

Metrological relationships between primary vacuum standards

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ABSTRACT

Over the past 20 years, despite poor reproducibility of the transfer standards, several international comparisons have suggested that agreement between the primary vacuum standards held at national laboratories may not be as robust as indicated by the associated measurement uncertainties. This report reviews the results of such comparisons and analyses the level of agreement between laboratories and the various types of primary standards used, and also details internal comparisons between NPL's primary vacuum standards. The report highlights weaknesses in the physical models and assumptions that underpin the different types of standard and proposes some refinements including experimental protocols. It also identifies areas where, historically, a lack of documentation may have masked short-comings in either the models or experimental practice.

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1. INTRODUCTION

The current effort to formally determine the equivalence of primary measurement standards has given renewed impetus to understanding the potential causes of discrepancies between vacuum standards. Over a number of years, several comparisons have implied that measurements made in different laboratories do not agree within their combined uncertainties [1, 2, 3].

In the high vacuum region, static-expansion and dynamic-expansion systems are used as primary standards, whilst in the medium vacuum region liquid column manometers and static-expansion systems are used. The equality of dynamic and static-expansion methods for the generation of vacuum has to some extent been taken for granted because of the difficulties in comparing remote standards and the majority of the national metrology institutes maintain vacuum standards based on just one of the principles. The few laboratories that have systems based on both techniques have only compared them in the traditional way, that is by the use of a transfer gauge, as they have not generally housed them close to each other, and the comparisons have been of a somewhat ad-hoc nature. Recent international comparisons have typically been performed according to more carefully designed protocols, and greater confidence may be placed upon their results. Although still plagued by persistent problems with transfer standards, these recent comparisons do suggest that there may be principle-related discrepancies that have possibly been unappreciated for decades.

A project was initiated to investigate whether the differences between vacuum standards are principle-related, specific to a particular standard or application of a principle, however established, or random in nature. Its aims were to confirm the extent of discrepancies between NPL standards based on different principles and to compare them with those of international comparisons, plus to develop a better understanding of the limitations of the long-standing physical models which underpin the different principles.

This report reviews both the internal and international comparisons of NPL's vacuum standards over the past 20 years or so. Despite the wealth of data obtained over years of development of high-vacuum standards it is difficult, if not impossible, to analyse some of it in a sufficiently coherent manner to enable the different principles to be compared with confidence. In some cases the original data were not corrected for certain systematic influences, whose effects have only been recognised more recently. In addition, vacuum calibrations are time-consuming and statistical analysis was often based on a very limited population sample. Although the data distribution was assumed to be Gaussian, on many occasions this assumption was probably flawed. One factor which hampered the analysis of all but the most recent calibrations was the lack of clarity about the associated measurement uncertainties. Commonly there were no statements concerning the coverage factors used [1], nor was it explained how the individual components were added. In some cases no uncertainties were given at all [9]. During the period in question the accepted practice for summing uncertainty components changed significantly, adding to the difficulty in comparing data. An attempt has been made to quote all uncertainties at a coverage factor $k=1$, but where it was not possible to derive standard uncertainty values from the published figures, it has been assumed that the information quoted referred to a confidence level of not less than 95%.

This report also attempts to analyse more thoroughly some of the theoretical models associated with both static-expansion and dynamic-expansion primary standards.

Unfortunately, much of the historical data is based on underlying assumptions which have not been documented fully and it is therefore difficult to interpret the data with sufficient clarity to analyse the influence of some parameters. Some parameters now considered important were only quantified in a cursory manner or, in a few cases, not even measured and retrospective application of corrections to the original results is often not possible. As with the comparison data, the uncertainties associated with the published values were not always given, and confidence levels rarely stated. Thus the amount of data available for examining the influence of mathematical models on the calculated pressures and their uncertainties is quite small. In several cases it has been found that relatively small changes in modelling assumptions made significant differences to the calculated final pressure values. In some cases the observed differences exceeded the quoted uncertainties. Some weaknesses in both models and assumptions relating to vacuum standards have been identified and improvements to the models and experimental protocols proposed. Areas where lack of documentation may mask short-comings in either the models or experimental practice have been identified. In addition details are also given of the measurements made following the improvements to the models.

2. COMPARISON OF PRIMARY VACUUM STANDARDS

2. METHODS FOR COMPARISON OF VACUUM STANDARDS

2. Principles

Static-expansion allows a small volume of gas at a known and relatively high pressure to 'expand' into a larger volume; the pressure of the expanded gas is calculated from knowledge of the ratio of the volumes, assuming that the Boyle-Marriott law is obeyed. Traceability to the SI is established through the calibration of gauges used to measure the initial pressure and the inter-stage pressures during determination of the ratios of the volumes of various vessels comprising the system.

Dynamic-expansion relies on balancing the rate of flow of gas injected into a chamber against the rate at which it is being evacuated through an orifice; under conditions of steady molecular flow, the pressure is calculated from knowledge of the gas flow-rate and the conductance of the orifice. Traceability comes from knowledge of the conductance of the orifice, obtained from dimensional metrology and gas kinetics, and the calibration of the flow-meter which is obtained through the calibration of gauges used to measure the flow-meter fill pressure and from the dimensional characteristics of the variable volume.

Static- and dynamic-expansion standards are pressure *generators* and hence can only be compared by indirect comparison using a transfer gauge calibrated in turn against each.

2.1.2 Transfer standards

Poor repeatability and reproducibility are unfortunate characteristics of vacuum transfer standards, and can lead to erroneous conclusions. The choice, handling and use of transfer gauges is therefore most important.

2.1.2.1 *Transfer standards in medium-vacuum*

Capacitance diaphragm gauges are commonly used as transfer standards in the medium vacuum region. When comparing calibration results, it is the gauge's *sensitivity* that is considered.

$$\text{CDG sensitivity} = \frac{P_{\text{CDG}}}{P_{\text{STD}}} \quad 1)$$

where P_{CDG} is the pressure indicated by the CDG
 P_{STD} is the reference pressure

2.1.2.2 *Transfer standards in high- and ultra-high vacuum*

The transfer standards widely used for high-vacuum comparisons are ionisation gauges (IGs) of various designs and, more recently, spinning-rotor gauges (SRGs). In the ultra-high vacuum region the only practical transfer standards are ionisation gauges.

When comparing calibration results of spinning-rotor gauges, it is the *effective accommodation coefficient* (σ) of the gauge's rotor which is considered.

$$\sigma = \frac{P_{\text{SRG}}}{P_{\text{STD}}} \quad (2)$$

where P_{SRG} is the pressure indicated by the SRG
 P_{STD} is the reference pressure

When comparing calibration results of ionisation gauges, it is the *sensitivity* of these gauges which is considered.

$$\text{Sensitivity} = \frac{(I_C - I_{C_0})}{P_{\text{STD}} I_e} \quad (3)$$

where P_{STD} is the reference pressure
 I_{C_0} is the collector current at residual pressure p_0
 I_C is the collector current at pressure p
 I_e is the emission current.

2.2 HISTORICAL PERSPECTIVE OF VACUUM COMPARISONS

2.2.1 Earliest vacuum comparisons

One of the earliest vacuum comparisons was initiated by the American Society for Testing Materials (ASTM) between five American laboratories. It is unlikely that there was a strict protocol for the measurements and gauge conditioning prior to calibration, but even so the results presented by Kalkbrenner[4] at the 1965 American Vacuum Society (AVS) meeting were disappointing as they differed by up to 34%.

At about the same time vacuum work started in Germany and in the UK. The laboratory of Leybold, Germany, built two vacuum systems, one operating on the static-expansion principle, and the other based on dynamic-expansion and when compared the two systems were found to agree within 4% [5], using argon gas, over the range 10^{-4} Pa to 10^{-1} Pa. This agreement was seen as extremely good in the light of the American results, particularly considering that the uncertainties claimed for the static and the dynamic-expansion systems were 3% and 7% respectively. At the National Physical Laboratory (NPL), UK, a static-expansion system was constructed during the mid-1960s and its performance, using argon and nitrogen gases, was compared with a McLeod gauge using a Bayard-Alpert ionisation gauge as a transfer standard. The results [6] agreed to better than 5% over the full range of the comparison (10^{-4} Pa to 10^{-1} Pa). As the agreement obtained in these two in-house comparisons was so much better than for comparisons between different laboratories, it was concluded that transportation of transfer standards significantly affected their characteristics and hence the outcome of comparisons [7]. In the meantime, a system based on static-expansion was developed at Physikalisch-Technische Bundesanstalt (PTB), Germany, and in

1975 the first bi-lateral NPL - PTB comparison took place in the form of a calibration of an ionisation gauge over the pressure range 2×10^{-3} Pa - 5×10^{-3} Pa using argon. A detailed comparison protocol, including instructions for baking the system and outgassing the gauge, was used and the results agreed to within 0.2%, whilst the uncertainty in measured gauge characteristics in either laboratory was quoted as 1% [7],[8]. This was a very good result, albeit only over a very small pressure range, but later comparisons showed less close agreement.

A three-stage comparison over the pressure range 7×10^{-5} Pa to 1.3×10^{-2} Pa and involving four laboratories took place in the USA between 1973 and 1979 [9]. Three laboratories used systems based on dynamic-expansion whilst the fourth laboratory used a static-expansion system, with none of the laboratories providing estimates of the uncertainties. The first stage was meant to serve as a test for a newly-developed hot-cathode ion gauge, although three standard commercial IGs were also included. However, the large differences in the results from the participating laboratories only served to show that disagreement between laboratories was much greater than the repeatability of measurements at an individual laboratory and that the performance of the specially developed gauge was not significantly better than that of any of the commercial gauges. Consequently, the studies shifted from the gauge study and concentrated on calibration systems and protocols. The practice review performed by each laboratory led to improvements so that the second part of the comparison showed some reduction in disagreements, but differences of up to 20% at the extremes of the range and up to 6% in the middle still remained. Due to problems in some laboratories the last cycle was limited to only two laboratories. In addition the three transfer gauges were also calibrated by NPL on two occasions. The results of 1979 show fairly good agreement for all three laboratories, for four out of five gauges.

With the exception of the NPL - PTB comparison it was unusual for the same measurement protocol to be followed by the participating laboratories, and the uncertainties associated with the standards were generally not quoted. Whilst there were large differences between the participating standards, the results were too crude to pinpoint any specific reasons, especially since the comparisons were influenced by poor characteristics of the transfer standards [10].

2.2.2 Second generation of international vacuum comparisons

By the mid 1970s it was appreciated that rigorous pre-conditioning of transfer standards was necessary and that pre-arranged comparison protocols were necessary [7]. In addition, work in this area generally became concentrated within national standards laboratories rather than commercial organisations and basic uncertainty budgets had been developed for most national standards.

2.2.2.1 *The Community Bureau of Reference (BCR) high-vacuum comparison*

The Community Bureau of Reference (BCR), a division of the Directorate-General for Research Science and Education of the Commission of the European Communities, arranged the first European vacuum comparison between NPL, PTB, Instituto di Metrologia "G. Colonnetti" (IMGC), Italy and Leybold-Heraeus-Sogev company (SOGEV), France; it was performed between 1976 - 78. NPL and PTB used static-expansion systems, whilst IMGC and SOGEV used dynamic-expansion systems. Three JHP high-pressure ionisation gauges were chosen as transfer standards following studies at SOGEV [11]. The calibrations

were performed over the range 8×10^{-5} Pa to 8×10^{-2} Pa with one calibration pressure per decade. The averaged results of the comparison, in terms of weighted means of differences between participants, and the measurement uncertainties claimed are given in Table 1 [1] (the coverage factors are unknown).

Laboratory	Difference / %	Uncertainty of standard / %			
		8×10^{-5} Pa	8×10^{-4} Pa	8×10^{-3} Pa	8×10^{-2} Pa
SOGEV	-1.5	1.5	1.5	1.5	1.5
NPL	1.2	0.8	0.65	0.65	0.5
IMGC	-1.9	2	2	2	2
SOGEV 2	-0.3				
PTB	0.2	1	1	1	1
SOGEV 3	-4.2				

Table 1: BCR comparison - averaged difference between participants

The intention was to circulate three gauges to all four participating laboratories but during the comparison several gauges malfunctioned and some had to be replaced, hence not all the laboratories calibrated the same gauges. Two gauges, numbered 18 and 20, were calibrated by all four participants and the results obtained for gauge 18, in terms of the weighted gauge sensitivity, are presented Figure 1.

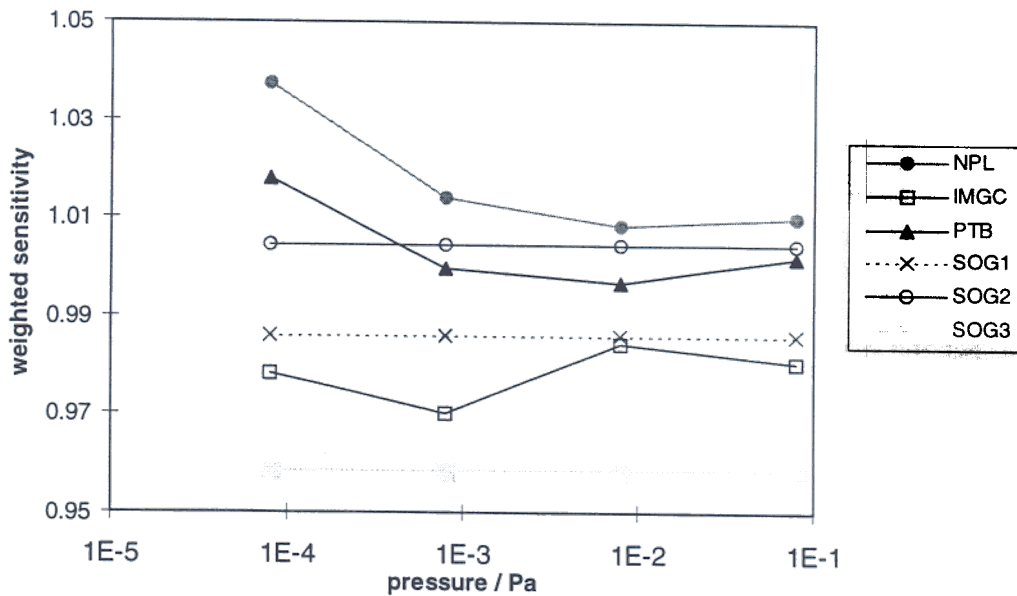


Figure 1: BCR comparison results for gauge no. 18

The NPL results for gauge no. 20 could be considered anomalous for although the sensitivity values showed similar characteristics to gauge no. 18, they were significantly higher than those of the other laboratories, unlike the NPL data for gauges 16 and 18. Shifts in gauge sensitivity measured at the pilot laboratory (SOGEV) were between 2% and 5%. Figure 2 shows the percentage difference between the standards of the participating laboratories and the weighted means of the differences, averaged over all pressures and gauges. The solid bars

represent the uncertainty calculated in the percentage differences and the dashed error bars represent the uncertainties quoted by each laboratory for its measurements, including the uncertainties in the measurement of the gauge potentials and the ion current [1].

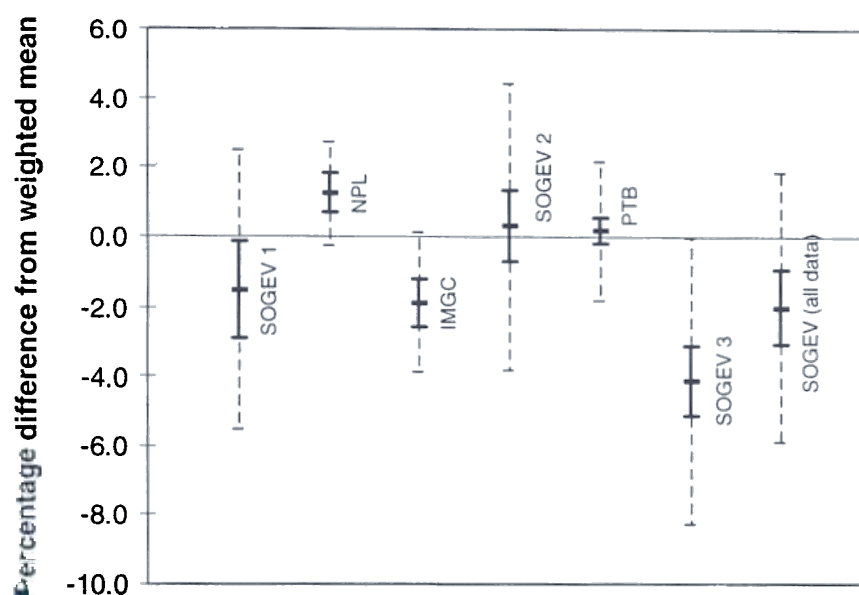


Figure 2: BCR comparison - percentage difference from weighted mean

Although the overlap between participants was considered satisfactory, particularly when the stability of the transfer standards was taken into account, differences of several percent were apparent. It was also evident that the pressures calculated at NPL and PTB were always lower than those calculated at SOGEV and IMG, with the NPL pressure being consistently lower than PTB's. With hindsight, this was the first evidence that there might be a principle-related systematic difference between primary vacuum standards but, if it was noticed, it was not investigated further.

2.2.2.2 The CCM very low pressure comparison

After consultations with national metrology institutes, the Low Pressure Working Group of the Comité Consultatif pour la Masse et les Grandeurs Apparentées (CCM) of the Comité International des Poids et Mesures (CIPM) initiated what was to be the first world-wide vacuum comparison in 1979.

Laboratories from nine countries took part in the project: Germany (PTB - pilot laboratory), UK (NPL), Italy (IMG), France (LNE), Czechoslovakia (CSMU), China (NIM), India (NPL(India)), USA (NIST) and Japan (ETL). The first generation of the then newly-developed spinning-rotor gauge was proposed as the transfer standard and a preliminary phase of the project, a bi-lateral comparison between PTB and LNE, was carried out in order to investigate transportation effects. The results obtained at the two laboratories agreed to within 3%.

The comparison started in 1981 using four SRGs as transfer standards over the pressure range 5×10^{-4} Pa to 1 Pa, using argon and where possible hydrogen. In 1984 the SRG electronics

and gauge ‘fingers’ were replaced by ones of a newer design but pilot laboratory results showed that this produced changes in the gauge coefficients of between -0.6% and 0.1% for argon and between -1.4% and -1.8% for hydrogen. After shipping to and from NIST changes of around 4% were detected, causing the original rotors to be discarded. A set of new rotors was used at NPL (calibration marked NPL(2)) and then ETL, while a second set was used at NIST.

A summary of the results obtained for argon gas at all the participating laboratories is given by Figure 3 [2]. The results are given in terms of the ratios of an average gauge accommodation coefficient obtained at a particular laboratory and the corresponding accommodation coefficient obtained at the pilot laboratory, averaged for all the gauges calibrated for the comparison.

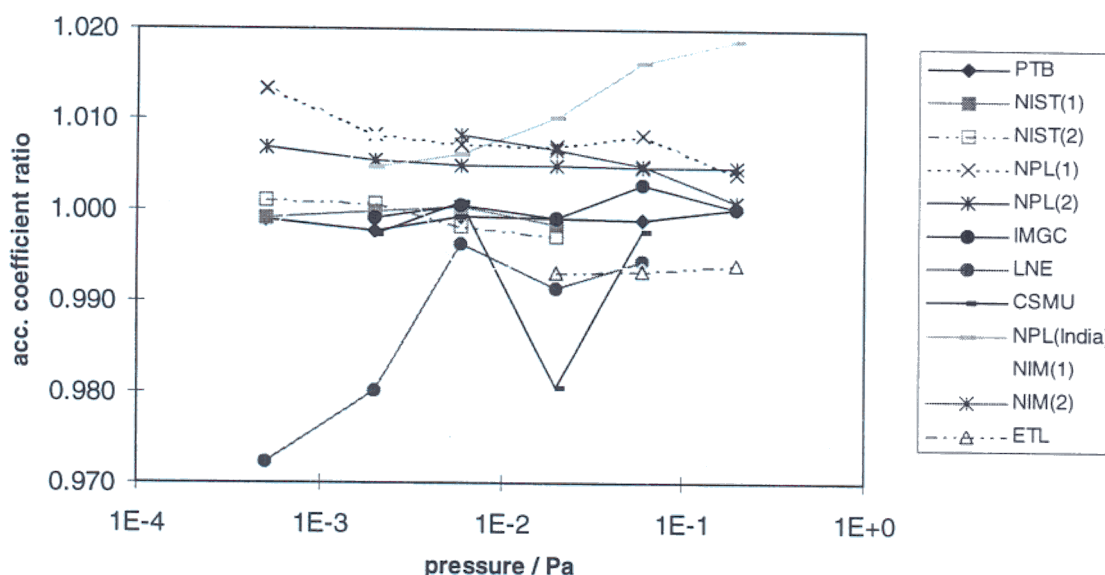


Figure 3: Results of CCM very low pressure comparison using argon

Excluding the results of the pilot laboratory (PTB), the results obtained by static-expansion (NPL(India) and NPL) were higher than those generated by dynamic-expansion (NIST, IMGIC, CSMU). Of the measurement standards used, however, not all were strictly ‘primary’ in nature and of those that were, two laboratories (CSMU and NPL(India)) experienced significant problems with them which significantly affected their results. Figure 4 shows data for measurements using argon gas, for the laboratories not categorised in this way.

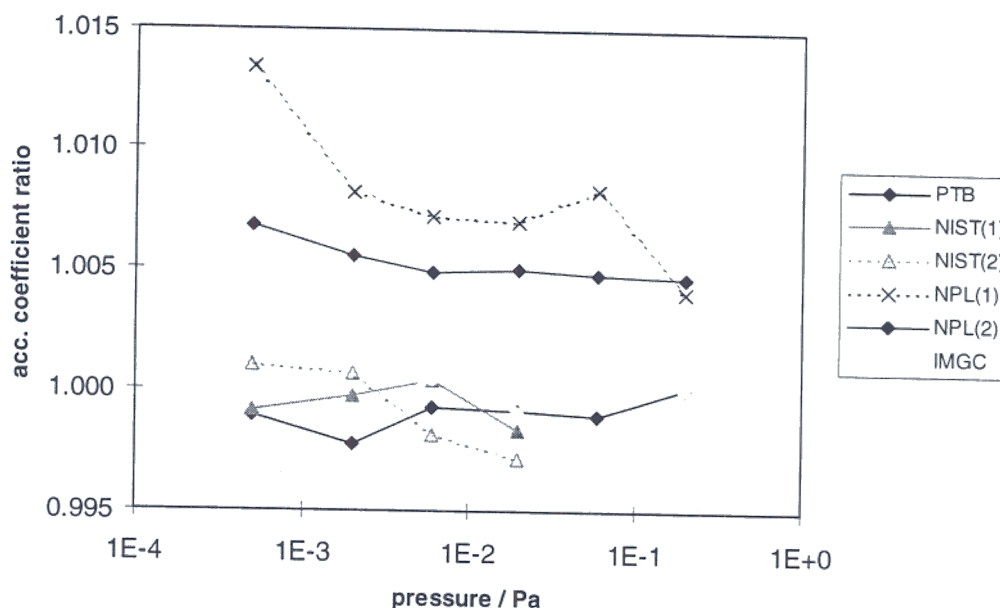


Figure 4: CCM very low pressure comparison of primary standards for argon

Only three laboratories, PTB, NPL and NIST carried out measurements using hydrogen and their results are shown in Figure 5. For both gases the NPL results are higher than those of the other laboratories, although the effect is less marked for the hydrogen data than for the measurements using argon.

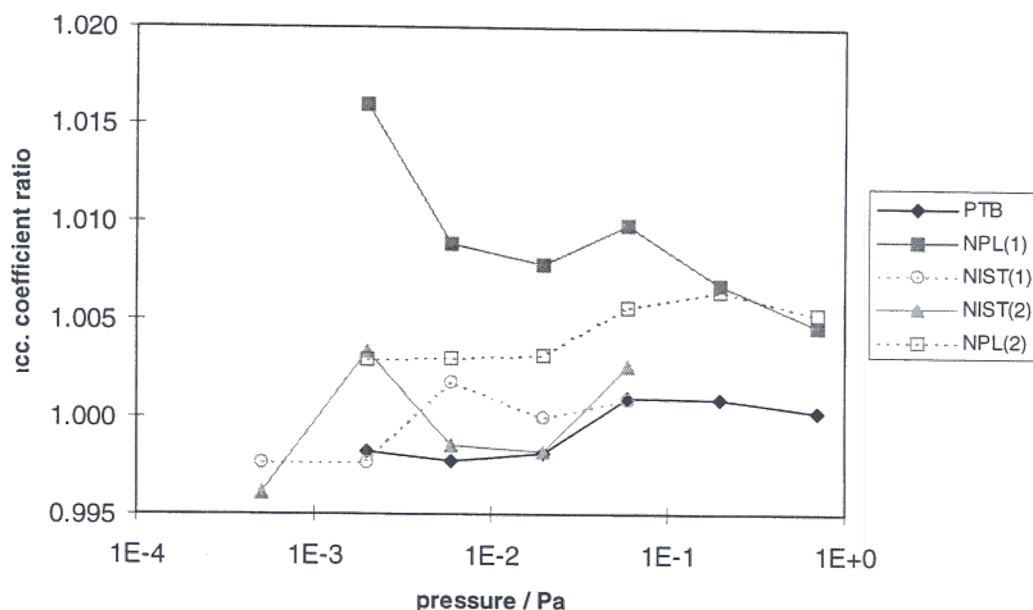


Figure 5: CCM very low pressure comparison - results for hydrogen

An additional difficulty in trying to re-analyse the published results was that the uncertainties quoted in the paper did not clearly state how they were calculated or how they varied with pressure. Table 2 is the authors' less-than-robust attempt to provide a ball-park comparison of the data expressed at standard uncertainties at $k=1$.

Pressure/Pa	Uncertainty/%			IMGC
	PTB	NPL	NIST	
1×10^{-4}	0.35	0.44	0.50	0.80
5×10^{-4}				
5×10^{-3}	0.20	0.33	0.53	
1×10^{-2}			0.87	
1×10^{-1}				
1×10^0				
principle	static	static	dynamic	dynamic

Table 2: CCM very low pressure comparison - uncertainties of four primary vacuum standards

2.2.2.3 PTB - NPL(India) - extension of the CCM very low pressure comparison

Following the CCM very low pressure comparison the volumetric ratios of the standard used by NPL(India) were re-determined and their values found to have changed by about 0.8%. This was significant in relation to the claimed standard uncertainty (0.65%), precipitating a repeat comparison between PTB and NPL(India) [12].

Two SRGs served as transfer standards and both laboratories calibrated them on static-expansion systems, in argon, over the pressure range 10^{-3} Pa to 2 Pa. The results agreed to within 0.7%, which was within the combined uncertainties of the two standards. However, NPL(India)'s values were higher than those from PTB for both gauges, consistent with the previous results. The average accommodation coefficients for each gauge calibrated are shown in Table 3.

Mean σ	PTB	NPL(India)	Difference / %
gauge 1	1.023	1.027	0.4
gauge 2	1.014	1.021	0.7
uncertainty / %	0.4	0.7	-

Table 3: PTB - NPL(India) comparison - averaged results

2.2.2.4 The CCM low pressure comparison

Dynamic-expansion vacuum standards are not generally used in the low pressure region, but comparisons can be made between liquid manometers and static-expansion systems.

With limited information available on the reproducibility of potential transfer standards, the CCM Low Pressure Working Group decided to use four commercial capacitance diaphragm gauges (CDG), two with 1 torr (133 Pa) range and two with 10 torr (1300 Pa) range. Considerable bi-lateral work was undertaken between 1980 and 1982 by NBS (subsequently NIST), who acted as a pilot laboratory, and NPL to evaluate the comparison protocol and to

characterise transportation effects. The results were worse than expected with the gauges showing instabilities of between 0.6% and more than 1% over two years. The largest shifts in two gauges, from the same manufacturer, occurred during shipping; the other two gauges, however, exhibited changes of similar magnitude without being transported.

Despite these characteristics, more suitable transfer standards were not found and in 1985 the comparison proceeded, including PTB and Australia (CSIRO).

Pressure	Deviation from the mean / %			
	PTB	NPL	NIST	CSIRO
<10 Pa	0.20	0.50	0.65	0.80
>10 Pa	0.05	0.20	0.10	0.20
principle	static	static	manometer	manometer

The pilot laboratory, NIST, and CSIRO both used mercury barometers, while NPL and PTB calibrated the CDGs using static-expansion systems. PTB used the same system used for the very low pressure comparison, while NPL used a different standard. The analysis of the results was complicated by non-linear characteristics of the transfer standards due to the effect of thermal transpiration [13]. Also, the calibration protocol asked each laboratory to follow their normal protocol; whilst this made the results more 'real', it made the analysis of the data more difficult. Using interpolation to generate data at common pressures, pilot laboratory reference sensitivities were calculated and a corresponding mean sensitivity ratio was determined at each pressure, from the four gauges, for each participant.

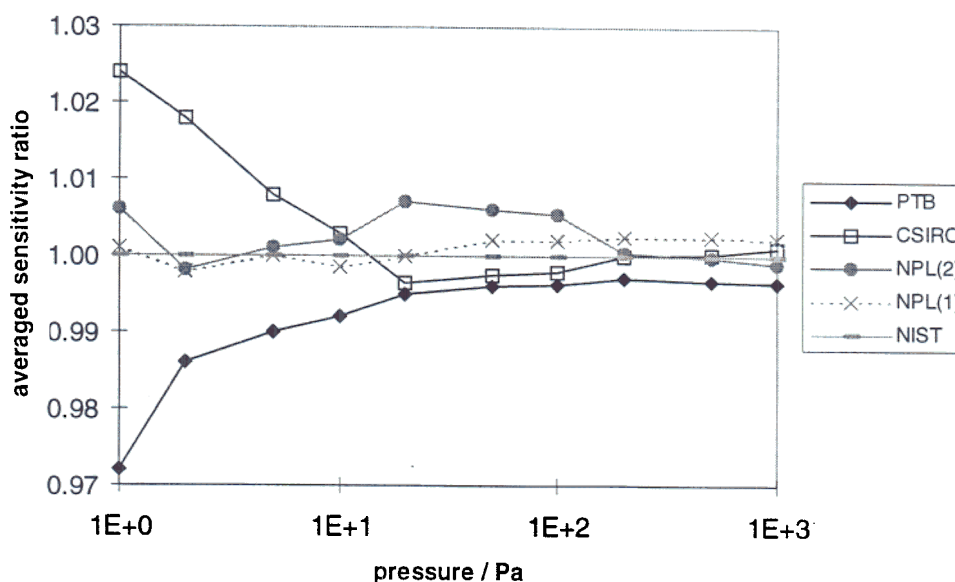


Figure 6: CCM low pressure comparison - averaged results

The results showed good agreement (between 0.1% and 0.3%) between NIST and NPL(1). For pressures above 20 Pa the CSIRO results also agreed with these two laboratories to

within 0.3%. The sensitivity values obtained at PTB however, remained consistently lower than those of the other laboratories by almost 0.5% in this range. It should be noted that for pressures about 10 Pa, the results obtained from the two static-expansion systems roughly straddle those obtained with the two liquid column manometers.

2.2.2.5 The CCM ultra-high vacuum comparison

In 1993 the CCM Low Pressure Working Group initiated the first comparison to include pressures below 10^{-5} Pa. It had not been attempted before as the stability of ionisation gauges, the only practical transfer standards at these pressures, was believed to be insufficient to enable meaningful interpretation of the results. The situation improved with the development of the spinning-rotor gauge. Although their lowest operational pressure was about 10^{-4} Pa, they could be used to normalise ion-gauge results at this pressure, thus removing effects of any pressure-independent systematic shifts in the IG characteristics and enhancing their ability to be used for comparisons at lower pressures.

A preliminary phase of the comparison was performed by three laboratories: PTB (pilot laboratory), NPL and NIST [3]. Three transfer standards were used, one SRG, one Bayard-Alpert ionisation gauge (BAG) and one extractor ionisation gauge (EXG). The pressure range was 3×10^{-7} Pa to 9×10^{-4} Pa, with three calibration pressures per decade. The argon pressures generated at 9×10^{-4} Pa were compared by comparing the SRG accommodation coefficients measured in each laboratory. These coefficients were also used together with the results obtained from the ionisation gauges at the same pressure to normalise the ion-gauges' results at lower pressures. The standard uncertainties quoted by the participating laboratories are given in Table 5.

Pressure / Pa	Uncertainty / %		
	NPL	NIST	PTB
	0.55		
	0.46		
	0.42		
	0.41		
	0.41		
	0.41		
	0.41		
	0.41		
	0.32		
	0.32		
	0.32		

Table 5: CCM UHV comparison - uncertainties of three primary standards

The first NPL results using the ionisation gauges were not self-consistent so the NPL - PTB comparison was repeated a year later by extending the comparison with three additional sets

of calibrations (PTB(4), NPL(2) and PTB(5)). However, the second set of results showed a trend similar to that observed in the first set, so both sets of the results were included in the analysis.

The argon pressures generated by the standards at 9×10^{-4} Pa were compared using the spinning-rotor gauge. The mean accommodation coefficient calculated by each participating laboratory i was normalised using the mean value obtained for the initial set of pilot laboratory results, PTB(1), and then divided by the corresponding reference value (the mean of PTB results from before and after calibration at each participating laboratory) to give the ratio:

$$R_i = \frac{\sigma_n^i}{\sigma_n^{\text{ref}(i)}} \quad (4)$$

Table 6 summarises the normalised mean effective accommodation coefficients at each laboratory.

Laboratory	Acc. coefficient σ normalised to PTB(1)	$\Delta\sigma$ as measured
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

Table 6: CCM UHV comparison - SRG results obtained at 9×10^{-4} Pa

Laboratory	$100 \cdot (P_{\text{LAB}} - P_{\text{PTB}}) / P_{\text{PTB}}$	Relative Uncertainty ($k = 1$)
NIST	-0.09	± 0.24
NPL(1)	-1.17	± 0.45
NPL(2)	-1.44	± 0.36

Table 7: CCM UHV comparison - differences between participants at 9×10^{-4} Pa

Table 7 shows the differences between the pilot laboratory and the two other participants at 9×10^{-4} Pa. The differences ascribed to both sets of NPL data are about three times greater than the relative standard uncertainties.

Pressures between 3×10^{-7} Pa and 9×10^{-4} Pa were compared by measuring the sensitivity of the two ionisation gauges. The average sensitivity obtained at 9×10^{-4} Pa $\langle S(9 \times 10^{-4} \text{ Pa}) \rangle$ was used to normalise the mean sensitivity values, obtained in one day, at each pressure p_j .

$$S_{nc}(p_j) = \frac{\langle S(p_j) \rangle}{\langle S(9 \times 10^{-4} \text{ Pa}) \rangle} \quad (5)$$

The normalised data from different days were then averaged at each calibration pressure to give $S_{nc}^i(p_j)$. Finally, for each participant i , the deviation is calculated between its averaged normalised sensitivity and the reference sensitivity of the pilot laboratory (corrected by the SRG data) for each pressure and each gauge:

$$\mu_{BAG/EXG}(p_j) = \frac{S_{nc}^{ref(i)}(p_j)}{R_i S_{nc}^i(p_j)} - 1 \quad (6)$$

At each calibration pressure the measured sensitivity values for the Bayard-Alpert gauge were averaged and normalised using the protocol described above. For each laboratory the average value at $9 \times 10^{-4} \text{ Pa}$ was normalised by the initial value obtained by the pilot laboratory, PTB(1). Table 8 shows the normalised sensitivity for the Bayard-Alpert gauge together with the associated standard deviations (ΔS).

laboratory	sensitivity S normalised to PTB(1)	ΔS as measured
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

Table 8: CCM UHV comparison - averaged B-A gauge results at $9 \times 10^{-4} \text{ Pa}$

Concentrating only on the five PTB sets of measurements, it is evident that significant changes occurred between calibration by the pilot laboratory. Pressure dependent changes (between PTB(1) and PTB(2)) in the normalised sensitivity of the Bayard-Alpert gauge were observed [3], with the largest change of 3.6% at the lowest pressures. If the NIST data are compared with PTB(2), rather than to the mean of PTB(1) and PTB(2), the agreement is well within the combined standard uncertainties.

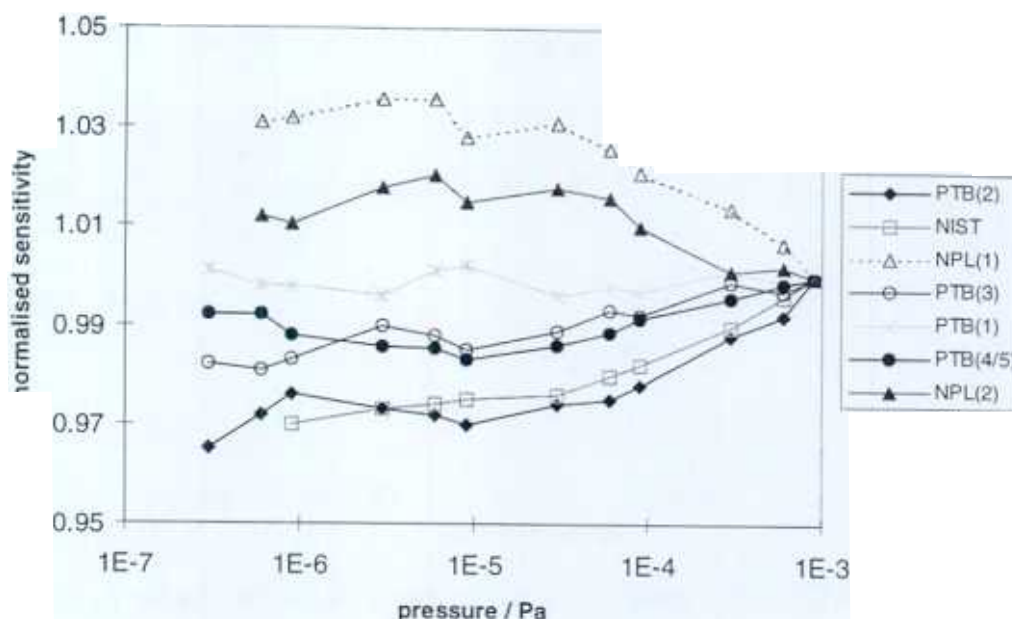


Figure 7: CCM UHV comparison - B-A gauge results

The mean normalised sensitivities of the Bayard-Alpert gauge for all pressures, at all participating laboratories are shown in Figure 7. The NIST and PTB standards (using PTB(2) data only) agree to within the average uncertainty of the comparison (0.7%), having the largest difference of over 1% at the lowest pressures, and the average measured relative difference over the whole measured pressure range of 0.24%. On the other hand, the NPL(1) data clearly indicate a pressure-dependent difference between the NPL and PTB standards, with the difference increasing with decreasing pressure to as much as 6%. NPL(2) results are closer to PTB results, and the difference does not show such a sharp pressure dependence. In both cases the measured differences significantly exceeded the combined standard uncertainty. However, subsequent to the comparison PTB observed that the Bayard-Alpert gauge emission current was occasionally around 5% higher than usual, hence the resultant sensitivity was reduced by 5%. It was therefore considered possible that the measurement of emission current at NPL was in error due to an erroneous additional current from the anode to ground or to the cathode. The failing isolation resistance may be pressure dependent and may also depend on the length of the exposure to the gas at a particular pressure before measurement.

Figure 8 represents the difference, measured by the Bayard-Alpert gauge and calculated using equation (6), between the NPL and NIST results, and the reference PTB results. For the NPL results the reference values were the mean of PTB 'before' and 'after' results. For NIST, the line (NIST) represents the difference between NIST and PTB(1), and the other line (NIST') is the difference between NIST and PTB(2) results.

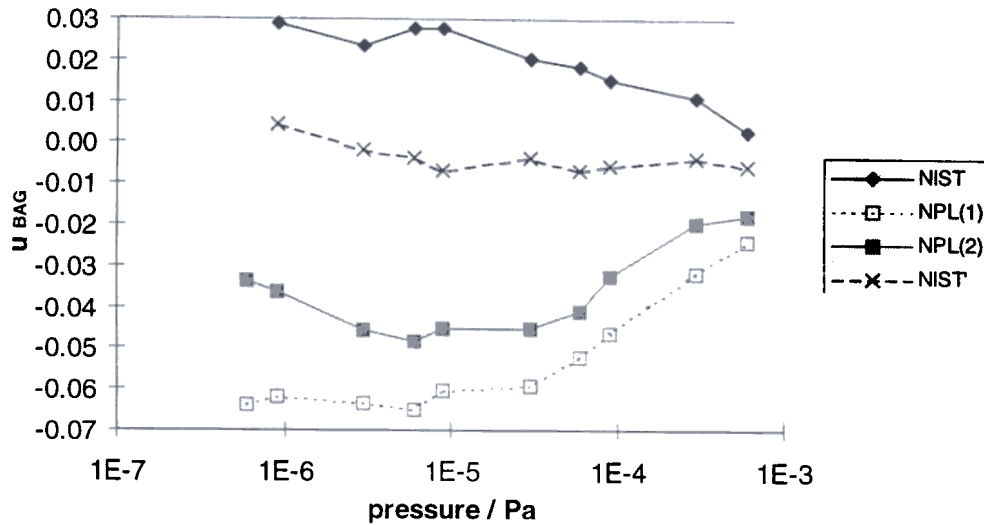


Figure 8: CCM UHV comparison - difference measured by the B-A gauge

The changes in the extractor gauge characteristic between the pilot laboratory calibrations were significantly larger than those observed for the Bayard-Alpert gauge - a change of 12.6% occurring between PTB(2) and PTB(3). Moreover, there was a large pressure-dependent difference between the normalised PTB(2) and PTB(3) characteristics below 9×10^{-5} Pa, although the standard deviations of PTB(2), NPL(1) and PTB(3) measurements did not show large changes during the course of the calibrations. This indicated that a significant change had occurred in the fundamental characteristics of the extractor ion-gauge before and/or after the first calibration at NPL, and therefore the first set of PTB - NPL comparison data was compromised. The average extractor gauge sensitivities at 9×10^{-4} Pa, normalised to PTB(1) are given in Table 9.

laboratory	sensitivity S normalised to PTB(1)	ΔS as measured
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

Table 9: CCM UHV comparison - averaged extractor gauge results at 9×10^{-4} Pa

The agreement between the NPL and PTB standards indicated by the extractor gauge (Figure 9) appeared much better than when compared using the Bayard-Alpert gauge. Also, the extractor gauge results, unlike the Bayard-Alpert gauge, did not indicate any significant pressure dependence. The agreement for the second set of the results was even better, with

differences within the overall uncertainty of the comparison (2%), even at the lowest pressures. The difference between the Bayard-Alpert and extractor gauge results raised a question of possible gauge-system interactions, related to gauge design.

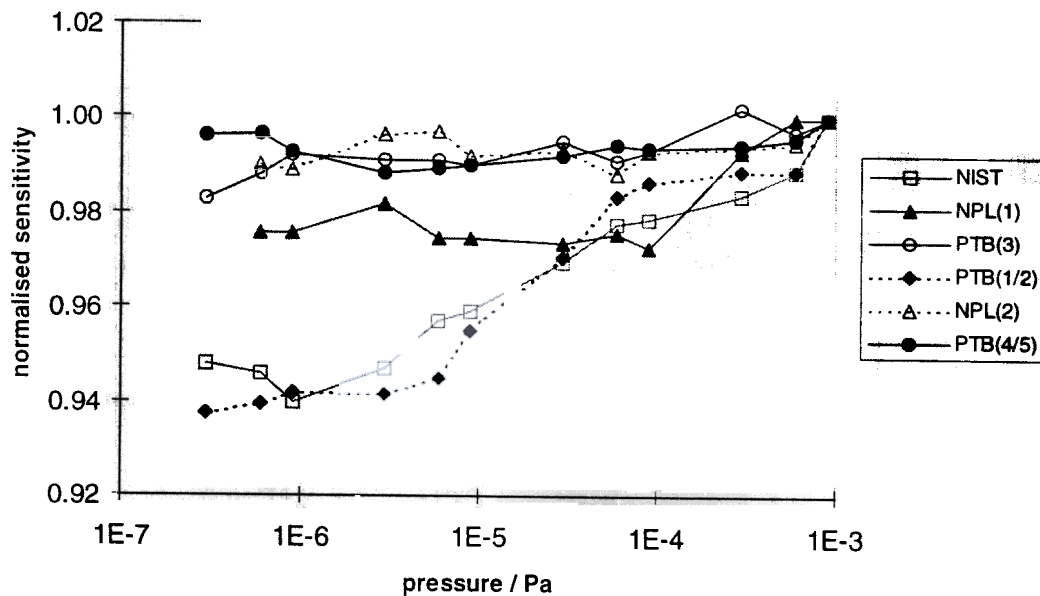


Figure 9: CCM UHV comparison - extractor gauge results

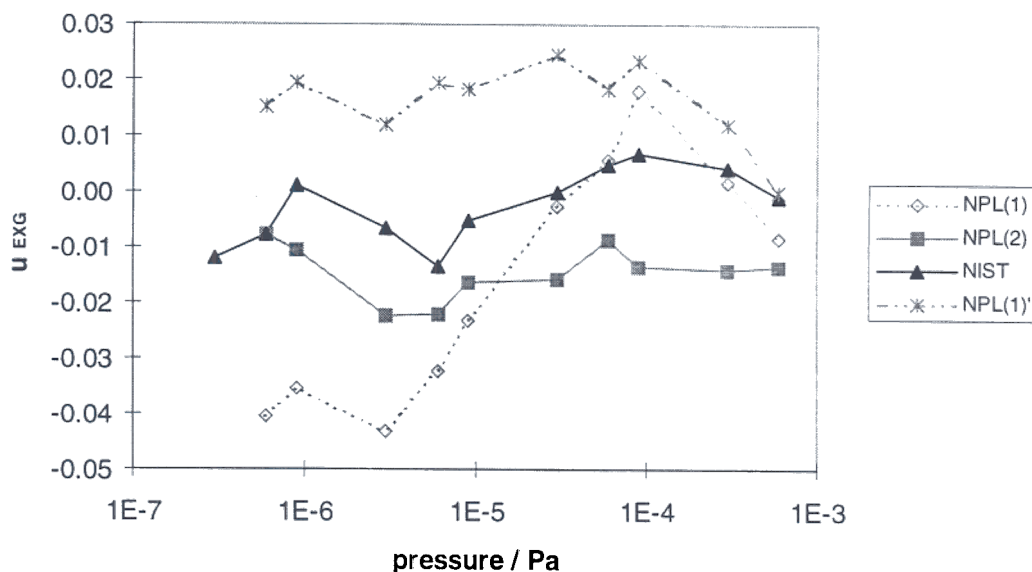


Figure 10: CCM UHV comparison - difference measured by the extractor gauge

Figure 10 illustrates the susceptibility of the comparison results to the choice of reference values. The line labelled NPL(1) represents the difference between the NPL(1) results and the mean of PTB(2) and PTB(3) results, whereas the line NPL(1)' represents the difference

between the NPL(1) and PTB(3) results. It can be seen that data represented by NPL(2) is of a different sign to NPL(1)'.

2.2.3 Recent bi-lateral comparisons

2.2.3.1 ETL - IMGC comparison

The vacuum standards of ETL, Japan and IMGC, Italy were compared in 1995 [14] using argon in the pressure range 10^{-4} Pa to 10 Pa with four spinning-rotor gauges as transfer standards. In the pressure range 10^{-4} to 10^{-1} Pa, ETL used their static-expansion system, while IMGC used their dynamic-expansion system. At higher pressures both laboratories used static-expansion systems. The results for the lower pressures are summarised in Table 10.

Gauge	Mean σ	
	ETL	IMGC
gauge 3	0.980	0.982
gauge 4	0.994	1.003
gauge 11	0.978	0.980
gauge 12	0.972	0.973
principle	static	dynamic

Table 10: ETL - IMGC comparison - averaged results

The results agree to within 0.2%, which is well within the combined standard uncertainties, except for gauge 4, but all the ETL results have lower values than the corresponding IMGC results at the same pressures. The static-expansion - dynamic-expansion differences are of opposite sign to those of earlier comparisons (see sections 3.2.1 3.2.2.1 and 3.3) but their magnitude is small and these results therefore do not counterbalance the apparent systematic nature of earlier observations.

2.2.3.2 KRISS - NPL comparison

The high vacuum standards of the Korean Research Institute of Standards and Science (KRISS) and NPL, UK were compared at the end of 1997 at four pressures in the range 4.5×10^{-3} Pa to 1.25×10^{-2} Pa using argon [15]. The standard used by KRISS was based on dynamic-expansion and had a standard uncertainty of 0.5%. NPL used its static-expansion system, with a standard uncertainty of 0.35%. The standards were compared by examining the values of the effective accommodation coefficients (σ_{eff}) determined in each laboratory using two SRGs.

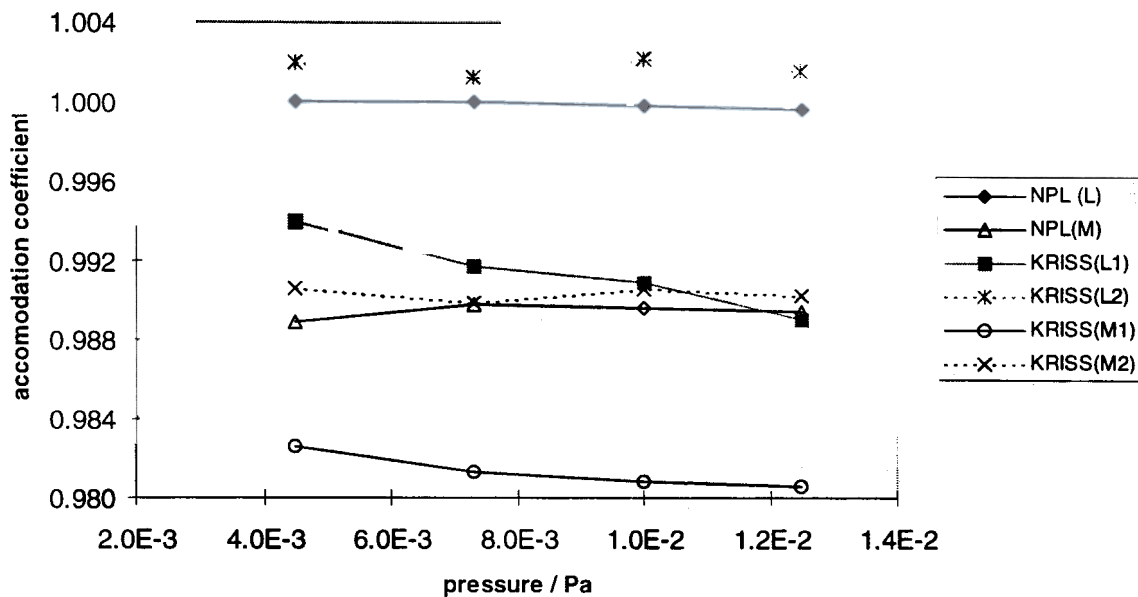


Figure 1: Results of bi-lateral KRISS - NPL comparison

The gauges were first calibrated at KRISS, then at NPL and finally re-calibrated at KRISS. First set of results obtained at KRISS showed a pressure dependence, which for one gauge approached 0.5% of the value of σ_{eff} . Such behaviour is not normally encountered at these pressures and it was not observed in later calibrations. Moreover, the shift between the first and the second data set obtained at KRISS was around 1%, in the same direction for both gauges; while this is rare and could indicate that there was a problem with the first KRISS calibration and/or standard, it is not impossible for SRGs to shift in such a way and without any further information, it was decided to keep both data sets.

The difference between KRISS(1) and NPL data varied between 0.6% and 1.1% depending on the gauge and the pressure considered, and was mostly outside the combined standard uncertainty. However, KRISS(2) and NPL results agreed to within 0.2%. If NPL results were compared with the mean of the two sets of data obtained by KRISS, then the maximum difference between the two laboratories was less than 0.4%, which was within the combined standard uncertainty.

RECENT INTERNAL NPL COMPARISONS

Comparison of static-expansion systems

Comparisons have been performed between the three NPL static-expansion systems, abbreviated as SEA II (high vacuum), SEA III (medium vacuum) and SEA V (high vacuum).

2.3.1.1 SEA II - SEA V comparison

An ionisation gauge and a spinning-rotor gauge (SRG) were calibrated a number of times against SEA II and SEA V using nitrogen gas.

The IG calibrations covered the range 9×10^{-7} Pa to 9×10^{-2} Pa and took place over a year. The mean gauge sensitivity value at each pressure was calculated for the SEA V and SEA II data; and the differences between the two systems is shown in Figure 12.

With the exception of one pressure point, the two systems agreed to within 0.6%, with an average difference around 0.4%, which is within the combined standard uncertainty for the two standards (0.7% to 0.4%). The difference between the systems appears to vary with pressure in a periodic fashion, although it should be noted that the magnitude of this effect is broadly in line with the repeatability and reproducibility of the transfer standard.

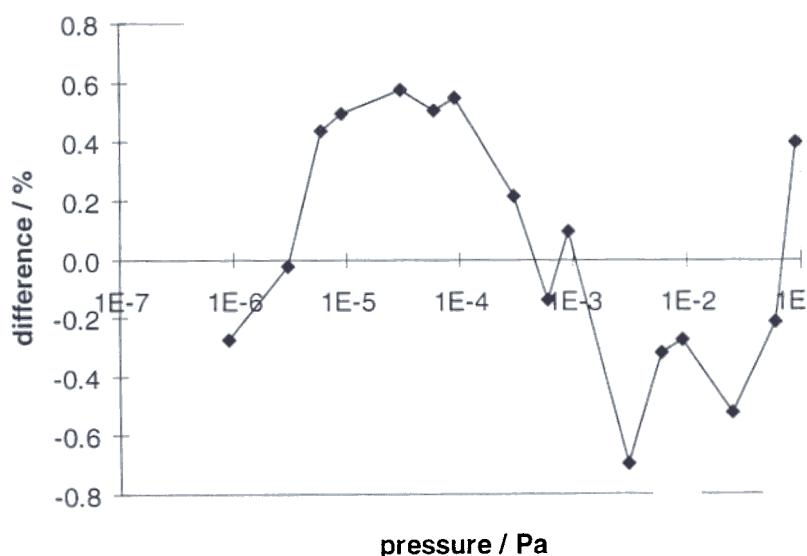


Figure 12: Difference between SEA II and SEA V using an ion gauge

The SRG calibrations covered the range 3×10^{-4} Pa to 3 Pa. Differences in the gauge's accommodation coefficient of between 0.1% and 0.6% were obtained, with the largest difference occurring at 3×10^{-4} Pa where the non-repeatability of the gauge's residual drag has the greatest influence. The average difference between the systems was around 0.3%, a figure substantially less than the combined uncertainty of the two standards (0.5% for $k = 1$) and less than the reproducibility associated with the spinning-rotor gauge (0.5%). The summary of the results is given in Figure 13 and Figure 14. The SRG accommodation coefficient was consistently higher when measured on SEA V indicating that the pressure in this system was higher than in SEA II.

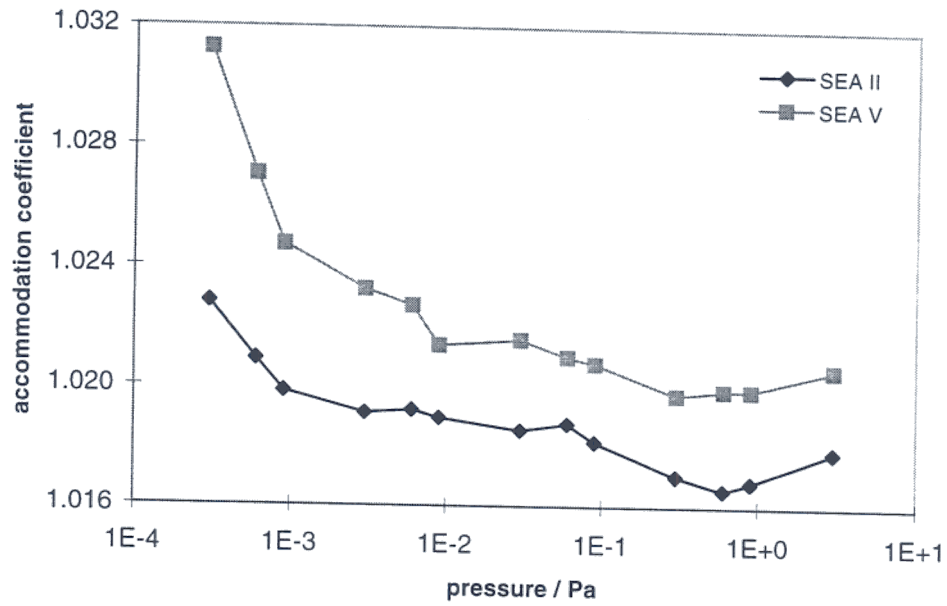


Figure 13: Comparison of SEA II and SEA V using an SRG

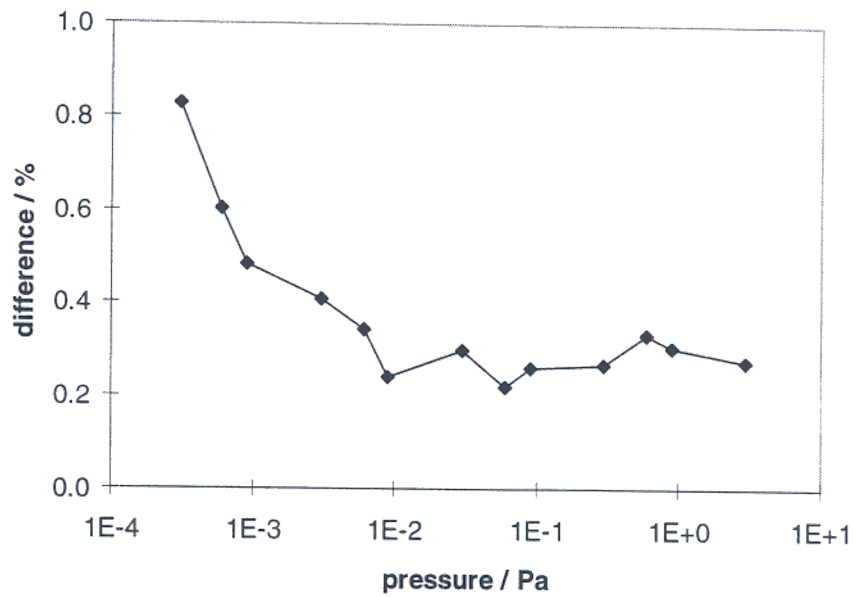


Figure 14: Difference between SEA II and SEA V using an SRG

2.3.1.2 SEA III - SEA V comparisons

A capacitance diaphragm gauge (CDG) with a full scale range of 133 Pa was calibrated a number of times against SEA III and SEA V using nitrogen gas. The calibrations covered the range 2×10^{-2} Pa to 70 Pa.

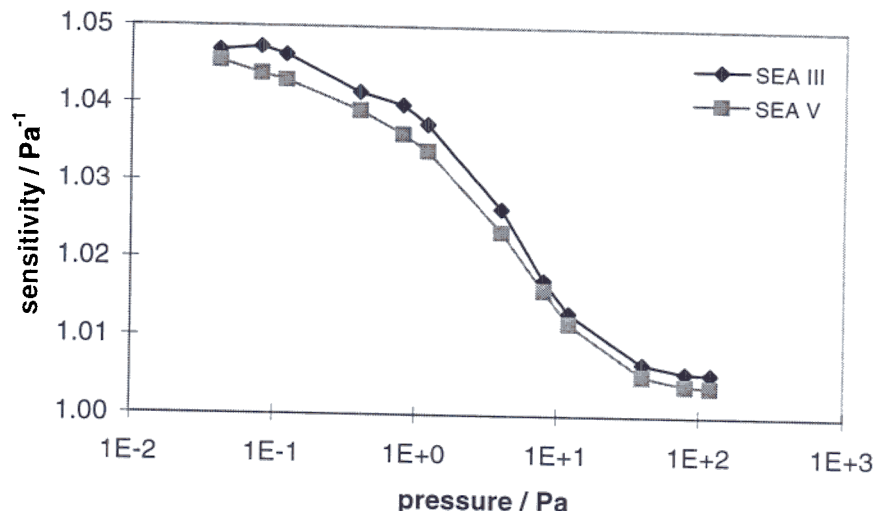


Figure 15: Comparison of SEA III and SEA V using a 133 Pa CDG

The results agreed to better than 0.3% with an average difference of around 0.2 %, which is within the combined standard uncertainties of the two standards (0.4% to 0.25%). The observed difference is in the opposite direction to the difference between SEA II and SEA V measured with the spinning-rotor gauge. However, the largest differences and variations occurred at the lowest pressures where the random scatter from the capacitance manometer is greatest and the differences at the higher pressures are comparable with the reproducibility of the capacitance manometer.

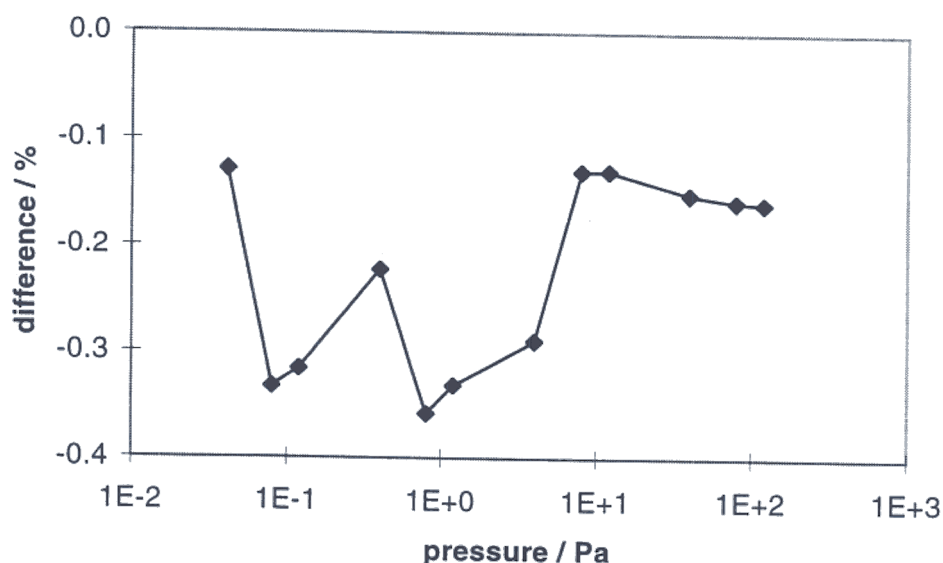


Figure 16: Difference between SEA III and SEA V using a CDG

2.3.2 Indirect comparison of static and dynamic-expansion systems

2.3.2.1 UHV system - SEA II comparison

The NPL ultra-high vacuum system (UHV) is based on the dynamic-expansion principle. However, due to the very low pressures generated within the UHV system the flow-rate is not established using a flow-meter; instead multiple stage pressure reduction is used. Gas from the inlet chamber flows through a very small orifice into the calibration chamber and then through a second orifice into a cryopump. The diameter of the first orifice is sufficiently small that the pressure downstream from the orifice may be considered negligible when compared to the upstream pressure. The flow-rate may therefore be calculated from the conductance of the first orifice and the pressure in the inlet chamber, measured by either a spinning-rotor gauge or an ionisation gauge which are traceable to the high vacuum static-expansion system. The uncertainty in the flow-rate is therefore very much higher than that associated with the dynamic-expansion system (see section 2.3.2.2).

A comparison between the UHV system and the high vacuum static-expansion system (SEA II) was performed using an ionisation gauge as a transfer standard. The calibrations covered the range 6×10^{-7} Pa to 9×10^{-4} Pa.

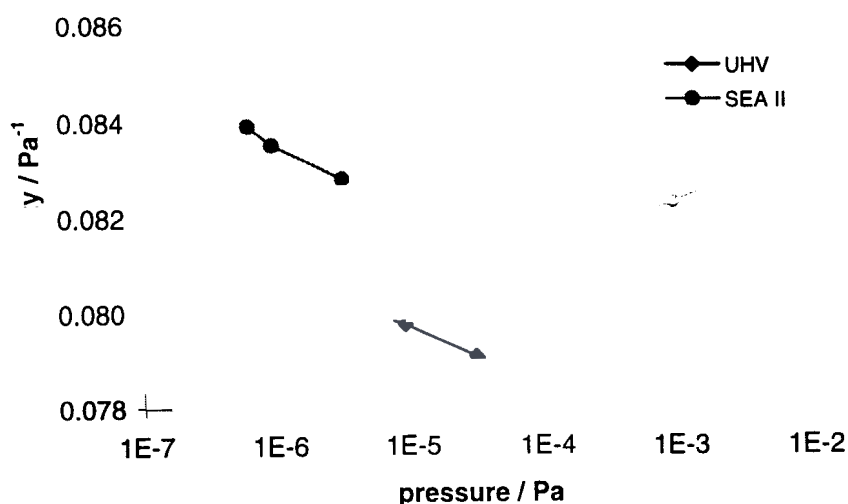


Figure 17: Comparison of UHV and SEA II using an ionisation gauge

Although the results agreed within the combined standard uncertainties (4%), it is notable that the sensitivity values obtained using the dynamic-expansion system were again lower than those obtained using the static-expansion system by about 3.5% over the most part of the comparison range. It should be noted however that the standard uncertainty associated with the UHV system (4%) dominated the combined value.

2.3.2.2 Dynamic-expansion system - SEA II comparison

The dynamic-expansion system (DES) uses a constant-pressure flow-meter, with two pistons, to measure the quantity of gas flowing into the calibration chamber. A 10 mm ‘small’ diameter piston was used for generating very low pressures (up to a few $\times 10^{-3}$ Pa), whilst a 25 mm ‘large’ diameter piston was used for establishing higher pressures.

The comparison between the DES and SEA II was performed using a spinning-rotor gauge as the transfer standard. Calibrations on the DES were made using the flow-meter pistons in turn.

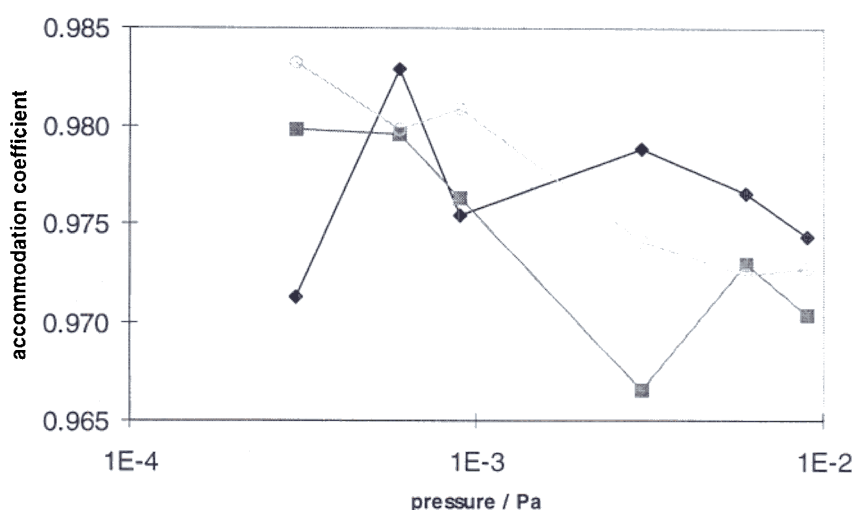


Figure 18: Calibration of an SRG using the DES small piston

The results of six calibrations on the DES, three performed using the small piston and three with the large piston, are shown in Figure 18 and Figure 19. The repeatability of the small piston results was worse than for the large piston.

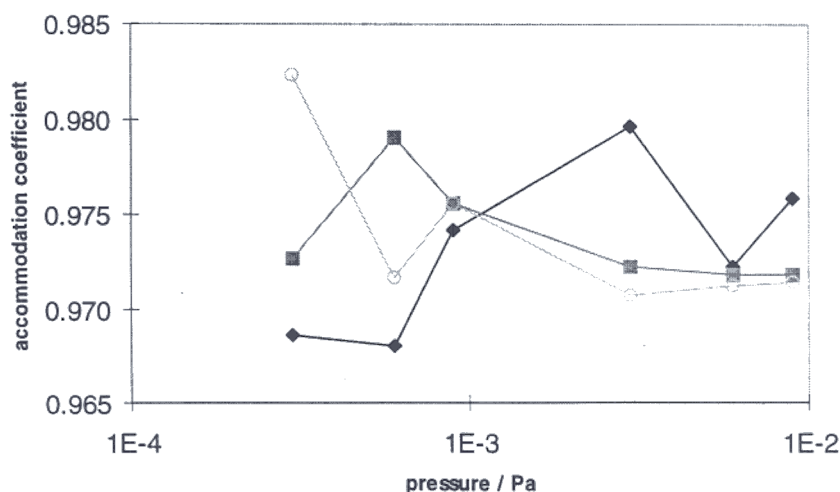


Figure 19: Calibration of an SRG using the DES large piston

Figure 20 shows a summary of the results obtained for both pistons, together with the data from SEA II. The combined uncertainty ($k = 1$) for the two standards was 0.5%.

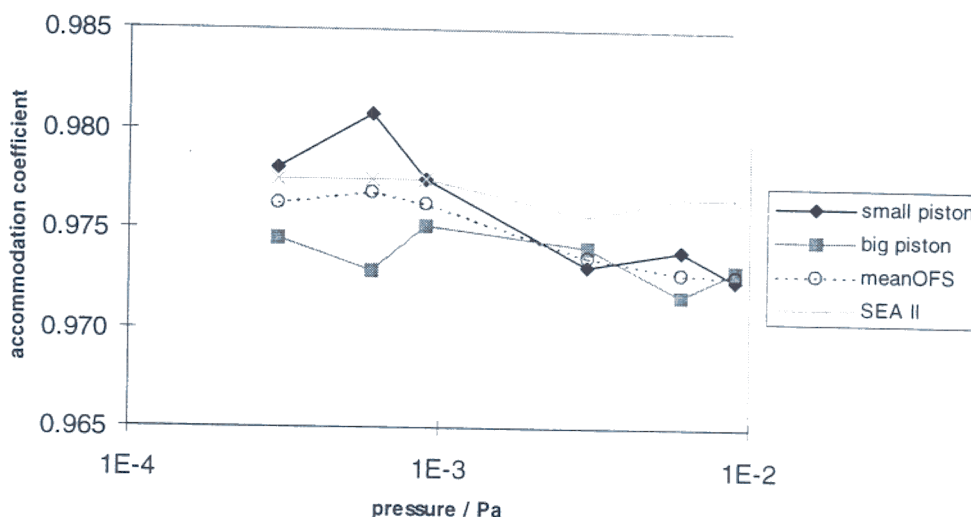


Figure 20: Comparison of static and dynamic systems using an SRG

2.3.3 Quasi-direct comparison of static and dynamic-expansion systems

2.3.3.1 Physical connection of two primary vacuum standards

The two types of primary standard, static and dynamic-expansion, both *generate* calculable pressures rather than *measuring* them; hence they cannot be compared directly but require the use of transfer gauges. As mentioned earlier in section 2.2, it is recognised that the integrity of international comparisons in the vacuum region can be severely limited by the stability and performance of the gauges used as transfer standards, particularly at lower pressures. Even for comparisons within an individual laboratory the stability of the transfer standards may be less than ideal.

One way in which the performance of the transfer standards may be improved is to avoid changes in their environment by, for example, not moving them, keeping them under vacuum and leaving them switched on. At NPL a unique experiment was developed which enabled the quasi-direct comparison of a high vacuum static-expansion system (SES) and the dynamic-expansion (DES). The two systems were installed next to each other, enabling them to be physically connected via a specially designed pressure-lock manifold (see Figure 21). The manifold enabled gauges to be calibrated against both standards in rapid succession without switching off or moving the gauge, and without exposing it to significant pressure changes - thus improving their reproducibility.

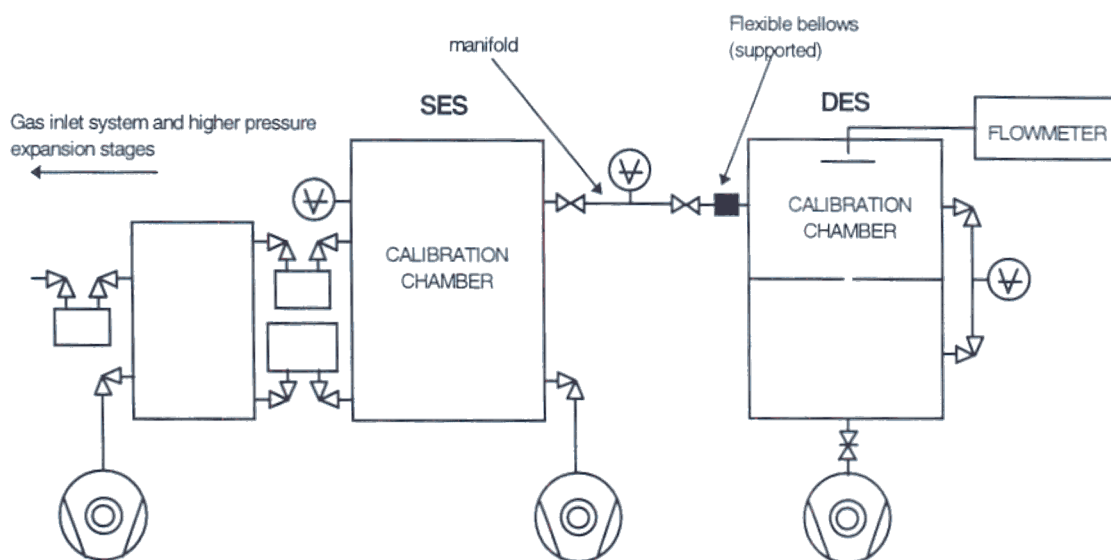


Figure 21: Schematic of quasi-direct comparison of static and dynamic-expansion systems

The manifold's design was dictated by the sometimes contradictory requirements imposed by both systems including the need to prevent gas migrating from one system to the other, to minimise disturbances in the gas distribution within each system and to minimise pressure gradients between the gauge and the calibration chamber. The isolating valves at both ends of the manifold and the flexible bellows used to align the two systems were positioned to minimise the variable volume (due to the internal bellows in the valves) connected to the static-expansion system. The manifold incorporated six gauge ports and had a volume of approximately 1.4 litre. The resultant increase in the volumetric ratios of the calibration chamber of the static-expansion system due to the presence of the manifold and isolating valves was measured and found to be around 1.7%.

2.3.3.2 Comparison protocol

The two systems were compared using a spinning-rotor gauge over the pressure range 10^{-4} Pa - 10^{-1} Pa. Measurements were made with nitrogen gas and three spinning-rotor gauges (SRGs) were used, one mounted on the calibration vessel of each standard and the third on the manifold. To avoid pressure-cycling of the gauges, the same nominal pressure was simultaneously generated in each standard. For each calibration point the manifold was first opened to the static-expansion system and the manifold-mounted SRG calibrated. The manifold was then isolated from the static-expansion system and opened to the dynamic-expansion system and the SRG was again calibrated. The chamber-mounted SRGs were used to confirm the repeatability of their respective systems. The time interval between completing a static-expansion system measurement and starting a corresponding dynamic-expansion system measurement was about 5 minutes but the preparation time between pressure changes was much longer, hence only about 5 measurement pairs could be completed per day, causing a 3-decade calibration to be split over two days.

2.3.3.3 Results

The results of two sets of measurements, in the pressure range 10^{-4} Pa - 10^{-1} Pa, are presented in Figure 22 and Figure 23 in terms of SRG effective accommodation coefficient. Figure 24 combines the data from the two previous figures and the error bars shown represent the standard uncertainties associated with the two primary standards. Figure 25 shows the spread in the accommodation coefficients for the manifold-mounted spinning-rotor gauge determined on each system for repeat measurements during individual calibration runs.

The dynamic-expansion system results have been corrected for the changes in the flow-meter pressures, measured at each calibration point. These changes had the largest effect at the lowest measured pressures. At the other end of the measured range, as the pressure approaches 10^{-1} Pa, the flow regime begins to change from molecular to transitional flow causing the conductance of the orifice to change.

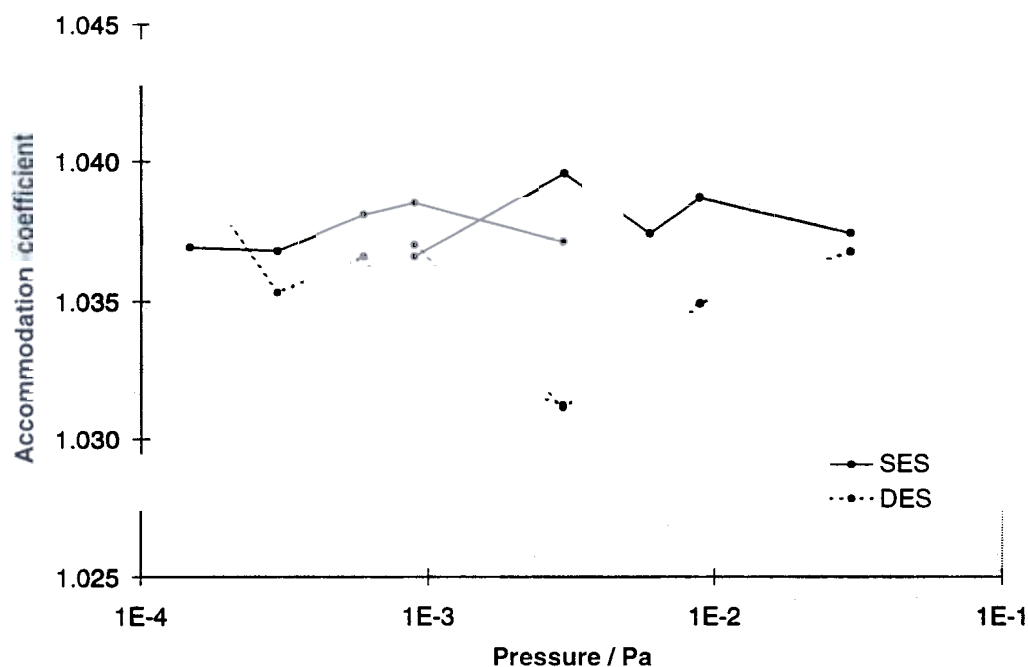


Figure 22: First set of measurements of the quasi-direct comparison

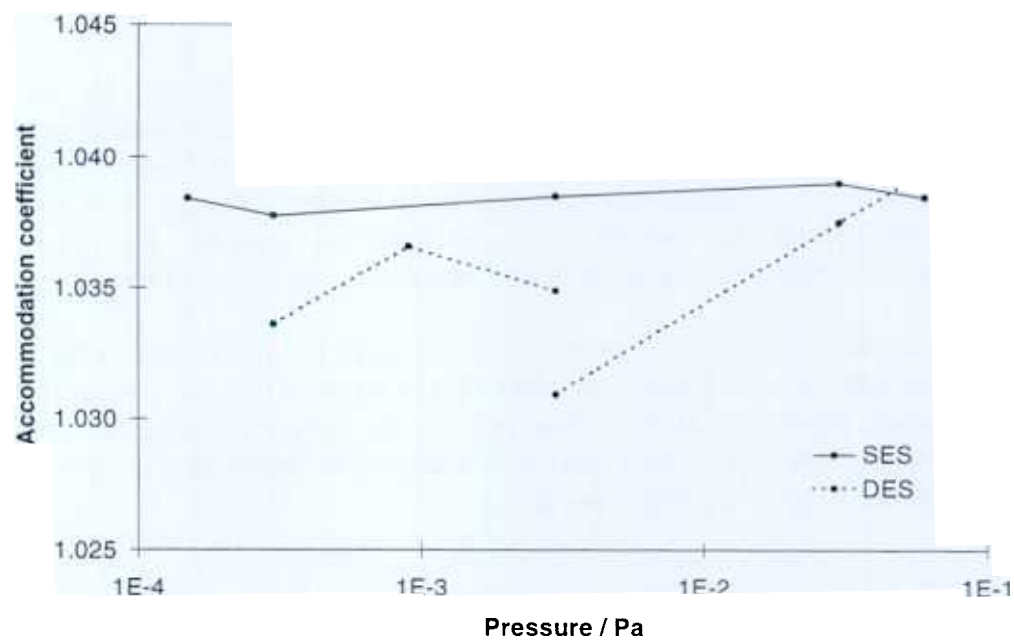


Figure 23: Second set of measurements of the quasi-direct comparison

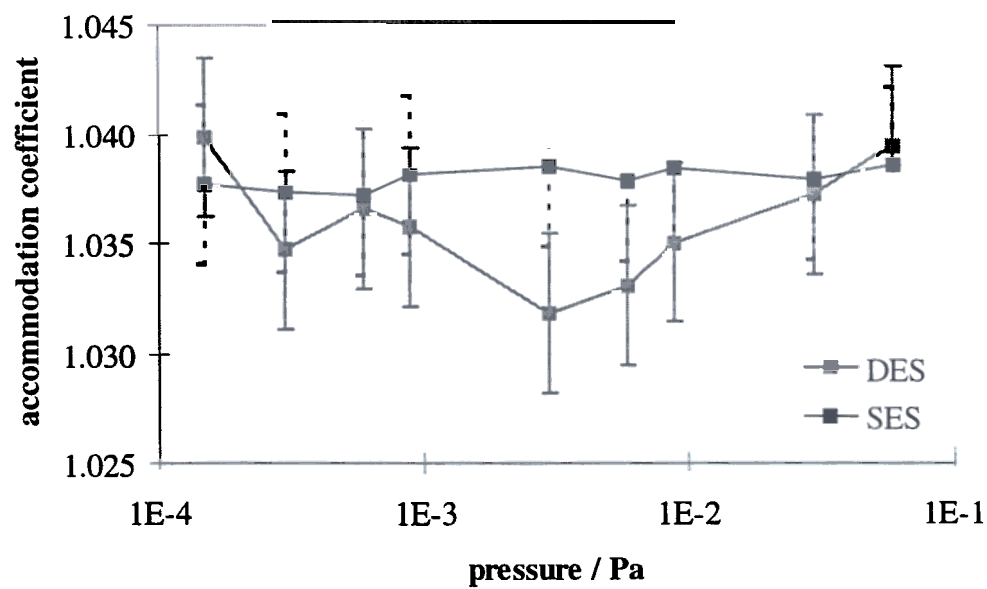


Figure 24: Combined results of the quasi-direct comparison

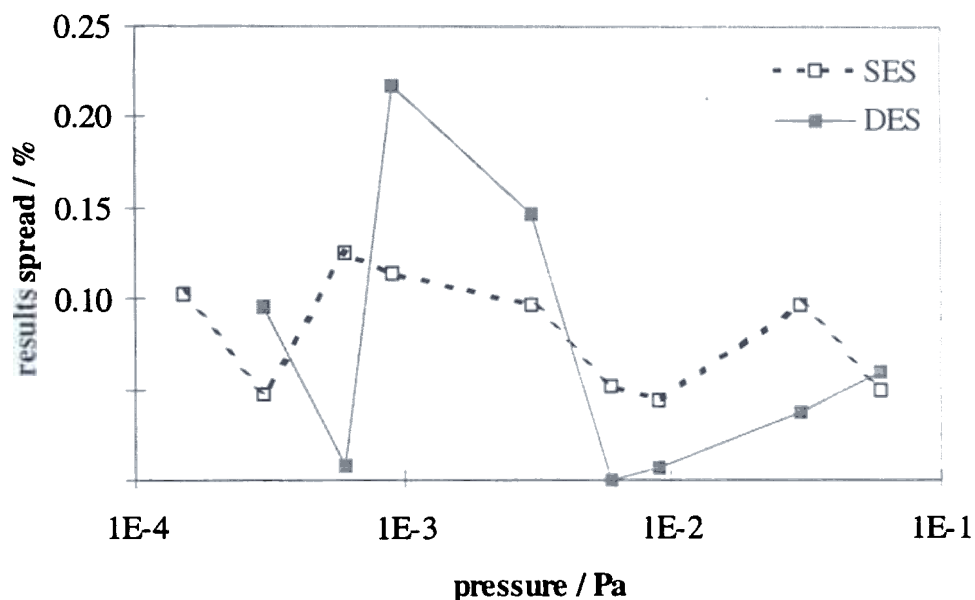


Figure 25: Spread of results obtained for the SRG accommodation coefficient

The median difference in results between the systems was about 0.3% which was comparable to the data scatter observed during the measurements, and was within the combined standard reference pressure uncertainties for a single measurement: 0.35% for the static-expansion system and 0.45% for the dynamic-expansion system. The maximum observed difference was 0.6%. Data from the calibration vessel mounted SRGs confirmed the apparent poorer reproducibility of the dynamic-expansion system. It is noticeable that spinning-rotor gauge accommodation coefficients obtained from measurements on the dynamic-expansion system were consistently lower than those obtained using the SES.

3. WEAKNESSES AND IMPROVED MODELLING OF VACUUM STANDARDS

3 DETERMINATION OF VOLUMETRIC RATIOS FOR STATIC-EXPANSION SYSTEMS

3.1 Overview

In the medium and high vacuum region, static-expansion systems are used as primary standards by a number of national metrology institutes [16]. As mentioned in 2.1.1, calculation of the pressures generated by these standards relies on knowledge of the volumetric ratios associated with the various combinations of vessels and these ratios form a critical component in the characterisation of the standards. In order to avoid disassembly of the system an in-situ protocol is used. Due to the irregular and complex geometry of the vessels, dimensional metrology is inappropriate for the measurement of the volumes. Gravimetric techniques, eg filling a volume with a liquid of known density and weighing, have been employed by some laboratories [17, 18], but these methods are not appropriate for the regular re-evaluation required to monitor changes in the ratios, for example due to wear in the valves.

A practical in-situ technique using multiple successive expansions was developed at NPL in the late 1970s [19, 20]. This technique was subsequently adopted by laboratories using static-expansion systems and has remained the prime experimental method since. The experimental protocol is described below.

3 .2 Experimental protocol

Consider a simple single-stage system comprising two vessels, one small and one large, with volumes V_S and V_L respectively, connected via a valve.

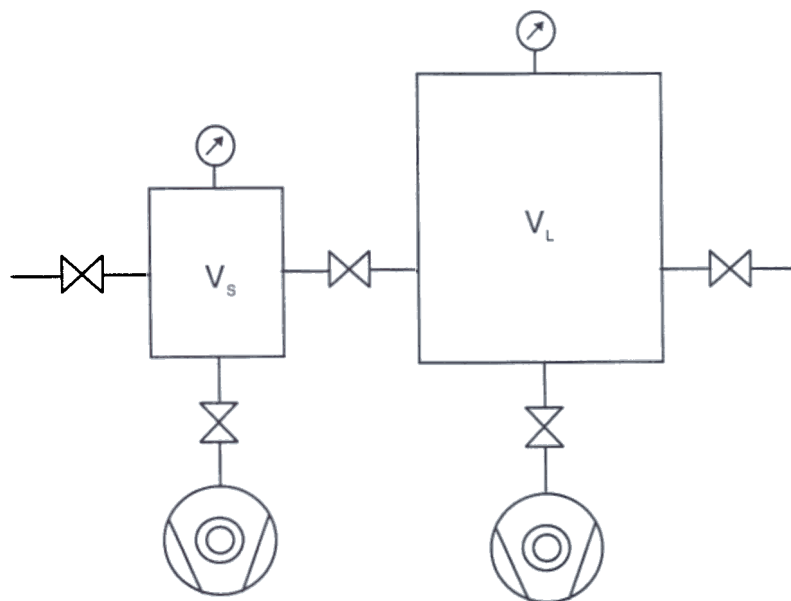


Figure 26: Experimental setup for volume ratio determination

Both vessels are evacuated to a low pressure and the valve connecting the small and large vessel is shut. The vessels are then isolated from the pumps and the small vessel filled with gas to a pressure p_s . The inter-connecting valve is then opened and the gas expanded into the large vessel. Using the ideal gas law the volumetric ratio ($R = (V_S + V_L)/V_S$) may be calculated from the initial pressure p_s , the pressure, p_L , in the large vessel after expansion and the initial and final temperatures T_S and T_L . However, after a single expansion the resulting pressure is often too low to be measured with adequate accuracy using a mechanical pressure gauge.

The solution developed at NPL [19, 20] was to accumulate successively higher pressures in the large vessel. This was achieved by repeatedly refilling the small vessel and expanding the gas into the large vessel, hence increasing the pressure. This protocol was repeated until the resultant pressure in the large vessel was high enough to be measured with sufficiently low uncertainty.

3.3 Original models for the calculation of volumetric ratios

3.1.3.1 Calculation of the volumetric ratio under isothermal conditions

The following expression for the volumetric ratio determined under isothermal conditions was derived by Elliott and Clapham [19, 20]:

$$R = \left\{ 1 - \left(\frac{(p_L)_N}{p_s} \right) \right\}^{1/N} \quad (7)$$

where the parameters related to the small vessel are indexed with S, and those related to the large vessel are indexed with L, hence $(p_L)_N$ is the pressure in the large vessel after the Nth expansion. The derivation assumed that the pressure in the large vessel, prior to the first expansion, was negligible and that the temperatures of the two vessels were identical and constant throughout the experiment.

3.1.3.2 Elliott and Clapham's method for temperature corrections

Unfortunately isothermal conditions are rarely encountered in practice and it is necessary to correct for the temperature gradient between the small and the large vessel and the drift in the vessel temperatures during the experiment. However, a simple explicit equation for the volumetric ratio, determined by successive expansions [19, 20], may only be derived under isothermal conditions. Various methods to account for the temperature gradients between the vessels and the drift in the vessel temperature during the experiment have been derived. Elliott et al [19, 20] accounted for the effect of the temperature gradient and drift separately, with the mean isothermal ratio multiplied by the two corrections following the completion of the determination. Their corrections for temperature drift and temperature gradient respectively are given below:

$$C_{\text{Tdrift}} = 1 + \left(\frac{(T_L)_N - (T_L)_i}{2(T_L)_i} \right)$$

$$C_{\text{Tgradient}} = \frac{(T_L)_i}{(T_S)_i}$$

3.1.4 Additional corrections to the volumetric ratio

In the above derivations it is assumed that the gas obeys the ideal gas law. For the gases commonly used, such as nitrogen and argon, at ambient temperature and the pressures encountered, the departures from the ideal gas law are small and can be corrected by multiplying the calculated ratio by the factor $(1-B/v_m)$, where B is the second virial coefficient and v_m is the molar volume of the gas in the small vessel prior to expansion. Secondly, the volume of the large vessel is changed from the normal configuration by the presence of the gauge used to measure the expanded pressure and this additional volume, V_G , may be accounted for by subtracting the term V_G/V_S from the measured ratio.

3.1.5 Improved models for volumetric ratio determination

Two alternative models for the calculation of volumetric ratios from multiple successive expansions have been developed at NPL [20], aimed at improving the corrections for non-isothermal temperatures. The two methods are described below; the first method is applicable to the calculation of the ratios during the experiment, whereas the second method is aimed at an improved estimation of the uncertainties associated with the calculation of the ratios.

3.1.5.1 Iterative method for temperature corrections

This method for correction of thermal effects utilises the fact that pressures at one temperature may be transformed to equivalent values at a different reference temperature, ie $(p_L/T_L) = (P_L/T_{\text{ref}})$. Here $(P_S)_i$ and $(P_L)_i$ represent the pressure values after correction to the reference temperature, whereas $(p_S)_i$ and $(p_L)_i$ relate to the values at the original temperatures. Unlike the method of Elliott and Clapham, the small vessel pressures are not assumed to remain constant. For each expansion the equation for the conservation of molecules is re-arranged to express the pressure in the large vessel in terms of P_S , V_S and V_L . This expression is then substituted into the equation derived for the next expansion, thus enabling the pressure after expansion to be expressed in terms of the volumes, and the pressures in the small vessel prior to expansion.

After the first expansion

$$\frac{(p_S)_i V_S}{(T_S)_i} = \frac{(p_L)_i (V_S + V_L)}{(T_L)_i}$$

but using the substitution $(P_L/T_L) = (P_L/T_{ref})$ and similar expression for the pressure in the small vessel we have

$$\frac{(P_S)_1 V_S}{(T_{ref})_1} = \frac{(P_L)_1 (V_S + V_L)}{(T_{ref})_1} \quad (11)$$

$$(P_L)_1 = \frac{(P_S)_1 V_S}{(V_S + V_L)} \quad (12)$$

After the second expansion

$$(P_S)_2 V_S + (P_L)_1 V_L = (P_L)_2 (V_S + V_L) \quad (13)$$

$$(P_L)_2 = \frac{(P_S)_1 V_S V_L}{(V_S + V_L)^2} + \frac{(P_S)_2 V_S}{(V_S + V_L)} \quad (14)$$

Continuing this protocol we obtain for the Nth expansion

$$(P_L)_N = \frac{V_S}{(V_S + V_L)} \left\{ (P_S)_1 \left(\frac{V_L}{V_S + V_L} \right)^{N-1} + (P_S)_2 \left(\frac{V_L}{V_S + V_L} \right)^{N-2} + \dots + (P_S)_N \right\} \quad (15)$$

$$(P_L)_N = \frac{1}{R} \sum_{i=1}^N \left\{ (P_S)_i \left(\frac{R-1}{R} \right)^{N-i} \right\} \quad (16)$$

hence

$$R = \frac{1}{(P_L)_N} \sum_{i=1}^N \left\{ (P_S)_i \left(\frac{R-1}{R} \right)^{N-i} \right\} \quad (17)$$

The temperature corrected ratio is independent of the reference temperature; at NPL the nominal laboratory temperature, 20 °C, is used. Equation (17) may not be solved explicitly so an iterative method is applied to solve equation (17) after each expansion. For the first iteration an estimate for R is entered into the equation; for the first expansion the value for the isothermal ratio is used but for subsequent expansions the temperature corrected ratio obtained from the previous expansion is used. The value of R calculated after each iteration is then re-entered into the right-hand side of the equation and the process repeated until the value obtained differs by less than 0.0001 from the previous value. The function converges rapidly and using the initial estimates described above only two iterations are typically required.

3.1.5.2 Generalised regression method for temperature corrections

A different approach was taken in a method developed at NPL [20], whereby the solution for R reflects the uncertainties associated with the input parameters. For each expansion the

virial equation of state, approximated to the second term, is used to express the number of molecules in each vessel before and after expansion, assuming conservation of molecules. Estimates for the five input parameters, $(p_S)_i$, $(p_L)_i$, $(T_S)_i$, $(T_L)_i$ and B (the second virial coefficient) are represented by expressions of the form $(p_L)_i^* = (p_L)_i + u_1 + u_2 + (u_3 + u_4)(p_L)_i + \varepsilon_i$. Here u_1 and u_3 represent the absolute and relative errors associated with the calibration of the gauge, u_2 and u_4 represent the absolute and relative drift in the characteristics of the gauge since its calibration and ε_i the uncorrelated random errors sampled from a normal distribution with mean zero. Unlike other methods, the term $(p_L)_i$ is not eliminated by substitution from the equation for the i^{th} expansion. An estimate of R , along with its associated uncertainty, is obtained by application of generalised regression techniques to the matrix of equations.

In this approach, the values of the model parameters (that is the volumetric ratio and the parameters describing the behaviour of sensors) are determined by minimising an error function that takes into account the major sources of measurement error, both systematic and random. Although the computation involves the solution of a moderately large non-linear least squares system, its potential advantages are that: a) the non-isothermal conditions and the non-ideal behaviour of the gas are incorporated directly into the model and b) the estimates and their uncertainties more closely reflect what is known about the measurement system. This technique is most applicable for the subsequent reprocessing of the data following completion of the measurements.

3. 6 Weaknesses in volumetric ratio models and experimental protocols

3.1.6.1 Mathematical models

As mentioned earlier, the equations developed by Elliott et al [19, 20] have been adopted and used by laboratories over many years. In their calculation the gradient between vessels was assumed to remain constant throughout the measurements, an assumption which is often a close approximation to reality, although errors can occur if temperature changes are significant or erratic. However, Elliott and Clapham's expression for the correction of temperature drift is flawed by assumptions about the quantity of gas which changes temperature after each expansion (due to the drift in temperature). The results obtained using Elliott's method was compared with the iterative method using data from the NPL static-expansion systems. Whilst the differences between results for the two methods were small, generally of the order of 0.02%, they could exceed 0.06% under unfavourable conditions, for example when the rate-of-change of temperature is rapid, variable or bi-directional. It is, of course, possible for even larger errors to have arisen at laboratories with less well controlled environmental conditions.

The effect on the repeatability of the ratios due to the improved corrections for temperature changes obtained using the iterative method is shown Table 11. The ratios were calculated using the same experimental data for both methods of calculation. With one exception, the repeatability of the ratios is significantly lower for the iterative method of calculation than for the Elliott and Clapham method, probably indicating that the iterative method more readily accommodates changes in environmental conditions both during individual measurement runs and also between determinations. The repeatability for the isothermal method was worse than that for both the Elliott and Clapham method and the iterative method.

System	Expansion ratio	Elliott et al method		Iterative method	
		ratio	<u>std dev</u> %	ratio	<u>std dev</u> %
SEA II	v2→v2+V2+v3	282.920	0.024	282.916	0.013
	v3→v3+V3+v4	450.436	0.008	450.438	0.001
	v4→v4+V4+v5	84.554	0.021	84.558	0.001
	v5→v5+V5	110.964	0.001	110.977	0.012
SEA V	v1→v1+V1+v2	196.518	0.012	196.514	0.005
	v2→v2+V2+v3	197.333	0.007	197.346	0.008
	v3→v3+V3+v4+v4'	196.895	0.009	196.885	0.007
	v4→v4+V4	295.397	0.047	295.364	0.027
	v4'→v4'+V4	99.097	0.031	99.085	0.010

Table 11: Repeatability of volume ratios calculated by two different methods

An alternative method by Jousten [18] utilises the isothermal ratio derived by Elliott et al to produce an approximate solution to the equations for the conservation of gas molecules to obtain a correction for the combined effect of temperature gradient and drift. Comparison between this method and the NPL iterative method showed agreement to better than 0.002%.

3.1.6.2 Measurement of the vessel temperature

In addition to the model induced errors, the importance of the measurement of the temperature of the vessels has not always been appreciated. Early determinations of volumetric ratios using successive expansions were made with minimal, if any, temperature measurements. Subsequent to the work of Elliott and Clapham the temperatures were sometimes measured, but often relied on only a single thermometer per vessel. Even as recently as the late 1980s the vessel temperature was not always measured [17]. Using data from recent volumetric ratio determinations with multiple thermometers on each vessel, the authors have studied the effect of the various thermal corrections. For the NPL determinations considered, the temperature gradient between the small and large vessels was typically a few tenths of a degree Celsius and gradients across the individual vessels typically ranged from 0.2 °C to 0.4 °C. A number of volumetric ratios were re-calculated using the iterative method using data from individual thermometers on each vessel and some typical results are presented in Table 12 and Table 13.

Isothermal ratio	Elliott et al method	Iterative method		
		multiple probes	single probe	single probe
450.00	450.46	450.44	450.70	450.26
282.56	282.97	282.94	282.83	283.06
110.88	110.96	110.97	111.09	110.85
84.52	84.57	84.56	84.52	84.60

Table 12: Temperature effects on volumetric ratio calculations for a typical set of measurement results

% differences from iterative method ratio using multiple probes			
Iterative method		Elliott et al method	Isothermal ratio
single probe	single probe	multiple probes	
-0.04	+0.06	+0.00	-0.10
-0.04	+0.04	+0.01	-0.13
-0.11	+0.11	-0.01	-0.08
-0.05	+0.05	+0.01	-0.05

Table 13: Errors due to poor correction for temperature for the data presented in Table 12

Differences exceeding 0.2% for a single ratio are apparent between results calculated using different probes. The errors caused by inadequate temperature measurement can exceed the errors of not applying any corrections for temperature effects and can exceed the claimed uncertainty in the determination of the volumetric ratios, showing the estimate to be over-optimistic. The isothermal ratio is usually smaller than the temperature corrected ratio because it is much less common for the temperature to fall during the measurements rather than rise, and the small vessel temperature is often lower than that of the large vessel. For these reasons, laboratories which have not applied temperature corrections to their measured ratio values tend to have lower ratio values and hence higher values for the calculated pressure generated within the static system and correspondingly lower sensitivity figures. An example of the effect of the various temperature corrections is shown graphically in Figure 27.

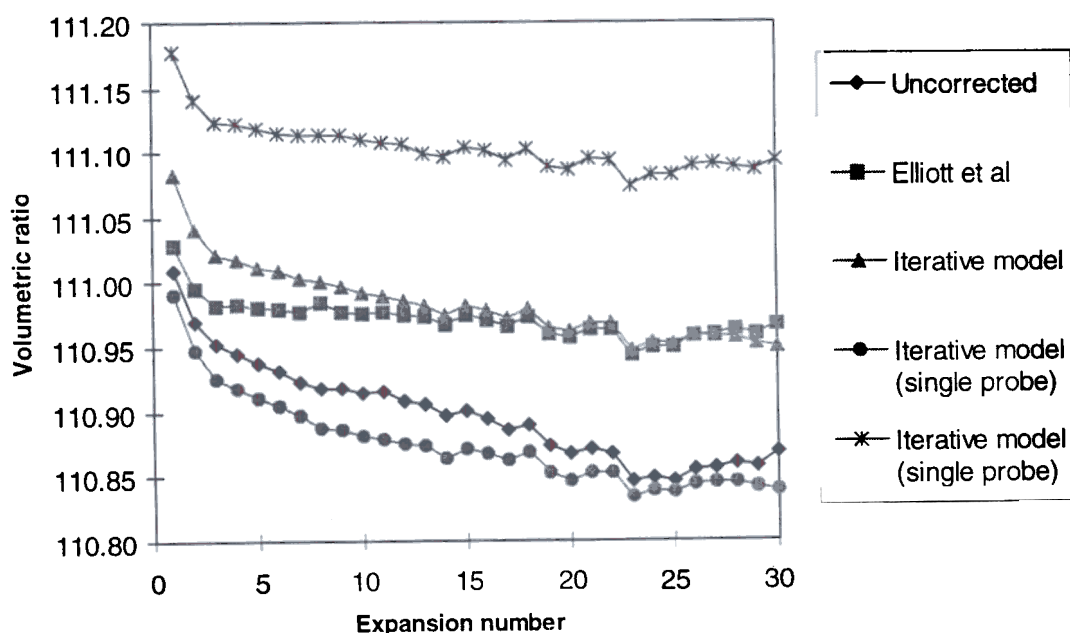


Figure 27: Volumetric ratios calculated by different methods using the same data

3.1.6.3 *Estimation of volumetric ratio stability*

Due to the time-consuming nature of volumetric ratio determinations, the statistical sample from which the stability of the volumetric ratios may be estimated is small. An estimate of the stability is obtained from historical data by considering the changes in the values of the ratios between consecutive calibrations, although re-determinations resulting from the replacement of a faulty component can further reduce the pool of usable data. It has sometimes been the case that researchers either overlooked or were unaware of the fact that the ratios may have been determined under different experimental conditions, using different models or less robust estimations of the temperatures of the vessels. As can be seen from the previous sections these errors can be significant and this can lead to over- and under-estimates of the stability of the volumetric ratios and in extreme cases the perceived direction of the drift of the ratio may be wrong. As a consequence of these failings, errors in the estimation of uncertainties can persist long after the faulty model or inadequate experimental protocol have been replaced. More robust estimates of the stability of volume ratios would therefore be obtained if the ratios were determined using methods which more adequately account for changes in the environmental conditions.

3.2 EXPERIMENTAL WEAKNESSES IN THE TEMPERATURE CORRECTION APPLIED TO PRESSURES GENERATED BY STATIC-EXPANSION SYSTEMS

The straightforward equation for the pressure generated by a static-expansion system assumes that the expansions are performed under isothermal conditions. However, as with the determination of volumetric ratios this scenario rarely encountered in practice and temperature differences between vessels and temperature gradients across vessels will influence the pressure generated within the system. In order to correct for the gradients between neighbouring vessels it is usual just to measure the temperature of the first small vessel and the calibration chamber and to multiply the calculated pressure by the correction factor $T_{L(N)}/T_{S(F)}$, where $T_{S(F)}$ is the temperature (in kelvin) of the first small vessel used for the expansion and $T_{L(N)}$ is the temperature of the last large vessel (calibration chamber). This simplified correction assumes that the temperature of the gas in each small vessel is equal to the temperature of the preceding large vessel. This will only be true if there is good mixing of the gas between the two vessels and neither vessel is affected by significant heat sinks or sources. Following isolation of the gas in the small vessel, the temperature of the gas will be affected by the length of time the gas is allowed to equilibrate prior to expansion.

Temperature gradients between neighbouring vessels during calibrations can be larger than those observed during volume ratio determinations due to the presence of additional heat sources, for example ionisation gauges and increased number of pumps in operation. Temperature gradients across the vessels can also be increased. The magnitude of the overall temperature correction to the generated pressures can often approach 0.2%. If the temperature gradients across the vessels are comparable with the gradients between the vessels, then inadequate temperature measurement (the use of a large number of thermometers may be impractical) and/or the unsuitable location of thermometers can cause significant errors in the temperature correction and in the extreme this can result in a correction of the opposite sign to that predicted. Like the determination of volume ratios, the errors caused by inadequate temperature measurement can exceed the error arising from non-application of the correction and may exceed the estimated uncertainty due to thermal effects.

3.3 INFLUENCE OF TIMING ON THE GENERATION OF PRESSURES BY STATIC-EXPANSION

3.3. Effect of timing of expansion sequence on the generated pressure

The expansion of gas during the generation of reference pressures within a static-expansion system is carried out according to a regular timing sequence. This sequence should be repeatable so that the influence of outgassing within the vessels can be determined and, if appropriate, a correction made. The timing of the sequence of valve operations should also be such that the gas reaches thermal and molecular equilibrium at each expansion stage and that the disturbance to the distribution of molecules due to the closure of the valves is minimised.

In order to investigate whether the timings used for both the expansion sequence and the operation of the valves were optimum and to see if the generated pressure could be significantly affected by the choice of these parameters, a number of experiments were performed. Measurements were made with nitrogen at a pressure of 6×10^{-3} Pa on a high vacuum static-expansion system (SEA II) using four spinning-rotor gauges. Spinning-rotor gauges were chosen rather than ionisation gauges due to their superior repeatability. This pressure was generated using three expansions and was a compromise between the reduction of the influence of instabilities in the residual drag of the SRGs yet being sufficiently low that outgassing and thermal effects might still contribute. The system has manually operated valves and under normal conditions the gas is trapped in the small vessel for 15 seconds before being expanded into the next large vessel and allowed to equilibrate for 45 seconds. The effects of both the time taken for each expansion and the rate of closure of the valves leading into the small vessels were investigated in the six experiments described below:

- Experiment 1: normal timing for expansions; all valves operated at normal speed.
- Experiment 2: total time per expansion stage halved - hence the gas was left in the small vessel for ~8 seconds and in the large vessel for ~22 seconds; all valves operated at normal speed.
- Experiment 3: total time per expansion stage doubled - hence the gas was left in the small vessel for ~30 seconds and in the large vessel for ~90 seconds; all valves operated at normal speed.
- Experiment 4: normal timing for expansions, the inter-volume valves were shut as quickly as possible.
- Experiment 5: where possible normal timing for the expansions was used; the valves leading into the small vessels were operated slowly - taking around 1 minute to close, other valves were operated at normal speed.
- Experiment 6: check measurements; repeat of experiment 1. These measurements were used to ensure that no significant changes had occurred during the course of the experiments detailed above.

With the exception of experiment 6, at least three measurements were made for each experiment. The data obtained are shown in Figure 28 and in order to compare the results obtained for the various gauges the data for each gauge have been scaled to the mean of the data for that gauge for experiment 1.

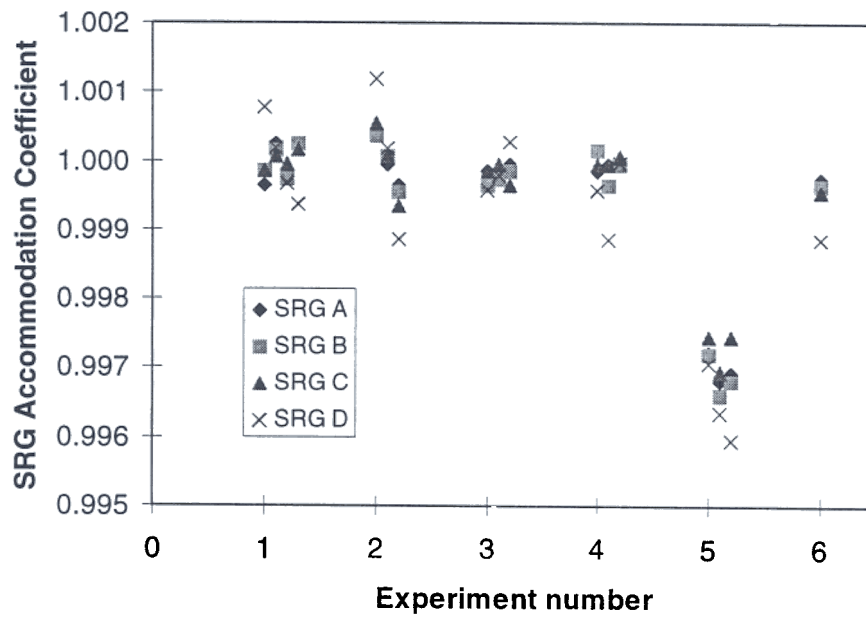


Figure 28: Scaled accommodation coefficients obtained during timing experiments

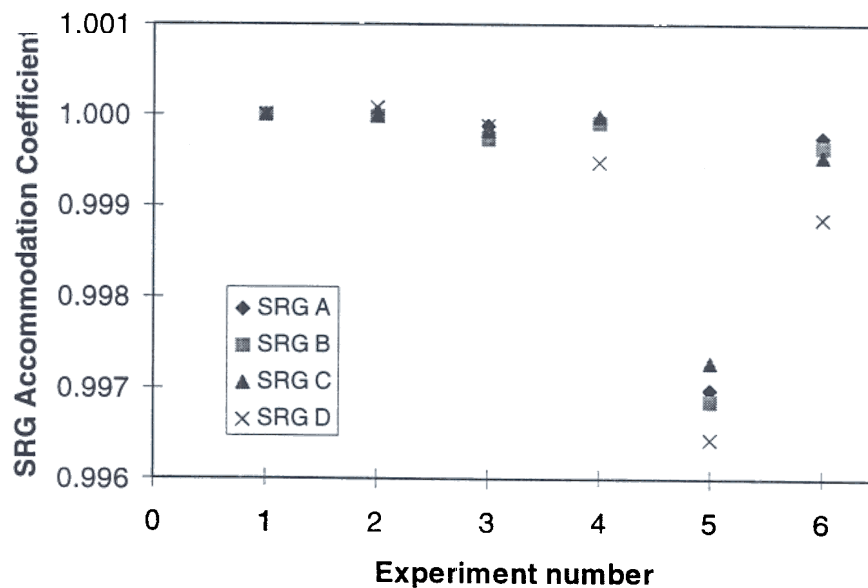


Figure 29: Mean scaled accommodation coefficients obtained during timing experiments

Figure 29 shows the mean scaled accommodation factor for each gauge for each experiment. In all the experiments the data for gauge D were considerably more scattered than those for the other three gauges. It is apparent that halving or doubling the time taken for each expansion stage had a negligible effect; the mean accommodation coefficient for experiment 2 (averaged over all gauges) was very similar to that of experiment 1, whilst the mean accommodation coefficient for experiment 3 was only 0.02% lower. The standard deviations of the accommodation coefficients for the each experiment were around 0.04%.

The results from experiments 2 and 3 indicate that the gas was in thermal and molecular equilibrium at each expansion stage and that within the repeatability of the measurements the timing of the expansion had a negligible effect on the pressure generated.

The results obtained for experiments 4 and 5, where the speed at which the valves were shut was modified, were significantly different. The accommodation coefficients obtained when the inter-volume valves were shut as quickly as possible were only 0.02% lower than those of experiment 1, and well within the standard deviation of the measurements. The comparability of the two sets of measurements may be due to the physical configuration of the systems which prevents the manual inter-volume valves being shut very quickly, hence the difference in the speed of closure may not have been very great. The accommodation coefficients for experiment 5, when the valves leading into the small vessels were shut very slowly, were 0.3% lower than those of all the other experiments. Further investigation of this phenomenon was therefore carried out.

3.3.2 Effect of speed of operation of valves

From 3.3.1 it was apparent that the rate at which valves were closed could have a significant influence on the pressure generated. Investigation concentrated on the speed of closure of the valves leading into the small vessels. The valves to the pumps were closed whilst the system was at base pressure prior to the expansion of the gas and hence would not affect the gas distribution, and the rate at which the valve leading out of the small vessel was opened would not influence the expansion of the gas. Reducing the speed of closure yet further did not result in an additional decrease in the accommodation coefficient, but only in increased scatter of the data. In order to investigate any pressure dependence, three spinning-rotor gauges were calibrated over the range 3×10^{-4} Pa to 3 Pa using nitrogen. Measurements were made in ascending pressure order and at each pressure the accommodation coefficient was determined for both normal and slow valve closure speeds.

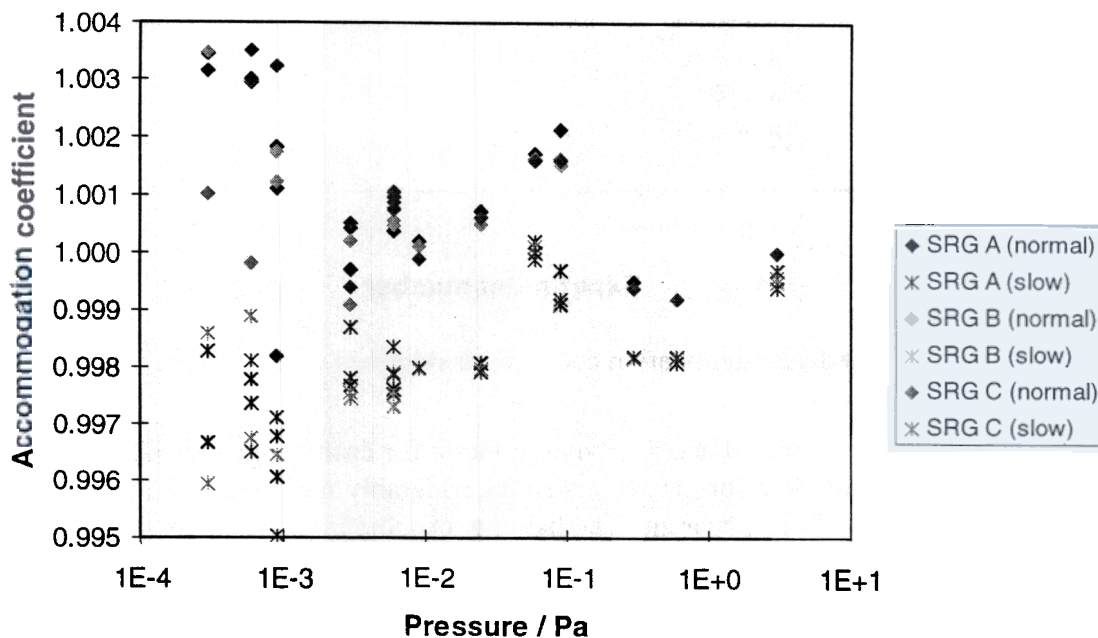


Figure 30: Effect of speed of valve operation on accommodation coefficients

Figure 30 shows the mean accommodation coefficients for three spinning-rotor gauges for both normal and slow speeds of closure of the valves leading into the small vessels. Figure 31 shows the mean decrease in the accommodation coefficients at each pressure for each gauge as a result of the reduction of the speed of closure of the valves leading into the small vessels.

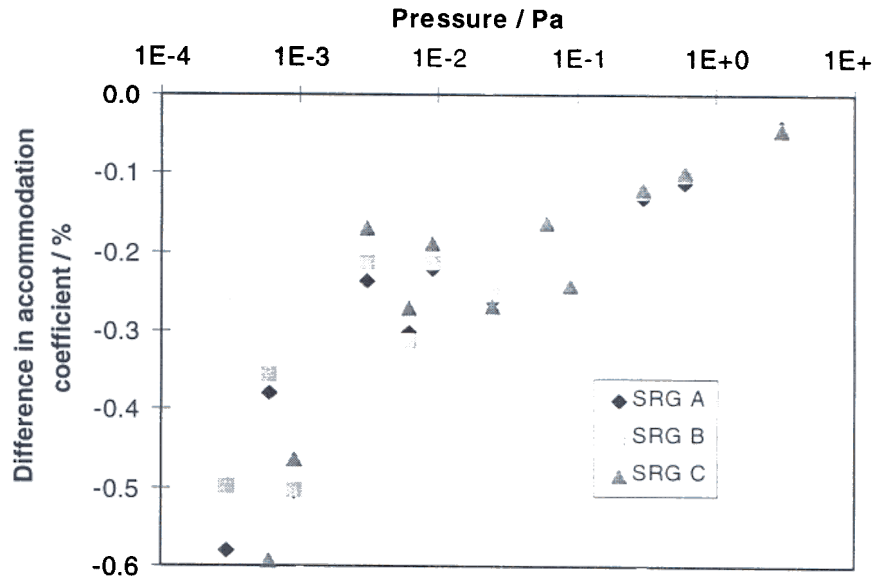


Figure 31: Difference between accommodation coefficients for normal and slow valve closure speeds

The accommodation coefficients determined when the valves leading into the small vessels were shut slowly were significantly lower than those for the normal speed of operation. The reduction in accommodation coefficient was pressure dependent and varied from an average value of 0.5% at 3×10^{-4} Pa, through 0.3% at 6×10^{-3} Pa, 0.2% at 9×10^{-2} Pa to 0.1% at 1 Pa.

The higher accommodation coefficient for the normal speed of closure is equivalent to the pressure within the small vessel being higher than predicted and hence the generated pressure being higher than calculated. This is consistent with the observations from various comparisons [1, 2, 3] that the pressure generated by static-expansion systems tends to be higher than that generated by dynamic-expansion systems. As shown in section 3.3.1, at these pressures the pressure generated is unaffected by the time taken to perform the expansions and in any case if the results were influenced by outgassing the expectation would be that the accommodation coefficient would increase as the time is increased rather than decrease.

As an inter-volume valve is shut the distribution of gas molecules is disturbed. The nearer the valve is to the fully closed position the more the molecular distribution is disturbed and the more difficult it becomes for the redistribution of molecules to occur. In addition, the quicker the valve is closed the less time there is for equilibrium to be reached. As the pressure is reduced the gas regime changes from viscous flow, through the transition flow to molecular flow and the mechanisms for molecule-molecule interactions reduce thus increasing the time required for equilibration. In order to reduce the effect of variable volumes within the small vessels, static-expansion systems are usually designed so that the valve seats are connected to the small vessel whilst the bellows (used to seal the valve actuation mechanism to atmospheric

pressure) are connected to the large vessel. Therefore as the valves are shut there is a tendency for the pressure in the small vessel to be increased with respect to that in the large vessel. The opportunity for the gas molecules to redistribute whilst the valve is being closed and for equilibrium to be reached may depend on the design of the valve and the configuration of the system. The pressure generated is linked directly to the volumetric ratios, however, this experiment showed that the effect decreases with increasing pressure and is small above 1 Pa. The effect of speed of valve closure on the determination of volume ratios is therefore likely to be negligible and only to influence the generated pressure.

3.3.3 Influence of response time to pressure changes on the calibration of ionisation gauges

During the first phase of the CCM UHV comparison [3] the results obtained by NPL using the Bayard-Alpert gauge and the extractor gauge were not consistent. The Bayard-Alpert gauge indicated a pressure dependent difference between the static-expansion system of NPL and the two dynamic-expansion systems of NIST and PTB, whereas the difference indicated by the extractor gauge was much smaller and pressure independent. The differences between the gauges were repeatable and whilst the gauges were not calibrated simultaneously the differences could not be attributed to variations in the generated pressure between the B-A gauge calibrations and the extractor gauge calibrations. Instabilities in the emission current of the B-A gauge were observed at NPL and shifts in the emission current of around 5% were observed at PTB during post comparison measurements. However, although the measurements at PTB suggested that this effect was of comparable magnitude to the pressure dependent difference of the NPL data, no pressure dependent effect was evident. If there was a problem with the emission current control/measurement circuit then it is possible that the transient step change in pressure generated by the static-expansion system resulted in a pressure dependent response in the emission current.

Another possibility is that ionisation gauges have a pressure dependent time response which is different for the steady state pressure generated by the dynamic-expansion systems compared with the step change in pressure generated by the static-expansion systems and that this response varies according to the design of the ionisation gauge. Four different ionisation gauge heads were mounted on the NPL high vacuum static-expansion system with the axis of their electrode structure vertical. Details of the gauges are given in Table 14. Gauges A, C and D had single filaments whilst gauge B had twin filaments. With the exception of gauge D which was already on the system, the gauges were located a comparable distance from the calibration chamber in order to replicate as far as possible the conductance between the calibration chamber and the transfer standards used in the CCM UHV comparison.

Following evacuation the system was baked and all the gauges operated for two days prior to the start of the measurements. Each gauge was operated overnight immediately prior to its measurements. In order to avoid interaction between the gauges, only one gauge was operated at a time, in a similar manner to the NPL measurements for the CCM comparison.

Gauge	Electrode geometry	Filament geometry	Grid diameter mm	Grid potential volts	Filament potential volts	Emission current mA	Sensitivity Pa ⁻¹
A	Extractor	ring	12	220	100	1.6	0.058
B	Bayard-Alpert (nude)	helical	23	220	80	10.0	0.156
C	Bayard-Alpert (glass tubulated)	hairpin (opposed)	18	150	50	1.0	0.082
D	Bayard-Alpert (glass tubulated)	hairpin	12	150	50	1.0	0.012

Table 14: Parameters of ionisation gauges used in the time response study

Gauges A and B were of identical designs to the two gauges used for the CCM UHV comparison. These gauges were operated from a more recent model of the commercial ion gauge control unit used for the comparison. Unfortunately design changes by the manufacturer to this type of the control unit meant that for these recent measurements the B-A gauge could not be operated at an emission current of 0.6 mA (the current used for the comparison) but instead a current of 10 mA was used. Gauges C and D were operated using an NPL designed control unit. The ion current from each gauge was measured using an electrometer. Whilst of a different design to the electrometer used for the comparison, any influence of the time response of the electrometer would affect all gauges equally.

Measurements were performed at three pressures; 9×10^{-6} Pa, 9×10^{-5} Pa and 6×10^{-4} Pa. To check the time response of the gauges each pressure was generated six times, three times using the normal timing for the calibration of ionisation gauges and then a further three times with an additional delay of 60 seconds between the end of the ion current measurements 'before' the expansion of the gas and the start of the ion current measurements 'after' the expansion. The sensitivity of the gauge was calculated for each measurement. For gauges B, C and D one measurement was also made with an additional delay of 120 seconds between the 'before' and the 'after' readings in order to check whether equilibrium had been reached.

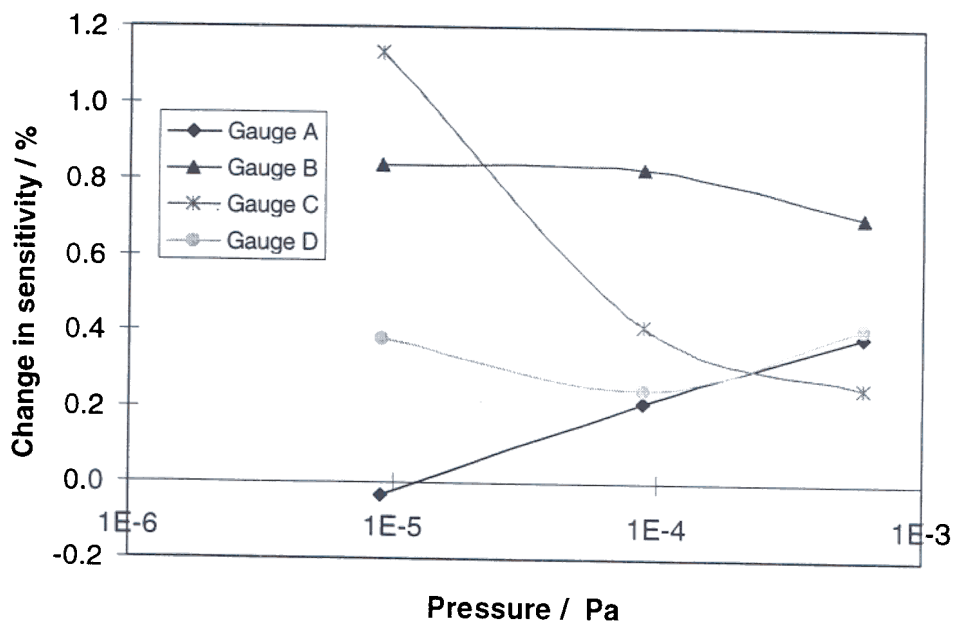


Figure 32: Effect on sensitivity due to the time response of ion gauges to pressure changes

Figure 32 shows the increase in sensitivity for measurements made with the additional 60 second delay between the 'before' and the 'after' measurements. The data points have been joined by smooth lines solely for clarity of presentation. The spread in the three sensitivity values for each experiment at each pressure varied between 0.1% and 1% with an average spread around 0.3%. Gauges A, B and D showed no significant pressure dependent time response. Measurements incorporating a 120 second delay indicated that the gauge indication of these three gauges reached equilibrium after the additional delay of just 60 seconds. Gauges A and D showed little time response at all, whereas the pressure independent difference of 0.8% between the two sets of measurements for gauge B suggested that equilibrium had not been reached during the normal timing schedule. The results for gauge C are less clear, the gauge showed a significant pressure dependence to the time response, particularly at the lowest pressures. Although not conclusive from the data, it is noticeable that the gauges with small electrode structures were less affected by timing and reached equilibrium more quickly.

These measurements show that determining the time taken for a gauge to reach equilibrium is not straightforward. If historical decisions were based on gauges similar to gauge A or D then it would be concluded that a delay of around 30 seconds between the end of the 'before' readings and the start of the 'after' readings would be adequate to ensure equilibrium was reached, whereas the information from gauge B shows that this is not the case for all gauges. During the CCM UHV comparison the extractor gauge data would have been unaffected by any timing influences, whilst potentially the B-A gauge (type B) would have experienced a shift in sensitivity. However, as the time response of gauge B was pressure independent and the comparison data was scaled to the SRG data at 9×10^{-4} Pa, this would not have affected the results of the comparison. The characteristics obtained for gauge B were very similar to the comparison data of PTB(2) and bore little resemblance to the NPL data for the B-A gauge transfer standard. It therefore appears that the NPL comparison results were the result of characteristics inherent in that gauge head and/or control electronics.

3.4 CALCULATION OF ORIFICE TRANSMISSION PROBABILITY FOR DYNAMIC-EXPANSION SYSTEMS

3.4.1 Overview

As described in 2.1.1, the dynamic-expansion technique for the generation of pressures relies on balancing the rate of flow of gas into the vessel against the rate at which gas passing through an orifice is evacuated. Under conditions of steady molecular flow, where the conductance of the orifice is independent of pressure and constant for a given gas species at a constant temperature, this technique permits the generation of steady calculable pressures across the orifice, providing that its conductance, which may be measured or calculated, is known.

Many national standards laboratories employ dynamic-expansion systems as their high vacuum primary standards [21, 22, 23, 24, 25]. Methods to measure conductance usually rely on the use of transfer gauges which must themselves be calibrated against another standard; therefore if the orifice is to form part of a primary vacuum standard this method is not normally appropriate and often does not provide an adequately low uncertainty. One exception is the original dynamic-expansion primary standard at IMGC which employs a

variable orifice whose conductance was determined using an oil manometer primary standard [26]. Most laboratories therefore rely on calculation of the transmission probability, and hence the conductance of the orifice. With the exception of a NIST publication [27] there is little detail within the published literature detailing how this calculation was carried out.

The work of Clausing showed that the rate of effusion of a gas through an orifice may be modelled as the product of the rate of effusion of molecules through an ideal aperture, of the same size as the inner surface of the orifice, and its ‘transmission probability’ [28]. Transmission probability is a dimensionless quantity that depends only on the shape of the aperture and is defined as a ratio of the number of molecules which pass through the orifice to the number entering the orifice. Practical orifices differ from the ideal orifice due to their finite thickness and it is necessary to take this into account when modelling the transmission probability. In calculating the transmission probability for the orifice used within the NPL dynamic-expansion system, a number of different approaches were considered.

3.4.2 Design of the NPL orifice

The orifice in the NPL dynamic-expansion system [29] was designed for ease of transmission probability calculation, whilst ensuring mechanical stability. The orifice dimensions were chosen to give a conductance of approximately 10 litres per second for nitrogen at room temperature, which provides a good compromise between the range of pressures to be established and pumping speed in the ‘upper’ chamber of the system. The orifice edge region was formed in the 12 mm thick central plate by lapping an 11 mm hole with a 16 mm diameter steel ball to nominally equal depths from above and below. This produced two concave spherical surfaces which met at a sharp circular edge. Prior to lapping, the upper and lower surfaces of the plate were machined to form two hollow truncated 84° half-angle cones, leaving 0.45 mm at the edge of the centre hole (see Figure 33). The transmission probabilities were calculated for the conical segments and the combined spherical surfaces.

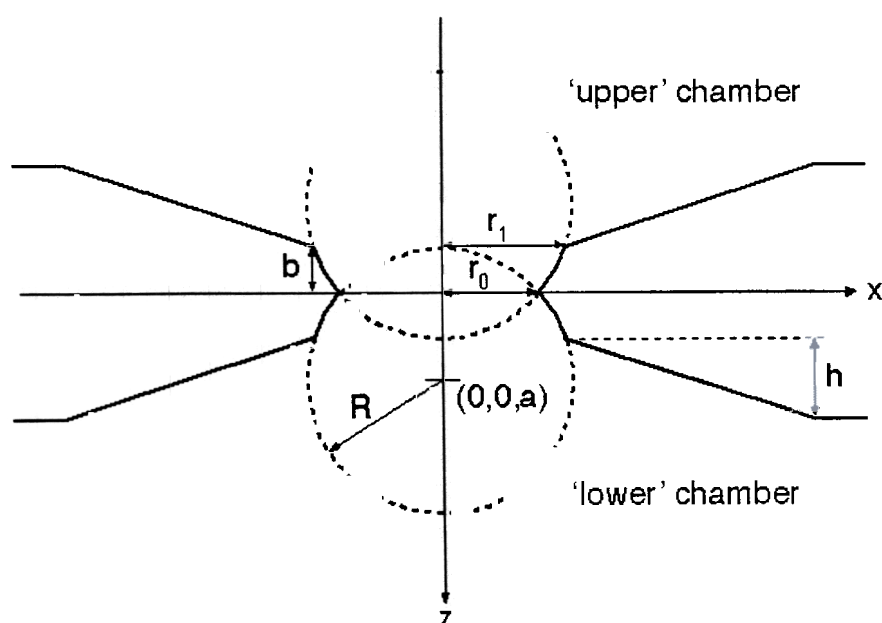


Figure 33: Schematic of the NPL orifice

3.4.3 Monte-Carlo simulation

Monte-Carlo methods simulate the interaction of a large number of molecules with the orifice, and an estimate of the orifice transmission probability is obtained from the ratio of molecules that pass through the orifice to the total number of molecules which enter it. Molecular paths are generated according to a uniform random distribution for incoming molecules and according to a cosine law for molecules reflected from the orifice walls (see section 3.4.6). This approach is conceptually relatively simple and the intensity of required computations does not represent a problem for modern computers. It is also believed to offer adequate numerical precision. However, in the published literature [28, 30, 31] the authors encountered differing numerical interpretations of the assumptions on which the calculations are based, for both starting and re-bounding (scattering conditions). Choices have to be made when determining both spatial and angular distribution of molecules entering the orifice and the distribution of the rebound direction following impacts with the orifice walls. The interpretation of the cosine law was found to vary from author to author, from considering the reflection directions to be entirely independent of incident directions to the pre-definition of the azimuth angle. The latter approach results in non-randomness of collision co-ordinates.

The simulations performed at NPL for simple conical and cylindrical orifices showed significant differences when using the two different distribution generators below:

Generator	Davis's [30] distribution for starting positions and directions, and Davis's cosine distribution for the rebound directions.
Generator 2	Uniformly distributed starting positions and Clausing's [28] cosine distribution (as derived directly from the theory) in both starting and rebound directions.

The use of Davis's distribution gave better agreement with published results [30, 32] than the use of Clausing's distribution, but even then the agreement was not as good as expected from the number of significant digits published. For both cylindrical and conical orifices, generator 1 gave values higher than expected from published data for short geometry and lower than expected for longer cylinders and cones, whilst the second algorithm consistently gave higher transmission probabilities for both geometries. The observed differences exceeded 20% for very long narrow cones. An illustration of model-induced differences for gas flow through conical ducts is given in Figure 34. The published data is taken from Iczkowski [32]. Moreover, the resulting distributions of generated angles vary significantly with the generators chosen, from approximately uniform azimuth angles and cosine distribution of angle with the normal for generator 2 (Figure 36) to the highly non-uniform azimuth obtained with generator 1 (Figure 35).

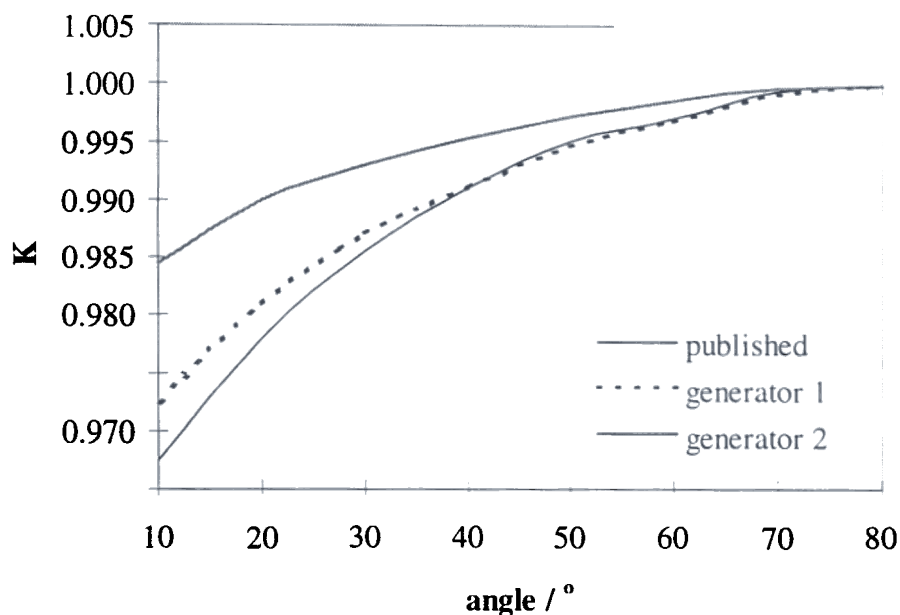


Figure 34: Comparison of the transmission probabilities (K) for wide cones ($L/R=0.1$)

Inspection of Davis's algorithm revealed some surprising features

- Davis's choice of random numbers is such that uniformly distributed random numbers are generated in pairs and the whole pair is either accepted or rejected, depending on the relationship between them. The purpose of this test, and the implications of this condition, are not clear.
- There is an implied link between the position of a point and the generation of the azimuth angle. It would appear that the same two random numbers are used in both cases, which would pre-determine the azimuth angle of the rebounding direction by the incident position for the molecules entering the orifice. Davis implies that the same considerations apply for molecules colliding with the wall, but does not give any details. This would result in non-randomness of collision co-ordinates and also in a deterministic azimuth angle of rebound molecules, implying that the molecules undergo partial specular reflection.

The azimuth angle generated by Davis has a distribution $|\cos(2\theta - \pi/2)|$, $-\pi \leq \theta \leq \pi$, while the 'generator 2' distribution of azimuth is approximately uniform. The two azimuth angle distributions are shown in Figure 35 and Figure 36.

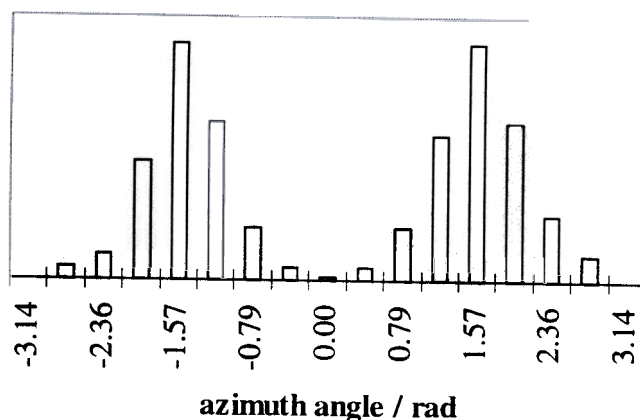


Figure 35: Distribution of azimuth angle obtained for generator

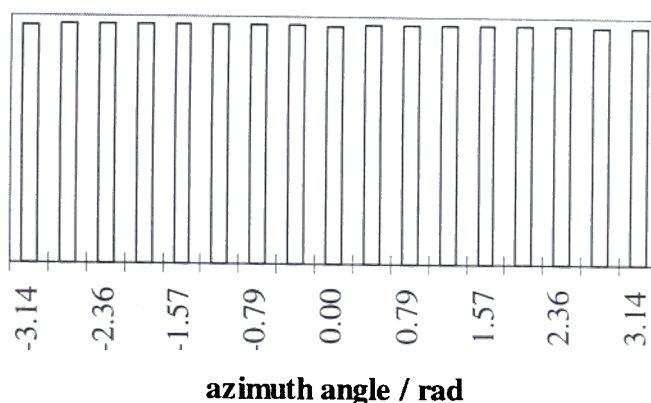


Figure 36: Distribution of azimuth angle obtained for generator 2

Subsequent to NPL's work, IMGC and PTB have also developed new dynamic-expansion systems and have been faced with the challenge of calculating the transmission probability for their fixed orifices. PTB's orifice [33] has a single sided conical profile with an orifice diameter 33 mm. IMGC's 11 mm diameter orifice [34] is based on the profile of the NIST orifice. The transmission probabilities for the orifices within the new PTB [33] and IMGC [34] dynamic-expansion systems have recently been calculated using Monte-Carlo techniques by Szwemin and co-workers at the Warsaw University of Technology (WUT). Their simulation was carried out from the central plane of the orifice outwards using 10^{10} molecules as this required fewer simulations than the reverse scenario. The molecules were apparently simulated using the protocols described by Davis [30]. The importance of performing simulations with an extremely high number of molecules was highlighted as they estimated that 10^{12} molecules would be required to obtain a relative statistical standard deviation of the order of 10^{-5} .

The diameter of the PTB orifice is a factor of three larger than those employed by NIST, IMGC and NPL and hence it has a larger effect on the gas flux and molecular distribution within the chamber. Szwemin et al [33] also modelled the gas flux distribution within the PTB chamber and calculated the deviation from the ideal case at each gauge port; the

maximum deviation amounted to -0.33%, a value comparable with the estimated expanded ($k = 2$) uncertainty of the PTB system.

The high level of agreement between published results is surprising given the limited number of molecules simulated (typically 10000 in the 1960s). The results quoted in some older papers would therefore appear to overstate the number of statistically valid digits for the number of trials which were probably performed. Uncertainties were rarely quoted indicating that unrealistically low estimates of the uncertainties may have been assumed. In addition, when comparing the transmission probabilities for differing parameters, the number of simulations performed should be varied such that the number of molecules transmitted remains constant. As a result, due to the large number of simulations required, accurate results from Monte-Carlo simulations may typically only be obtained for transmission probabilities close to 1. Significant deviations are therefore likely for structures with lower transmission probabilities, unless a very high number of simulations are performed.

When comparing the results of different estimation methods it is clear, even for the relatively simple and well-studied case of the cylinder, that the difference between models is higher than expected from the estimated uncertainties. Kurepa and Lucas [35] reported that none of several theoretical solutions gave a satisfactory description of end-effects for density distribution in a capillary tube and that it was unclear whether the approximate solutions were insufficiently accurate, or the theoretical treatment, derived from the classical kinetic theory of gases, was flawed by invalid assumptions. Davies and Lucas [36] suggested that the differences observed may be due to invalidity of the assumption of the cosine distribution, as they found evidence of specular reflection and surface diffusion in some cases.

3.4.4 Integral equation method

Transmission probability is a dimensionless quantity that depends only on the shape of the aperture and is defined as a ratio of the number of molecules passing through the orifice to the number entering the orifice. If diffuse scatter is assumed for all molecules, it is possible to find a solution for any orifice, although, except in special cases, the conductance calculations are complex and time consuming. This method also requires regularity conditions to be derived for each given geometry, and by its very nature any integral equation solution introduces approximations which adds to the overall uncertainty in the result.

McCulloh [27] calculated the transmission probability for the orifice in the NIST high vacuum standard using the integral equation approach. Despite a well documented report a crucial step, set of assumptions or approximations has been omitted. There is little doubt that the results he obtained are valid, but nobody from a number of laboratories (NIST, IMGC, NPL and WUT) has been able to replicate the derivation. The dimensions of the IMGC orifice are close to those of the NIST orifice and Szwemin et al [34] used the inequalities derived by McCulloh and substituted the dimensions of the IMGC orifice in order to compare the results with his Monte-Carlo estimates.

3.4.5 Analytical method

By careful design of the orifice employed in a dynamic-expansion vacuum standard it is possible to ensure its mechanical stability whilst retaining a transmission probability very near to unity. If spherical geometry is chosen the integral equation method calculations can be reduced to simple analytical expressions, and the introduction of approximations connected with evaluating integrals avoided completely. This not only significantly reduces the complexity of the calculations but also gives an exact solution which does not contain an additional method-induced uncertainty. The analytical method provides direct traceability to dimensional data and achieves extremely low uncertainties. Details of the method as applied to the orifice within the NPL system are presented.

3.4.5.1 Transmission probability for a spherical orifice

Edwards and Gilles [37] showed that the integral equation, which expresses the molecule impingement rate on a unit area, possesses a simple, closed-form solution for spherical orifices (Figure 37) and they derived the transmission probability for such orifices.

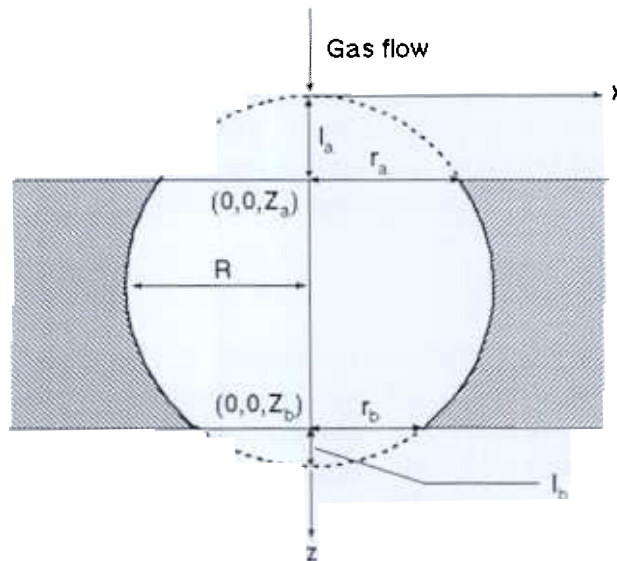


Figure 37: Schematic of a spherical orifice

Starting from the assumptions that:

- the molecules arriving at the orifice entrance are uniformly distributed according to the cosine law
- molecules re-bound off the orifice walls in directions determined by the cosine law
- each molecule striking the orifice wall is reflected from the point at which it strikes

they derived the *special transmission probability* as:

$$W' = \frac{N_b}{N_0} = \frac{N_0 2\pi R \frac{l_b}{4\pi R^2} + \int_{Z_a}^{Z_b} v(z) 2\pi R dz \frac{2\pi R l_b}{4\pi R^2}}{N_0}$$

where: N_0 is the rate at which molecules pass into the entrance of the orifice
 N_b is the rate at which they effuse from the orifice
 $v(z)$ is the incident molecular flux on the incremental ring at z , on the orifice wall

By substituting $N_0 = \mu_0 2\pi R l_a$ and $\psi(z) = v(z)/\mu_0$, where μ_0 is the incident molecular flux on the orifice entrance, the following equation is obtained:

$$W' = \frac{l_b}{l_a + l_b} + \frac{l_b}{2R l_a} \int_{Z_a}^{Z_b} \psi(z) dz$$

where: l_a denotes the distance between the point at the top of the sphere and the orifice entrance
 l_b denotes the distance between the orifice exit and the bottom of the sphere
 R is the radius of the sphere
 z_a and z_b are co-ordinates along z axis
 r_a and r_b are the radii of the entrance circle and the exit circle of the orifice respectively.

The first term on the right in equations (18) and (19) gives the ratio of the rate of transmission directly from the orifice entrance through the exit, without collisions with the orifice wall, to the rate at which molecules pass into the spherical segment which constitutes the orifice entrance. The second term gives the ratio of the transmission between the orifice wall and its exit to the rate of entry through its entrance. Edwards et al [37] proved that the incident molecular flux in a sphere is constant for a given spherical segment, thus giving a constant special transmission probability. The transmission probability (W') for a spherical segment orifice entrance/exit may then be modified to consider a circle as the orifice entrance/exit (W).

$$\psi(z) = \psi = \frac{\mu_0}{l_a + l_b}$$

and

$$W' = \frac{l_b}{l_a + l_b}$$

hence

$$W = W' \frac{2\pi R l_a}{\pi r_a^2} = \frac{l_b}{l_a + l_b} \frac{2R}{2R - r_a^2}$$

3.4.5.2 Calculation of the correction for the conical segments

The transmission probability of a cone can be calculated using the integral equation approach of Iczkowski et al [32]. However, due to the dimensions of the NPL cone (the cone vertex half-angle $\gamma = 83.87^\circ \pm 0.15^\circ$ and $r_1/h = 1.015 \pm 0.020$) the difficulties in solving Fredholm integral equations were avoided by bilinear interpolation of the data from Iczkowski et al [32]. The correction factor K_{cone} was calculated to be equal to unity, to six significant figures.

The dimensions of the cones above and below the central orifice are such that the overall transmission probability of the orifice is unchanged by their presence and the uncertainty of the cone correction is negligible.

3.4.5.3 Calculation of the transmission probability of the spherical segments

Rectangular co-ordinate axes were chosen for the calculation, with the origin at the centre of the 'throat', where the throat is defined by the plane in which the two spherical surfaces meet. The z-axis is the axis of rotational symmetry and the axial co-ordinate increases in the direction of flow. The plane of symmetry for the orifice was assumed to lie within the plane of the throat and the uncertainty assigned to dimension b incorporates errors arising from this assumption. The entrance and exit apertures refer to the cross-sections in the planes b and -b, where the lapped surfaces terminate. The points (0,0,-a) and (0,0,a) are the centres of curvature for the two spherical surfaces, respectively.

For the orifice comprising two spherical segments it is necessary to calculate the transmission probabilities for the 'upper' and 'lower' parts separately. Using the notation of Figure 37 the values of the entry and exit lengths are given by

$$\begin{aligned} l_a^{\text{up}} &= l_a = R + a - b = R + \sqrt{R^2 - r_0^2} - b \\ l_b^{\text{up}} &= l_b = R - a = R - \sqrt{R^2 - r_0^2} \\ l_b^{\text{lo}} &= R + a - b = R + \sqrt{R^2 - r_0^2} - b = l \\ l_a^{\text{lo}} &= R - a = R - \sqrt{R^2 - r_0^2} = l_b \end{aligned}$$

and hence

$$W_{\text{upper}} = \frac{l_b}{l_a + l_b} \frac{2R}{2R - l_b}$$

and

$$W_{\text{lower}} = \frac{l_a}{l_a + l_b} \frac{2R}{2R - l_b}$$

As a result of gas equilibrium conditions W_{lower} may also be calculated from the relationship

$$A_0 W_{\text{upper}} = A_1 W_{\text{lower}}$$

where $A_0 = \pi r_0^2$,

r_0 is the radius of the throat

$A_1 = \pi r_1^2$,

$r_1^2 = R^2 - (a - b)^2 = r_0^2 - b^2 + 2b\sqrt{R^2 - r_0^2}$ is the radius of the entrance to the upper orifice and exit of the lower orifice

Hence

$$W_{\text{lower}} = W_{\text{upper}} \frac{r_1^2}{r_0^2}$$

The combined transmission probability for the complete lapped aperture can be found using the expression of Oatley [38]

$$W = \frac{W_{\text{upper}} W_{\text{lower}}}{W_{\text{upper}} + W_{\text{lower}} - W_{\text{upper}} W_{\text{lower}}}$$

Exploiting the symmetry and gas equilibrium conditions, the above equation may be re-written as

$$W = \frac{r_1^2 W_{\text{upper}}^2}{(r_0^2 + r_1^2) W_{\text{upper}} - r_1^2 W_{\text{upper}}^2} = \frac{r_0^2 W_{\text{lower}}^2}{(r_0^2 + r_1^2) W_{\text{lower}} - r_0^2 W_{\text{lower}}^2}$$

Finally, the combined transmission probability is transformed so that it refers to the throat of the orifice enabling its conductance to be calculated as a simple product of throat transmission probability and the conductance of an ideal aperture of the same radius:

$$W_{\text{throat}} = W \frac{r_1^2}{r_0^2}$$

and, following Poulter [16], the conductance of the orifice is easily calculated as the product of its combined transmission probability, the area of the equivalent ideal orifice and the mean molecular velocity calculated for a particular gas species ($\bar{c}$):

$$C = r_0^2 \pi \frac{\bar{c}}{4} W_{\text{throat}} K_{\text{cone}}^2 = r_1^2 \pi \frac{\bar{c}}{4} W K_{\text{cone}}^2$$

For a less accurate estimate of the transmission probability, symmetry of the orifice about the centre plane makes it possible for the calculations to be performed for just one side of the orifice and the transmission probability for the whole orifice to be derived simply by squaring the one-sided result multiplied by the ratio of the radii.

The uncertainty in the computed cone transmission probability was obtained by Monte-Carlo sampling of the space of cone parameters, assuming a multivariate normal distribution of these parameters, and was proved to be negligible. The distribution of the computed result is taken as being the distribution of the cone transmission probability. The measured quantities were assumed to be unbiased, uncorrelated and normally distributed. The validity of the

expressions for all derived contributing factors was checked numerically by computing their finite difference approximations. The uncertainties in the derived quantities and the combined transmission probability were computed in accordance with the Guide to the Expression of Uncertainty in Measurement [39].

Realisation of an orifice with a spherical geometry enables a simple analytical solution to be obtained. Since this was achieved by lapping, which is a self-centring method, the hole is well centred and corrections for departure from centrality or circularity are negligible. If the orifice comprised only conical segments it would be difficult to avoid misalignment of throat plane with respect to the z axis, departure from a circular centred hole, or the need for a short tube correction. In addition, the transmission probability of a spherical orifice exceeds that of a conical one for the same dimensions.

Clarification of the cosine distribution

3.4.6.1 Overview

In Monte Carlo modelling of vacuum systems the interpretation of the phrase “cosine distribution” and the correct simulation of a “cosine distribution” of emitted particles is crucial. The authors have found that variations in interpretations can occur and incorrect definitions exist in the published literature [40]. This section first describes the cosine law and one possible method of “generating” the correct distribution. The distribution that results from the most common misinterpretation of the “cosine law” is then described.

The “cosine law

In terms of gas molecules leaving a surface, the “cosine law” (sometimes called the Knudsen Cosine Law or Lambert’s Law) can be stated as follows:

the molecular flux leaving a plane surface element A , due to all molecules having velocity vectors with directions within a small solid angle $d\omega$ whose axis makes an angle θ with the normal to A , is given by the cosine law formula

$$dn = N A v_a \cos \theta \frac{d\omega}{4\pi} \quad (35)$$

where N is the number density of molecules and $v_a = \langle v \rangle$, the average molecular velocity.

Although the individual processes that result in molecules leaving a surface do not necessarily produce Maxwell-Boltzmann velocity distributions, for equilibrium conditions the *overall* distribution of molecular flux will obey the cosine law [41].

In the system of spherical-polar co-ordinates the solid angle, $d\omega$ is given by

$$d\omega = \sin \theta \, d\theta \, d\phi \quad (36)$$

so the expression can be written in terms of the polar and azimuthal angles (θ, ϕ):

$$dn = \frac{N A v_a}{4\pi} \cos \theta \sin \theta d\theta d\phi$$

From kinetic theory we have the result that the total molecular flux, N_o , from A is:

$$N_o = \frac{N A v_a}{4}$$

so the cosine law can conveniently be written as:

$$dn = \frac{N_o}{\pi} \cos \theta \sin \theta d\theta d\phi$$

Angular probability distributions

Monte Carlo modelling of the molecules in a vacuum system involves following the progress of a molecule from its “origin” until some other meaningful event occurs, eg the molecule leaves the system or is captured. For realistic modelling the parameters describing the molecule’s behaviour must be chosen so that they are representative of the real behaviour of molecules in the system. In terms of the molecule’s scattering behaviour this means that the angular distribution of the molecules should be chosen so that the overall flux distribution obeys the cosine law. The angular probability distributions for molecules scattered with a cosine distribution from a surface element A can be determined from equation (39) as follows:

Azimuthal angle

Molecules with a particular azimuth, ϕ , can occur with a range of polar angles, θ , therefore, to discover the azimuthal-angular-probability-distribution, $f(\phi)$, equation (39) should be integrated over all θ , ie

$$dn_\phi = \frac{N_o}{\pi} d\phi \int_0^{\pi/2} \cos \theta \sin \theta d\theta$$

therefore

$$f(\phi) = \frac{1}{N_o} \frac{dn_\phi}{d\phi} = \frac{1}{\pi} = \text{constant}$$

Polar angle

Molecules with a particular polar direction, θ , can occur with a range of azimuthal angles, ϕ , therefore, to discover the polar-angular-probability-distribution, $g(\theta)$, equation (39) should be integrated over all ϕ , ie

$$dn_{\theta} = \frac{N_o}{\pi} \cos \theta \sin \theta d\theta \int_0^{2\pi} d\phi$$

ie

$$dn_{\theta} = 2 N_o \cos \theta \sin \theta d\theta$$

therefore

$$g(\theta) = \frac{1}{N_o} \frac{dn_{\theta}}{d\theta} = 2 \cos \theta \sin \theta = \sin 2\theta$$

3.4.6.3 Generation of angular distributions

Random number generation of the angular distributions to produce a “cosine distribution” of molecular flux can be achieved in a number of ways.

Azimuthal angle

Generating the azimuthal angle is mathematically trivial. The simplest approach is to generate a uniformly distributed random number, y , in the range $(0 < y \leq 2\pi)$ then to assign $\phi = y$. (Alternatively, if the random number can only be generated in a limited range, eg $(0 < y \leq 1)$ then $f(\phi)$ can be generated by scaling y ie $\phi = y \times 2\pi$).

Polar angle

The polar angle can be generated from a single, uniformly distributed random number, say x , in the range $(0 \leq x \leq 1)$ by integrating the polar-angular-probability-distribution [42]:

$$x = \frac{\int_0^{\theta} g(\theta).d\theta}{\int_0^{\pi/2} g(\theta).d\theta} = \frac{\int_0^{\theta} \sin 2\theta.d\theta}{1} = 1 - \cos^2 \theta = \sin^2 \theta \quad (45)$$

ie

$$\theta = \sin^{-1}(\sqrt{x}) \quad (46)$$

3.4.6.4 Incorrect interpretation of the cosine distribution law

The common misunderstanding associated with the cosine law arises when it is *erroneously* supposed to state that

the probability of a molecule leaving the surface within a narrow angle $d\theta$, in a direction making an angle θ to the surface normal, is $\cos\theta$

The misinterpretation occurs most often when a system is being modelled in 2-dimensions. This *incorrect* statement of the cosine law fails to take into account the 3-dimensional nature of the real molecular distribution and the inherent fact that for given $d\theta$ *the solid angle varies as a function of θ* .

The misinterpretation effectively states that the polar-angular-probability-distribution, is $h(\theta)$ where $h(\theta) = \cos\theta$. This distribution can be generated from a single, uniformly distributed random number in the range ($0 \leq x \leq 1$) by integrating the distribution as follows:

$$x = \frac{\int_0^\theta h(\theta) \cdot d\theta}{\int_0^{\pi/2} h(\theta) \cdot d\theta} = \frac{\int_0^\theta \cos \cdot d\theta}{1} = \sin \theta$$

so in this case

$$\theta = \sin^{-1}(x)$$

An associated azimuthal angle is usually generated using the approach mentioned above.

To see what effect the choice of polar angle generator has on the molecular flux distribution (ie on the amount of flux in a small solid angle), the angular distribution needs to be normalised to take account of the changing solid angle (as a function of polar angle) ie

$$dn(\theta, d\omega) \propto \frac{p(\theta)}{d\omega(\theta)}$$

where

$$d\omega = \sin \theta \, d\theta \, d\phi$$

For the correct polar distribution $p(\theta) = g(\theta)$, this gives

$$dn_g(\theta, d\omega) \propto \frac{g(\theta)}{\sin \theta \, d\theta \, d\phi} = \frac{2 \cos \theta \sin \theta}{\sin \theta \, d\theta \, d\phi}$$

ie

$$dn_g \propto \cos \theta$$

the correct “cosine distribution” as defined at the beginning of this section.

For the *incorrect* polar distribution $p(\theta) = h(\theta)$, this gives

$$dn_h(\theta, d\omega) \propto \frac{h(\theta)}{\sin\theta \, d\theta \, d\phi} = \frac{\cos\theta}{\sin\theta \, d\theta \, d\phi}$$

ie

$$dn_h \propto \cot\theta$$

This ($\cot\theta$) distribution is grossly different to the cosine distribution, being very strongly peaked along the surface normal ($\theta = 0$).

3.4.6.5 Summary

The correct interpretation of the cosine law distribution *for molecular flux* results in a $\sin 2\theta$ polar-angle-probability-distribution. This distribution can be generated from uniformly distributed random numbers in the range ($0 \leq x \leq 1$) using the relation $\theta = \sin^{-1}(\sqrt{x})$.

Incorrect interpretation of the cosine law usually arises when it is *erroneously* supposed to state that the *polar-angle-probability-distribution* is a $\cos\theta$ distribution. This results in a $\cot\theta$ distribution for molecular flux which is very strongly peaked along the surface normal. Use of a $\cos\theta$ distribution for the polar-angle-probability-distribution could therefore produce very different results to those generated with the correct $\sin 2\theta$ polar-angle-probability-distribution.

3.5 FEED-BACK CONTROL EFFECTS ON CALCULATED THROUGHPUT FOR DYNAMIC-EXPANSION SYSTEMS

3.5.1 Overview

In the molecular flow regime the pressure obtained in a dynamic-expansion system is directly proportional to the gas flow-rate which can be measured by flow-meters of various designs [43]. The principle of operation of a constant pressure flow-meter, such as the one used at NPL, relies on reducing the flow-meter volume by driving the piston into it in such a way that the pressure within the flow-meter is kept constant. This is achieved by regulating the piston speed so that the differential pressure between the two reservoirs of the flow-meter is kept equal to zero. In practice, it is only possible to control that pressure within some limits around zero. This may be realised in different ways resulting in different levels of pressure fluctuations as well as different pressure change gradients. The calculated gas throughput is therefore dependent on both the chosen driving mechanism (which affects the piston speed changes) and the chosen flow-rate (which dictates the average piston speed) and errors can arise from an inappropriate control algorithm.

3.5.2 Choice of control mechanism

Commonly used piston-driving algorithms are variations of closed-loop control techniques [44] and varying levels of performance can be achieved depending upon the way the feed-back signal is processed by the controller. The simplest type of feedback is

proportional control which exaggerates the error and reacts immediately to correct it; however, the system usually overshoots and oscillates around the desired value, often becoming unstable if damping is insufficient. Due to the nature of this mechanism, even stable values of proportional gain can not eliminate errors completely, resulting in a non-zero steady-state error.

Proportional control was initially implemented within the software used to control the NPL flow-meter and measure the throughput. The results obtained with such control were mostly unsatisfactory, due to the steady-state error which, although very small, meant that significant gradients in differential pressure were allowed (see Figure 38).

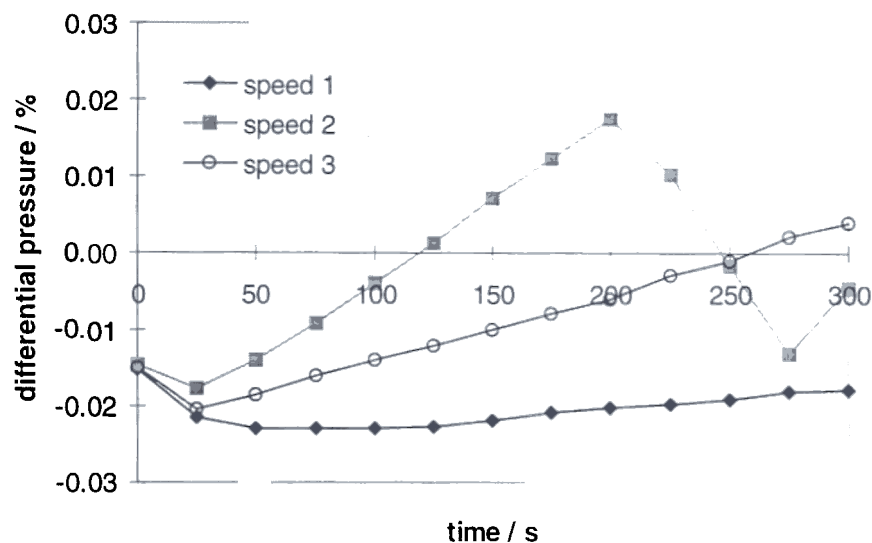


Figure 38: Relationship between pressure change and piston speed for proportional control

The cumulative errors can be overcome by use of integral control. Since this technique accumulates the error signal over time and corrects the motion by the integral error, the correction factor does not become negligible as the immediate error approaches zero. The combination of these two techniques, proportional-integral (PI) control, generally provides good control. However, in order to provide optimal control, the gains may have to be varied throughout the duration of the piston travel.

The next step in development of the NPL dynamic-expansion system was to use proportional-integral control. This algorithm provided much better control, but requires the selection of appropriate gain settings (see Figure 39). The gains vary not only with the pre-set flow rate, which effectively mean that they depend on both the chosen piston and the chosen speed, but should also vary with time if the best control is to be achieved

Derivative control uses the rate of change of the error as a corrective signal and so stabilises the transient response but fails to affect the steady-state errors. It can be combined with proportional and integral elements (PID) to further improve the control mechanism. However, the feedback elements are interactive and require tuning for each specific application if optimum dynamic system performance is to be achieved; increasing the number of elements increases the complexity of their interactions and the difficulty of choosing

optimal gains.

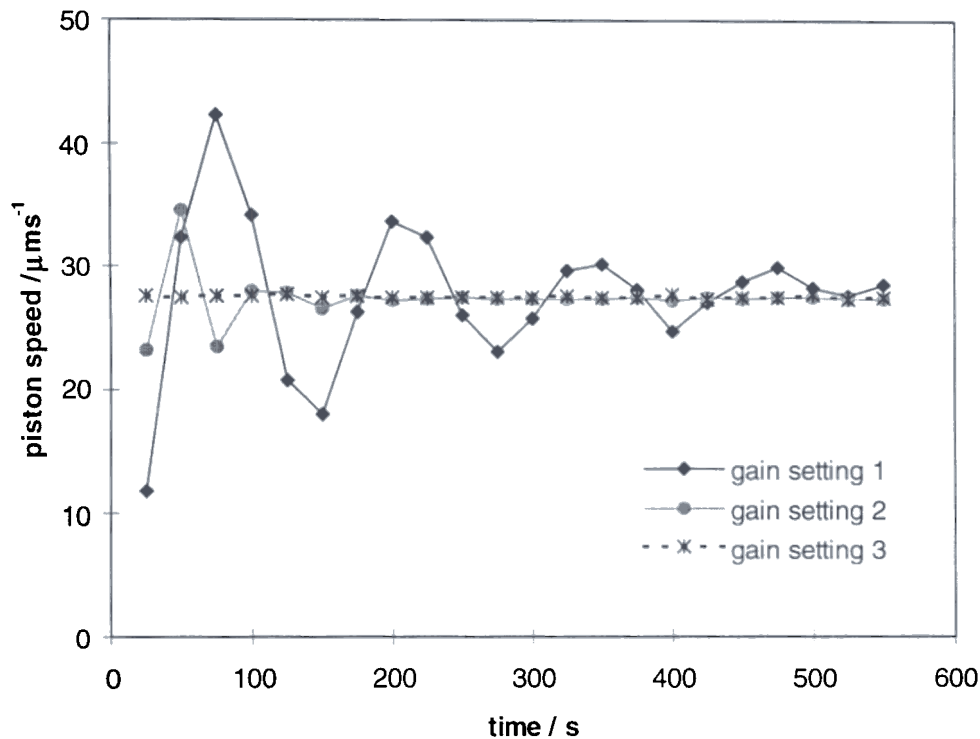


Figure 39: Fluctuations in piston speed due to PI control gains settings

Differences in calculated flow-rates of about 0.2% for the PI control mechanism and up to 2% for the proportional control were measured, while use of both techniques succeeded in controlling the flow-meter pressure to within very small limits. Care had to be exercised when implementing the control and the error signal changes were closely monitored. The inappropriate choice of the control algorithm and gain settings can cause errors which exceed the total uncertainty associated with the measured throughputs.

3.6 STATISTICAL LIMITATIONS

In much work within the vacuum region the measurement data population consists of remarkably few samples because the measurements are time-consuming and difficult to perform. The assumption that a coverage factor 'k = 2' uncertainty corresponds to 95% confidence is often flawed and the very few data sets may result in a poorer confidence level. It would also seem that in some cases the useable data has been further reduced by subjective practices - such as discarding 'rogue' data points when results have been regarded intuitively as untypical of normal system performance. Such practices can artificially enhance the perceived confidence in results, by altering their statistical distribution from the Gaussian and significantly reducing the perceived limits associated with that distribution.

4. SUMMARY AND CONCLUSIONS

In this report an attempt has been made to analyse the data from a number of comparisons of vacuum standards carried out over the last twenty years or more. The possible causes of the apparent discrepancies between standards in the vacuum region has also been investigated.

The historical data from many previous international comparisons of vacuum standards presents a strange mixture of inconsistent discrepancies. A main factor was the inadequacy of transfer standards, but with hindsight many protocols were weak - demanding questionable post-hoc adjustment of data. This report has attempted to collate the data and to interpret their significance. It was impeded by the manner in which earlier data was reported - the majority of published results reduced data to a single number for each participating laboratory. Reduction of data from several gauges, over a number of calibration pressures, to a single figure obscured repeatability, reproducibility and pressure-dependent effects. Individual results often deviated from the averaged figures by around 1%, a figure which often exceeded the uncertainties claimed for many of the primary standards.

The reproducibility of the transfer standards was generally poor, making analysis difficult and limiting the ability to measure the respective equivalence of the participating standards. Nevertheless, the impression remains that systematic differences do exist between standards and, taking the data as a whole, significantly more results (sensitivity values or accommodation factors) obtained using static-expansion systems lie above those of dynamic-expansion systems than vice versa.

In attempting to understand the possible causes of discrepancies between primary vacuum standards, this work has identified a number of areas where the modelling of the standards is either incorrect or inadequate and has proposed more robust models. In addition weaknesses in some experimental practices have also been highlighted and the influence of the improvements to the models described.

A summary of findings for static-expansion systems is given below:

- Under most experimental conditions, errors will arise if the equation for isothermal conditions is used to calculate the volumetric ratio.
- It is important that volumetric ratios are corrected to account for temperature effects during the determination. The iterative method provides more robust corrections for temperature effects than those proposed by Elliott et al. Whilst differences between the two methods are generally small, under unfavourable conditions they can approach the standard uncertainty associated with the determination of the ratio.
- Inadequate measurement of the vessel temperature during determination of the ratios can lead to errors which significantly exceed the standard uncertainty for the determination. These errors may even exceed those due to not applying any corrections for temperature effects.
- Poor experimental protocols and inadequate modelling in the past may still affect present estimates for the stability of the volumetric ratios.
- Inadequate measurement of the vessel temperature can lead to significant errors in the calculation of the temperature correction to the generated pressure and this can exceed the error due to non application of the correction.

- Closing the inter-volume valves too quickly can disturb the molecular distribution leading to a significant increase in the generated pressure and consequent increase in the corresponding sensitivity values, particularly at low pressures.
- Following a step change in pressure the time taken for an ionisation gauge to respond and its indication to reach equilibrium depends on the design of the gauge. Equilibration times based on historical data may be flawed depending on the type of gauge used to establish the data and errors in the calculated sensitivity values may result.

A summary of findings for dynamic-expansion systems is given below:

- Consideration should be given to the design of an orifice to be incorporated into a primary standard in order to obtain a mechanically stable structure with a transmission probability close to one which may be determined accurately.
- Integral equation methods are not generally applicable to orifices used within practical systems.
- If Monte-Carlo methods are used for transmission probability calculations it is crucial that the cosine distribution is correctly generated.
- A large number of simulations are required if Monte-Carlo methods are to provide an accurate estimate of the transmission probability.
- The use of spherical geometry enables analytical methods to be applied to the calculation of the transmission probability, reducing model induced uncertainties.
- The combination of spherical geometry and conical segments provides a rugged structure and if the angles of the cones are selected correctly, the conical segments have a negligible effect on the transmission probability.
- The appropriate feed-back algorithm and suitable gain setting selection are important for the correct calculation of flow-rate.

Whilst the influence of some the findings described above will be quite small, other effects will have a more significant impact although the exact magnitudes will depend on the environmental conditions and the experimental set-up specific to a particular standard. The use of the more robust temperature correction to the volumetric ratios should produce more consistent data. Improved measurement of temperature, both during the determination of volumetric ratios and the generation of reference pressures would also improve the consistency of results. A more appropriate speed of closure of the inter-volume valves on static-expansion systems would reduce the differences between the results of static and dynamic-expansion systems. It is therefore hoped that consideration of the points discussed above may lead to more harmonised results between vacuum standards in future comparisons and a reduction in the discrepancies between the standards.

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