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Development of a new high-stability transfer standard based on resonant silicon gauges for the range 100 Pa to 130 kPa

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Abstract
The National Institute of Standards and Technology (NIST) has developed a new transfer standard capable of absolute-mode and differential-mode operation in the range 100 Pa to 130 kPa. This newly built transfer standard relies on resonant silicon gauges (RSGs) of the same type used to provide superior long-term calibration stability in NIST piloted international key comparisons CCM.P-K4 and CCM.P-K5, which covered absolute and differential pressure standards pressures up to 1 kPa (Miiller *et al* 2002 *Metrologia* **39** *Tech. Suppl.* 07001 and 07002). The new transfer standard package differs from the previous packages in that it fully covers the atmospheric pressure range (100 Pa to 130 kPa). This was made possible by the addition of a pair of 130 kPa RSGs to complement a pair of 10 kPa RSGs. The RSG transfer standard package has demonstrated good short-term zero stability and pressure resolution, and has demonstrated long-term instability of only a few ppm at 130 kPa, increasing to 0.01% at 100 Pa. The long-term instability is nominally commensurate with that associated with piston gauge standards, which are limited to pressures of nominally 10 kPa and higher. The main advantage of the new package is that it operates easily at lower pressures than piston gauges while still covering the atmospheric pressure range 100 Pa to 130 kPa.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

This paper describes the development and testing of a newly constructed RSG-based transfer standard package capable of both absolute and differential mode operation in the extended pressure range 100 Pa to 130 kPa. The objective was to develop a transfer standard with long-term stability that is commensurate with the exceptionally low measurement uncertainties of the NIST ultrasonic interferometer manometer (UIM) primary standards [1–8]. The newly built transfer standard consists of a pair of 130 kPa RSGs and a pair of 10 kPa RSGs that enable the use of Youden analyses [1,2] to be applied to the data.

The new transfer standard is expected to find applications in national ‘round robins’ and international comparisons of pressure standards that operate in the atmospheric pressure range 100 Pa to 130 kPa. In addition, the RSG-based transfer standards may become the basis for new calibration services in which the transfer standards are circulated among customer laboratories between periodic calibrations against primary standards at NIST. This could provide customers with calibrations at lower cost and greater convenience at the expense of a small increase in uncertainty.

2. Sensor selection and performance considerations

The sensors used in this transfer standard were Yokogawa[1] model 265381-2653-S15 for the 130 kPa gauges and model

[1] Identification of commercial equipment does not imply recommendation or endorsement, nor does it imply that the equipment identified are necessarily the best available for the purpose.

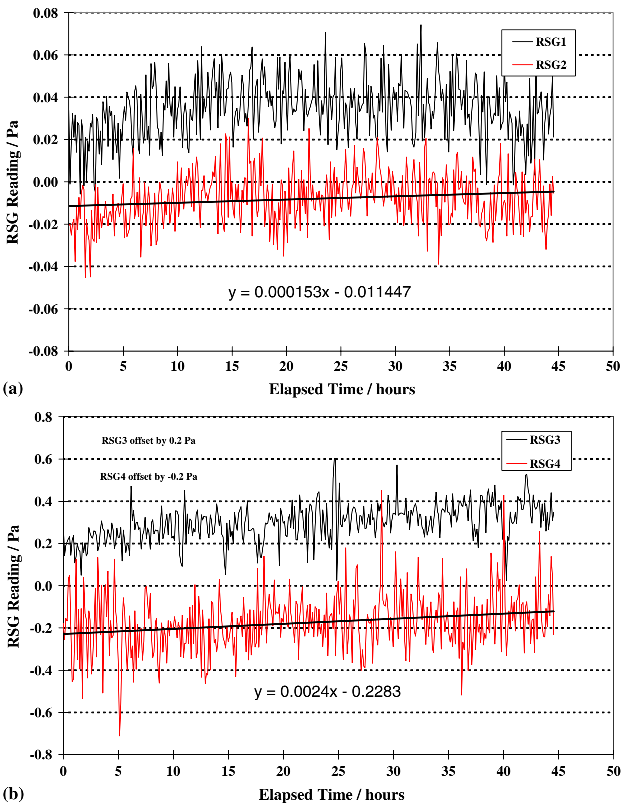

Figure 1. (*a*) Plot of zero stability and random noise of the two 10 kPa RSGs in the RSG transfer standard package over a 45 h time period. RSG2 is shown in the lower trace. (*b*) Plot of zero stability and random noise of the two 130 kPa RSGs in the RSG transfer standard package over a 45 h time period. RSG4 is shown in the lower trace.

265381-S2 for the 10 kPa gauges. These gauges make use of micro electro mechanical systems (MEMS) type sensors that are manufactured by silicon micromachining techniques to produce silicon diaphragms nominally a few millimetres square by a fraction of a millimetre thick. Two single-crystal silicon resonators each encapsulated in a vacuum microcavity are micromachined onto the surface of the diaphragm. Changes in pressure on the diaphragm are determined by measuring strain-induced changes in the two resonant frequencies. The two frequencies are also used for temperature compensation to minimize thermal effects. Since each resonating element is encapsulated in a vacuum, the most critical part of the sensor is never in direct contact with the gas whose pressure it is sensing [9].

The RSG sensors used in the transfer standard package were evaluated at NIST for zero instability, random noise and long-term stability (see sections 4–6). Figures 1(*a*) and (*b*) show the results for zero instabilities and random noise. The zero instability was monitored via the RSGs digital data port (RS-232) over the short term (45 h) and was also evaluated to determine the random noise produced by the sensors which ultimately limits the pressure resolution. The 10 kPa gauges showed a maximum drift rate over 45 h of 0.2 mPa h^{-1} while the 130 kPa gauges demonstrated a maximum drift rate of 2.4 mPa h^{-1}. The noise-limited pressure resolution average of the two 10 kPa RSGs (given by twice the standard deviation of repeated readings at a stable pressure) was 2.4 parts in 10^6 of full scale (FS), while the 130 kPa gauges demonstrated a noise-limited pressure resolution average of the two gauges of 1.9 parts in 10^6 of FS. The results of the zero instability and noise limited pressure resolutions are summarized in table 1. The resolution of the different transducers tends to scale linearly with their FS ranges. A tilt sensitivity of 0.4 Pa mrad^{-1} was determined, which is consistent with the findings of a previous study[2] [10]. Sensitive bubble levels were installed on the transfer standard package so that it could be re-levelled to correct for any changes in tilt during the course of measurements.

Table 1. Table of zero instability and pressure resolution due to random noise of transducers used in the transfer standard package.

Transducer	Zero instability (typical)/mPa h^{-1}	Resolution due to random noise (2 × stdev)
RSG1 (10 kPa)	0.2	2.4 parts in 10^6 FS
RSG2 (10 kPa)		2.3 parts in 10^6 FS
RSG3 (130 kPa)	2.4	1.4 parts in 10^6 FS
RSG4 (130 kPa)		2.0 parts in 10^6 FS

3. Transfer standard package

The design of the transfer standard package was based on the design previously described in previous publications [1, 2] but briefly consists of a pressure transducer package (PTP), a support electronics package (SEP) and a laptop computer (see figure 2). The PTP is shown in figure 2(*a*) and is a temperature controlled enclosure containing a total of four differential transducers (two 10 kPa RSGs, two 130 kPa RSGs). The transfer standard package is designed for shipment between national metrology institutes (NMIs) in containers that are specifically designed to isolate vibration and shock that inevitably occurs during shipment.

3.1. The pressure transducer package (PTP)

The differential RSG pressure transducers were encased in an insulated enclosure that was temperature controlled with a thermoelectric module (a Peltier device), which acted as a heat pump to maintain the interior at a preset temperature of 23.0 °C. The RSG sensors have the ability to sense their internal temperature and report the temperature via RS-232 communication. These temperatures were monitored during the data collection process. An 8 L s^{-1} ion pump along with a reference-pressure vacuum gauge maintained and monitored the reference vacuum during the measurements, which were performed in absolute mode. Stainless steel plumbing was used throughout the vacuum system. The valves (see figure 2(*a*)) included external isolation valves V1 and V2, an internal isolation valve V4 for the 10 kPa RSGs and an internal bypass valve V3 between the reference and pressure sides of the gauges. Two 10 kPa RSGs were mounted above the two 130 kPa RSG sensors. The isolation valve V4 was closed at pressures greater than 10 kPa to prevent over-pressurization of the lower range gauges at higher pressures. During the data collection, the tilt orientation of the RSG was monitored by

[2] Erratum: the tilt sensitivity, given in [10] as 400 Pa mrad^{-1}, should read 0.4 Pa mrad^{-1}.

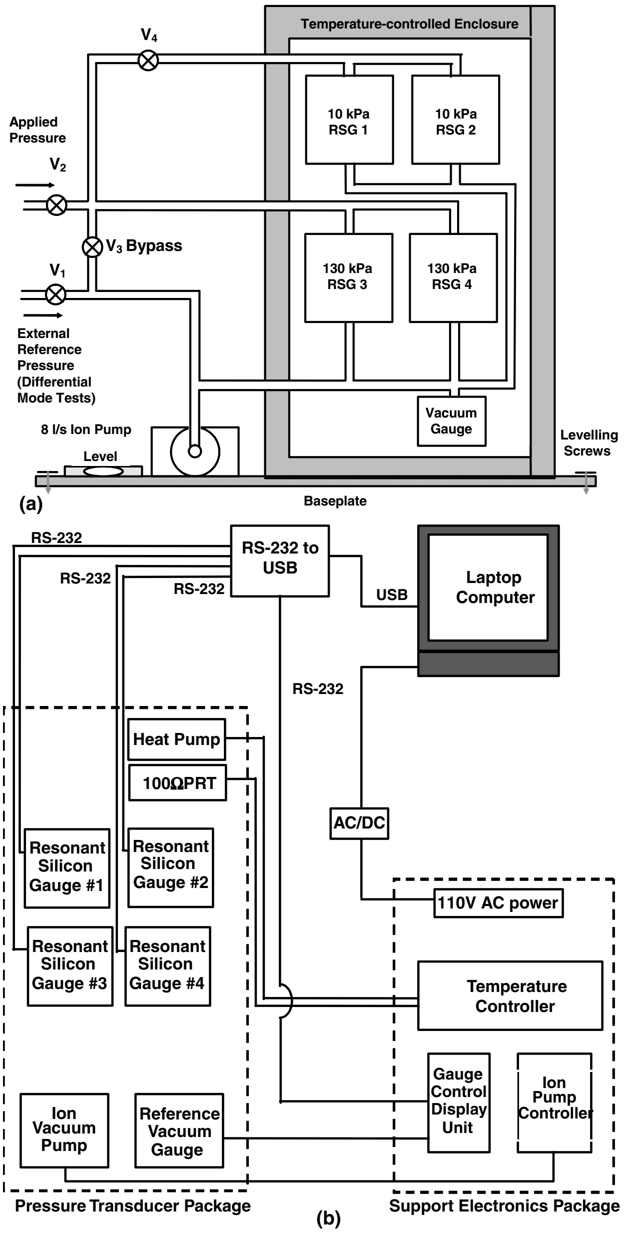

Figure 2. Schematic diagram of (*a*) the PTP and (*b*) the electrical connections between the PTP, the SEP and the laptop computer.

means of sensitive bubble levels. Small changes in tilt were observed at pressures above 30 kPa and were corrected using levelling screws attached to the base plate.

3.2. The support electronics package (SEP)

The SEP shown in figure 2(*b*) contained all the electronics to control, operate and acquire data from the PTP. These electronics operated the heat pump, an ion pump and the reference vacuum gauge. The temperature control circuit for enclosure consisted of a Wheatstone bridge mounted inside the enclosure where the bridge included a PRT and an adjustable resistor in two of its arms. NIST-built electronics supplied power to the heat pump using a proportional integrated differential (PID) control scheme. This arrangement maintained temperature stability of the enclosure to better than 0.1 °C even while the exterior room temperature changed by 2.0 °C during a 24 h period. A portable computer was connected through a USB hub to RS-232 communication lines for data acquisition and control of the four RSGs and the reference vacuum gauge during calibration. The RSG pressure data were read using a digital RS-232 interface provided by the manufacturer. The NIST designed software was set to $n = 50$, where n is the number of data points averaged per recorded pressure. For pressures from 100 Pa to 10 kPa, the RSG gauges were read in sequence from RSG1 to RSG4. The time to acquire one set of readings from each RSG gauge was approximately 20 s (integration time for the 50 averaged data points). At pressures greater than 10 kPa, only the 130 kPa gauges (RSG3 and RSG4) were recorded.

4. Collection of data for long-term stability study

A study was undertaken to evaluate the long-term stability of the transfer standard package in which the transfer standard package was repeatedly calibrated (with nitrogen) by comparison with the NIST 160 kPa UIM primary standard[3]. A single calibration consisted of five runs, with each run taken on a different day, usually on five successive days. Within each run (always less than 12 h), five repeated readings of the RSGs and the NIST primary standard were recorded at each target pressure in the following ascending order: 100 Pa, 300 Pa, 1000 Pa, 3000 Pa, 10 000 Pa, 30 000 Pa, 100 000 Pa and 130 000 Pa. A single calibration, which yielded 25 data points at each target pressure, was usually completed within a period of approximately one week. Finally, calibrations were repeated every few months over a period nominally spanning a year, resulting in multiple sets of data that enabled the long-term stability of the transfer standard package to be evaluated.

5. Data analysis of long-term stability study

The data reduction procedure outlined in this section was used to generate Youden plots [11], and ultimately, to determine the long-term stability of the transfer standard package. The data analysis was performed similar to that used in the previous NIST piloted key comparisons CCM.P-K4 and CCM.P-K5 [1, 2] with the major difference being that this long-term stability study involved only one primary standard, the NIST 160 kPa UIM. The data analysis presented below, while based on the previous work, was altered to reflect the use of only one standard to evaluate long-term stability of the transfer standard package.

5.1. Corrections for zero-pressure offsets

The readings of each gauge i were corrected for its zero-pressure offset. The index i is equal to either 1 or 2 and refers to either the *pair* of 10 kPa RSGs (RSG1, RSG2) or the *pair* of 130 kPa RSGs (RSG3, RSG4) shown in figure 2. At a given target pressure during calibration run k, the corrected reading of gauge i for repeat set l is given by

$$p_{ikl} = p_{\mathrm{G}ikl} - \langle p_{\mathrm{G}ik0} \rangle_{10}, \qquad (1)$$

[3] The expanded ($k = 2$) stated uncertainty of the NIST 160 kPa UIM due to systematic effects has been evaluated as $[(6 \times 10^{-3})^2 + (5.2 \times 10^{-6}\, P)^2]^{1/2}$ where P is the pressure.

where p_{Gikl} is the uncorrected gauge reading and $\langle p_{Gik0}\rangle_{10}$ is the mean of ten zero-pressure readings taken just prior to the start of calibration run k.

5.2. Corrections for thermal transpiration effects

The temperature of the transfer gauges was maintained at 23.0 °C to better than 0.1 °C whereas the equilibrium temperature of the primary standard was dependent on the temperature of the laboratory on a particular day. The effect of different operating temperatures between the primary standard and the transfer standard gauges was minimized by determining the pressure, P_j, that the primary standard would measure if it were operating at the same temperature as the transfer standard gauges:

$$P_j = P_{ju} f_{tt}, \tag{2}$$

where P_{ju} is the uncorrected reading of the primary standard and f_{tt} is the thermal transpiration correction factor calculated from the Takaishi–Sensui equation [1, 12, 13].

5.3. Calculation of calibration ratios

The RSGs in the transfer standard are nominally linear devices. Therefore, the ratio of a RSG reading to that of the primary standard will be essentially independent of pressure for a small range of pressures near each target value (within 2% of target). Once determined, these calibration ratios are used to correct the gauge readings for deviations of the primary standard from the target pressure.

At each target pressure during a calibration run, k, the mean ratio of the five repeat sets of readings of transfer standard gauge i (RSG) and primary standard j (NIST 160 kPa UIM) is given by

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

where p_{ikl} and P_{jkl} are the 'simultaneous' readings of the gauge and primary standard respectively. The mean of the a_{ijk} for five calibration runs (conducted over a period of about one week) defines a *calibration ratio* given by

$$a_{ij} = \frac{1}{5}\sum_{k=1}^{5} a_{ijk}. \tag{4}$$

The calibration ratio expressed as

$$a_{ij} = \frac{p_i}{P_j} \tag{5}$$

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

5.4. Calculation of predicted gauge readings

The calibration ratios were used to predict the RSG readings that would have been observed if the 160 kPa UIM primary standard had been set to the target pressure.

At each target pressure up to and including 130 kPa there are a *pair* of gauges ($i = 1, 2$). Thus, for the transfer standard package, there will be two gauge readings for each pressure measured by the primary standard j and, according to equation (5), the predicted gauge readings, p_{ij}, may be expressed as

$$p_{ij} = a_{ij} p_t, \tag{6}$$

where a_{ij} is the calibration ratio for gauge i in the RSG transfer standard package, and the primary standard reading equals the target pressure, i.e. $P_j = p_t$.

5.5. Estimates of uncertainties in the normalized gauge readings

The combined standard uncertainty ($k = 1$) in the predicted gauge readings may be estimated from the root-sum-square of two component uncertainties [14] given by

$$u_c(p_{ij}) = \sqrt{u_{std}^2(p_{ij}) + u_{rdm}^2(p_{ij})}, \tag{7}$$

where $u_{std}(p_{ij})$ is the uncertainty in p_{ij} due to systematic effects in the NIST 160 kPa primary standard, j, and $u_{rdm}(p_{ij})$ is the uncertainty in p_{ij} due to the combined effect of short-term random errors of the transfer standard RSG gauge i and the primary standard j during calibration. It follows from equation (6) that the component relative uncertainties in the predicted gauge readings are equal to the component relative uncertainties in the corresponding calibration ratios, e.g. $u_{rdm}(p_{ij})/p_{ij} = u_{rdm}(a_{ij})/a_{ij}$, etc.

5.6. Long-term shifts in gauge response

At a given target pressure, the variation due to long-term shifts was modelled by a normal distribution such that the best estimated value is the mean, $u_{lts}(a_{ij}) = \big((a_{ij})_{max} + (a_{ij})_{min}\big)/2$, and there is a 2 out of 3 chance the calibration ratio lies in the interval between maximum and minimum values of a_{ij} obtained from five repeat calibrations over a period of nominally a year. Then the standard uncertainty due to this source of error equals one-half the difference between the maximum and minimum values:

$$u_{lts}(a_{ij}) = \big((a_{ij})_{max} - (a_{ij})_{min}\big)/2. \tag{8}$$

This estimate is unaffected by any systematic bias in the NIST 160 kPa primary standard, which would be present in all five calibrations. This estimate assumes that the observed shifts in the calibration ratios are primarily due to the gauges and not the NIST 160 kPa primary standard.

6. Results

The results for the predicted gauge readings, p_{ij}, and their standard ($k = 1$) uncertainties (error bars shown on plots), $u_c(p_{ij})$, are presented in figures 3 and 4 in the form of Youden plots [11] in which differences $p_{2j} - p_t$ are plotted as a function of $p_{1j} - p_t$. The y- and x-axes are labelled RSG2 − STD and RSG1 − STD, or RSG4 − STD and RSG3 − STD, where RSG1 and RSG2 are the 10 kPa RSG gauges, RSG3 and RSG4 are the 130 kPa gauges, and STD is the NIST 160 kPa UIM primary pressure standard set to the target pressure.

The long-term stability of the transfer standard package was evaluated using equation (8) with the results shown in

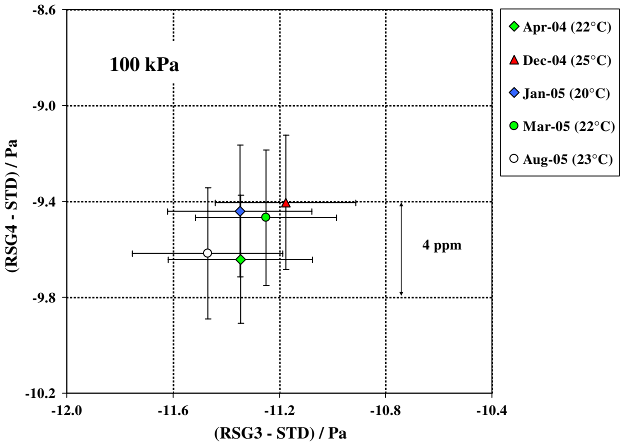

Figure 3. Youden plot of differences between predicted pressure readings of 130 kPa RSGs and pressures measured by the NIST 160 kPa UIM primary standard when set equal to a target pressure of 100 kPa. The error bars refer to combined standard ($k = 1$) uncertainties.

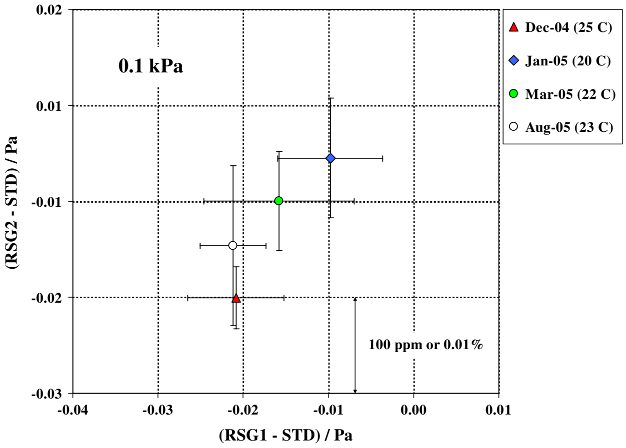

Figure 4. Youden plot of differences between predicted pressure readings of 10 kPa RSGs and pressures measured by the NIST 160 kPa UIM primary standard when set equal to a target pressure of 100 Pa. The error bars refer to combined standard ($k = 1$) uncertainties.

figure 5. Also shown in the plot are the relative uncertainties due to systematic effects for the NIST UIMs [1–8]. This plot shows that the long-term stability of the transfer standard package will enable an excellent comparison of primary standards. Specifically, the demonstrated long-term stability of the transfer standard package is commensurate with the relative uncertainties due to systematic effects in the NIST primary standards.

7. Discussion

In studies that involve one primary standard, as in the present case, Youden plots have the potential of revealing any systematic effects arising from different operating conditions for the primary standard. A comparison of results obtained at laboratory temperatures between 20 °C and 25 °C (see figures 3 and 4) reveals no systematic temperature effect at the different target pressures.

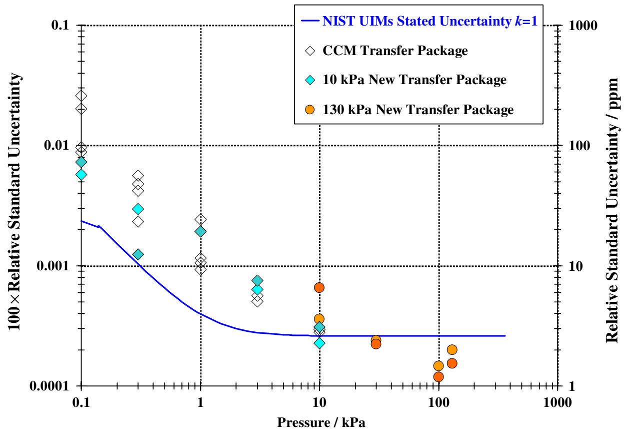

Figure 5. Comparison of the relative uncertainty due to long-term shifts in the RSG transfer standard gauges (symbols), to the relative uncertainties due to systematic effects in the NIST primary standards (solid line). This plot shows that the RSG transfer standard package has demonstrated excellent long-term instability of only a few ppm at 130 kPa, increasing to 0.01% at 100 Pa. The data were collected over the nominal period of a year as a series of calibration trials. Each trial consisted of five repeat runs, with each run lasting approximately one day. The solid circles refer to the 130 kPa RSGs and the solid diamonds refer to the 10 kPa RSGs. The open diamonds refer to 10 kPa RSG data collected in the NIST piloted key comparison CCM.P-K4 [1].

In this study, the transfer-standard package was evaluated by looking at the long-term stability of repeat weeklong calibrations against the NIST 160 kPa UIM primary standard. The error bars in the Youden plots are given by equation (7) where $u_{\rm std}(p_{ij})$ is the uncertainty ($k = 1$) due to systematic effects in the NIST primary standard, and $u_{\rm rdm}(p_{ij})$ is the uncertainty ($k = 1$) in p_{ij} due to the combined effect of short-term random errors of the RSG transfer standard gauge and the NIST primary standard during calibration. As previously noted, the long-term stability estimate is unaffected by any systematic bias in the NIST 160 kPa primary standard, as it is present in all repeat calibrations. With the assumption that the NIST 160 kPa primary standard is stable over time, then the observed scatter in the Youden plots (see figures 3 and 4) reflects the long-term stability of the RSG transfer standard package. Applying equation (8), $u_{\rm lts}(a_{ij})$ was calculated for the transfer standard (for values of a_{ij} at pressures of 130 kPa, 100 kPa, 30 kPa, 10 kPa, 3 kPa, 1 kPa, 300 Pa and 100 Pa) with the results shown in figure 5. This figure clearly shows that the long-term relative standard uncertainty of the RSG transfer standard package varies from a few parts in 10^6 at 130 kPa to as high as 0.01% at 100 Pa. Also shown in figure 5 are results (open diamonds) from the NIST piloted key comparison [1], which show that the long-term stability of 10 kPa RSG gauges (of the same model and type as used in this study) had very similar results to this study over the common range of pressures.

8. Conclusions

NIST has developed and tested a self-contained, portable RSG transfer standard suitable for intra-laboratory or international comparisons of pressure standards operating in the range of 100 Pa to 130 kPa (nominally 1 Torr to 1000 Torr). The transfer standard package has demonstrated a long-term calibration stability of a few parts per million (ppm) at 130 kPa, increasing to 0.01% at the lowest pressure of 100 Pa. This level of stability is nominally commensurate with piston gauges at pressures from 100 kPa down to 10 kPa, with the advantage of this transfer standard package being that it can extend the measurement capability down to 100 Pa.

Acknowledgments

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