# Comparison of the standards for high and ultrahigh vacuum at three national standards laboratories

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A preliminary phase of an international comparison of standards for high and ultrahigh vacuum was carried out by the National Institute of Standards and Technology (NIST), the National Physical Laboratory-Teddington (NPL-UK), and the Physikalisch-Technische Bundesanstalt (PTB). A spinning rotor gauge (SRG), a Bayard-Alpert gauge (BAG) and an extractor gauge (EXG) were chosen as transfer standards. The comparison was carried out in a star-like pattern with PTB as pilot laboratory. The argon pressures generated by the standards at  $9 \times 10^{-4}$  Pa were compared by measuring the accommodation coefficient of the SRG, the argon pressures from  $3 \times 10^{-7}$  Pa to  $9 \times 10^{-4}$  Pa by measuring the sensitivity of the two ionization gauges. The accommodation coefficients determined at NIST and PTB indicate a difference between the pressures generated by the NIST and PTB standards of  $(\mathcal{P}_{\text{NIST}} - \mathcal{P}_{\text{PTB}})/\mathcal{P}_{\text{PTB}} = (0.09 \pm 0.11)\%$  (standard or one-sigma uncertainty). For the pressures between  $3 \times 10^{-7}$  Pa and  $9 \times 10^{-4}$  Pa the results obtained at NIST and PTB had a mean difference of  $\langle (\mathcal{P}_{\text{NIST}} - \mathcal{P}_{\text{PTB}}) / \mathcal{P}_{\text{PTB}} \rangle = (0.24 \pm 0.12)\%$ , with a maximum difference of (1.2±0.6) at the lowest pressure. The NIST-PTB differences are all within the combined uncertainties of the two standards. Large transfer standard instabilities and inconsistent results in a first NPL-PTB comparison prompted a repeat set of measurements. An average of the two sets of SRG measurements indicates a significant difference between NPL and PTB standards of  $(\mathcal{P}_{NPL} - \mathcal{P}_{PTB})/\mathcal{P}_{PTB} = (1.31 \pm 0.14)\%$ . The two sets of ionization gauge measurements effectively repeated, but the results for the individual gauges are inconsistent. The EXG results indicate no significant pressure dependence in the difference between the standards for pressures below  $9 \times 10^{-4}$  Pa. The BAG results indicate a significant increase in the difference between the standards as the pressure is reduced, with  $(\mathcal{P}_{NPL} - \mathcal{P}_{PTB})/\mathcal{P}_{PTB}$  becoming as large as 5% or 6% at the lower pressures. Several potential problems with the BAG operation were identified, which might indicate that more confidence should be placed in the EXG results. However, there is no reliable evidence that the BAG comparison results were actually affected, and the results for each gauge are so internally consistent that neither can be rejected. Further work should be directed towards resolving this discrepancy. © 1997 American Vacuum Society. [S0734-2101(97)01604-7]

#### INTRODUCTION

During the past twenty years several comparisons<sup>1-5</sup> between vacuum standards have been performed (Table I). But, none of these comparisons extended to pressures below  $10^{-5}$  Pa. One difficulty with low pressure comparisons is the relatively large instability of ionization gauges (IGs), the only practical transfer standards below about  $10^{-4}$  Pa. However, the development of the spinning rotor gauge (SRG) as a suitable transfer standard in the high vacuum regime<sup>6</sup> allows the possibility of comparing standards with improved precision at some pressure above the SRG's lower operating limit, about  $10^{-4}$  Pa. Ion gauge data obtained at this same pressure can then be used to normalize lower-pressure ion gauge data, removing the effects of any pressure-independent systematic shifts in the ion gauge.

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For these reasons, the Low-Pressure Working Group of the CCM (Comite consultatif pour la masse et les grandeurs apparentees) decided at a 1993 meeting to carry out a comparison for pressures between  $3 \times 10^{-7}$  Pa and  $9 \times 10^{-4}$  Pa. To check the comparison procedure and the suitability of the chosen transfer standards a preliminary phase of the comparison was carried out by three laboratories with extensive experience with high and ultrahigh vacuum gauge calibrations: National Institute of Standards and Technology (NIST), National Physical Laboratory-Teddington (NPL-UK), and Physikalisch-Technische Bundesanstalt-Berlin (PTB). The PTB served as the pilot laboratory and a star-like series of measurements was chosen with an initial calibration sequence: PTB(1), NIST, PTB(2), NPL(1), PTB(3). The NPL results appeared to be internally inconsistent, so the NPL-PTB comparison was repeated one year later by extending the calibration sequence with PTB(4), NPL(2), and PTB(5). The PTB also analyzed the data and provided the

Table I. Published international comparisons of vacuum standards since 1975. CMU—Ceskoslovenský Metrologický Ústav, formerly CSSR: ETL—Electrotechnical Laboratory, Japan; IMGC—Istituto di Metrologia "G. Colonetti." Italy: LIP—Lanzhou Institute of Physics, China; LNE—Laboratoire National d'Essais, France; NIM—National Institute of Metrology, China; NIST—National Institute of Standards and Technology, USA; NPL—National Physical Laboratory, Great Britain; NPL/I—National Physical Laboratory, India; PTB—Physikalisch-Technische Bundesanstalt, Germany; SOGEV—Laboratoire de Metrologia de L.H. Sogev, France.

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Date of final publication	Pressure range (Pa)	Participating laboratories	Reference
1975	$2 \times 10^{-3}$ to $5 \times 10^{-3}$	NPL, PTB	1,2
1978	$8 \times 10^{-5}$ to $8 \times 10^{-2}$	NPL, IMGC,	2
		SOGEV, PTB	
1989	$10^{-4}$ to 1	PTB, LNE, IMGC	
		NPL, CMU, NIM,	
		NPL/I, NIST, ETL	3
1989	$10^{-3}$ to 1	PTB, NPL/I	4
1992	$2 \times 10^{-2}$ to 0.2	LIP, PTB	5

transfer standards: one SRG, one Bayard-Alpert ionization gauge (BAG) and one extractor ionization gauge (EXG).

#### II. DETAILS OF COMPARISON

The standards to be compared are pressure generators. A value, p, of the true generated pressure,  $\mathcal{P}$ , can be calculated from the operating parameters of the standards, as explained in the references cited in Section II A. The calculated pressure p is only an approximation to the generated pressure P, with a probable limit of error given by the specified uncertainty of the standard. While P can never be known exactly, the transfer standards used for this comparison, ion and spinning rotor gauges, described in Section II B, respond directly to  $\mathcal{P}$  with an observable ion current or deceleration rate determined by  $\mathcal P$  and a true gauge characteristic or sensitivity. With appropriate corrections for operating parameters (e.g., temperature, emission current), calibration data for these gauges, obtained with a particular standard, can be used to calculate a sensitivity or gauge characteristic, as described in Section II C, that is directly proportional to the generated pressure and inversely proportional to the calculated pressure for that particular standard. Thus, from the differences between sensitivity values obtained with different standards at the same value of the calculated pressure, we can determine the differences between the true pressures generated by the different standards.

It is not possible, of course, to perform the calibrations with the different standards at exactly identical values of the calculated pressure, but since the gauge sensitivities or characteristics are slowly varying functions of the pressure, we can readily extrapolate to equivalent values at the specified calculated pressures. Ideally, the true gauge characteristics should not change between the calibrations at the different standards. In practice, such changes are a significant prob-

lem, and several steps, described in the text, were taken to minimize such changes and to determine their effect in evaluating the data.

It can also be noted that, as in any comparison of standards, we can only determine differences and not absolute values. Therefore, there is some probability that standards found to be in "good" agreement might share a significant common systematic source of error. The probability of such undetected sources of error is decreased as the number and design variety of compared standards increase.

# A. The primary standards under comparison

The NIST and PTB standards for the range of this comparison are based on the continuous-expansion method (also called dynamic-expansion or orifice-flow method); the NPL standard is based on the series-expansion method (also called the static- or volume-expansion method).

The NIST ultrahigh vacuum standard (full calibration range  $1\times10^{-7}$  Pa to  $1\times10^{-1}$  Pa) was used in the present comparison. It is geometrically similar to, but somewhat larger than, the NIST high vacuum standard. The constant-pressure flowmeter, which is common to both NIST vacuum standards, is described in Ref. 8. Subsequent improvements have resulted in reduced uncertainties compared to the uncertainties given in Refs. 7 and 8.

The PTB standard is described in Refs. 9 and 10, and the constant-pressure flowmeter in Ref. 11. Although similar in design and operation, the PTB and NIST standards differ in several details, principally the pump (PTB uses a cryopump, while NIST uses a turbomolecular pump), base pressure (see below), and the size of the orifices (the PTB orifice is 34 mm in diameter, the NIST orifice is 11 mm in diameter). The calibration range of the PTB continuous expansion standard starts at  $3 \times 10^{-7}$  Pa and ends at  $1.5 \times 10^{-3}$  Pa.

The NPL high vacuum series-expansion standard uses four expansion stages, the first two of which are part of an earlier standard described in Ref. 12. This standard is normally used to generate pressures in the range  $1 \times 10^{-6}$  Pa to 10 Pa, although it has the potential for operation at both higher and lower pressures. For this comparison purposes its operation was extended to pressures below  $1 \times 10^{-6}$  Pa.

Table II gives the uncertainties of the calculated pressures over the relevant range as specified by each laboratory at the time of the comparison. The values in Table II include only uncertainties due to systematic effects; uncertainties due to random effects were omitted (they will be reflected in the standard deviations of the comparison data). Following current recommendations, <sup>13</sup> quadratic addition of the individual uncorrelated uncertainties is used throughout this article and, in keeping with the convention for reporting the results of comparisons of primary standards, all uncertainties are standard or one-sigma uncertainties.

The pressures calculated from the operation of these standards are additive to a residual or base pressure. Typical residual pressures measured with the transfer extractor gauge

TABLE II. Relative standard uncertainties (one sigma) due to systematic effects in the calibration pressures of the standards included in this comparison, as stated by each laboratory (quadratic addition of individual uncorrelated uncertainties). Uncertainties due to random effects are not included.

Pressure	Re	elative uncertainty (	%)
(Pa)	NPL	NIST	PTB
3×10 <sup>-7</sup>	***	0.61	0.24
$5 \times 10^{-7}$	0.55	0.58	0.24
$9 \times 10^{-7}$	0.46	0.54	0.24
$3 \times 10^{-6}$	0.42	0.54	0.24
$5 \times 10^{-6}$	0.41	0.43	0.24
9×10 <sup>-6</sup>	0.41	0.33	0.24
$3 \times 10^{-5}$	0.41	0.21	0.13
$5 \times 10^{-5}$	0.41	0.20	0.13
$9 \times 10^{-5}$	0.41	0.20	0.13
$3 \times 10^{-4}$	0.32	0.17	0.13
5×10 <sup>-4</sup>	0.32	0.17	0.13
9×10 <sup>-4</sup>	0.32	0.17	0.13

were  $5 \times 10^{-9}$  Pa at PTB (after 300 °C bakeout for 50 h),  $2 \times 10^{-8}$  Pa at NIST (260 °C, 16 h), and  $1 \times 10^{-7}$  Pa at NPL (250 °C, 18 h).

#### B. The transfer standards

The primary focus of this work is a comparison in the high and ultrahigh vacuum range where the only practical transfer standards are ionization gauges. As previously mentioned, two different types of ionization gauges were used as transfer standards: a Bayard-Alpert gauge and an extractor ionization gauge; both gauges have yttrium oxide coated iridium cathodes. The EXG allows a better measure of the base pressures since its residual (zero-pressure) collector current is one to two orders of magnitude smaller than that of a BAG. However, the stability of ion gauges is less than desirable for this comparison. Therefore, an additional transfer standard, the spinning rotor gauge with better demonstrated stability was included. This was used to establish the difference between the standards at  $9 \times 10^{-4}$  Pa, a pressure that can be conveniently measured by all the gauges, and, in effect, recalibrate or renormalize the ion gauges at this pressure with respect to the reference laboratory's standard.

Each nude ionization gauge head was mounted into a standard vacuum plumbing elbow and each gauge/elbow unit attached symmetrically to an all metal "T" valve. This assembly, which remained intact for the entire duration of the comparison, ensured that the electric potential distribution around each gauge and the thermal and molecular conductances between each gauge and the vacuum chamber were identical in each lab. This assembly was evacuated and sealed (T valve closed) when transporting the gauges.

The pressures in the isolated T valve and gauge assembly were estimated after each transportation using the SRG. The pressures were typically between 0.1 Pa and 1 Pa, however higher pressures (100 Pa), caused by leaks of the valves during transportation, were found after the NIST-PTB transfer.

There is no apparent correlation between the pressure after transport and changes in the calibration of the transfer gauges.

The ionization gauges were mounted on each standard with their axes vertical. A commercial ion gauge controller supplied all necessary voltages and currents. The applied potentials with respect to ground were 220 V for the anode, 80 V (100 V) for the BAG (EXG) cathode, and 205 V for the ion repeller of the EXG. Emission currents in each ion gauge (BAG: 0.6 mA, EXG: 1.6 mA) were measured with a precision ammeter inserted into the anode circuit. All ion currents were measured with the same picoammeter. All these instruments, including cables, were part of the transfer set to ensure the largest possible consistency between the laboratories

The SRG rotor and thimble assembly included a special transport device<sup>14</sup> that ensured that the rotor (stainless steel, 4.76 mm diameter) was kept under vacuum and could not move in the thimble during transportation. Each laboratory used its own rotor suspension head and controller to operate the SRG. A considerable body of experience in our laboratories indicates that, in the range of this comparison, interchanging suspension heads and control units does not affect the reliability of the readings.

# C. Comparison procedure

#### 1. Spinning rotor gauge

Comparison of the standards at  $9 \times 10^{-4}$  Pa was accomplished by comparing the values determined in each laboratory for the pressure-independent accommodation coefficient  $\sigma$  of the SRG rotor, <sup>15</sup> which is the calibration constant of the SRG. It is also known as the *effective* accommodation coefficient since it accounts for the surface roughness of the ball as well as the tangential-momentum accommodation coefficient. The measured accommodation coefficient is given by

$$\sigma = \frac{\pi}{20} \left( \frac{-\omega}{\omega} \right) \frac{\rho d}{p_{\text{std}}} \overline{c}(T), \tag{1}$$

where  $\rho$  and d denote the density and diameter of the rotor,  $p_{\rm std}$  the calculated value of the calibration pressure,  $-(\omega/\omega)$  the measured fractional rate of change of the rotor's angular speed due to molecular drag (the measured deceleration rate corrected for a pressure-independent residual drag) and  $\overline{c}(T)$  is the average molecular speed given by

$$\overline{c}(T) = \sqrt{\frac{8RT}{\pi M}}. (2)$$

Each laboratory used the same parameter values for  $\rho$ , d and M (the argon test gas molecular weight) and the universal gas constant R. The local gas temperature T was measured by each laboratory. For each standard, the accommodation coefficient was determined at  $9 \times 10^{-4}$  Pa each time either ionization gauge was calibrated at this pressure. This served to detect any anomalous behavior of the standards or ionization gauges.

#### 2. Ionization gauges

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For the ion gauges the measurand to be compared is the argon sensitivity S (also called ion gauge constant), defined as

$$S = \frac{(I_c - I_{c0})}{(p - p_0)I_e},\tag{3}$$

where  $p_0$  is the pressure due to residual gases in the calibration chamber, p is the pressure due to the residual gases plus standard's calculated calibration  $p_{\text{std}}(p=p_0+p_{\text{std}})$ ,  $I_c$  is the collector current at pressure p,  $I_{c0}$  the collector current at residual pressure  $p_0$ , and  $I_e$  the emission current. NIST and PTB made an emission current measurement with each ion current measurement; NPL measured the emission current at base pressure, just prior to the generation of a calibration pressure. Since the standards operated at different temperatures, and ion gauges are sensitive to gas density, it was necessary to multiply the calculated sensitivities by the ratio of the absolute temperature of the standard divided by 296.15 K to determine the equivalent sensitivity values at a common reference temperature of 23 °C. In so doing, we are assuming that the temperature difference between the IGs and the standards (due to cathode heating) is the same in each laboratory.

Sensitivities were determined in a sequence of increasing calibration pressures starting at  $3 \times 10^{-7}$  Pa for PTB and NIST (extractor gauge), at  $5 \times 10^{-7}$  Pa for NPL, and  $9 \times 10^{-7}$  Pa for NIST (BA gauge) and continuing up to  $9 \times 10^{-4}$  Pa in a three-points-per-decade pattern  $(3 \times 10^{-j}, 5 \times 10^{-j}, \text{ and } 9 \times 10^{-j}, \text{ with } j = 7, 6, 5, \text{ and } 4)$ . The sensitivity was measured at least twice at each calibration pressure. The calibrations were repeated at least once on another day at a minimum of four pressures, including  $3 \times 10^{-6}$  Pa,  $3 \times 10^{-5}$  Pa,  $9 \times 10^{-5}$  Pa, and  $9 \times 10^{-4}$  Pa.

Detailed calibration procedures are determined in part by the operating procedures of the primary standards, which differ significantly. In particular, volume-expansion standards start each calibration point at base pressure with a measurement of  $I_{c0}$  and reach the calibration pressure in a fraction of a minute after the initiation of the gas expansion. However, at low pressures the generated pressure changes at a significant rate due to surface adsorption, outgassing, and gauge pumping, so that the calibration data must be obtained within 1 or 2 min from the initiation of the expansion, and only one gauge is calibrated at a time. Orifice-flow systems typically take 5-15 min to change calibration pressures and another 5-20 min to determine the pressure but, once achieved, can maintain the pressure stable to 0.1% or better almost indefinitely. Typically, multiple gauges are calibrated at the same time, and NIST operated both the EXG and BAG at all times, although the electronics used with the BAG and EXG permitted the calibration of only one gauge at a time. Orifice-flow calibrations start with a determination of  $I_{c0}$  at base pressure, followed by the generation of one or more calibration pressures; PTB returned to base pressure after each calibration point, while NIST generated a series of increasing calibration pressures through the course of a day. without any intervening return to base pressure. These operating characteristics, and the consequent calibration procedures, are in effect a part of the standards to be compared. Therefore, in general, each laboratory was expected to followits normal operating and data taking procedures.

One exception is that all laboratories agreed to adopt a gauge-conditioning procedure developed by the pilot laboratory (PTB). 16,17 Each laboratory initially baked its vacuum standard, with transfer gauges attached and isolation valve open, to at least 250 °C or higher, according to its normal procedure. During cooldown, between 150 °C and 100 °C, the EXG was degassed by electron bombardment of the anode (grid) with 45 mA of 480 eV electrons for 3 min; for the BAG the current was increased to 90 mA. On the day before the first calibration, the ion gauges were degassed a second time, while at room temperature, in the same manner. After this degassing the two ion gauges were operated simultaneously at  $10^{-3}$  Pa argon pressure for 1 h. The electron bombardment primarily cleans the anode, whereas the highpressure operation cleans the ion collector surface by ion bombardment to achieve a stable secondary electron production at the collector. 18,19 After this initial treatment, no further degassing was performed. Following this procedure, at least 12 h elapsed before the calibration began. The efficacy of this procedure in stabilizing ion gauge sensitivity is discussed in Section III, and is reflected in the overall ion gauge stability observed during the comparison.

This change of conditioning procedure is of concern principally in its effect on the stability of  $p_0$  and  $I_{c0}$ . It can be seen from Eq. 3 that  $p_0$  and  $I_{c0}$  must be stable relative to pand  $I_c$  over the time between the measurements at base pressure and the completion of a calibration point. This is of little concern for the NPL volume-expansion standard since only a few minutes elapse between the measurement of  $I_{c0}$  and the completion of a calibration point. However, for the NIST orifice-flow standard it can be 1 h between the measurement of  $I_{c0}$  and the completion of the first point of a calibration sequence, with additional elapsed times of the same magnitude for subsequent points. Since the gauge-conditioning procedure used in this comparison differs from that normally used at NIST, NIST monitored  $I_{c0}$  before the generation of calibration pressures and found that the rate of change never exceeded the equivalent of 2×10<sup>-11</sup> Pa/h, which will not cause a significant error for even the lowest-pressure points. At any pressure, electron-stimulated desorption (ESD) of ions from the anode (grid) can contribute to the measured collector current  $I_c$  . We believe that under our operational conditions ESD is a negligible effect. However, if ESD is a significant effect, it could be pressure dependent, with a consequent time dependence as the surface coverage of the grid equilibrates with the ambient pressure. At each calibration pressure NIST monitored the total collector current  $I_c$  and found that it never drifted by more than 0.1%/h, which can be explained by instabilities of the ambient temperature. These results are effectively the same as those observed at NIST after the normal NIST conditioning procedure, so we

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#### p. Evaluation of experimental data and uncertainties

As noted eariler, we are comparing the standards by comparing calculated transfer standard characteristics or sensitivities, specifically, the accommodation coefficient  $\sigma$  of the spinning rotor gauge and the sensitivities S of the two ionization gauges. In general, the gauge characteristics as determined by each participating laboratory can differ for three reasons: (i) differences of the pressures generated by the standards at the same specified pressure, (ii) random errors, and (iii) instabilities or differing responses of the transfer standards. Lacking other information, the measured difference in gauge characteristics is ascribed to (i)-a difference in the standards. But this indicated difference must be interpreted within the context of the possible errors caused by (ii) and (iii). The resultant uncertainties can be both large and difficult to evaluate. Vacuum calibrations are slow and difficult so that the repetition of data to reduce the effects of random errors is limited by practical considerations. Further, significant shifts in ion gauge sensitivity are possible. These problems prompted efforts to estimate the instability of the transfer standards and develop a meaningful uncertainty analysis from the limited data available.

The instability of the transfer gauges under laboratory conditions was examined in the pilot laboratory before the comparison and is described in Section III. However transportation and exposure to different environments can cause even larger instabilities and can be estimated only from the comparison data itself. For the SRG this estimate and the uncertainty analysis is relatively straightforward due to the smaller magnitude of the instabilities and larger amounts of data, and is described in Section IV A. For the ionization gauges, however, we first make some general comments and then outline the procedure in the following paragraphs.

At the time of the calibration there exists a value of the sensitivity, that we will call the "true" value  $S^{\text{true}}$ . We can only approximate this value by a calculated value S, which differs from  $S^{\text{true}}$  due to systematic and random errors. It is also possible that  $S^{\text{true}}$  can change with time and use; in particular,  $S^{\text{true}}$  might change between the time it is calibrated in one laboratory and the time it is calibrated at another. One type of change, sometimes observed in ionization gauge calibration data, is the change of the sensitivity by a pressure-independent factor. To minimize the effects of this type of change (and reduce the data to a readily comprehensible form), the sensitivity data were normalized by the sensitivity at  $9 \times 10^{-4}$  Pa,

$$S_{nc}(p) = \frac{\langle S(p) \rangle}{\langle S(9 \times 10^{-4} \, \text{Pa}) \rangle},\tag{4}$$

where  $S_{nc}$  is the "normalized characteristic." Each  $\langle S(p) \rangle$  is the mean of repeated values obtained at a given pressure during a calibration procedure (typically one day's data). Note that the SRG data provide an independent comparison of the standards at  $9 \times 10^{-4}$  Pa and, to the extent that the

SRG is stable, transfer the pressure scale at  $9 \times 10^{-4}$  Pa of one laboratory (a) to another (b). Thus, simultaneous SRG and ion gauge data taken at (b) can be used to correct the original calibration of the ion gauge at (a) for any changes in the ion gauge true sensitivity that have occurred since leaving (a). To within the stability of the SRG, this eliminates pressure-independent shifts in the ion gauge sensitivity.

For pressures below  $9 \times 10^{-4}$  Pa the relative standard deviation of  $S_{nc}(p)$  is approximated by

$$\frac{\Delta S_{nc}(p)}{S_{nc}(p)} = \sqrt{\frac{\left[\Delta \langle S(p)\rangle\right]^2}{\langle S(p)\rangle^2} + \frac{\left[\Delta \langle S(9\times10^{-4}\,\text{Pa}\rangle\right]^2}{\langle S(9\times10^{-4}\,\text{Pa}\rangle^2}},\quad(5)$$

where  $\Delta \langle S(p) \rangle$  are the experimental standard deviations of the mean.

Because of the limitations in obtaining repeated data it is difficult to obtain a "representative" value of the standard deviation  $\Delta S(p)$  at any given pressure; in many cases there are only two data points at a given pressure and the calculated standard deviations have large variations as a function of pressure. The values used in this report are "smoothed" values obtained from zero to third order polynomials fitted as a function of pressure to the calculated standard deviations for each laboratory's data.

In each laboratory at least two characteristics were measured on different days. The normalized data [Eq. (4)] for the different days were averaged at each target calibration pressure, and are indicated as

$$S_{nc}^{i}(p) = \langle S_{nc}(p) \rangle. \tag{6}$$

The index "i" stands for the final result of the calibration sequence i (i=PTB(1), NIST, PTB(2), NPL(1), PTB(3), PTB(4), NPL(2), or PTB(5).

At a later stage in the analysis, the data at  $9 \times 10^{-4}$  Pa are further normalized to the initial pilot laboratory calibration, PTR(1)

As will be seen in Sections IV B and IV C, for some of the data the dominant uncertainties were caused by systematic changes in the ion gauge characteristics. The effects of these changes are much more difficult to quantify than the imprecision of repeated data.

# III. PRE-COMPARISON TESTING OF THE TRANSFER GAUGES

The stability of the transfer gauges was investigated at the pilot laboratory before undertaking the comparison. While the IGs were new at the start of this investigation, calibration data for the SRG rotor have existed since 1986, when this rotor was used in an earlier CCM comparison.<sup>3</sup> The history of the accommodation coefficient values determined at PTB is shown in Table III, where all data have been normalized to the accommodation coefficient measured at PTB(1) in November 1993. Over this seven year period the rotor changed its accommodation coefficient by only 0.7% relative to the value determined in November 1993 [PTB(1)]. If the first measurement of the freshly prepared rotor is omitted, a sys-

TABLE III. Historical values of the accommodation coefficient of the transfer rotor since 1986, all normalized to PTB(1). All values except PTB(1) were measured on the same PTB standard.

Date	$\sigma/\sigma_{\text{PTB(I)}}$
9/86	1.0071
10/86	1.0049
6/87	1.0050
7/87	1.0041
7/88	1.0034
9/93	1.0001
11/93ª	1.0000

<sup>a</sup>PTB(1).

tematic drift of only about  $7 \times 10^{-4}$  per year could be estimated and therefore this rotor appeared to be suitable as a transfer standard.

To check the stability of the ion gauges, they were mounted onto a static expansion standard and their sensitivities were repeatedly determined over a two month period at  $5 \times 10^{-5}$  Pa, a pressure in the middle of the agreed pressure range of the comparison. During this testing period, the effects on the gauges of several conditioning treatments were investigated. During this time the gauges were not removed from the system nor exposed to atmospheric air.

The sensitivities were normalized to the average of selected values (see below) and the results for the normalized sensitivities are shown in Figs. 1 and 2. It can be seen that the BA gauge had a systematically higher sensitivity after degassing. Ion bombardment of the collector (operating the gauge at higher pressure, see Section II C) brought the sensitivity back to the original value. This is the reason why we chose not to repeat degassing alone between calibrations. Degassing with succeeding ion bombardment, however, was allowed to recondition the gauge. After recalibration or ion bombardment the sensitivity was sufficiently stable. Excluding those results obtained after degassing, the relative standard deviation about the mean is 0.21% for the BAG.

Since the extractor gauge did not show this effect after degassing, the value (0.45%) of the relative standard devia-

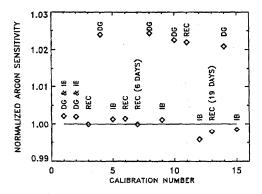


Fig. 1. Normalized sensitivities (see the text) of the Bayard-Alpert gauge over a two month period before the comparison. DG: degassing; IB: ion bombardment of collector (see the text); REC (recalibration on the same day or several days later).

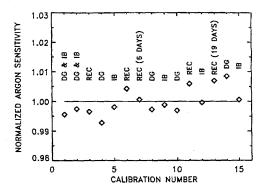


Fig. 2. Normalized sensitivities (average=1.00) of the extractor gauge over a two month period before the comparison. For inscriptions see explanations to Fig. 1.

tion about the mean is based on the entire set of data. These values were used as estimates of the instability in these gauges before the comparison.

We believe the differing behaviors of the BAG and EXG are due to their different geometries; the collector of the BAG is fully exposed to the grid, whereas the EXG collector is shielded from its grid. During electron bombardment there is a significant probability that material outgassed from the BAG grid can contaminate its collector, whereas the probability of this happening in the EXG is much smaller.

The pilot laboratory also performed five calibrations [PTB(1), PTB(2), PTB(3), PTB(4), and PTB(5)] during the course of the comparison for each transfer standard. These data can be used to estimate long-term instabilities including transportation effects (see Section IV). These data indicate that during the course of the comparison each transfer standard showed transportation-related changes whose magnitude clearly exceeded both short-term random instabilities and the changes observed during laboratory testing. The magnitudes of these changes were large enough that it was difficult to establish a direct comparison between NIST and NPL. Therefore, we will present this comparison as two bilateral comparisons, NIST-PTB and NPL-PTB, instead of one multilateral comparison.

# IV. REDUCTION OF DATA AND ASSIGNMENT OF UNCERTAINTIES

# A. Spinning rotor gauge data at $9 \times 10^{-4}$ Pa

Each laboratory determined the rotor's accommodation coefficient  $\sigma$  at least eight times at  $9\times10^{-4}$  Pa. For each set of measurements i, where again i=PTB(1), NIST, PTB(2). NPL(1), PTB(3), PTB(4), NPL(2), PTB(5), the mean value  $\langle \sigma^i \rangle$  and the standard deviation of the mean were calculated. The mean values, normalized by the mean value for the initial set of results at PTB(1), are given by

$$\sigma_n^i = \frac{\langle \sigma^i \rangle}{\langle \sigma^{PTB(1)} \rangle},\tag{7}$$

and presented in Table IV.

TABLE IV. The mean accommodation coefficients  $\sigma_n^i$  and their standard deviations as measured in the eight calibration sequences. All values are normalized to PTB(1).

Calibration sequence i	$\sigma_n^i$	$(\Delta \sigma_n^i)$
PTB(1)	1.0000	0.0007
NIST	1.0001	0.0005
PTB(2)	0.9984	0.0007
NPL(1)	1.0072	0.0004
PTB(3)	0.9927	0.0003
PTB(4)	0.9932	0.0004
NPL(2)	1.0075	0.0006
PTB(5)	0.9932	0.0012

To compare the generated pressures at  $9 \times 10^{-4}$  Pa between the different laboratories a procedure similar to that described in Ref. 3 was adopted. In the absence of other information, the most plausible reference value  $\sigma_n^{\rm ref(i)}$  for laboratory i is the arithmetic mean of the calibrations at the pilot laboratory before and after the transport. That is,

$$\sigma_n^{\text{ref(NIST)}} = \frac{(\sigma_n^{\text{PTB}(1)} + \sigma_n^{\text{PTB}(2)})}{2},$$

$$\sigma_n^{\text{ref(NPL}(1/2))} = \frac{(\sigma_n^{\text{PTB}(2/4)} + \sigma_n^{\text{PTB}(3/5)})}{2},$$
(8)

where the second line indicates that the reference for the NPL(1) data is derived from PTB(2) and PTB(4), and that the reference for the NPL(2) data is derived.

The uncertainty of these reference values depends in part on the standard deviations  $\Delta \sigma_n$  of the before and after values determined at the pilot laboratory. However, if the differences between the before and after pilot laboratory calibrations significantly exceed the standard deviations of the pilot lab calibrations, then it is probable that a significant systematic change has occurred in the characteristic of the gaugemost likely due to transportation between the pilot and participant laboratories. Unfortunately, such changes were not unusual during this comparison. However, since we do not understand the mechanisms that cause these changes, we cannot say how much change occurred between the pilot laboratory and the participating laboratory, and how much on the return transport, i.e., there is no guarantee that the mean value [Eq. (8)] is the actual value of the characteristic during the participant laboratory calibration. Therefore, in evaluating the uncertainty of the reference value we must also take into account a transport uncertainty. Unfortunately, we have only one relevant data point, the difference between the before and after calibrations, so we can only make a plausible assumption as to the likely changes in the gauge. We assume that the standard deviation of the mean value—half the difference between the before and after calibration valuesrepresents a one-sigma transportation uncertainty. Similar uncertainties were assumed in an earlier comparison.3 Therefore the variances of the reference values  $\sigma_n^{\text{ref}(i)}$  are

TABLE V. Results of the comparisons at  $9 \times 10^{-4}$  Pa. Calculated from Eq. (8),  $\sigma_n^{\text{ref}}$  are the pilot laboratory reference values and  $\Delta \sigma_n^{\text{ref}}$ , from Eq. (9), are their standard deviations, including an allowance for transfer instabilities.  $R_i$  are the ratios of the accommodation coefficients measured at  $9 \times 10^{-4}$  Pa, calculated using Eq. (10), and  $\Delta R_i$  are the standard deviations of those ratios, including transport instabilities.

Comparison	$\sigma_n^{\mathrm{ref}}$	$\Delta  \sigma_n^{\mathrm{ref}}$	$R_i$	$\Delta R_i$
NIST-PTB	0.9992	0.0010	1.0009	0.0011
NPL(1)-PTB	0.9956	0.0029	1.0117	0.0029
NPL(2)-PTB	0.9932	0.0006	1.0144	0.0009

$$(\Delta \sigma_{n}^{\text{ref(NIST)}})^{2} = \frac{1}{4} \left[ (\Delta \sigma_{n}^{\text{PTB}(1)})^{2} + (\Delta \sigma_{n}^{\text{PTB}(2)})^{2} + (\sigma_{n}^{\text{PTB}(2)} - \sigma_{n}^{\text{PTB}(1)})^{2} \right],$$

$$(\Delta \sigma_{n}^{\text{ref[NPL(1/2)]}})^{2} = \frac{1}{4} \left[ (\Delta \sigma_{n}^{\text{PTB}(2/4)})^{2} + (\Delta \sigma_{n}^{\text{PTB}(3/5)} - \sigma_{n}^{\text{PTB}(2/4)})^{2} \right],$$

$$(9)$$

$$+ (\sigma_{n}^{\text{PTB}(3/5)} - \sigma_{n}^{\text{PTB}(2/4)})^{2} \right],$$

where  $\Delta$  indicates a standard deviation. The normalized mean result for each participating laboratory [Eq. (7)], is divided by the appropriate reference value [Eq. (8)] to obtain the ratio

$$R_i = \frac{\sigma_n^i}{\sigma_n^{\text{ref}(i)}}. (10)$$

The relative standard deviation of this ratio is approximated by

$$\frac{\Delta R_i}{R_i} \approx \sqrt{\left(\frac{\Delta \sigma_n^i}{\sigma_n^i}\right)^2 + \left(\frac{\Delta \sigma_n^{\text{ref(i)}}}{\sigma_n^{\text{ref(i)}}}\right)^2}.$$
 (11)

Table V summarizes the data and uncertainties for the comparison using the SRG transfer standard.

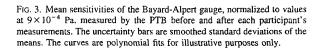
# B. Bayard-Alpert ionization gauge data

As outlined in Sections II C and II D, the measured ion gauge sensitivity values at each pressure are averaged and normalized by the sensitivity value at  $9 \times 10^{-4}$  Pa [Eq. (4)]. For each laboratory, the average value at  $9 \times 10^{-4}$  Pa is normalized by the initial value obtained by the pilot laboratory, PTB(1), i.e.,  $\langle S^i(9 \times 10^{-4} \text{ Pa}) \rangle / \langle S^{\text{PTB}(1)}(9 \times 10^{-4} \text{ Pa}) \rangle$ . These normalized values for the BA gauge are presented in

TABLE VI. The average Bayard-Alpert gauge sensitivities  $S_n$  at  $9 \times 10^{-4}$  Pa, normalized to PTB(1), and their experimental standard deviations as measured in the eight calibration sequences.

Calibration sequence	$S_n$	$(\Delta S_n)$
PTB(1)	1.000	0.001
NIST	1.002	0.000
PTB(2)	1.008	0.010
NPL(1)	0.975	0.004
PTB(3)	0.993	0.002
PTB(4)	0.986	0.009
NPL(2)	0.991	0.005
PTB(5)	0.999	0.004

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Table VI, along with their standard deviations. Assuming stability of the pilot laboratory standard, the five PTB values give an indication of the absolute stability of the sensitivity at this pressure; the differences between these values are three to eight times larger than the preliminary test results (0.21%) described in Section III, the largest changes apparently reflecting the effects of transport between laboratories.

The averaged and normalized BAG sensitivities obtained from Eq. (6) for the five PTB measurement sequences are shown in Fig. 3. For easier recognition of the data, polynomials of third or fourth order were fitted to the  $S_{nc}^{l}$  vs log(p) data. It is evident that significant pressure dependent changes occurred. A difference as large as 3.6% in the normalized sensitivity is observed at the lowest pressures between PTB(1) and PTB(2). These differences again significantly exceed the standard deviations obtained during preliminary testing and the standard deviations of the calibration data, indicated by the vertical bars in Fig. 3 [the standard deviations of PTB(2) are higher because of a power supply failure at PTB that necessitated reconditioning of the ion gauge during data collection for PTB(2)]. In summary, between PTB(1) and PTB(2) there was a systematic change in the characteristic  $S_{nc}^{i}(p)$  from practically pressure independent to a small but significant pressure dependence.

The change in the fundamental characteristic of the BAG between the time it left PTB, was shipped to NIST, and returned to PTB clearly challenges its reliability as a transfer standard. However, when the normalized characteristics for PTB(2) and NIST are compared, as illustrated in Fig. 4 (for purposes of clarity, only the fitted lines are included for the PTB data, but all data analyses are based on actual data), it can be seen that there is very good agreement at all pressures, well within the combined standard deviations of the two calibrations. This agreement is possible only if two conditions are met; the two standards are in essential agreement, and there was little or no change in the transfer standard between NIST and PTB(2). In other words, it is extremely unlikely that at all pressures the shifts in the gauge characteristic between NIST and PTB(2) almost exactly cancel any

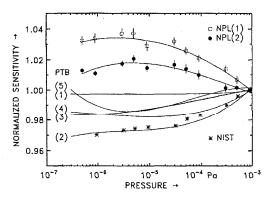


Fig. 4. Mean sensitivities of the Bayard-Alpert gauge, normalized to values at  $9\times10^{-4}$  Pa, measured by the participating laboratories. Uncertainty bars are smoothed standard deviations of the means; the NIST standard deviations are less than the symbol size. Pilot laboratory results are indicated by the fitted lines from Fig. 3.

differences between the standards. In effect, apart from a change in the normalizing factor, the gauge appears to have the same characteristic when calibrated at NIST and at PTB(2). This argument is buttressed by the good agreement between PTB and NIST obtained using the EXG, as presented in Section IV C. Therefore, we conclude that the large changes in the BAG occurred between the PTB(1) and NIST, and that PTB(2) is a much more valid reference for comparison with NIST than is the average of PTB(1) and PTB(2).

Accepting PTB(2) as the reference to be compared with NIST, we still must allow for the possibility of a small change in the BAG between NIST and PTB(2) beyond those accounted for by the normalizing process, i.e., it is still necessary to estimate the transport uncertainty. We believe that the differences between PTB(1) and PTB(2) are anomalous, so for lack of any better information, we will assume that one-half the difference between PTB(3) and PTB(2) represents an equivalent one-sigma limit on changes in the gauge characteristic between NIST and PTB(2). Since this difference (Fig. 3) is relatively constant for most of the pressure range, we will use a value averaged for all pressures, i.e.,

$$\Delta_{\text{trans}} = \left(\frac{1}{2N}\right) \sum_{j=1}^{N} 2 \frac{\left|S_{nc}^{\text{PTB}(2)}(p_j) - S_{nc}^{\text{PTB}(3)}(p_j)\right|}{S_{nc}^{\text{PTB}(2)}(p_j) + S_{nc}^{\text{PTB}(3)}(p_j)}.$$
 (12)

The average difference between the normalized characteristics for PTB(3) and PTB(2) for the BAG is 1.3%, so  $\Delta_{trans} = 0.65\%$ .

In the case of the NPL(1) data, there is no indication that either PTB(2) or PTB(3) is a superior representative of the gauge characteristic when it was calibrated by NPL. Therefore, we use the average of PTB(2) and PTB(3) as the reference to be compared with NPL(1). The transport relative uncertainty for this reference is derived from the previously discussed average difference, and from Eq. (12)  $\Delta_{\text{trans}} = 0.65\%$ . For NPL(2) we use the average of PTB(4) and PTB(5) as the reference. However, the differences between PTB(4) and PTB(5) have a significant pressure dependence: therefore we use one-half the difference at each pressure as the transport uncertainty at that pressure.

TABLE VII. The extractor gauge sensitivities  $S_n$  at  $9 \times 10^{-4}$  Pa, normalized to PTB(1), and their experimental standard deviations as measured in the eight calibration sequences.

Calibration sequence	$S_{n}$	$(\Delta S_n)$
PTB(1)	1.000	0.003
NIST	1.008	0.003
PTB(2)	1.016	0.005
NPL(1)	1.012	0.005
PTB(3)	0.888	0.005
PTB(4)	0.947	0.003
NPL(2)	0.920	0.001
PTB(5)	0.923	0.006

#### C. Extractor ionization gauge

Table VII presents the average extractor gauge sensitivities at  $9 \times 10^{-4}$  Pa of the various sequences normalized to the value determined by PTB(1). The changes between the pilot laboratory calibrations are significantly larger than those observed for the BAG, in particular, a 12.6% change occurred between PTB(2) and PTB(3).

The averaged and normalized pilot laboratory EXG sensitivities are presented in Fig. 5, and Fig. 6 includes the normalized NIST and NPL data. The differences between the normalized characteristics are relatively small and pressure independent for PTB(1) and PTB(2), and comparable to or slightly larger than the combined standard deviations. There is no evidence that either PTB(1) or PTB(2) is more representative of the gauge characteristic while it was at NIST, therefore we use the average of the normalized characteristics PTB(1) and PTB(2) as the reference for the NIST comparison. Again as in Section IV A, we use the average difference between the normalized characteristics PTB(1) and PTB(2), 0.65%, to calculate the transport equivalent relative standard deviation for this reference,  $\Delta_{trans} = 0.33\%$ .

In addition to the previously noted large change at  $9 \times 10^{-4}$  Pa, there is a large pressure-dependent difference between the normalized PTB(2) and PTB(3) characteristics

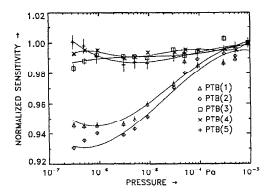


Fig. 5. Mean sensitivities of the extractor gauge, normalized to values at  $9 \times 10^{-4}$  Pa, measured by the PTB before and after each participant. Uncertainty bars are smoothed standard deviations of the means; curves are for illustrative purposes only.

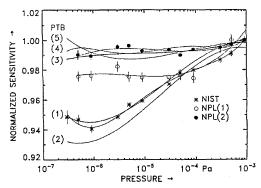


Fig. 6. Mean sensitivities of the extractor gauge, normalized to values at  $9 \times 10^{-4}$  Pa, measured by the participating laboratories. Uncertainty bars are smoothed standard deviations of the means; curves are for illustrative purposes only. Pilot lab results are shown by the fitted lines from Fig. 5.

below 9×10<sup>-5</sup> Pa, indicating a significant change occurred in the fundamental characteristics of this gauge before and/or after the first calibration at NPL. The standard deviations of PTB(2), NPL(1), and PTB(3) do not indicate large changes during the course of the calibrations. Again we have no basis for discarding either PTB(2) or PTB(3) as a reference, therefore we will use the average of the normalized characteristics. It is apparent that the difference between PTB(2) and PTB(3) is strongly pressure dependent, therefore we use half the difference at each pressure as the estimate of the transport uncertainty at that pressure. The agreement between PTB(4) and PTB(5) is much better, therefore as a reference for NPL(2) we use the average of the normalized characteristics, PTB(4) and PTB(5). The average difference between these characteristics can be used to calculate the transport uncertainty,  $\Delta_{\text{trans}} = 0.17\%$ .

# V. ANALYSIS OF RESULTS

As noted before, the results of this comparison must be considered in the context of their uncertainties. In so doing, it should be kept in mind that the uncertainty indicates a limit within which there is some probability that the "true" value lies. It is sometimes assumed that a standard or one-sigma uncertainty implies a probability or level of confidence of 68%. Strictly speaking, this assumption is valid only in the limiting case of an infinite data set, and in the case of our very finite data sets the level of confidence is generally significantly smaller.

#### A. Comparison of standards at $9 \times 10^{-4}$ Pa

The accommodation coefficients at  $9 \times 10^{-4}$  Pa can be used in a straightforward manner to compare the pressures generated by the standards. Since the calculated accommodation coefficient is proportional to the measured deceleration rate, which in turn is proportional to the generated pressure,  $\mathscr{P}$ , the ratio of the generated pressures equals the  $R_i$  in Table V. Thus, for NIST and PTB,  $(\mathscr{P}_{\text{NIST}} - \mathscr{P}_{\text{PTB}})/\mathscr{P}_{\text{PTB}} = R_i - 1 = 0.09\%$ , with a standard deviation,  $\Delta R_i$ , of 0.11%. Quadratically combining this uncertainty with the uncertainties of the two standards at  $9 \times 10^{-4}$  Pa, 0.13% for PTB and

0.17% for NIST (see Table II), the one-sigma limit for expected differences is calculated to be 0.24%. Clearly the measured difference is well within this limit.

From Table V  $R_{\rm NPL(1)}=1.0117$  and  $R_{\rm NPL(2)}=1.0144$ , indicating differences between the pressures generated by the standards of  $(\mathcal{P}_{\rm NPL}-\mathcal{P}_{\rm PTB})/\mathcal{P}_{\rm PTB}=1.17\%$  and 1.44%, with standard uncertainties of 0.29% and 0.09%. The difference between the results of these two comparisons, 0.27%, is less than their combined standard deviations, so we summarize these results by their mean value,  $\langle (\mathcal{P}_{\rm NPL}-\mathcal{P}_{\rm PTB})/\mathcal{P}_{\rm PTB} \rangle = (1.31\% \pm 0.14\%)$ . We believe the uncertainty of  $R_{\rm NPL(2)}$  is fortuitously small and a weighted mean is not warranted, therefore, we report a simple mean and its standard deviation. Taking into account the uncertainties of the standards (Table II; 0.32% for NPL and 0.13% for PTB), the one-sigma limits for the expected differences are 0.45% and 0.36%. Thus, both of the measured differences significantly exceed the combined uncertainties.

# B. Comparison of standards below 9×10<sup>-4</sup> Pa

The BAG and EXG are used to compare the standards below  $9 \times 10^{-4}$  Pa. As discussed in Sections IV B and IV C, for each gauge normalized and averaged sensitivities,  $S_{nc}^{i}(p)$ , were calculated for each participant i, including the

five pilot laboratory calibrations, and presented in Figs. 3-6. With one exception, previously discussed, the average of the before and after calibrations of the pilot laboratory were used as the reference sensitivity,  $S_{nc}^{\rm ref(i)}(p)$ . For each participant, i, the deviation is calculated between its normalized sensitivity and the reference sensitivity for each pressure and each gauge. As noted before, the SRG data are used to correct the reference sensitivity for any pressure-independent changes, so the difference between the generated pressures,  $\mu_{\rm BAG}(p_j)$  or  $\mu_{\rm EXG}(p_j)$ , as indicated by the BAG and EXG, respectively, is given by

$$\mu_{\text{BAG/EXG}}(p_j) = \frac{R_i S_{nc}^i(p_j)}{S_{nc}^{\text{ref}(i)}(p_j)} - 1,$$
 (13)

where the notation  $\mu_{BAG/EXG}$  indicates that the same equation is used for both sets of data.

The standard deviations of these measured differences are calculated from the square root of the quadratic sum of the standard deviations of the ion gauge calibration results obtained by each standard, the standard deviation of the comparison at  $9\times10^{-4}$  Pa,  $\Delta R_i$ , and the equivalent standard deviation  $\Delta_{trans}$  related to the transportation as described in Sections IV B and IV C

$$\Delta\mu_{\text{BAG/EXG}}(p_j) = \sqrt{\left(\frac{R_i \Delta S_{nc}^i(p_j)}{S_{nc}^{\text{ref}(i)}(p_j)}\right)^2 + \left(\frac{R_i S_{nc}^i(p_j) \Delta S_{nc}^{\text{ref}(i)}(p_j)}{\left[S_{nc}^{\text{ref}(i)}(p_j)\right]^2}\right)^2 + \left(\frac{S_{nc}^i(p_j) \Delta R_i}{S_{nc}^{\text{ref}(i)}(p_j)}\right)^2 + (\Delta_{\text{trans}})^2}.$$
(14)

Since most of the factors in this equation are close to 1, we can derive the following approximation to better illustrate the significant factors determining the total uncertainty,

 $\Delta \mu_{\text{BAG/EXG}}(p_j)$ 

$$\sim \sqrt{[\Delta S_{nc}^{i}(p_{j})]^{2} + [\Delta S_{nc}^{\text{ref}(i)}(p_{j})]^{2} + \Delta R_{i}^{2} + (\Delta_{\text{trans}})^{2}}.$$
(15)

The data of Fig. 7 indicate that the NIST and PTB standards agree to within the uncertainty of the comparison, with the largest measured difference of  $(1.2\pm0.6)\%$  at  $3\times10^{-7}$  Pa. The average magnitude of the uncertainties (represented by the vertical lines attached to the points) over the entire pressure range for both gauges is 0.7%. Presenting both sets of results,  $\mu_{BAG}(p)$  and  $\mu_{EXG}(p)$ , the same plot allows a visual evaluation of any correlation between the BAG and EXG data. It is evident that the data for the two gauges are not correlated to any significant extent, and the calculated correlation coefficient, -0.09, is virtually zero, indicating that observed differences are random in nature and any systematic deviations between the standards are within the scatter of the data. The lack of correlation in the data justifies a further reduction of the results by averaging them over pressure. The average measured relative difference between the pressures generated by the NIST and PTB standards from  $3\times10^{-7}$  Pa to  $9\times10^{-4}$  Pa is  $\langle (\mathcal{P}_{\text{NIST}}-\mathcal{P}_{\text{PTB}})/\mathcal{P}_{\text{PTB}}\rangle = (0.24\pm0.12)\%$ .

The NPL-PTB results in Fig. 8 are less satisfactory. The first set of EXG results are compromised by large gauge instabilities, indicated both by the large change (13%) of the

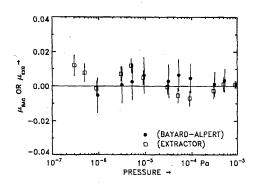


FIG. 7. Relative differences between the generated pressures. ( $\mathcal{N}_{NIST} - \mathcal{N}_{PTB}$ ).  $\mathcal{N}_{PTB}$ , as given by  $\mu_{BAG}$  and  $\mu_{EXG}$  [from Eq. (13)]. Uncertainty bars are standard uncertainties from Eq. (14), including an allowance for transfer standard instabilities. To distinguish the uncertainty bars for the two gauges, the points for the BA gauge are slightly shifted to the right on the pressure axis.

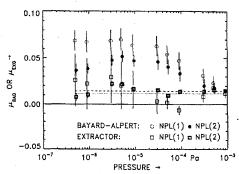


Fig. 8. Relative differences between the generated pressures, ( $\mathscr{N}_{NPL} - \mathscr{N}_{PTB}$ ).  $\mathscr{N}_{PTB}$ , as given by  $\mu_{BAG}$  and  $\mu_{EXG}$  [from Eq. (13)]. Uncertainty bars are as described in Fig. 7.

normalization factor at  $9 \times 10^{-4}$  Pa, and a large pressure-dependent shift at lower pressures. Much smaller EXG instabilities were observed during the second NPL-PTB comparison. However, taking into account the different offsets at  $9 \times 10^{-4}$  Pa, both sets of EXG results indicate virtually the same conclusion; to within the uncertainty of the comparisons there is no significant pressure dependence of the difference between the NPL and PTB standards for pressures below  $9 \times 10^{-4}$  Pa.

The BAG results are quite different. The repeatability of the pilot lab reference calibrations indicate better stability for the BAG than for the EXG. However, taking into account the indicated uncertainties and the offsets at  $9 \times 10^{-4}$  Pa, the two sets of BAG results are not as well correlated at the lower pressures as the two sets of EXG data. Nonetheless, the BAG data clearly indicate a pressure-dependent difference between the two standards, with the difference increasing with decreasing pressure to an offset as large as  $(\mathcal{P}_{\text{NPL}} - \mathcal{P}_{\text{PTB}})/\mathcal{P}_{\text{PTB}} = 6\%$ , for the NPL(1) results, and 5% for the NPL(2) results. In both cases the indicated differences significantly exceed the combined standard uncertainties of the two standards, which is a maximum of 0.60% at the lowest pressure.

# VI. DISCUSSION

The instabilities of the transfer standards created problems in the data analysis and compromised the results. However, in spite of these difficulties, and because of the use of redundant transfer standards and the repeat of the NPL-PTB data, several useful conclusions can be drawn from this comparison.

First, the NIST and PTB standards agree to well within the combined uncertainties over the entire range of the comparison. In view of the absence of systematic trends in the ion gauge results, the average indicated relative difference between the pressures,  $((\mathcal{P}_{\text{NIST}} - \mathcal{P}_{\text{PTB}})/\mathcal{P}_{\text{PTB}}) = (0.24 \pm 0.12)\%$ , can be considered representative of the differences over the entire range,  $3 \times 10^{-7}$  Pa to  $9 \times 10^{-4}$  Pa. At the highest pressure, the SRG data indicate an even

smaller relative difference,  $(\mathscr{N}_{\text{NIST}} - \mathscr{N}_{\text{PTB}})/\mathscr{N}_{\text{PTB}} = (0.09 \pm 0.11)\%$ , which is comparable to the difference observed eight years earlier.<sup>3</sup>

The results of the NPL and PTB comparisons are quite different. At  $9 \times 10^{-4}$  Pa, the average of the relative differences observed in the two comparisons,  $\langle (\mathcal{P}_{\text{NPL}} - \mathcal{P}_{\text{PTB}})/\mathcal{P}_{\text{PTB}} \rangle = (1.35 \pm 0.14)\%$ , significantly exceeds the combined uncertainties of the two standards, and is also significantly larger than the differences,  $(0.6 \pm 0.2)\%$  and  $(0.5 \pm 0.3)\%$ , found using SRGs in an earlier comparison at this pressure.<sup>3</sup> Two earlier comparisons, using ionization gauge transfer standards, involved a different PTB standard and had much larger uncertainties, but they did find the pressure generated at NPL to be higher than the PTB pressures. It appears that at this pressure there is persistent evidence for a significant difference between the NPL standard and the PTB standard (and by extension the NIST standard).

At lower pressures the situation is less clear. The EXG data indicate no pressure dependence of the difference between the standards. The BAG data are quite different; they indicate a significant pressure-dependent difference between the two standards. The consistency of the results for each gauge from the two comparisons makes it difficult to dismiss the results for either gauge, and has prompted an examination of possible systematic, pressure-dependent problems with one or both of the ionization gauges.

Most of this examination has focused on the BAG, in part because there is a general inclination to place more weight on results that consistently indicate no pressure dependence over the entire pressure range (the EXG results), since it is unlikely that pressure-dependent shifts in a gauge will cancel pressure-dependent errors in the standards at all pressures. In addition, there is some evidence of a possible problem with the BAG emission current control/measurement circuit. In the later stages of the comparison NPL noted instabilities in the measured BAG electron emission current. Postcomparison testing of the BAG at PTB found, on one occasion, shifts of comparable magnitude (about 5%) in the measured current. The data indicate that the actual emission current was stable and the instability was in the current measurement, which will cause corresponding errors in the calculated sensitivity. If a pressure-dependent effect of this type occurred during both NPL calibrations, but not during the pilot laboratory calibrations, it could explain the pressure dependence of the NPL-PTB results. However, the observed instabilities at PTB were not pressure dependent, and from the available evidence we cannot determine if this type of instability occurred throughout the NPL calibrations. The possibility of a pressure-dependent time response of the ionization gauges was examined, since the NIST and PTB standards generate a steady-state pressure, whereas the NPL standard generates a transient step function. However, experiments at PTB using both their orifice-flow and volumeexpansion standards found no significant difference in the response of the gauges.

Thus, there is no hard evidence of systematic effects in either ion gauge that could explain the difference between

the two sets of results. And, even if the EXG results are accepted as more reliable and we were to conclude that there is no significant difference between the NPL and PTB standard (and by extension the NIST standard), beyond that determined at  $9 \times 10^{-4}$  Pa, the BAG results still must be explained. They are consistent enough that at a minimum they indicate a calibration problem with this type of gauge.

In an attempt to resolve these ambiguities, a new set of transfer standards is being assembled and tested. In an attempt to avoid the problems with the first set of transfer standards, it will include different types of ionization gauges and multiple SRGs.

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