impurity, etc.

Like manometers and static expansion systems, pressure generation in a dynamic expansion system imposes well known conditions, the most significant of which are\(^ {41,2,42,43}\):

- High pumping speed compared to conductance of the orifice (100:1)
- Constant value of conductance in the operating pressure range
- Low ultimate pressure in the calibration chamber (by a factor of \(10^{-2}\) than the lowest calibration value of the test gauge)
- Constant reference pressure with time, &
- The temperature of all parts of the system must remain constant.
- The lowest calibration pressure generated with dynamic expanders which is determined by the lower limit of acceptable performance of the flowmeter and the base pressure of the vacuum chamber

#### 2.3.1 The KRISS Dynamic Expansion System

The KRISS dynamic expansion system (DES) which is a calibration system for high and ultrahigh vacuum is

| Pressure (Pa) | \(u_{CDG1}\) | \(u_{CDG2}\) | \(d\) | \(u_d\) | \(E_n(p)\) |
|--------------|-------------|-------------|-----|-------|----------|
| 3            | 0.178       | 0.137       | -0.00501 | 0.116 | -0.022   |
| 6            | 0.116       | 0.086       | 0.00366 | 0.075 | 0.024    |
| 10           | 0.025       | 0.023       | 0.00374 | 0.031 | 0.061    |
| 30           | 0.011       | 0.005       | 0.00240 | 0.068 | 0.018    |
| 100          | 0.002       | 0.001       | 0.00152 | 0.230 | 0.003    |

Fig. 12 The basic operational principle of a dynamic expansion system\(^ {2}\).
basically a two-stage flow-divider system, as shown in Fig. 13. It can be used for the calibration of high as well as ultrahigh vacuum gauges in the range from $10^{-5}$ Pa to $10^{-2}$ Pa and $10^{-7}$ Pa to $10^{-5}$ Pa, respectively. These gauges include SRGs, IGs (hot cathode), and extractor gauges. The system consists of two similar orifice-flow calibration systems, one for HV and the other for UHV connected by two paths one of which contains a restriction/porous plug having comparatively small conductance ($6.36 \times 10^{-3}$ L $s^{-1}$ for N$_2$ at 23°C). The HV system is evacuated by a TMP with a nominal pumping speed of 345 L $s^{-1}$ for N$_2$ and the UHV system by a helium-refrigerator-type cryopump bakeable up to 150°C with 1500 L $s^{-1}$ N$_2$ nominal pumping speed. It is worth to mention here that the ultimate pressures of the calibration chambers were $10^{-7}$ Pa and $10^{-9}$ Pa for the high and ultra high vacuum systems, respectively. Although, the effect of pumping speed of the main TMP has not been calculated, but their overall pumping speeds are stable at the high and UHV sides.

The pressure in the flowmeter, $p_f$ is about 1 kPa but it is changeable according to the standard pressure to be generated in the calibration chambers of the system. The flowmeter is the variable volume type in which the moving length is measured by a digital micrometer with elapsed time by counter of HP Company. Besides, the conductance of the porous plug installed in between the HV and UHV systems was measured by using “small porous plug conductance” measuring system which is maintained at the KRISS standards’ lab. It is worth mentioning here that outgassing rate of both the calibration chamber and the flowmeter were not so large to affect to generated standard pressures and pressure of flowmeter seriously.

All chambers and plates are made from stainless steel 316, and demountable oxygen-free high-conductivity (OFHC) seals are used for the conflate flanges (CF). The respective diameters of the orifices were measured using optical microscope at length lab. such that for nitrogen gas, the calculated values of conductance of the orifices were 12.30 L/s for high vacuum side and 15.82 L/s for UHV at 23°C. The conductances of the orifices $O_1$, $O_2$, and $O_p$ are respectively $C_1$, $C_2$, and $C_p$. The calibration pressures in the upper chambers of the HV, $p_1$, and UHV, $p_3$, are then given by;

$$p_1 = \frac{Q}{[C_1(1-R_{ph})]}$$
$$p_3 = \frac{(C_p/C_2)[1/(1-R_{pu})]}{p_1}$$

where $R_{ph} = p_2/p_1$ and $R_{pu} = p_4/p_3$ are the pressure ratios of the lower and upper chambers of the HV and the UHV systems, respectively. The pressure ratio, $R_p$, can be obtained from $R_p = Q_f/Q_0$. For argon gas, $R_{pu}$ was measured to be 0.03889, and $R_{ph}$ of the HV system to be 0.08131. Also, the stability of the pressure in the flowmeter was approximately less than $10^{-4}$ of generated pressure in the flowmeter.

The expanded uncertainty over the pressure range of $4.57 \times 10^{-3} - 1.26 \times 10^{-2}$ Pa for the high vacuum DES was between $4.18 \times 10^{-3}$ and $4.13 \times 10^{-3}$, and over the pressure range of $3.01 \times 10^{-6} - 9.02 \times 10^{-4}$ Pa for the ultrahigh vacuum standard DES the uncertainty was between $1.37 \times 10^{-2}$ and $9.10 \times 10^{-3}$.

### 2.3.1.1 Comparison with other Standards

Bilateral comparisons of the KRISS dynamic expansion system are given in Fig. 14. In Fig. 14 (a) the KRISS and the ETL (NMIJ) while in Fig. 14 (b) the KRISS and the NPL (UK) standards’ comparison are shown. In both comparison processes, two spinning rotor gauges were used as transfer standards.

### 3. Uncertainties Data Sheet

Type B uncertainties (also known as systematic uncertainties are usually fixed and depend on the design of the system as well as history of measuring instruments used)
Table 2  For UIM at 1.3125 kPa, the combined standard uncertainty was determined from the quadratic addition of the individual sources.

| Source of uncertainty | Value $x_i$ | Standard uncertainty $u(x_i)$ | Sensitivity coefficient $c_i$ | Uncertainty contribution $|c(q_i)| \cdot u(q_i)$ |
|------------------------|-------------|-------------------------------|-----------------------------|---------------------------------|
| $\rho_1$               | 13548.1969 kg/m$^3$ | $6.8 \times 10^{-2}$ kg/m$^3$ | $9.686769 \times 10^{-2}$ Pa/(kg/m$^3$) | $6.587 \times 10^{-2}$ |
| $\beta_1$              | $1.8115 \times 10^{-4}$/K | $3 \times 10^{-5}$/K | $-1.232325 \times 10^4$ PK | $-3.6998 \times 10^{-5}$ |
| $\beta_2$              | $7.7552 \times 10^{-5}$/K | $1 \times 10^{-5}$/K | $1.157154 \times 10^3$ PK | $1.15715 \times 10^{-9}$ |
| $\Delta T$             | 19.061°C | $5 \times 10^{-3}$°C | $2.377215 \times 10^{-1}$ P/°C | $1.18861 \times 10^{-3}$ |
| $g$                    | 9.79831 m/s$^2$ | $2 \times 10^{-4}$ m/s$^2$ | $1.33910 \times 10^2$ Pa/(m/s$^2$) | $2.67820 \times 10^{-4}$ |
| $L_1$                  | $-2.32789 \times 10^{-4}$ m | $1 \times 10^{-3}$ m | $-6.637464 \times 10^3$ Pa/m | $-6.63746 \times 10^{-3}$ |
| $L_2$                  | $9.65063 \times 10^{-1}$ m | $1 \times 10^{-1}$ m | $1.32749 \times 10^3$ Pa/m | $1.32749 \times 10^{-2}$ |
| $p_{N_1}$              | $1.51352 \times 10^{-2}$ kg/m$^3$ | $1 \times 10^{-2}$ | $-4.33314$ Pa/(kg/m$^3$) | $-6.55830 \times 10^{-4}$ |
| $p_{N_2}$              | $4.65815 \times 10^{-6}$ kg/m$^3$ | $1 \times 10^{-2}$ | $4.23629$ Pa/(kg/m$^3$) | $1.97333 \times 10^{-7}$ |
| $R_1$                  | $4.46 \times 10^{-2}$ mm | $5 \times 10^{-1}$ mm | $-1.48254 \times 10^{-1}$ Pa/mm | $-7.4126 \times 10^{-5}$ |
| $p_{back}$             | $4.0397 \times 10^{-1}$ Pa | $1 \times 10^{-3}$ | 1 | $4.03966 \times 10^{-4}$ |

Combined standard uncertainty $u_c$ (Pa) $1.4908 \times 10^{-2}$

Table 3  Combined standard uncertainty contribution for the SES at 10 Pa.

| Source of uncertainty | Value $x_i$ | Standard uncertainty $u(x_i)$ | Sensitivity coefficient $c_i$ | Uncertainty contribution $|c(q_i)| \cdot u(q_i)$ |
|------------------------|-------------|-------------------------------|-----------------------------|---------------------------------|
| $p_1$                  | 10.6192 kPa | 6.9223 Pa | $8.0501 \times 10^{-6}$ | $5.5725 \times 10^{-5}$ |
| $p_{X11}$              | 66.7973 kPa | 0.0288 Pa | $1.4955 \times 10^{-4}$ | $4.2996 \times 10^{-5}$ |
| $p_{X22}$              | 0.5716 kPa | 47.3814 Pa | $-1.2797 \times 10^{-6}$ | $-6.0638 \times 10^{-5}$ |
| $p_{Y11}$              | 113.5958 Pa | 0.1568 Pa | $-8.0625 \times 10^{-2}$ | $-1.2638 \times 10^{-2}$ |
| $p_{Y12}$              | 112.5310 Pa | 0.1568 Pa | $7.9870 \times 10^{-2}$ | $1.2520 \times 10^{-2}$ |
| $p_{Y21}$              | 113.4886 Pa | 0.1568 Pa | $7.5325 \times 10^{-4}$ | $1.1807 \times 10^{-4}$ |
| $p_{Y22}$              | 113.4889 Pa | 0.1568 Pa | $-7.5325 \times 10^{-4}$ | $-1.1807 \times 10^{-4}$ |
| $T_A$                  | 294.58 K | 0.05 K | $2.9019 \times 10^{-7}$ Pa/K | $1.4501 \times 10^{-8}$ |
| $T_C$                  | 294.77 K | 0.05 K | $-2.9001 \times 10^{-7}$ Pa/K | $-1.4500 \times 10^{-8}$ |

Combined standard uncertainty $u_c$ (Pa) $1.779 \times 10^{-2}$
data sheet for the three primary standard systems ($k = 1$ confidence level = 66%) is given in Tables 2–5 and Fig. 15.

4. Summary

The Vacuum Measurement Lab. at Korea Research Institute of Standards and Science maintains primary vacuum gauge calibration standards namely: the mercury ultrasonic interferometer manometer (1 Pa to 100 kPa), the static expansion system (3 Pa to 100 Pa), and dynamic expansion system (10⁻⁷ Pa to 10⁻² Pa) which cover the pressure range from 10⁻⁷ Pa to 133 kPa. Vacuum secondary standard gauges like capacitance diaphragm gauges (CDGs), spinning rotor gauges (SRGs), and hot cathode ionization gauges (IGs) are routinely calibrated on these standards. KRISS, in the past, has participated in various key as well as bilateral comparisons to validate their primary calibration standards for the realization of vacuum pressures in the rough, medium, and high as well as ultrahigh vacuum range. In these

| Source of uncertainty | Value $x_i$ | Standard uncertainty $u(x_i)$ | Sensitivity coefficient $c_i$ | Uncertainty contribution $|\delta q_i|/u(q_i)$ |
|-----------------------|-------------|-------------------------------|-----------------------------|------------------|
| $p_i$                 | 532.59 Pa   | $5.326 \times 10^{-1}$ Pa     | $1.392 \times 10^{-9}$      | $7.417 \times 10^{-10}$ |
| $A$                   | 7.06195 cm² | $1.65 \times 10^{-3}$ cm²     | $1.050 \times 10^{-7}$ Pa/cm² | $1.731 \times 10^{-10}$ |
| $dL$                  | $6.02 \times 10^{-1}$ cm | $1.204 \times 10^{-3}$ cm | $1.232 \times 10^{-6}$ Pa/cm | $1.483 \times 10^{-9}$ |
| $dt$                  | 173.32 s    | $7.541 \times 10^{-3}$ s      | $-4.28 \times 10^{-9}$ Pa/s  | $-3.227 \times 10^{-9}$ |
| $T_{ch}$              | 296.0 K     | $5.0 \times 10^{-2}$ K        | $2.506 \times 10^{-9}$ Pa/K  | $1.253 \times 10^{-10}$ |
| $T_i$                 | 296.8 K     | $5.0 \times 10^{-2}$ K        | $2.499 \times 10^{-9}$ Pa/K  | $1.249 \times 10^{-10}$ |
| $C_j$                 | 9.8159 L/s  | $5.0 \times 10^{-3}$ L/s      | $7.557 \times 10^{-8}$ Pa/L/s | $3.778 \times 10^{-10}$ |
| $R_{ph}$              | $7.625 \times 10^{-2}$ | $5.0 \times 10^{-4}$ Pa      | $8.031 \times 10^{-7}$ Pa  | $4.015 \times 10^{-10}$ |
| $C_p$                 | $5.559 \times 10^{-3}$ L/s | $4.73 \times 10^{-3}$ L/s | $1.334 \times 10^{-4}$ Pa/L/s | $6.312 \times 10^{-9}$ |
| $C_2$                 | 11.202 L/s  | $5.0 \times 10^{-3}$ L/s      | $-6.622 \times 10^{-8}$ Pa/L/s | $-3.311 \times 10^{-10}$ |
| $R_{pu}$              | $3.889 \times 10^{-2}$ | $5.0 \times 10^{-4}$ Pa      | $-7.719 \times 10^{-7}$ Pa  | $-3.859 \times 10^{-10}$ |

Combined standard uncertainty $u_c$ (Pa) $= 7.324 \times 10^{-9}$
Fig. 15 Based on Table 5, the graphs for generated pressures vs. expanded uncertainty for all the three systems are shown above.

Fig. 16 Summary of expanded uncertainties for the KRISS standards. However, in between 0.1 Pa to 1 Pa range no good reference exists.

Comparisons, the KRISS systems have good degree of equivalence with the other international standards in the same pressure range. Figure 16 reflects the summary of the KRISS primary vacuum gauge calibration standards.

Since, KRISS is the national metrology institute of...
Rep. of Korea; these primary standards are used for calibration of secondary standards which in turn are used on vacuum comparison systems for calibration of vacuum gauges received from the industrial sector within the country as well as abroad.

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