Establishment of Thailand’s national primary vacuum standard by a static expansion method

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Abstract. A novel primary standard system for a vacuum pressure calibration has been established at the National Institute of Metrology, Thailand (NIMT). The system, which covers the pressure range from $1.3 \times 10^{-1}$ Pa up to $1.3 \times 10^{3}$ Pa is based on the static expansion method. By the series of re-expansion, the range could be extended to lower calibration pressures. The facility consists of two small and one large vessels, which provide an expansion ratio by a factor of approximately $10^{2}$ to $10^{3}$ for a single expansion and more for a multiple one. Six platinum resistance thermometers are attached to the vessel to ensure the correction of the non-isothermal effect during an expansion. The system could deliver the expanded ($k = 2$) relative uncertainties under 0.6% of reading at the lowest operating pressure. The measurement results of the system were verified by comparing with the calibration results of the capacitance diaphragm gauge (CDG) from Physikalisch-Technische Budensanstalt (PTB). The $E_n$ ratios represent an excellent agreement between NIMT and PTB calibration results.

1. Introduction and historical aspects
Pressure and Vacuum Laboratory, Fluid Mechanics Group, Mechanical Metrology Department, National Institute of Metrology (Thailand), NIMT has been providing the pressure and vacuum calibration services under ISO17025 since the establishment of the institute in 1998. More and more measurement parameters have been developing since then, and now NIMT services cover from $10^{-4}$ Pa up to $5 \times 10^{8}$ Pa of pressure measurement. Form a low vacuum (where the absolute pressure is in the range from $1 \times 10^{-3}$ to $1 \times 10^{-5}$ Pa) until high pressure, the national primary pressure standards by dimensional measurement method [1-2] are available. These Thailand’s standards were established by Priruenrom in 2012 [2]. Unfortunately, due to the theoretical and the technical limitations the dimensional measurement method is not valid for a measurement of a pressure range under the low vacuum. Therefore, the calibration services in the medium ($1 \times 10^{-1}$ Pa to $1 \times 10^{3}$ Pa), the high ($1 \times 10^{4}$ Pa to $1 \times 10^{6}$ Pa) and the very high ($1 \times 10^{7}$ Pa to $1 \times 10^{9}$ Pa) pressure range have to be traceable to Physikalisch-Technische Budensanstalt (PTB), the National Metrology Institute of Germany.

In order to reduce the annual export cost of recalibration of the national standards in vacuum measurement, the primary standard has been developed. The standard is based on the static expansion method which was first employed by Knudsen in 1910 and later become one of the most widely used method for a primary national standard in many National Metrology Institutes (NMIs) [3-6]. The principle of the static expansion method (also called volume of series of expansion method) is originally basing on the Boyle-Mariotte law where the product of pressure and volume of a confined gas held at a constant temperature is constant. Therefore, if the gas is expanded from a knowing low initial volume,
$V_1$, at a knowing high initial pressure, $p_1$, to a larger knowing volume, $V_2$, the final pressure or the calibration pressure, $p_{cal}$, can be calculated from

$$p_{cal} = p_1 \left( \frac{V_1}{V_1 + V_2} \right).$$

(1)

In the calibration, non-isothermal conditions and real gas behaviour shall be taken into account. Then, equation (1) is derived to [7]:

$$p_{cal} = p_1 \left( \frac{V_1}{V_1 + V_2} \right) \left( 1 - \frac{B}{R_m T} p_1 \right) \left( 1 - \frac{\Delta T}{T_1} \right)$$

(2)

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

Various vacuum ranges of the calibration pressure, $p_{cal}$, can be performed by an appropriate configuration of $p_1$, $V_1$, and $V_2$. The pressure range could be covered from $10^{-6}$ to $10$ Pa with relative uncertainty ranging from a few parts in $10^{2}$ to a few parts in $10^{3}$ [7-8].

A novel repeat-stage static expansion system for the generation of calibration pressures in the medium vacuum from $1.3 \times 10^{-1}$ Pa up to $1.3 \times 10^{3}$ Pa has been established at NIMT. The detail of this novel primary standard, known as SE1 is described in this paper.

2. Design of the primary standard

The volume expansion system SE1 is a repeat-stage static expansion system. Figure 1 shows the configuration of the system. The system consists of two small initial vessel ($V_1$ and $V_2$) and one large vessel ($V_f$) where the valves separate them from each other.

**Figure 1.** Schematic of NIMT’s static expansion system SE1, which is used as the primary standard for a vacuum calibration in the pressure range from $1.3 \times 10^{-1}$ Pa to $1.3 \times 10^{3}$ Pa.

2465: Pressure balance – Fluke Ruska 2465
PPC4: Quartz Reference Pressure Transducer (Q-PRT) – Fluke PPC4 A100K
UUT: Unit under test
Each vessel have the following nominal volumes: \( V_1 \approx 1 \text{ liter} \), \( V_2 \approx 0.1 \text{ liter} \), and \( V_f \approx 100 \text{ liter} \). The small vessels are employed as initial volumes for the expansion process. The large vessel is the main calibration vessel, which has a passage to connect with a unit under test (UUT). The vessels and all connecting tubes are made from stainless steel and all connections are vacuum-tight connectors (VCR and CF types with a copper gasket). The system is built using vacuum technique. All valves are vacuum valves with following types: VB1 and VB2 are ball valves, VN1 and VN2 are needle valves, VG1A and VG2 are gate valves and VA1 to VA5 are pneumatic-controlled all metal angle valves.

The desired initial pressure is generated by an automatic pressure control of the PPC4 and the PPC4 itself is used to measured that pressure. The PPC4 has been calibrated using the Ruska 2465 pressure balance as a standard with an uncertainty below 0.06% of reading. As temperature effects play an important role in the expansion process, 6 calibrated PT100 sensors are attached to the vessels to measure the pressure during the calibrations. All PT100 sensors are calibrated annually against the reference sensor in the water bath.

The calibration pressure can be achieved by various expansion sequences both in a single expansion and a repeat expansion. Three sequences are presented in this paper as described in the table 1.

### Table 1. Expansion schemes and its correspond expansion ratio, initial and calibration pressure

| Expansion Sequence | Expansion Scheme | Nominal Expansion Ratio | True Expansion Ratio, \( f \) | Initial Pressure (Pa) | Calibration Pressure (Pa) |
|--------------------|------------------|-------------------------|-----------------------------|-----------------------|---------------------------|
| 1                  | \( V_1 + V_2 \rightarrow V_1 + V_2 + V_f \) | \( 1 \cdot 10^2 \) | 1.1234 \( \cdot 10^2 \) | 2.7 \( \cdot 10^3 \) to 1.2 \( \cdot 10^5 \) | 3.0 \( \cdot 10^4 \) to 1.3 \( \cdot 10^3 \) |
| 2                  | \( V_2 \rightarrow V_2 + V_f \) | \( 1 \cdot 10^3 \) | 1.3769 \( \cdot 10^3 \) | 9.4 \( \cdot 10^2 \) to 2.2 \( \cdot 10^4 \) | 1.3 \( \cdot 10^9 \) to 3.0 \( \cdot 10^4 \) |
| 3                  | \( V_1 + V_2 \rightarrow V_1 + V_2 + V_f \) \( V_2 \rightarrow V_2 + V_f \) | \( 1 \cdot 10^5 \) | 1.5499 \( \cdot 10^5 \) | 8.4 \( \cdot 10^3 \) to 8.4 \( \cdot 10^4 \) | 1.3 \( \cdot 10^4 \) to 1.3 \( \cdot 10^3 \) |

### 3. Volume and expansion ratio determination

In order to determine the absolute volume of vessels and the expansion ratios the gravimetric method and the cumulative gas expansion method were employed respectively.

In the gravimetric method, the vessels are weighed empty and filled with distilled water. The weight difference is then used to calculate the volume from the knowing water density. The absolute volume can be accurately determined using this method and the expansion ratio can be found from the ratio of an initial volume by a final volume [5]. However, problems arise with the large 100 liter vessel that requires a disassembling of the whole apparatus. Moreover, the vessel is too large to perform the measurement using the gravimetric method in the laboratory. To avoid a dis- and a re-assembling of the system, the cumulative method was employed to determine the ratios of the expansions from a small initial vessel to a large final vessel. In this method, the expansion process is repeated several times without an intermediate evacuation of the final vessel. Thus, the gas is accumulated and the generated pressure in the final vessel approaches the initial setting pressure of the initial vessel. Initial pressures are chosen in order to obtain accumulated generate pressures in the range where accurate calibration pressure gauges are available. From the direct measurement of the initial pressure and the final generated pressure, the volume or expansion ratio can be determined [5].

### 4. Measurement verification

To verify measurement results of the system, they are compared with those performed by Physikalisch-Technische Bundesanstalt (PTB), the National Metrology Institute of Germany. Capacitance diaphragm
gauges (CDG) with the range of 1 Torr and 10 Torr was used as a unit under test (UUT) or a measurement artefact. The measurement results are shown in figures 2 and 3 and tables 2 and 3.

Figure 2 represents the comparison between NIMT’s and PTB’s calibration results of the same 1 Torr CDG. Figure 3 represents the comparison between calibration results of 10 Torr CDG that was calibrated by the standard which could be traceable to PTB with those by NIMT’s SE1. The percentage error of each CDG at selected pressures are presented. Both comparisons show that NIMT’s calibration results are in an excellent agreement with those performed by PTB.

The agreement of both comparisons could be qualitatively considered by using the degree of equivalent, which is expressed as the normalized error ratios ($E_n$) as follow:
\[ E_n = \frac{|E_{PTB} - E_{NIMT}|}{\sqrt{U^2_{PTB} - U^2_{NIMT}}} \]  

where \( E_{PTB} \) and \( E_{NIMT} \) are the measurement error of UUT performed by PTB and NIMT respectively, and \( U_{PTB} \) and \( U_{NIMT} \) are the measurement uncertainty of UUT performed by PTB and NIMT respectively.

The calibration results and \( E_n \) of 1 Torr CDG and 10 Torr are given in table 2 and 3 respectively. All \( E_n \) ratios are far less than 1 which represent an excellent agreement in calibration results of NIMT’s SE1 and those traceable to PTB.

### Table 2. Calibration results and \( E_n \) ratios of 1 Torr CDG by NIMT vs PTB

| Nominal Cal. Pressure (mbar) | NIMT’s SE1 | Cal. Pressure (mbar) | Error (%) | Relative Uncertainty (%) | Cal. Pressure (mbar) | Error (%) | Relative Uncertainty (%) | \( E_n \) Ratio |
|-----------------------------|------------|----------------------|------------|-------------------------|----------------------|------------|-------------------------|----------------|
| 0.0013                      | 0.001301   | 0.001370             | 5.27       | 0.52                    | 0.001130             | 5.44       | 0.34                    | 0.28           |
| 0.002                       | 0.002010   | 0.002115             | 5.22       | 0.50                    | 0.001990             | 5.28       | 0.30                    | 0.10           |
| 0.003                       | 0.003109   | 0.003174             | 5.12       | 0.50                    | 0.002990             | 5.09       | 0.28                    | 0.06           |
| 0.005                       | 0.005036   | 0.005286             | 4.97       | 0.50                    | 0.004990             | 4.99       | 0.26                    | 0.03           |
| 0.009                       | 0.009042   | 0.009484             | 4.89       | 0.50                    | 0.008980             | 4.74       | 0.24                    | 0.27           |
| 0.013                       | 0.013016   | 0.013636             | 4.76       | 0.35                    | 0.012970             | 4.55       | 0.24                    | 0.50           |
| 0.02                        | 0.019995   | 0.020910             | 4.57       | 0.35                    | 0.019960             | 4.41       | 0.24                    | 0.39           |
| 0.03                        | 0.029961   | 0.031208             | 4.16       | 0.35                    | 0.029930             | 4.02       | 0.24                    | 0.33           |
| 0.05                        | 0.050052   | 0.051871             | 3.63       | 0.35                    | 0.049860             | 3.52       | 0.24                    | 0.27           |
| 0.09                        | 0.092028   | 0.092911             | 3.00       | 0.35                    | 0.089770             | 2.88       | 0.24                    | 0.27           |
| 0.13                        | 0.130414   | 0.133856             | 2.64       | 0.35                    | 0.129650             | 2.59       | 0.17                    | 0.13           |
| 0.2                         | 0.20669    | 0.20524              | 2.27       | 0.35                    | 0.200060             | 2.22       | 0.16                    | 0.12           |
| 0.3                         | 0.29796    | 0.30549              | 1.91       | 0.25                    | 0.259800             | 1.91       | 0.16                    | 0.00           |
| 0.5                         | 0.49922    | 0.50803              | 1.77       | 0.25                    | 0.499200             | 1.74       | 0.16                    | 0.09           |
| 0.9                         | 0.88939    | 0.91465              | 1.70       | 0.25                    | 0.898800             | 1.65       | 0.16                    | 0.16           |
| 1.3                         | 1.29790    | 1.31971              | 1.68       | 0.25                    | 1.298100             | 1.66       | 0.16                    | 0.07           |

### Table 3. Calibration results and \( E_n \) ratios of 10 Torr CDG by NIMT vs PTB

| Nominal Cal. Pressure (mbar) | NIMT’s SE1 | Cal. Pressure (mbar) | Error (%) | Relative Uncertainty (%) | Cal. Pressure (mbar) | Error (%) | Relative Uncertainty (%) | \( E_n \) Ratio |
|-----------------------------|------------|----------------------|------------|-------------------------|----------------------|------------|-------------------------|----------------|
| 0.0013                      | 0.013017   | 0.01347              | 3.49       | 0.35                    | 0.013000             | 3.48       | 0.69                    | 0.02           |
| 0.02                        | 0.020000   | 0.02066              | 3.31       | 0.35                    | 0.020060             | 3.24       | 0.67                    | 0.09           |
| 0.03                        | 0.029962   | 0.03087              | 3.02       | 0.35                    | 0.030020             | 2.97       | 0.67                    | 0.07           |
| 0.05                        | 0.050026   | 0.05126              | 2.48       | 0.35                    | 0.049980             | 2.53       | 0.66                    | 0.07           |
| 0.09                        | 0.090233   | 0.09195              | 1.91       | 0.35                    | 0.090030             | 1.93       | 0.66                    | 0.03           |
| 0.13                        | 0.130417   | 0.13243              | 1.54       | 0.35                    | 0.130000             | 1.58       | 0.64                    | 0.04           |
| 0.2                         | 0.207025   | 0.20524              | 1.25       | 0.35                    | 0.200060             | 1.22       | 0.64                    | 0.04           |
| 0.3                         | 0.299938   | 0.30259              | 0.88       | 0.25                    | 0.300030             | 0.98       | 0.64                    | 0.14           |
| 0.5                         | 0.499793   | 0.50359              | 0.76       | 0.25                    | 0.500010             | 0.81       | 0.64                    | 0.07           |
| 0.9                         | 0.899499   | 0.90559              | 0.68       | 0.25                    | 0.900020             | 0.73       | 0.64                    | 0.08           |
| 1.3                         | 1.298524   | 1.30706              | 0.66       | 0.25                    | 1.300000             | 0.72       | 0.29                    | 0.16           |
| 2                           | 1.99746    | 2.0101               | 0.63       | 0.23                    | 2.000020             | 0.70       | 0.29                    | 0.17           |
| 3                           | 2.99410    | 3.0127               | 0.62       | 0.23                    | 3.000000             | 0.68       | 0.29                    | 0.15           |
| 5                           | 4.98543    | 5.0156               | 0.61       | 0.22                    | 5.000000             | 0.61       | 0.29                    | 0.01           |
| 9                           | 8.97873    | 9.0172               | 0.43       | 0.22                    | 9.001100             | 0.41       | 0.29                    | 0.05           |
| 13                          | 12.98860   | 13.0151              | 0.20       | 0.22                    | 12.996400             | 0.16       | 0.29                    | 0.11           |
5. Conclusion
The novel primary standard static expansion vacuum system, having repeat expansion stage, for the calibration of vacuum gauge in the range of $1.3 \times 10^{-1}$ Pa to $1.3 \times 10^3$ Pa has been established at NIMT. The expanded ($k = 2$) relative uncertainty of pressure of the system range from 0.22% to 0.52%. The generated pressure below $1.3 \times 10^{-1}$ Pa can also be achieved by the system. An extension of the calibration to a wider pressure range will be performed in the future.

This primary standard has been verified by comparing its measurement results with those of PTB by using 1 Torr and 10 Torr CDGs as a measurement artefact. The comparisons via $E_n$ ratios show an excellent agreement where $E_n$ ratios lie within 1.

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