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Results of the key comparison in absolute pressure from 1 Pa to 1000 Pa

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Abstract

This paper describes a CCM key comparison of low absolute-pressure standards at seven National Measurement Institutes that was carried out during the period March 1998 to September 1999 in order to determine their degrees of equivalence at pressures in the range 1 Pa to 1000 Pa. The Korea Research Institutes of Standards and Science(KRISS) participated from 10 Pa to 1000 Pa pressure range in 1999. The primary standards, which represent two principal measurement methods, included five liquid-column manometers and four static expansion systems. The transfer standard package consisted of four high-precision pressure transducers, two capacitance diaphragm gauges to provide high resolution at low pressures, and two resonant silicon gauges to provide the required calibration stability.

1. Introduction

At its 6th meeting held May 29th-30th, 1996, the Comite Consultatif pour la Masse et les grandeurs apparentees (CCM) approved proposals by the pressure working groups that identified six key comparisons in pressure, the relevant ranges, the transfer standards to be used, and the pilot laboratories. The objective of each comparison was to determine the degrees of equivalence [1] of primary measurement standards at major National Measurement Institutes(NMIs) and to test the principal measurement methods in the field.

One of the six key comparisons identified was in absolute pressure covering the range 1 Pa to 1000 Pa, which it was agreed would be piloted by the National Institute of Standards and Technology(NIST) using high-precision pressure transducers as transfer standards. This paper summarizes the calibrations of the transfer standards carried out at seven NMIs during the period March 1998 and September 1999. Two nominally identical transfer standard packages were used in this comparison to reduce the time required to complete all measurements, with Package A being circulated among laboratories in the European region(IMGC, NPL-UK, and PTB) and Package B being circulated among laboratories in the Asia-Pacific region(CSIRO, KRISS, and NPL-I). Results from the two regions were normalized by using data obtained during simultaneous calibrations of the two packages at the pilot laboratory. The following sections briefly describe of the primary standards, the design and construction of the transfer standard packages, the general calibration procedure required by the protocol, and the reduction and analysis of the data.

2. Primary Standards

The principal measurement methods tested by this comparison involved two types of primary standards: static expansion systems, which are pressure generators, and liquid-column manometers, which are pressure measurers.

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Four participants(IMGC, NPL-I, NPL-UK, and PTB) used static expansion systems as their primary standards and four participants used different types of manometers in which liquid-column heights were measured either by laser interferometry(CSIRO and IMGC) or by ultrasonic interferometry(KRISS and NIST).

2.1 Static Expansion System at the IMGC

The static expansion system at the IMGC is a modification of that described in reference [2], the principal difference being the addition of a third volume as described in reference [3]. The system consists of three volumes nominally, 10 mL, 500 mL and 50 L, the largest volume being the calibration chamber. The different expansion ratios are periodically measured by using the multiple-expansion method. The initial pressures between 1 kPa and 100 kPa are measured by secondary transfer standards directly traceable to the HG5 mercury manometer. The base pressure, which is obtained by a turbo pump, is in the range of 10^{-6} Pa. The relative combined uncertainty of the system for the pressure range 0.1 to 100 Pa is 2.1×10^{-3} when volumes added to the system by gauges to be calibrated can be disregarded. In the present comparison, however the additional volume of the transfer standard gauges and associated plumbing could not be disregarded and so the added volume was measured using a spinning rotor gauge. This procedure increased the relative combined uncertainty to 3×10^{-3}.

2.2 Static Expansion System at the NPL-I

The primary standard at the NPL-I used in the key comparison is a static expansion system in which there are two initial volumes v_1, v_2(nominally 25 mL and 384 mL, respectively) and a large chamber V_L with a nominal volume of 72 L [4-7]. VL can be evacuated to base pressures of 10^{-7} Pa using a diffusion pump and a liquid nitrogen trap equipped with an isolation valve. The initial pressures for the comparison were measured by means of a 110 kPa quartz spiral Bourdon gauge calibrated against an ultrasonic interferometer manometer. For generating the target pressure 1 Pa. 3 Pa, 10 Pa, and 30 Pa the expansion scheme v_1 to$(v_1 + V_L)$ was adopted with initial pressures ranging from 2800 Pa to 85000 Pa. The pressure points 100 Pa and 300 Pa were generated by using the expansion scheme v_2 to $(v_2 + V_L)$ with initial pressures of 18900 and 57000 Pa, respectively. The final pressure point of 1000 Pa was generated by using the successive expansion method with two expansions from v_2 to$(v_2 + V_L)$. Platinum resistance thermometers inserted into the different chambers were used to measure the temperature of the gas before and after expansion. The volume ratios of the different chambers have been determined both by gravimetry (filling the different chambers with triple distilled water) and also by the method of successive expansion [4,8].

2.3 Static Expansion System at the NPL-UK

The medium vacuum standard at the NPL-UK is a three-stage non-bakeable static expansion system with a 50-L calibration chamber. By varying the initial pressure and the number of stages of expansion, one may generate calculable pressures between 1.5×10^{-2} Pa and 2×10^{3} Pa may be generated. There is a choice of two small vessels from which gas may be expanded into the calibration chamber and this enables a greater range of pressures to be generated from a given range of initial pressures. The pressure of the initial gas sample is measured using a quartz Bourdon tube gauge. The pressure generated is calculated from knowledge of the initial pressure, the ratio of the volumes and the gas temperatures. The ratios of the volumes are determined using Elliott's [8] experimental procedure of repeated expansions and are calculated using the iterative method described by Redgrave et al [9].

2.4 Static Expansion System at the PTB

The primary standard of the PTB is a static expansion system, called SE2, in which pressures are generated by expanding gas of known pressure from two alternative small volumes of nominally 0.1 L and 1 L directly into a volume of 100 L. It is also possible to carry out two expansions in series with intermediate nominal volumes of 100 L and 1 L. The regular operational range of SE2 is 0.1 Pa up to 1 kPa. The system is described in detail in references [10-13].

2.5 Laser Interferometer Manometer at the CSIRO

The manometer uses a mercury U-tube in which the surfaces are the reflectors of a Michelson interferometer [14]. To determine the surface position, tungsten-weighted cat's eye floats are used as reflectors for the laser light. Sloping sides in the float are used to produce a flat mercury surface.

2.6 HG5 Laser Interferometer Manometer at the IMGC

The HG5 mercury manometer is the primary pressure standard of the IMGC in the barometric range up to 120 kPa and it can operate in both absolute and relative modes. A full description of HG5 and the discussion of the uncertainties are given in reference [15]. The HG5 is a laser interferometer manometer that essentially consists of two interconnected 60-mm bore, 1-m long glass tubes forming the U-tube, which is placed in a temperature-controlled water bath. The mercury temperature is measured by two platinum resistance thermometers (PRTs) installed coaxially at the base of the columns. The vertical displacements of the mercury menisci are measured with a single-beam interferometer. Corner cube reflectors mounted on very lightweight floats, thin glass disks that float on both mercury meniscus, reflect the two vertical laser beams. Increased accuracy at pressures up to 13 kPa is obtained by focusing and directly reflecting the laser beams from the mercury menisci using lenses mounted on the floats in a cat's-eye arrangement. Such floats were used for all measurements during the present comparison.

2.7 Ultrasonic Interferometer Manometer at the KRISS

The primary standard at the KRISS for this key comparison is a mercury ultrasonic interferometer manometer that was assembled and evaluated as an international cooperation project between the KRISS and the NIST beginning in 1988. The manometer [16] has an operating range of 0.5 Pa to 133 kPa and its design and operation are based on the mercury ultrasonic manometers developed at the NIST [17,18], which are described in the next section.

2.8 Ultrasonic Interferometer Manometers at the NIST

The primary standards at the NIST used in this key comparison are two Ultrasonic Interferometer Manometers (UIMs), a mercury UIM with a full-scale range of 160 kPa [17,18] and an oil UIM [19] with a full-scale range of 140 Pa. The unique feature of these manometers is that changes in height of the liquid columns are determined by an ultrasonic technique. A transducer at the bottom of each liquid column generates a pulse of ultrasound that propagates vertically up the column, is reflected from the liquid-gas interface, and returns to be detected by the transducer. The length of the column, which is proportional to the change in phase of the returned signal, is determined from the phase change and the velocity of the ultrasound [20]. The manometers have large-diameter(75 mm - Hg UIM; 100 mm - oil UIM) liquid surfaces to minimize capillary effects, thermal shields to stabilize the temperature and minimize its gradients, and high-vacuum techniques to minimize leaks and pressure gradients. The mercury UIM employs a "W" or three-column design to correct for possible tilt. The oil UIM uses a four-column design equivalent to two parallel manometers that also function as orthogonal tilt meters.

3. Transfer Standards

On the basis of earlier comprehensive reviews of pressure transducer performance [21,22], two types of gauges were selected as the transfer standards, namely, resonant silicon gauges(RSGs) for their good long-term stability and capacitance diaphragm gauges(CDGs) for their high-precision. The RSGs are a new type of MEMS (MicroElectroMechanical Systems) sensor that have excellent calibration stability, are resistant to mechanical shock, and are only moderately susceptible to overpressure although they are rather sensitive to tilt (~ 0.4 Pa/mrad). The two RSGs selected for the comparison had full-scale ranges of 1000 Pa and 10,000 Pa and were combined with two CDGs each with a full-scale range of 133 Pa. The transfer standard package consisted of three parts, a pressure transducer package(PTP), a support electronics package(SEP), and a laptop computer(see Fig. 1 to 2). The PTP included four differential transducers housed in a temperature-controlled enclosure, a calibrated 100-ohm platinum resistance thermometer(PRT) to measure the interior temperature of the enclosure, and an ion vacuum pump and reference-pressure vacuum gauge for the absolute mode calibrations. The tilt orientation of the PTP during calibration of the RSGs was monitored by means of sensitive bubble levels mounted on the PTP base plate and any observed changes were corrected using the leveling screws.

The SEP included a temperature controller for the transducer enclosure, signal conditioning electronics for the CDGs, a controller for the ion vacuum pump, and a digital voltmeter(DVM) for digitizing analog signals from the CDGs, the calibrated PRT, and the reference vacuum gauge. A laptop computer was used for controlling the acquisition of data from the RSGs and the DVM during calibration. The time required to obtain one set of readings was approximately 55 seconds. Because of their accuracy(~ 1 part in 10^4), the readings of the RSGs were multiplied by a scale factor before display and storage on the laptop computer in order to increase the level of confidentiality for the pilot laboratory data.

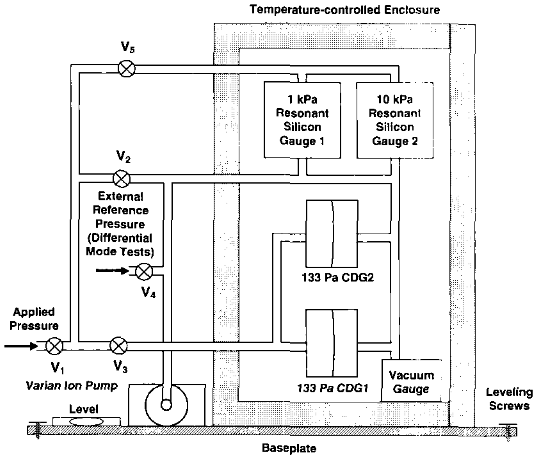

Fig. 1. Schematic diagram of the pressure transducer package(PTP).

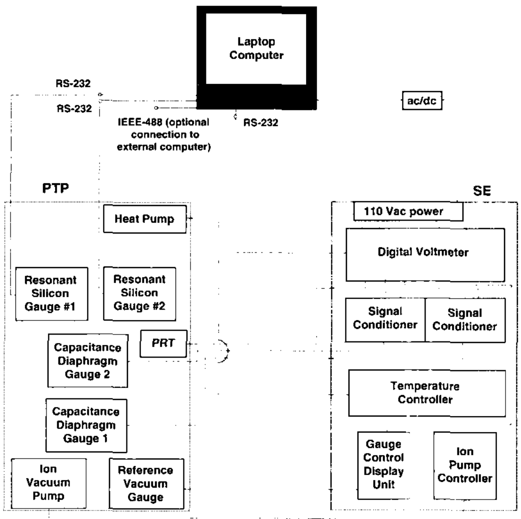

Fig. 2. Schematic diagram of the electrical connections between the PTP, the support electronics package(SEP) and the laptop computer.

4. General Calibration Procedure

A total of five calibration runs were required, with each run taken on a different day. Within a calibration run, five repeat sets of pressure and temperature readings of the transfer standard and primary standard were required at each target pressure. At the beginning of each calibration run, ten repeat sets of zero-pressure readings for the transfer standard gauges were required to be taken with the PTP isolated from the participant's calibration system(valves V_1 and V_4 closed) and with internal isolation valves V_3 and V_5 and bypass valve V_2 open. These data were needed to correct calibration data obtained with liquid-column manometers for zero-pressure offsets. An additional ten repeat sets of zero-pressure readings were to be taken at the end of each run in order to monitor zero drift in the four transducers during calibration. The calibration procedure also included the option of recording five sets of zero-offset readings for the gauges just prior to establishing each target pressure. These readings, which were taken with the external and internal isolation valves of PTP open and bypass valve V_2 closed, were needed to correct zero offsets in calibration data obtained with static expansion systems.

The format for reporting calibration data followed the measurement sequence dictated by the data acquisition software. All calibration data were transmitted to the pilot laboratory in the form of spreadsheet files, which greatly facilitated the analyses of a rather voluminous amount of data.

5. Reduction and Analysis of the Data

The reduction and analysis of the key comparison data required that several factors be addressed. These included zero-pressure offsets, thermal transpiration effects, deviations of the actual pressures from target pressures, relatively large calibration shifts in the capacitance diaphragm gauges, and normalization of data from two different transfer standard packages [24].

5.1 Corrections for Zero-pressure Offsets

The first step in reducing the comparison data was to correct the readings of each gauge i for their zero-pressure offset. The index i is equal to either 1 or 2 and refers to either, CDG1 and CDG2, or RSG1 and RSG2(see Fig. 1). At a given target pressure during calibration run k, the corrected reading of gauge i for repeat set l is given by:

$$p_{ikl} - p_{Gikl} - \langle p_{Gik0} \rangle_{10} + p_{REFkl}$$

for liquid-column manometer data (1a)

and

$$p_{ikl} = p_{Gikl} - \langle p_{Gik0} \rangle_{5}$$

for static expansion system data (1b)

where P_{Gikl} is the uncorrected gauge reading, $\langle P_{Gik0} \rangle_{10}$ is the mean of 10 zero-pressure readings taken just prior to the start of calibration run k, p_{REFkl} is the reference pressure reading during repeat set l, and $\langle p_{Gik0} \rangle_5$ is the mean of 5 zero-offset readings taken just prior to realizing each target pressure.

5.2 Calculation of Calibration Ratios

The transfer standard gauges are nominally linear devices and so the ratio of transfer standard reading to primary standard reading will be essentially independent of pressure for a range of pressures about each target value. These ratios form the basis for the comparison of primary standards from different NMIs.

At each target pressure during calibration run k the mean ratio of 5 sets of repeat readings l of transfer standard gauge i and primary standard j is given by

$$a_{ijk} = \frac{1}{5} \sum_{l=1}^{5} \frac{p_{ikl}}{P_{jkl}} \tag{2}$$

where p_{ikl} and P_{jkl} are the "simultaneous: readings of the gauge and primary standard, respectively. The mean of the a_{ijk} for 5 calibration runs defines a *calibration ratio* given by

$$a_{ij} = \frac{1}{5} \sum_{k=1}^{5} a_{ijk} = \frac{1}{25} \sum_{k=1}^{5} \sum_{l=1}^{5} \frac{p_{ikl}}{P_{jkl}} \tag{3}$$

The calibration ratio, if expressed as

$$a_{ij} = \frac{p_i}{P_j} \tag{4}$$

may be used to calculate a gauge reading p_i from the pressure being measured/generated by primary standard j, P_j, or vice-versa.

5.3 Degrees of Equivalence of the Primary Standards

Figure 3 to 7 presents final results for the pilot and participant NMIs as a function of nominal target pressures [23,24]. D_j is the deviation of the corrected mean gauge reading p_j obtained by NMI_j from the reference value P_R and U_j is the expanded uncertainty of this deviation at a 95 % level of confidence. The degrees of equivalence of individual NMIs with respect to key comparison reference values are presented graphically in Fig. 3 to 7 as plots of deviations, $D_j = p_j - p_R$, versus NMI. The results of the CSIRO for pressures between 10 and 300 Pa are considerably higher the D_j than those from other NMIs and excluded from calculating P_R and plotting of the D_j.

6. Conclusions

This results revealed no significant relative bias between the KRISS low vacuum standard and other participants

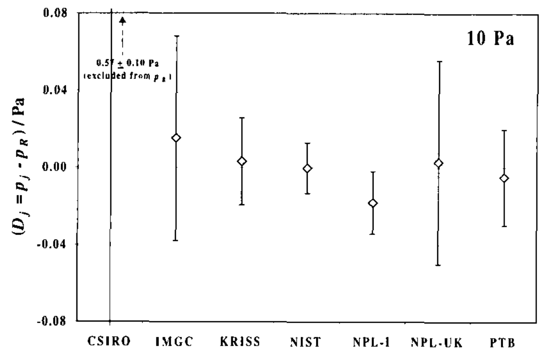

Fig. 3. Degrees of equivalence expressed as the deviation of corrected mean gauge readings from the key comparison reference values at 10 Pa. The error bars refer to expanded uncertainties (Uj) of the deviations at a 95 % level of confidence.

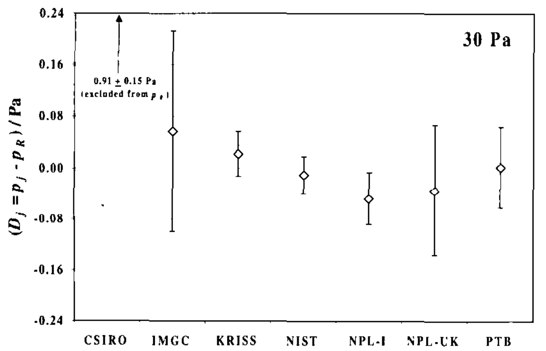

Fig. 4. Degrees of equivalence expressed as the deviation of corrected mean gauge readings from the key comparison reference values at 30 Pa. The error bars refer to expanded uncertainties (Uj) of the deviations at a 95 % level of confidence.

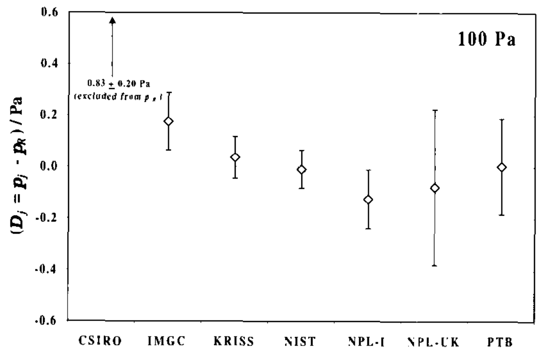

Fig. 5. Degrees of equivalence expressed as the deviation of corrected mean gauge readings from the key comparison reference values at 100 Pa. The error bars refer to expanded uncertainties (Uj) of the deviations at a 95 % level of confidence.

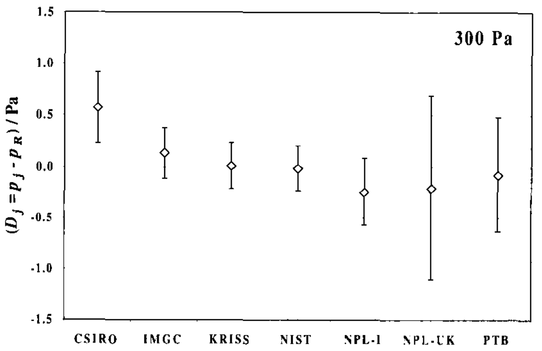

Fig. 6. Degrees of equivalence expressed as the deviation of corrected mean gauge readings from the key comparison reference values at 300 Pa. The error bars refer to expanded uncertainties (Uj) of the deviations at a 95 % level of confidence.

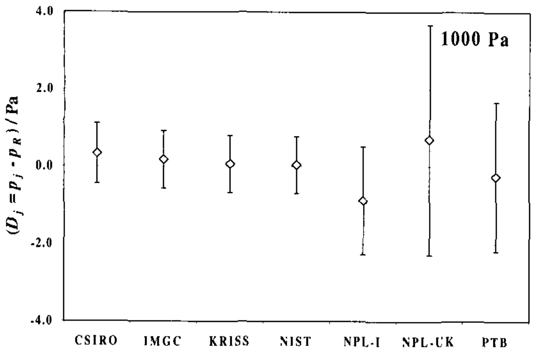

Fig. 7. Degrees of equivalence expressed as the deviation of corrected mean gauge readings from the key comparison reference values at 1000 Pa. The error bars refer to expanded uncertainties (Uj) of the deviations at a 95 % level of confidence.

standards by this key comparison CCM.P-K4 in absolute pressure in the range 10 Pa to 1000 Pa.

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