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Comparison of NPL High Vacuum Standards SEA2 and SEA5

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Comparison of NPL High Vacuum Standards SEA2 and SEA5

John Greenwood & Paul Carroll
DEPC

ABSTRACT

This report describes a comparison between two of NPL's high vacuum pressure standards, SEA2 and SEA5, in the pressure range 3×10^{-4} Pa to 0.9 Pa, using unbaked spinning rotor gauges as transfer standards. The structure of the comparison and the subsequent data analysis is based on that of the (bilateral) comparison SIM-EUROMET.M.P.BK3 and of the wider comparison, EUROMET.M.P-K1.b in which SEA2 took part.

The results demonstrate that SEA5 is equivalent to SEA2 in the measured range and in addition, demonstrates that SEA5 is equivalent to the EUROMET key comparison reference values in the range 9×10^{-4} Pa to 0.9 Pa.

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1 INTRODUCTION

The Static Expansion technique can be used to generate calculable pressures as low as 10^{-6} Pa. In its simplest form the technique requires a vacuum system consisting of two vessels separated by a valve and a means of measuring the pressure in the first of the vessels. Gas isolated in the first vessel is then shared between the two vessels by opening the connecting valve. The pressure after expansion is usually calculated from knowledge of the initial pressure, the temperature of each vessel, and the ratio between the final volume and the initial volume occupied by the gas. Pressure standards [1] based on the technique are maintained by a number of National Measurement Institutes [2].

Static Expansion was first used by Knudsen [3,4] in the development of his radiometer gauges. Interest in the technique increased in the 1960s with the requirement for more accurate calibration of ionisation gauges than was possible with the prevailing McLeod gauge standards [5]. At that time Static Expansion systems were largely constructed from glass [6,7,8] with the appearance of the first metal systems in the mid 1960s [5,9]. The papers of Schumann [7], Smetana [9], and Poulter [10] provide interesting descriptions of Static Expansion at that time and are useful sources for references to earlier work. The present state of the art is described in the recent papers of Jousten [11] and Jitschin [12,13].

This report describes a comparison of the performance of two of NPL's high vacuum standards in the pressure range 10^{-4} Pa to 1 Pa. The standards are known as "SEA2", which dates from the 1960s, and its replacement, "SEA5" which was constructed in the 1990s. The standards are briefly described in the next section. The following section of this report describes the technique used for comparing the standards and details the data processing that has been performed. It then presents an analysis of the results. The final section presents some concluding remarks and recommendations and is followed by an Appendix in which various aspects of the comparison are explained in further detail.

2 HIGH VACUUM STANDARDS

2.1 SEA2

SEA2 was built in the early 1970s when the existing standard SEA1 (dating from 1965) was upgraded. Since then it has been the UK's standard for pressures in the region from 10^{-6} Pa up to about 1 Pa. The system is shown in Figure 1 the original parts of the system (SEA1) being located to the right of the image.

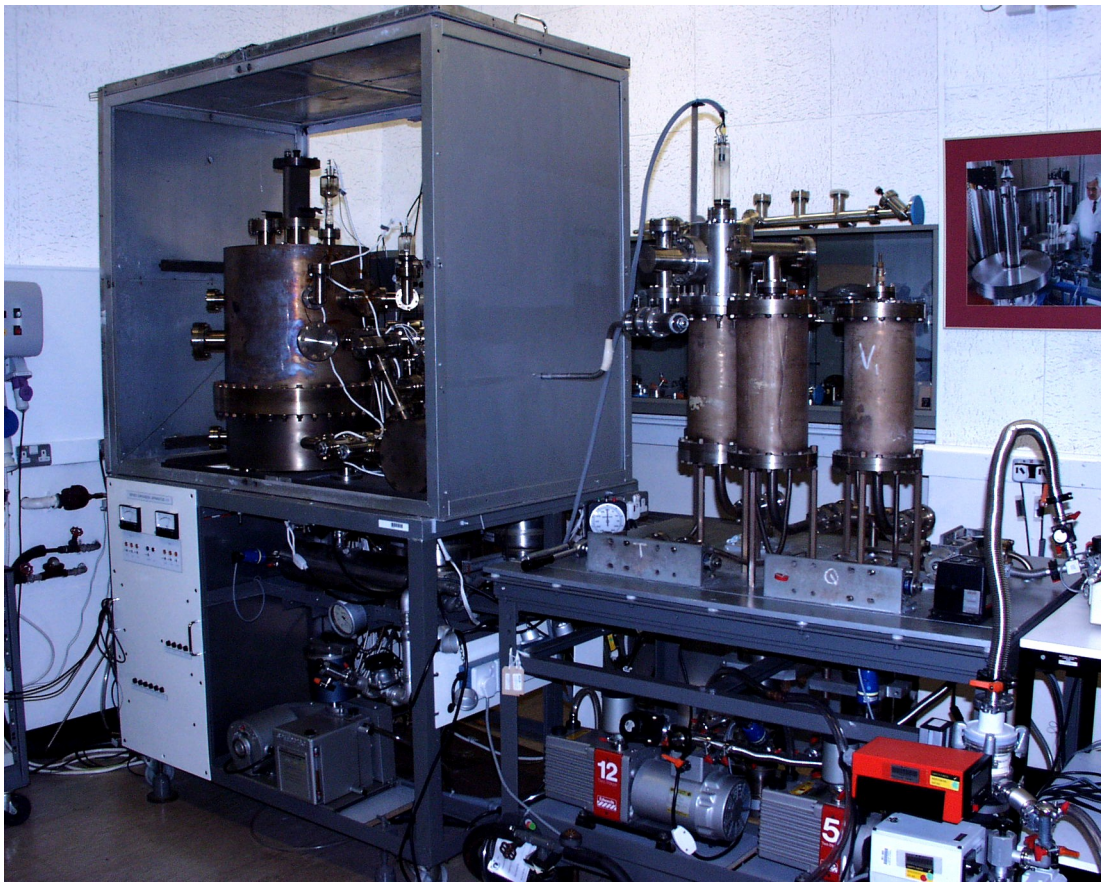


Figure 1. High vacuum standard SEA2

SEA2 is an all-metal system consisting of stainless steel chambers connected by manually operated, metal sealed angle valves. It is pumped using a system of oil diffusion pumps supported by liquid nitrogen cold traps. To achieve an ultimate pressure in the UHV region ($<10^{-8}$ Pa) the high vacuum end of the system can be baked to temperatures in excess of 250°C.

SEA2 is one of the standards supporting the UK's CMC table entries, summarised in part in Table 1.

Table 1: Summary of the UK's Calibration and Measurement Capability (CMC) Table entry in the vacuum region, December 2003.

Instrument Type or Method	Range Min /Pa	Range Max /Pa	Expanded uncertainty (k=2), Pressure p in Pa
SEA2, "Q" expansion, BAG	1×10^{-6}	1×10^{-4}	$1.2\times10^{-2}\,p+5.7\times10^{-9}$
SEA2, "T" expansion, SRG	1×10^{-4}	2.5×10^{-2}	$7.0\times10^{-3}\,p+3.3\times10^{-7}$
SEA3 "T" expansion, 1 torr CDG	2.5×10^{-2}	6×10^{-2}	$8.0\times10^{-3}\,p+9.0\times10^{-5}$
SEA2 "D" expansion, SRG	6×10^{-2}	$1\times10^{+1}$	$6.0\times10^{-3}\,p+6.0\times10^{-5}$
SEA3 "S" expansion, 10 torr CDG	$1\times10^{+1}$	$1\times10^{+3}$	$3.7\times10^{-3}\,p+6.0\times10^{-3}$

Besides being very labour intensive (typically taking up to 8 hours and involving a walk of several kilometres with over 600 manual valve operations) there are a number of features of SEA2 that limit its capability to deliver better uncertainties than are currently achieved. In particular: there is very little space for air circulation around the system's component parts resulting in measured inter- and intra-vessel temperature gradients of up to 2K. Also, the requirement to maintain fluid levels in liquid nitrogen traps and the lack of automation precludes any possibility for extended data collection to support continuous evaluation of the system. Finally, the physical condition of various (now obsolete) components of the system has made its operation increasingly unreliable. For these reasons a replacement system, SEA5 was constructed.

2.2 SEA5

Besides the age and fragility of SEA2 there are also concerns about the operability and flexibility of such a manual-intensive instrument in a modern laboratory environment. Modern products from our customers can often be handled in a near-automatic fashion and the collection and analysis of large amounts of calibration and characterisation data is now routinely achievable. For these reasons two replacements for SEA2 were constructed.

A series expansion standard, SEA4 was built in the early 1980s. Originally, it was intended that this system would act as a test bed for different interconnecting valves before eventually forming the first three stages of another system. However, the system experienced significant

problems. In particular, the volume ratio measurements did not converge. This was attributed to the large temperature gradients (several degrees) across the system. SEA4 was therefore abandoned in 1993 after retrieving the inter-volume valves for use on the recently designed, SEA5.

The most recent standard, SEA5 was constructed in 1993. SEA5 is a four-stage expansion system where the final two stages are bakeable. In particular, SEA5 is designed to allow improved air circulation around the system thus avoiding the large temperature gradients across vessels that are characteristic of SEA2.

Like SEA2, SEA5 is an all-metal system consisting of stainless steel chambers connected by metal sealed angle valves. SEA5 uses turbomolecular pumps, permitting around-the-clock operation, and employs electro-pneumatic valve actuators allowing remote, PC controlled operation of the system. The system is shown in Figure 2, illustrating the more open and accessible layout of the system.

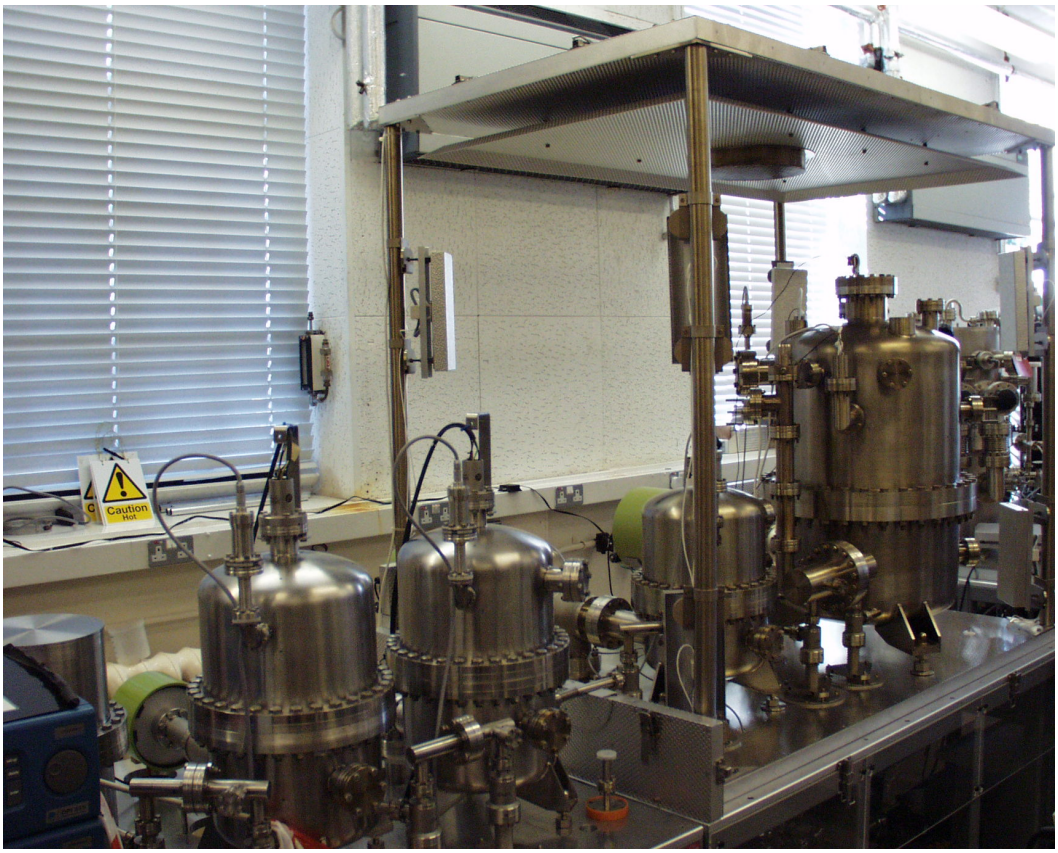


Figure 2. High vacuum standard SEA5

In common with SEA2 a quartz bourdon tube gauge is used to measure the initial pressure of gas prior to expansion.

3 MEASUREMENTS

3.1 TRANSFER STANDARDS

The comparison between SEA2 and SEA5 took place in the pressure range 3×10^{-4} Pa to 0.9 Pa using unbaked spinning rotor gauges as transfer standards.

A summary of the properties assigned[1] to the rotors is given in Table 2 and Table 3 (Logbook "SEA5", vol 5, p54)

Table 2: Rotors used in this comparison

Name	Serial number
SRG1	191375
SRG2	SRG03
SRG3	MKS90602

Table 3: Properties assigned to the rotors for this comparison

Property	Assigned value
Mol. weight	28.013
Viscosity	0.00
Accomodation factor	1.00
Diameter	4.5
Density	7.715
Temperature	293.1

3.2 CHRONOLOGY

The timescale for the measurements is summarised in Table 4. During each period of measurements 5 repeat runs were performed on 5 separate days.

Table 4: Chronology of data collection.

Dates	System
13-17 Feb 2006	SEA5
21-24,28 Feb 2006	SEA2
3,6-9 Mar 2006	SEA5

[1] The exact values for the parameters are not important in terms of the comparison of equivalence between the primary standards. The important point is that the same values were used throughout.

3.3 PROTOCOL

The structure of the comparison and the subsequent data analysis is based on that in the report of the (bilateral) comparison SIM-EUROMET.M.P.BK3 and the report of wider comparison, Euromet.M.P-K1.b [14,15].

In the comparisons cited here [14,15] the residual drag for each rotor is calculated from rotor frequency measurements and pre-determined frequency-drag characteristics. Although this technique can be applied to static expansion measurement, a more appropriate technique in this case is to directly measure the residual drag immediately prior to each expansion[2]. The only significant deviation this introduces is in the calculation of accommodation coefficient and the associated uncertainties, therefore, in place of equations (1) and (7)-(10) of [15] we instead have:

Accommodation coefficient, σ, defined as

$$\sigma = \frac{\Delta p_{ind}\sqrt{\frac{T_{rotor}}{T_{control}}}}{\Delta p_{ref}} \qquad (1)$$

where

T_{rotor} is the rotor temperature

$T_{control}$ is the temperature programmed into the SRG control unit

Δp_{ind} is the change in SRG indication after exposure to the change of Δp_{ref} in the reference pressure

Uncertainty in the mean value for n measurements of σ is therefore

$$u_\sigma^{\,2} = \frac{n-1}{n-3} s_\sigma^{\,2} + \left(u_{ind} S_{ind}\right)^2 + \left(u_{rot} S_{rot}\right)^2 + \left(u_{ctl} S_{ctl}\right)^2 + \left(u_{ref} S_{ref}\right)^2 \qquad (2)$$

where

s_σ is the standard deviation for the n repeated measurements

u_{ind} is the uncertainty in the indicated difference, calculated from the displayed resolution of the A and B indications

u_{rot} is the uncertainty in the rotor temperature

[2] This approach is not possible with the continuous expansion technique of [14], adopted by [15] for uniformity of approach and analysis.

u_{ref} is the uncertainty in the reference pressure change introduced by expansion

u_{ctl} is the uncertainty in the programmed controller temperature, = 0

S are the relevant sensitivity coefficients

$$S_{ind} = \frac{\sqrt{\frac{T_{rotor}}{T_{control}}}}{\Delta p_{ref}} \tag{3}$$

$$S_{rot} = \frac{\sigma}{2T_{rotor}} \tag{4}$$

$$S_{ref} = \frac{-\sigma}{\Delta p_{ref}} \tag{5}$$

3.4 DATA PROCESSING

All data has been processed using Excel workbooks to implement the methods outlined above and in the associated references.

Besides the measured data the following inputs were required:

3.4.1 SEA2 reference uncertainty

The uncertainty in the generated change in pressure over residual pressure for SEA2 was calculated from the relations:

$$u_{SEA2}{}^2 = \left(0.00297 \times p\right)^2 + \left(1.9 \times 10^{-7}\right)^2 \text{ for } p \le 0.02 \text{ Pa} \tag{6}$$

$$u_{SEA2}{}^2 = \left(0.00239 \times p\right)^2 + \left(8.55 \times 10^{-5}\right)^2 \text{ for } p > 0.02 \text{ Pa} \tag{7}$$

These values were used for Euromet.M.P-K1.b [15] and are slightly smaller than the CMC entries as they represent only the uncertainty associated with the reference standard. Uncertainties are expressed at coverage factor, $k = 1$.

3.4.2 SEA5 reference uncertainty

Estimation of the uncertainty in the generated change in pressure over residual pressure for SEA5 is described in the Appendix to this report. The uncertainty can be represented by:

$$u_{SEA5} = a \times p + b \tag{8}$$

where, a and b are given in Table 5. Uncertainties are expressed at coverage factor, $k = 1$.

Table 5: Coefficients of equation for evaluating estimated uncertainty in generated change in pressure over residual for SEA5.

Pressure Range, /Pa	a	b /Pa
$4.4 \times 10^{-7} < p < 1.3 \times 10^{-4}$	0.0024	3.5×10^{-10}
$1.3 \times 10^{-4} < p < 2.6 \times 10^{-2}$	0.00175	0
$2.6 \times 10^{-2} < p < 5.1$	0.0010	0
$5.1 < p < 1000$	0.0002	4×10^{-3}

3.4.3 Temperature

In the analysis of comparisons [14,15] followed here, a single fixed value has been assumed to represent the uncertainty associated with the ability to determine the temperature of any component in the system.

$u_T = 0.2\,\mathrm{K}$, ($k = 1$)

3.4.4 Drift during the comparison

The data collected indicates either a small systematic change in the accommodation factor for all three transfer-standards, or the presence of a small systematic difference associated with the operation of SEA5 at the times of the measurements. In either case, the effect is small (comparable to the sum of the standard deviations for each rotor) allowing a “zero-drift” model to be assumed in the data analysis, i.e. the SEA5 data is assumed to be sampled from a single population.

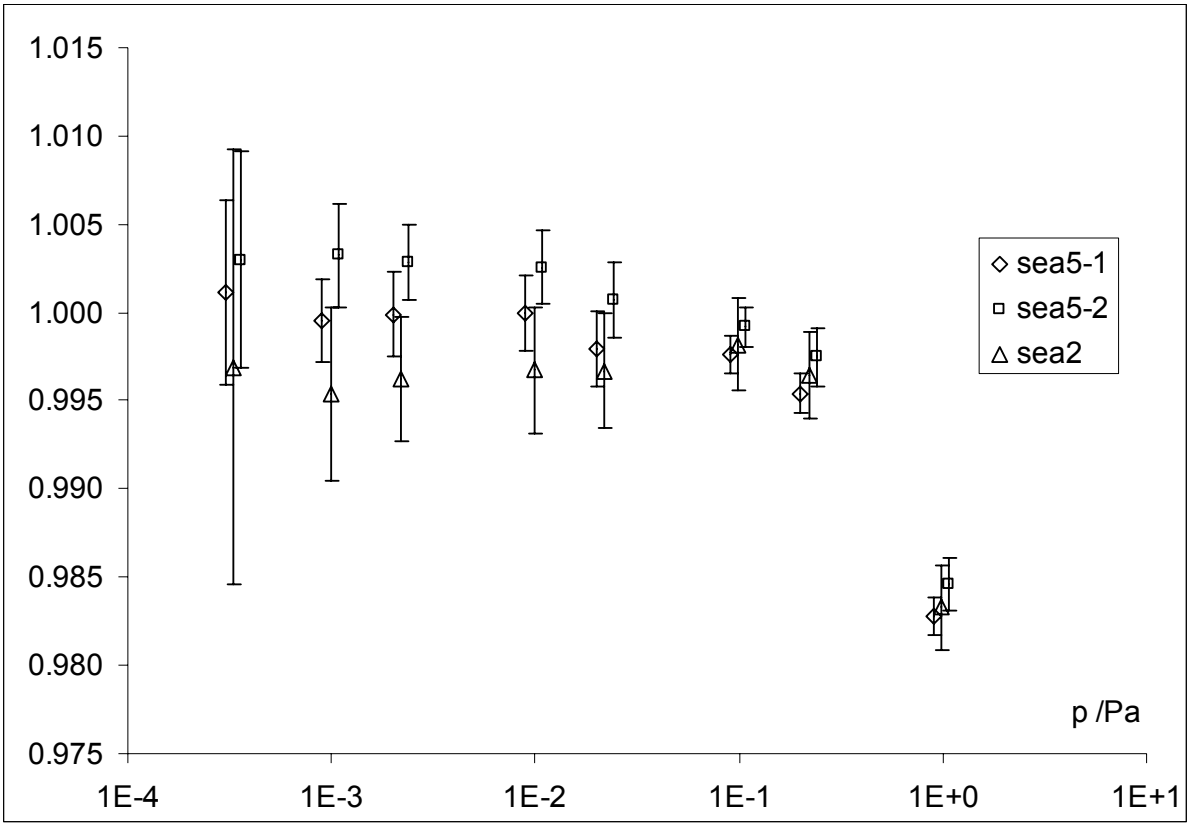


Figure 3: Average Accommodation Factor for SRG1. Uncertainty bars at *k*=1.

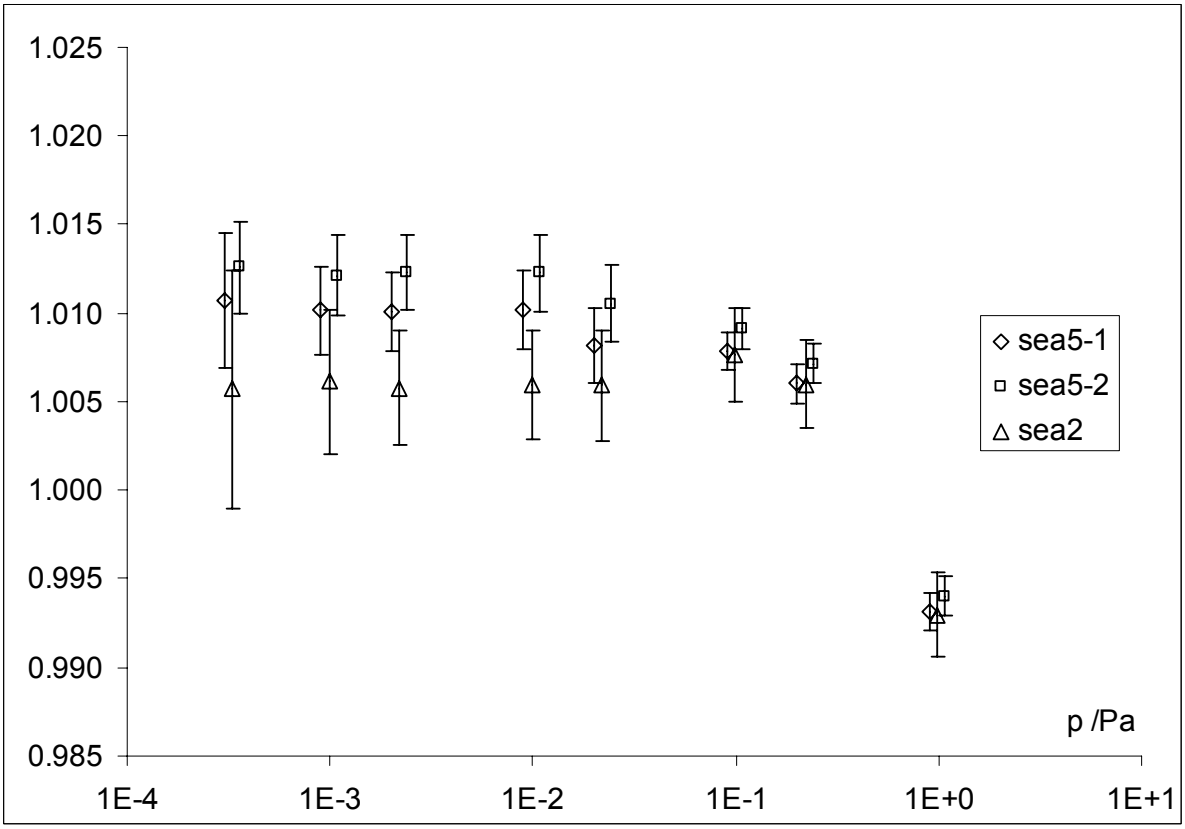


Figure 4: Average Accommodation Factor for SRG2. Uncertainty bars at *k*=1.

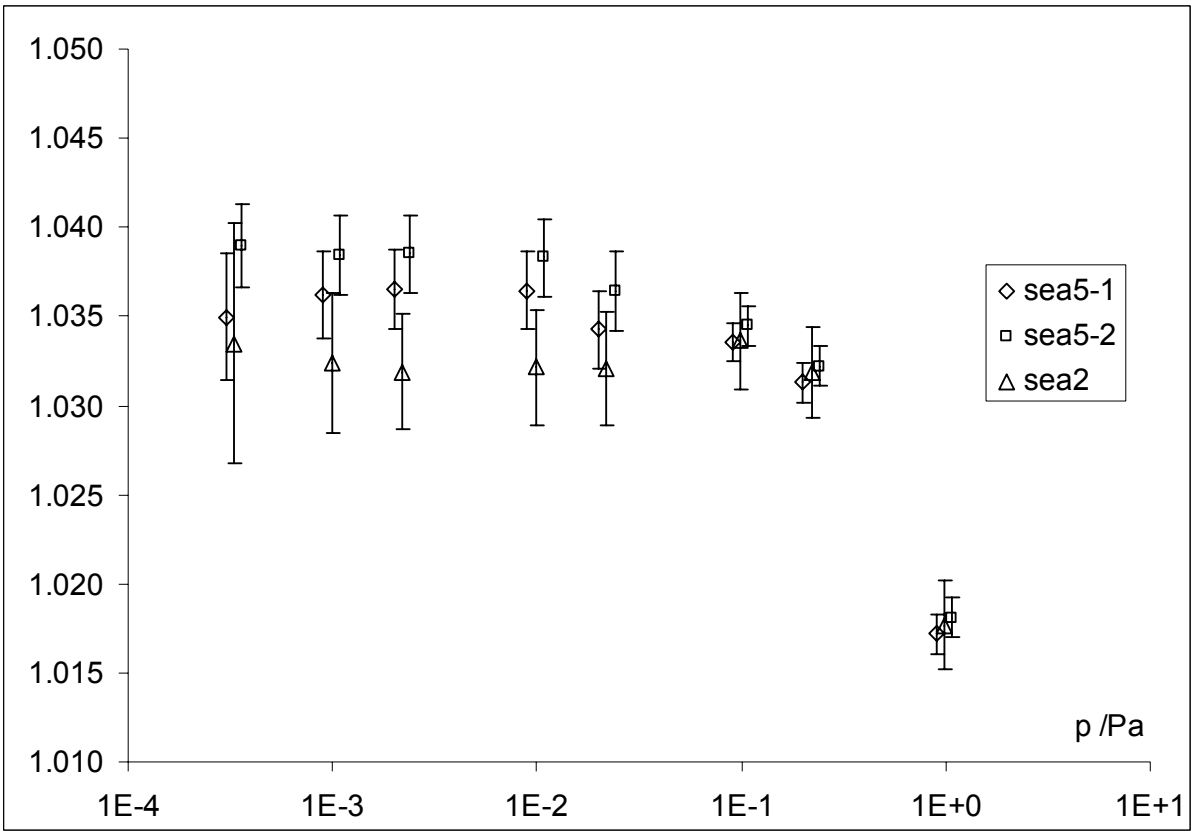


Figure 5: Average Accommodation Factor for SRG3. Uncertainty bars at *k*=1.

3.5 EQUIVALENCE ANALYSIS

The equivalence analysis was performed in line with previously published comparisons. The Excel worksheet where the final equivalence was evaluated is reproduced in Figure 6 and Figure 7.

3.5.1 Equivalence of SEA2 and SEA5

The "*En*" values for the comparison are tabulated in Table 6 below

Table 6: *En* (*k*=2) ratios for SEA2/SEA5 comparison

Pressure /Pa	*En* (*k*=2)
0.0003	0.23
0.0009	0.46
0.002	0.63
0.009	0.61
0.02	0.38
0.09	0.08
0.2	0.03
0.9	0.05

3.5.2 Equivalence of SEA5 with the EUROMET reference values

SEA5 can be linked to the EUROMET reference values since SEA2 took part in that comparison [15]. Following the same arguments linking SIM and EUROMET [14] we are able to calculate equivalence at the common pressures as summarised in Table 7.

Table 7: Summary of calculations for linkage between SEA5 and EUROMET reference values.

$p_{sea2\text{-}eur}$ /Pa	u_{peur} /Pa	$p_{SEA5\text{-}eur}$ /Pa	$u(p_{SEA5\text{-}eur})$ /Pa	En,eur (k=2)
9.0020×10^{-4}	1.8×10^{-6}	8.9569×10^{-4}	2.99×10^{-6}	0.551
9.0000×10^{-3}	1.6×10^{-5}	8.9545×10^{-3}	2.30×10^{-5}	0.715
9.0100×10^{-2}	1.5×10^{-4}	9.0056×10^{-2}	1.24×10^{-4}	0.165
9.0040×10^{-1}	1.1×10^{-3}	9.0012×10^{-1}	1.28×10^{-3}	0.035

where

$p_{sea2\text{-}eur}$ is the SEA2 EUROMET value, given in table 13 of the report describing Euromet.M.P-K1.b [15].

u_{peur} is the standard uncertainty associated with $p_{sea2\text{-}eur}$

$p_{SEA5\text{-}eur}$ is the equivalent SEA5 pressure corresponding to the EUROMET reference value.

$u(p_{SEA5\text{-}eur})$ is the uncertainty associated with $p_{SEA5\text{-}eur}$

Analysis of the equivalence of the high vacuum standards, sea2 and sea5, assuming zero drift in transfer standards.

Analysis is based on that in the report of the (bilateral) SIM-EUROMET.M.P.BK3 and the report of the Euromet.M.P-K1.b

Jousten, K., Santander Romero, L.A., Torres Guzman, J.C. Metrologia 2005; 42 No 1A (Technical Supplement 2005) 07002

Jousten, K et al. Metrologia 2005; 42 No 1A (Technical Supplement 2005) 07001

References to equations in the bilateral report are identified as (B1), (B2), ... for equations 1,2,...

References to equations in the main report are identified as (C1), (C2), ... for equations 1,2,...

All pressures in Pa

	(B11)				(B12)			(B7)			(B14)					(B17)			(B18)		
sea5 combined data					**"indicated pressure" at p_{nom}**			**overall uncertainty**			**uncertainty in indicated pressure**			**mean indication**		**pressure ratio**			**uncertainty in ratio**		
	p_{nom}	$\sigma 1_{sea5}$	$\sigma 2_{sea5}$	$\sigma 3_{sea5}$	$p1_{sea5}$	$p2_{sea5}$	$p3_{sea5}$	$u(\sigma 1)_{sea5}$	$u(\sigma 2)_{sea5}$	$u(\sigma 3)_{sea5}$	$u(p_1)_{sea5}$	$u(p_2)_{sea5}$	$u(p_3)_{sea5}$	p_{sea5}	$u(p_{sea5})$	r_1	r_2	r_3	$u(r_1)$	$u(r_2)$	$u(r_3)$
	3E-4	1.002038	1.011655	1.036994	3.006E-4	3.035E-4	3.111E-4	0.00549	0.00328	0.00361	1.648E-6	9.856E-7	1.084E-6	3.051E-4	1.239E-6	0.99489	0.99411	0.99662	0.01354	0.00747	0.00743
	9E-4	1.001373	1.011126	1.037309	9.012E-4	9.100E-4	9.336E-4	0.00325	0.00255	0.00259	2.929E-6	2.296E-6	2.335E-6	9.149E-4	2.520E-6	0.99402	0.99505	0.99529	0.00590	0.00479	0.00454
	2E-3	1.001369	1.011196	1.037501	2.003E-3	2.022E-3	2.075E-3	0.00275	0.00249	0.00245	5.498E-6	4.974E-6	4.892E-6	2.033E-3	5.121E-6	0.99488	0.99464	0.99461	0.00446	0.00406	0.00391
	9E-3	1.001268	1.011221	1.037386	9.011E-3	9.101E-3	9.336E-3	0.00250	0.00241	0.00239	2.253E-5	2.174E-5	2.155E-5	9.150E-3	2.194E-5	0.99546	0.99478	0.99494	0.00436	0.00387	0.00387
	2E-2	0.999339	1.009362	1.035354	1.999E-2	2.019E-2	2.071E-2	0.00260	0.00251	0.00248	5.208E-5	5.012E-5	4.955E-5	2.029E-2	5.059E-5	0.99734	0.99658	0.99681	0.00420	0.00395	0.00391
	9E-2	0.998365	1.008503	1.034022	8.985E-2	9.077E-2	9.306E-2	0.00138	0.00129	0.00121	1.240E-4	1.162E-4	1.088E-4	9.123E-2	1.163E-4	0.99981	0.99914	0.99962	0.00296	0.00291	0.00287
	2E-1	0.996439	1.006578	1.031745	1.993E-1	2.013E-1	2.063E-1	0.00176	0.00124	0.00120	3.523E-4	2.484E-4	2.409E-4	2.023E-1	2.805E-4	1.00002	0.99943	1.00006	0.00306	0.00276	0.00273
	9E-1	0.983687	0.993598	1.017628	8.853E-1	8.942E-1	9.159E-1	0.00161	0.00118	0.00120	1.447E-3	1.060E-3	1.078E-3	8.985E-1	1.195E-3	0.99958	0.99938	1.00004	0.00294	0.00270	0.00270
sea2 data																					
	p_{nom}	$\sigma 1_{sea2}$	$\sigma 2_{sea2}$	$\sigma 3_{sea2}$	$p1_{sea2}$	$p2_{sea2}$	$p3_{sea2}$	$u(\sigma 1)_{sea2}$	$u(\sigma 2)_{sea2}$	$u(\sigma 3)_{sea2}$	$u(p_1)_{sea2}$	$u(p_2)_{sea2}$	$u(p_3)_{sea2}$	p_{sea2}	$u(p_{sea2})$						
	3E-4	0.996917	1.005698	1.033492	2.991E-4	3.017E-4	3.100E-4	0.01234	0.00676	0.00678	3.703E-6	2.028E-6	2.034E-6	3.036E-4	2.588E-6						
	9E-4	0.995385	1.006121	1.032419	8.958E-4	9.055E-4	9.292E-4	0.00490	0.00410	0.00391	4.407E-6	3.689E-6	3.520E-6	9.102E-4	3.872E-6						
	2E-3	0.996242	1.005773	1.031905	1.992E-3	2.012E-3	2.064E-3	0.00350	0.00325	0.00322	7.002E-6	6.498E-6	6.446E-6	2.023E-3	6.649E-6						
	9E-3	0.996719	1.005943	1.032136	8.970E-3	9.053E-3	9.289E-3	0.00356	0.00306	0.00320	3.207E-5	2.759E-5	2.884E-5	9.104E-3	2.950E-5						
	2E-2	0.996683	1.005907	1.032055	1.993E-2	2.012E-2	2.064E-2	0.00328	0.00309	0.00319	6.564E-5	6.179E-5	6.374E-5	2.023E-2	6.373E-5						
	9E-2	0.998171	1.007641	1.033631	8.984E-2	9.069E-2	9.303E-2	0.00262	0.00264	0.00271	2.357E-4	2.374E-4	2.439E-4	9.118E-2	2.390E-4						
	2E-1	0.996455	1.006001	1.031808	1.993E-1	2.012E-1	2.064E-1	0.00249	0.00248	0.00255	4.977E-4	4.962E-4	5.098E-4	2.023E-1	5.012E-4						
	9E-1	0.983275	0.992978	1.01767	8.849E-1	8.937E-1	9.159E-1	0.00240	0.00241	0.00247	2.164E-3	2.169E-3	2.226E-3	8.982E-1	2.186E-3						

Figure 6: Equivalence analysis for SEA2/SEA5 comparison (p1/2)

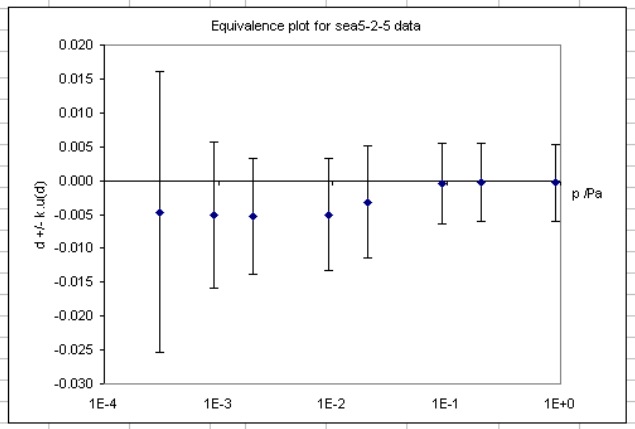
k = 2

Excluding u(ΔPref), to account for correlations in r_i when averaged ...

(B7), excluding u(ΔPref) restricted overall uncertainty			(B14), excluding u(ΔPref) restricted uncertainty in indication			(B18), excluding u(ΔPref) restricted uncertainty in ratio			(B19)	(B21)	(B20),(B22)	(B23)	(B25)
$u'(\sigma 1)_{sea5}$	$u'(\sigma 2)_{sea5}$	$u'(\sigma 3)_{sea5}$	$u'(p_1)_{sea5}$	$u'(p_2)_{sea5}$	$u'(p_3)_{sea5}$	$u'(r1)$	$u'(r_2)$	$u'(r_3)$	Equivalence calculations ... r	d	$u(d)$	$k.u(d)$	E_n
0.00511	0.00259	0.00296	1.534E-6	7.765E-7	8.880E-7	0.013042	0.00652	0.006477	0.995322	-0.00468	0.0103505	0.020701	0.225987
0.00256	0.00155	0.00156	2.308E-6	1.399E-6	1.403E-6	0.004678	0.003177	0.002783	0.994989	-0.00501	0.0053932	0.010786	0.464533
0.00188	0.00145	0.00129	3.767E-6	2.894E-6	2.590E-6	0.002656	0.00191	0.001574	0.994664	-0.00534	0.0042657	0.008531	0.625491
0.00150	0.00132	0.00119	1.353E-5	1.189E-5	1.076E-5	0.002492	0.001471	0.001462	0.994948	-0.00505	0.0041244	0.008249	0.612416
0.00167	0.00148	0.00136	3.339E-5	2.970E-5	2.720E-5	0.002193	0.001665	0.001561	0.996839	-0.00316	0.0041309	0.008262	0.382595
0.00095	0.00080	0.00062	8.548E-5	7.255E-5	5.642E-5	0.001081	0.000933	0.000791	0.999513	-0.00049	0.0029622	0.005924	0.082134
0.00145	0.00073	0.00062	2.905E-4	1.455E-4	1.243E-4	0.001567	0.000835	0.000748	0.999805	-0.00019	0.0028885	0.005777	0.033701
0.00127	0.00063	0.00063	1.145E-3	5.692E-4	5.682E-4	0.001385	0.000749	0.000748	0.999693	-0.00031	0.0028185	0.005637	0.054394

EUROMET equivalence calculations ...		(B26)	(B27)	(B28)
$p_{sea\,2\text{-}eur}$	u_{peur}	$p_{sea\,5\text{-}eur}$	$u(p_{sea5}\text{-}eur)$	$E_{n,eur}$
9.002E-04	1.800E-06	8.957E-4	2.993E-6	0.5508963
9.000E-03	1.600E-05	8.955E-3	2.303E-5	0.7146879
9.010E-02	1.500E-04	9.006E-2	1.243E-4	0.1649051
9.004E-01	1.100E-03	9.001E-1	1.278E-3	0.0354134

$u'(\sigma 1)_{sea2}$	$u'(\sigma 2)_{sea2}$	$u'(\sigma 3)_{sea2}$	$u'(p_1)_{sea2}$	$u'(p_2)_{sea2}$	$u'(p_3)_{sea2}$
0.01196	0.00603	0.00601	3.589E-6	1.809E-6	1.803E-6
0.00390	0.00280	0.00242	3.507E-6	2.517E-6	2.176E-6
0.00187	0.00127	0.00099	3.736E-6	2.545E-6	1.979E-6
0.00198	0.00068	0.00093	1.784E-5	6.126E-6	8.359E-6
0.00141	0.00078	0.00087	2.832E-5	1.569E-5	1.741E-5
0.00051	0.00048	0.00052	4.618E-5	4.371E-5	4.724E-5
0.00057	0.00042	0.00045	1.145E-4	8.436E-5	9.136E-5
0.00049	0.00039	0.00042	4.396E-4	3.536E-4	3.821E-4



Conclusions:

Under the zero-drift assumption

1. sea2 and sea5 are equivalent at k=2 level. (k_{min}=1.25)
2. sea5 is equivalent at the k=2 level with the Euromet reference values. (kmin=1.43)

Note:

From knowledge of the fundamental characteristics of SRGs and the previous operation of sea2, it seems that there may be a step discontinuity in the sea2 data. between the higher pressure, double expansion data and the lower pressure triple expansion data. This is reflected in the equivalence plot.

Figure 7: Equivalence analysis for SEA2/SEA5 comparison (p2/2)

4 CONCLUSION

A comparison was performed between NPL's two high vacuum standards, SEA2 and SEA5, based upon the protocol and methodology of two key comparisons: bilateral SIM-EUROMET.M.P.BK3 and regional, Euromet.M.P-K1.b [14,15]

The high vacuum standards SEA2 and SEA5 were been shown to be equivalent in the pressure range 3×10^{-4} Pa to 0.9 Pa. According to the computed "*En*" value, the systems remain equivalent down to a coverage factor of $k = 1.25$. In making this comparison the uncertainties for SEA5 have been evaluated to be about half as large as the uncertainties for SEA2. This is not surprising since the uncertainty evaluation for SEA2 used a very conservative approach whereas the SEA5 evaluation uses a "best estimate" approach.

SEA2 has been used to link SEA5 to the EUROMET reference values at the common pressures in the range 9×10^{-4} to 9×10^{-1} Pa. According to the computed "*En*" value, it remains equivalent down to a coverage factor of $k = 1.43$

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6 APPENDIX: SEA5 UNCERTAINTY BUDGET

This section of the Report describes the estimation of the uncertainty in the generated change in pressure over the residual pressure for SEA5. By necessity this section of the report refers to various unpublished Excel and Mathcad workbooks. The section is designed to stand alone and so a certain amount of repetition from the preceding sections is unavoidable.

6.1 BACKGROUND

Series Expansion Apparatus 5 (SEA5) is the replacement for the original NPL high vacuum standard, sea2. The new system is fully automated and incorporates a number of enhancements including the use of turbomolecular pumps to allow around the clock operation, and improved airflow around the vessels to reduce the thermal gradients between vessels.

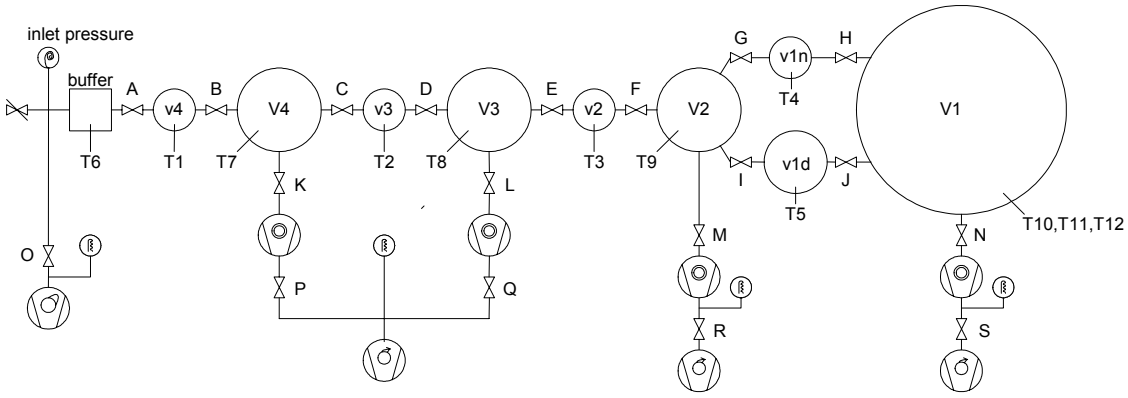

Figure 8. Schematic of the high vacuum standard, SEA5

The system consists of alternate small and large vessels. The pressure of the initially isolated gas is measured using a high quality transfer standard - a Ruska 7050i, quartz bourdon gauge. Calculable reference pressures are generated in the vessel V_1. Expansion into V_1 is performed from either v_{1d} or v_{1n} and can be:

"sd" - *Single Expansion* from v_{1d} - where initially valves J and H are closed and the other inter-vessel valves are all open so that an appropriate initial pressure can be established throughout the connected system. Volume v_{1d} is then isolated by slowly closing valve I, before the pressure of the gas trapped in v_{1d} is measured (by the inlet gauge), and then expanded into the connected volumes v_{1d}+V_1. Alternatively, it can be:

"sn" - *Single Expansion* from v_{1n} - where it is v_{1n} that is isolated by slowly closing valve G, before the pressure of the gas trapped in v_{1n} is measured, and then expanded into the connected volumes $v_{1n}+V_1$.

Expansion can also be:

"dd" ("dn") - *Double Expansion* - where the initial isolation takes place in v_2, before expansion into $v_2+V_2+v_{1d}+v_{1n}$, followed by isolation in v_{1d} (or v_{1n}) and then expansion into the connected volumes $v_{1d}+V_1$ (or $v_{1n}+V_1$). Or expansion can be:

"td" ("tn") - *Triple Expansion* - where the initial isolation takes place in v_3, before expansion into $v_3+V_3+v_2$, followed by isolation in v_2 and subsequent completion of the process as for a double expansion. Or:

"qd" ("qn") - *Quadruple Expansion* - where the initial isolation takes place in v_4, before expansion into $v_4+V_4+v_3$, followed by isolation in v_3 and subsequent completion of the process as for a triple expansion.

Calibrations are performed by exposing the unit under calibration (UUC) to the calculated reference pressure and then measuring its response.

For spinning rotor gauges (SRGs) the results are usually presented in terms of a *correction factor*, *CF*, defined in terms of the *change* in output corresponding to a calculated *change* in reference pressure.

CF = (change in reference pressure) / (change in output of UUC)

For ionisation gauges (IGs) the results may be presented in terms of CF, or it may be expressed in terms of a *calibration correction*, Δ_{cal}, defined in terms of the difference between the calculated absolute reference pressure and the UUC output

Δ_{cal} *= (reference pressure) – (output of UUC)*

Where the UUC provides an indication in pressure units the reference pressure is usually converted to the same units using standard conversion factors[3]. Where the output of the UUC is not measured using pressure units, it is usually converted to the reference pressure units using a nominal conversion given by the manufacturer.

The following sub-sections of this document describe the uncertainty budget for the pressure changes generated using this system.

[3] 1 mbar = 100 Pa = 1.333224 torr

6.2 UNCERTAINTY IN GENERATED CHANGE IN PRESSURE ABOVE THE RESIDUAL PRESSURE IN SEA5

6.2.1 Error Model for generated pressure

Following a static expansion sequence the total pressure, p_{tot}, in the calibration vessel of SEA5 is given by

$$p_{tot} = p_{cal} + p_{res} + \Delta p_{og} \tag{A1}$$

where

p_{cal} is the total pressure of calibration gas in the calibration vessel, given by

$$p_{cal} = p_{ME} + \delta p_{MEcalc} + \delta p_{ME} + \delta p_{ad} \tag{A2}$$

in which:

p_{ME} is the pressure calculated using the chosen *measurement equation*: Taking into account the non-ideal nature of a real gas, the simplified measurement equation[4] for static expansion based on expansion ratios can be written:

$$\rho_f = \rho_i \times E \tag{A3}$$

where E is the overall expansion ratio, ρ_f is the final molar density and ρ_i is the initial molar density. The density can be calculated using

$$\rho = \frac{1}{2RTB_T}\left(\left(R^2T^2 + 4RTB_T p\right)^{1/2} - RT\right) \tag{A4}$$

which is a solution of the second order virial equation of state for pressure

$$p = \rho\ R\ T\left(1 + \rho\ B_T\right) \tag{A5}$$

where R is the gas constant and B_T is the second virial coefficient for the particular gas species, evaluated at temperature T.

Hence the expanded pressure is calculated from the final density and temperature, (ρ_f,T_f) using

$$p_{ME} = p(\rho_f, T_f) \tag{A6}$$

The initial gas density, ρ_i, for initial pressure and temperature, (p_i,T_i) is

$$\rho_i = \rho(p_i, T_i) \tag{A7}$$

where the initial pressure is calculated from the measured pressure, p_m, after adding a calibration correction, Δp_m, and a correction for a systematic, valve-closing effect, Δp_{sys}.

$$p_i = p_m + \Delta p_m + \Delta p_{sys} \tag{A8}$$

δp_{MEcalc} is the error in p_{ME} associated with errors in the input quantities (p_m, T_i, T_f, E, B, Δp_m, Δp_{sys})

δp_{ME} is the error in p_{ME} associated with inadequacies of the model underlying the measurement equation for static expansion based on expansion ratios.

δp_{ad} is the change in calibration gas pressure due to adsorption on the surfaces of the calibration vessel.

p_{res} is the residual pressure in the calibration vessel immediately prior to expansion, consisting of residual gases from earlier operations and outgassing products that originate in the vessel in the time since it was isolated from the pumping system. It is given by

$$p_{res} = p_{resm} + \delta p_{res} \tag{A9}$$

where p_{resm} is the measured residual pressure and δp_{res} is the error associated with the measurement.

Δp_{og} is the change in pressure due to outgassing products, consisting of a fixed quantity of gas transported into the calibration vessel by the expansion process. It is given by

$$\Delta p_{og} = \Delta p_{ogm} + \delta p_{og} \tag{A10}$$

where Δp_{ogm} is the measured pressure change due to outgassing and δp_{og} is the error associated with the measurement.

[4] An *implicit* assumption of this simplified approach is that the molar density is the *same* throughout a system of connected vessels. This assumption holds under "isothermal" conditions where the temperature of the gas is

The total pressure can therefore be written

$$p_{tot} = p_{ME} + p_{resm} + \Delta p_{ogm} + \delta p_{MEcalc} + \delta p_{ME} + \delta p_{ad} + \delta p_{res} + \delta p_{og} + \delta p_o \qquad \text{(A11)}$$

where the additional term, δp_o, is introduced to take account of all other random errors.

6.3 UNCERTAINTY FOR GENERATED PRESSURE CHANGES

Assuming that the errors, δp, are uncorrelated, the uncertainty for the generated pressure can therefore be written as

$$u_{tot} = \sqrt{u_{MEcalc}{}^2 + u_{ME}{}^2 + u_{ad}{}^2 + u_{res}{}^2 + u_{og}{}^2 + u_o{}^2} \qquad \text{(A12)}$$

The uncertainty estimates are derived as follows:

6.3.1 Uncertainty associated with errors in the input quantities to the Measurement Equation, u_{MEcalc}

The uncertainty associated with errors in the input quantities to the measurement equation is estimated using the Mathcad workbook *"sea5 uncertainty in reference pressure.xmcd"*.

The workbook implements a Monte Carlo simulation of the measurement equations (A3) to (A8) in which errors are sampled from distributions describing the standard uncertainty for each of the input quantities, as summarised in Table 8 with N=50000 cycles.

constant throughout. See [16] *Greenwood, J.C. Vacuum (2006)* ***80****, 548-553.*

Table 8. Input distributions for Monte Carlo estimation of uncertainty associated with errors in the input quantities to the ME, u_{MEcalc}, calculated by Mathcad workbook *"sea5 uncertainty in reference pressure.xmcd"*

Input quantity	p.d.f. (normal distributions)
$p_m+\Delta p_m$	$\mu = 0$ mbar; $\sigma = 0.00319533 + (0.0000902075)\, p + (6.99726\times10^{-9})\, p^2$ /mbar Source: "*ruska ppi 7050i 59240 & 59241.xls*"
T_i, T_f	$\mu = 293$ K; $\sigma = 0.0135$ K Source: "*Edale CD 192.xls*"
E_{qn}	$\mu = 2281682379$, $\sigma = 1162110$
E_{qd}	$\mu = 764953077$, $\sigma = 412989$
E_{tn}	$\mu = 11604162$, $\sigma = 4331.8$
E_{td}	$\mu = 3890392$, $\sigma = 1571.2$
E_{dn}	$\mu = 58741$, $\sigma = 11.895$
E_{dd}	$\mu = 19693$, $\sigma = 4.58970$
E_{sn}	$\mu = 298.31$, $\sigma = 0.023780$
E_{sd}	$\mu = 100.01$, $\sigma = 0.011029$ Source: "*SA simulation results.xls*"
B	$\mu = 0$ Pa^{-1}, $\sigma = 0.1\times B_T$ / Pa^{-1}; $B_T = 2.4\times10^{-7}\, T - 7.62\times10^{-5}$ / Pa^{-1} Source: fit to data in [4]
Δp_{sys}	$\mu = 0$ Pa, $\sigma = u_v \times p$ /Pa $u_v = 2\times10^{-5}$ for "sd" expansions $u_v = 5\times10^{-5}$ for "sn" expansions $u_v = 0$ for directly generated pressures $u_v = 1\times10^{-4}$ for all other expansions Source: *"valve effects.xls"*

The results are summarised in Figure 9.

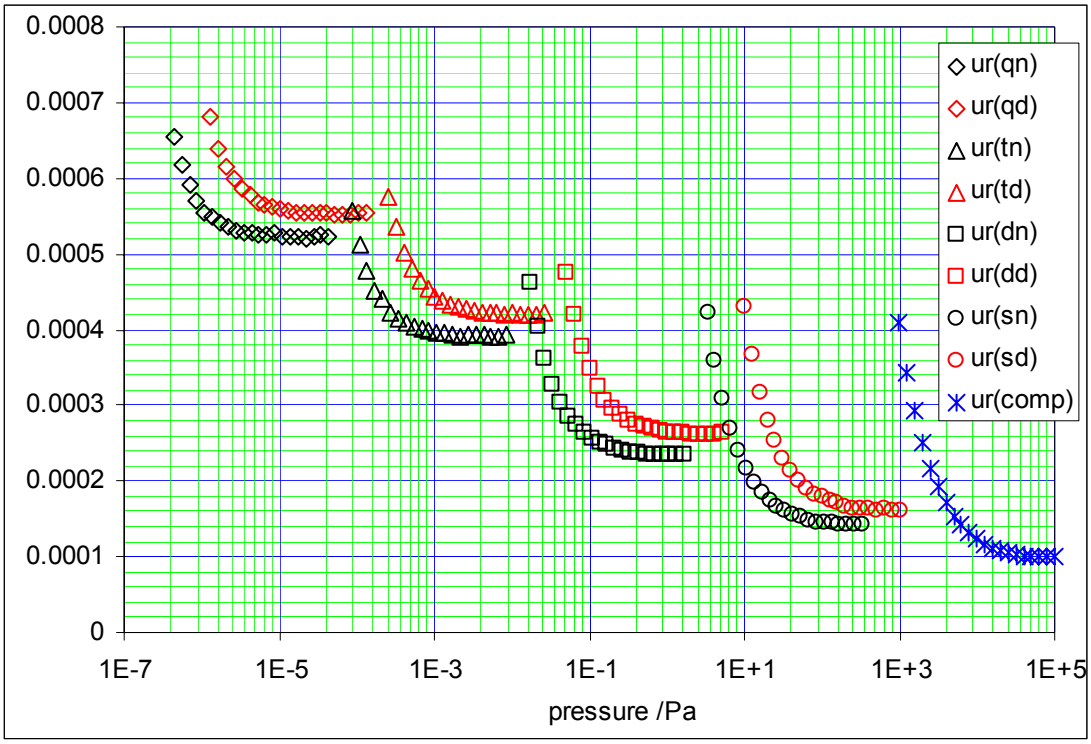

Figure 9. Summary of relative uncertainties in the calculated pressure, u_{MEcalc}, associated with errors in the input quantities to the ME for a range of expansion regimes. The figure also shows the uncertainty for pressures established by comparison "c".

It should be noted that the relative uncertainty calculated by the model is about a factor of 10 smaller than the expected *total* uncertainty.

The "d" expansions show a larger uncertainty than the "n" expansions due to the larger relative uncertainty of E_{sd} compared with E_{sn}.

6.3.2 Uncertainty associated with the model underpinning the ME, u_{ME}

The measurement equation based upon expansion ratios is used by necessity because of the difficulty in measuring the system volumes. Unfortunately the model relies on a number of assumptions that are known to be weak. The errors associated with the ME are simulated in a Mathcad workbook *"SEA5 expansion equation v1.xmcd"*.

Errors are calculated as the relative difference between the pressures calculated using the ME and the "true" pressure calculated using a sophisticated gas distribution model [1]. The model

uses estimates for the system volumes that are calculated [5] using the measured ratio data and estimates for small vessel volumes from earlier work.[6]

The validity of the model is demonstrated here by considering Figure 10, which shows the modelled errors for operation with v_{1d} operating at a temperature difference of 0.6 K compared with the remaining vessels [7]. This condition was experienced in practice during collection of the data shown in Figure 11 (where the air circulation around v_{1d} was inadvertently restricted). The agreement between model and measurement is extremely close, showing an error in the calculated pressure at 2 Pa of about 0.15%.

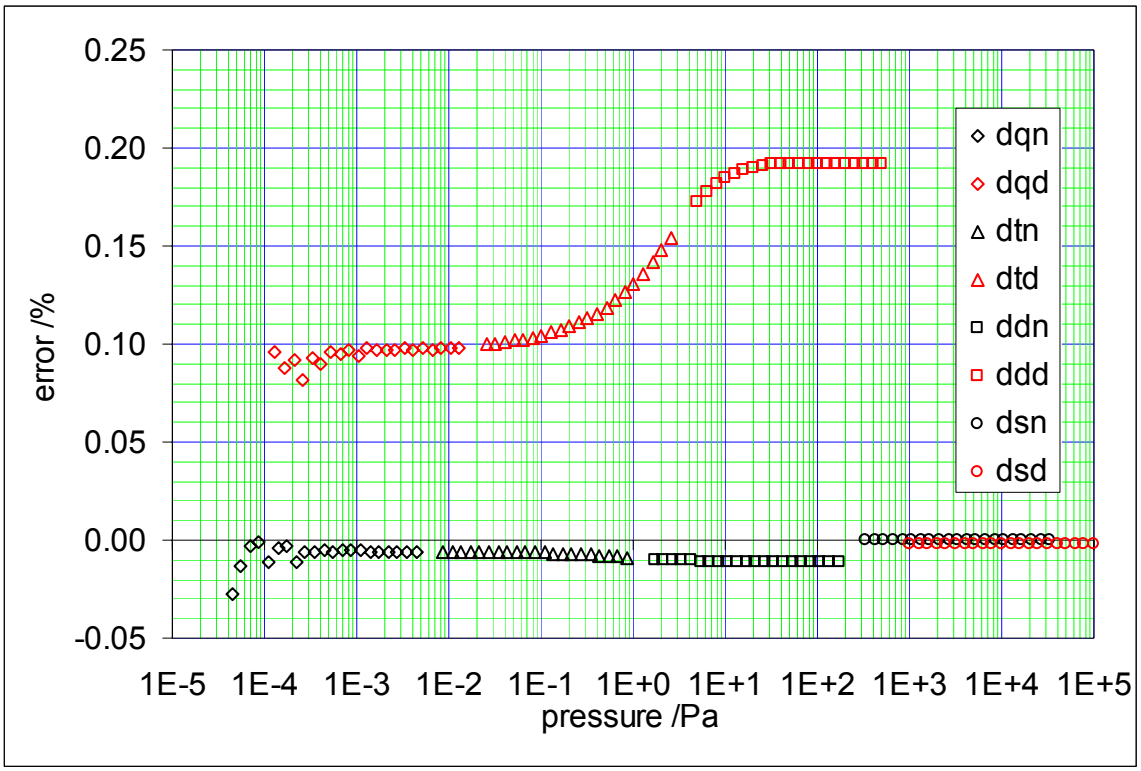

Figure 10. ME errors calculated for SEA5 with ΔT_{1d} = 0.6 K

[5] Worksheet "*measured ratio data*" in workbook "*sea5 uncertainty in expansion ratios.xls*"
[6] sea5 logbook vol.1, p200
[7] The measurement equation takes no account of such intermediate temperature differences.

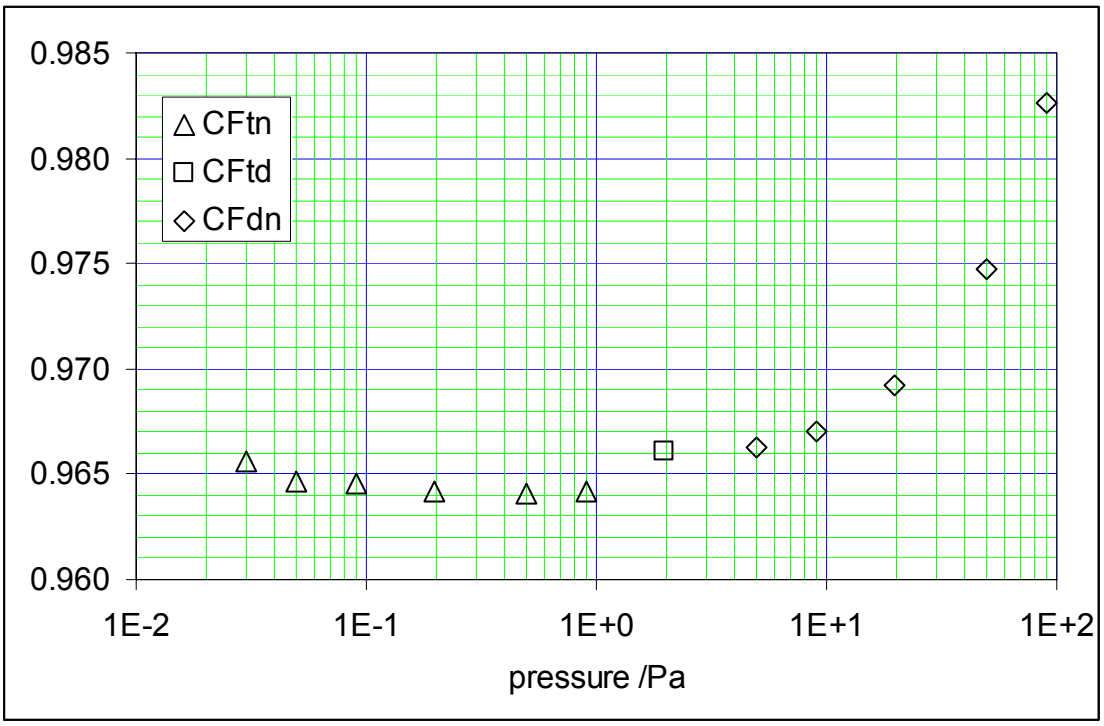

Figure 11. CF data for an SRG calibrated on SEA5 when temperature of v_{1d} was 0.6 K higher than other vessels. (Data are average of 12 measurements except lowest point, which is average of 18 points. See "*0602 SEA5 SRG summary.xls*")

There are several ways in which this type of result could be used: a correction could be applied to the measured data – a process that would be applied on a case by case basis; alternatively a "typical" temperature distribution could be used to determine corrections; or the errors could remain uncorrected and an uncertainty envelope representing the "most probable error" could be calculated.

The first approach is the best from a metrological standpoint, however the resulting uncertainties are likely to be much lower than currently accepted values and further supporting evidence would be required in justification.

The second approach would also require additional evidence before it was accepted, even though the residual uncorrected errors would result in a higher uncertainty than the first approach.

For the above reasons, until further supporting evidence becomes available, the third approach is taken (even though it is the least robust in metrological terms).

Figure 12 shows the ME errors associated with a 0.5 K temperature difference between small and large vessels. Although this distribution appears to be somewhat artificial it still represents realistic temperature differences in a poorly controlled temperature environment. The uncertainty associated with the ME errors is estimated by the envelope of these errors, which is taken to represent the limits of a rectangular distribution.

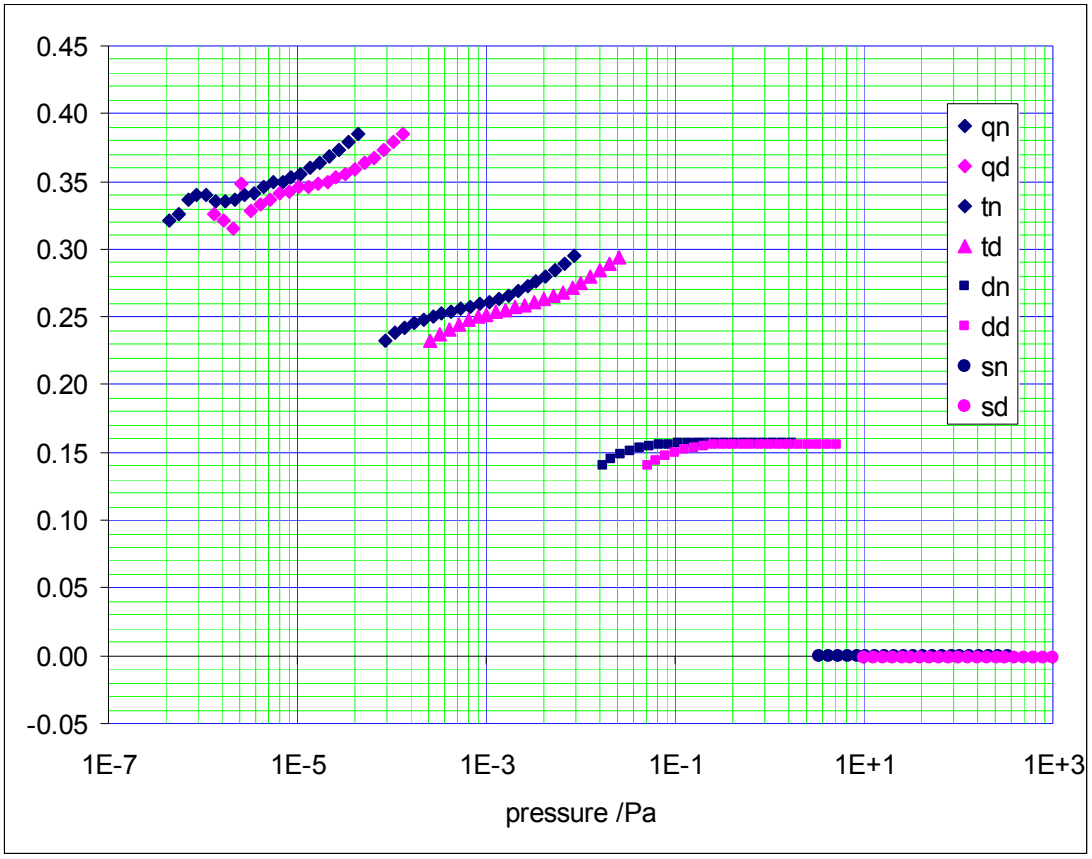

Figure 12. ME errors associated with a 0.5 K temperature difference between small and large vessels, shown as a percentage of the reference pressure.

6.3.3 Uncertainty associated with errors due to adsorption of calibration gas, u_{ads}

When following historical recommendation, the vacuum system is "preconditioned" following bakeout whilst the system IG and UUCs are switched ON. The purpose of the conditioning is to 'occupy the adsorption sites therby reducing adsorption of N_2 by the system. Following this procedure adsorption is expected to be very small and a correction is not made. Historically, in NPL uncertainty budgets a figure of 40 ppm has been used, though there is no reference to where this figure originates[8].

In a simple model, to calculate the number of molecules, n_{eq}, adsorbed onto unit area of a surface in equilibrium conditions we equate rates of adsorption and desorption to give [17]

$$n_{eq} = p^{1/j}\left(\frac{S_{eq}\tau_{eq}}{\left(2\pi\ m\ k_B T\right)^{1/2}}\right)^{1/j} \qquad \text{(A13)}$$

where

p is the pressure of the gas

S is the sticking probability

τ is the mean time of residence

m is the molecular mass

k_B is the Boltzmann constant

T is the thermodynamic temperature of the gas

j is the order of the desorption process

eq indicates that the values correspond to equilibrium conditions

The simplest adsorption model assumes that: $j = 1$; S and τ are independent of n, yielding a linear dependence of n with p (Henry's Law). This model is reasonable at low pressures but not so good at higher pressures since it predicts that, contrary to observed behaviour, there is an ever-increasing adlayer thickness as pressure is increased.

[8] First mention is on p24 of SEA vol.1 1977. No reference is supplied.

A more realistic model requires the use of an appropriate *adsorption isotherm*. The simple model can however be used to calculate an upper limit on the amount of adsorption.

Assuming that $S = 1$, and taking a value[9] of $\tau = 1\times10^{-11}$ s we can calculate the adsorption coefficient, η, to be

Table 9: Estimated adsorption coefficient.

gas	$\eta \times 10^{-11}$ /(Pa^{-1} m^{-2})
N_2	2.9
Ar	2.4
CO_2	2.3

where

$$\eta = \frac{S_{eq}\tau_{eq}}{\left(2\pi\ m\ k_B T\right)^{1/2}} \tag{A14}$$

For any given vessel of volume, V, and surface area, A, containing gas at a pressure, p, we can estimate the proportion of the gas adsorbed on the surface, from

$$n_{ads} = p\ A\eta \tag{A15}$$

and the total amount of gas molecules in the system

$$n_{in} = \frac{p\ V}{k_B T} \tag{A16}$$

hence

$$\frac{n_{ads}}{n_{in}} = \frac{A}{V} k_B T \times \sigma \tag{A17}$$

for expansions using N_2 in SEA5 we can calculate the proportion of gas adsorbed during each expansion stage

[9] Value appropriate for Ar, N_2, CO_2 from Table 10.1 in [17]. See also Steinherz [18] who gives 2×10^{-11} s for N_2 adsorption on Fe.

Table 10: Estimated proportion of N_2 adsorbed on system vessels

Expansion	vessels	V /m^3	A /m^2	$n_{ads}/n_{in} \times 10^6$
E_{1d}	$v_{1d}+V_1$	0.1137	1.3850	0.012
E_{1n}	$v_{1n}+V_1$	0.1129	1.3530	0.011
E_2	$v_2+V_2+ v_{1n}+ v_{1d}$	0.0212	0.5323	0.024
E_3	$v_3+V_3+ v_2$	0.0211	0.4668	0.021
E_4	$v_4+V_4+ v_3$	0.0212	0.4670	0.021

This simple calculation indicates that adsorption effects are virtually zero for SEA5. This is supported by recent data collected from hot filament ionisation gauges.

The data demonstrates that it is the IG itself that is conditioned, not the system, i.e. N_2 is not adsorbed by the system but does affect the IG sensitivity. This is clear from the results of Figure 13 and Figure 14, which show the measured correction factor, *CF*, for a Stabil-Ion gauge having two filaments, calibrated in ascending order of pressure with no system conditioning. The data for the first filament indicate that a change occurs at some point during the first run, '070117' and that the change is completed by the time of the following runs '070118' and '070119'. The key point is that the *same* behaviour is observed for the second filament. If the behaviour had been caused by conditioning of the vacuum system rather than the filament then no difference would be expected between the runs for the second filament.

For this reason no term due to adsorption will be included in the uncertainty budget.

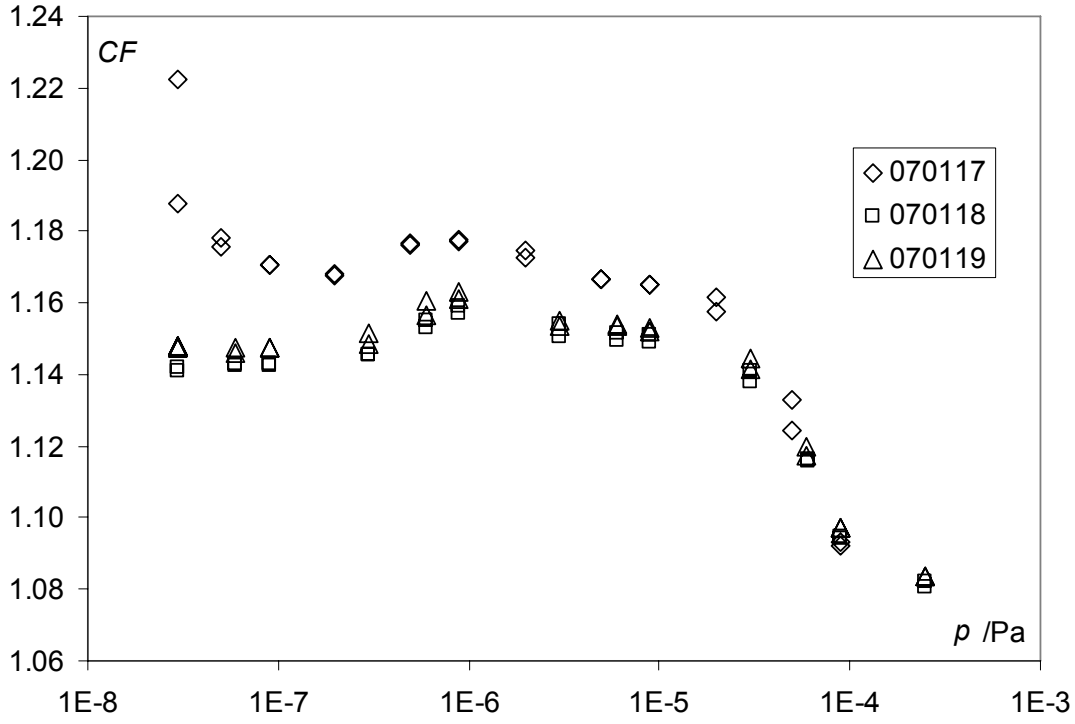

Figure 13: IG correction factor for first filament of a Stabil-Ion gauge, measured on three separate days with no "conditioning" of the system.

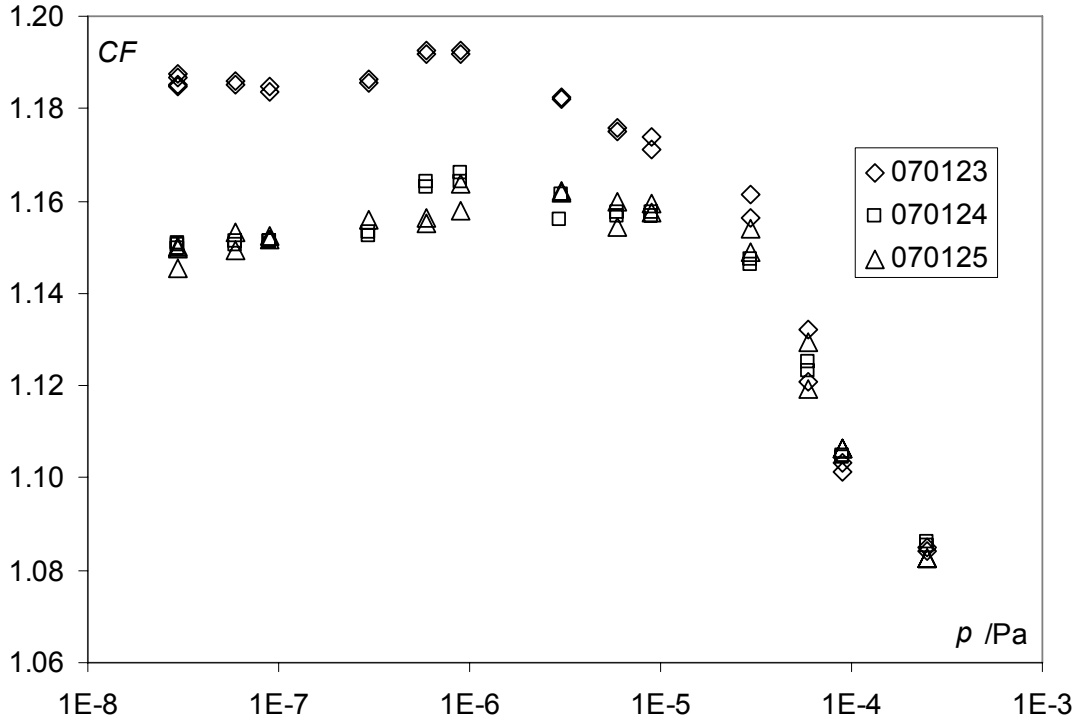

Figure 14: IG correction factor for second filament of a Stabil-Ion gauge, measured on three separate days with no "conditioning" of the system. Measurements follow those of first filament.

6.3.4 Uncertainty associated with residual pressure, u_{res}

For *CF*-type calibrations the residual pressure is not required.[10]

For Δ_{cal} –type calibrations requiring the uncertainty in the *total system pressure* a contribution of $u_{res} = 0.25\ p_{resm}$ is applied. This is based on the estimated uncertainty for a measurement made using an IG that has been calibrated at higher pressures and whose characteristics have been extrapolated back down to the residual pressure. The figure is relatively large to account for extrapolation and fitting errors and for the possible additional complication of the IG x-ray limit.

6.3.5 Uncertainty associated with outgassing, u_{og}

The outgassing product that is transported into the system is measured (using the UUC) by performing dummy expansions. These are performed for all types of UUC and all types of calibration.

When computing the *total system pressure* the uncertainty contribution is estimated from the scatter in repeated dummy expansion measurements. An upper estimate of 1×10^{-8} Pa is derived from data in "*0511 sea5 sea2 sea5StabilIon IG summary.xls*"

6.3.6 Uncertainty associated with other effects, u_o

The uncertainty due to random errors in the reference pressure is difficult to isolate since it is only observed in the random repeatability of measurements made by a UUC when is exposed to the pressure change. In this case the random effects associated with the UUC and the measuring system also influence, and probably dominate, the repeatability. In this sense therefore the uncertainty due to random errors of the reference pressure *on its own* is not a particularly meaningful concept. For this reason, no estimate is made. Strictly speaking any statement of the uncertainty in the established pressure change should state that the estimate *excludes* estimates of uncertainty due to short-term repeatability, though this is usually taken

[10] It is an implicit assumption in CF-type calibrations that the change in response of the UUC for a change in reference pressure is independent of the underlying system pressure. In practice, at low reference pressures where the residual pressure represents a significant proportion of the total pressure, all of the UUCs likely to be calibrated on sea5 have an essentially linear response over the range covered by the reference pressure change, hence the assumption is valid. At higher pressures where the response of the UUC may become non-linear the residual pressure is in any case negligible compared with the reference pressure and again the assumption is valid.

to be an implicit condition when single measurements (as opposed to mean values) of a quantity are reported.

6.3.7 Total uncertainty in reference pressure

Taking into consideration all of the above comments, for CF-type calibrations, the total uncertainty in the *change in system pressure* is

$$u_{tot} = \sqrt{u_{MEcalc}{}^2 + u_{ME}{}^2 + u_{og}{}^2} \qquad \text{(A18)}$$

whereas for Δ_{cal}-type calibrations the uncertainty in the absolute system pressure is

$$u_{tot} = \sqrt{u_{MEcalc}{}^2 + u_{ME}{}^2 + u_{res}{}^2 + u_{og}{}^2} \qquad \text{(A19)}$$

In fact the difference between these two equations is negligible except at the lowest of pressures at which *CF*-type calibrations are not usually performed. For this reason only equation (19) is evaluated. For the conditions outlined above, and a residual pressure of 4×10^{-8} Pa, the uncertainty has been evaluated[11] and is summarised in Figure 15.

For ease of statement, a functional fit to the data has been made. The functional fit is of the form $u = (a \times p) + b$; where the coefficients for each pressure range are summarised in Table 11

Table 11. Coefficients of fit to uncertainty in total pressure

Pressure Range, /Pa	*a*	*b* /Pa
$4.4 \times 10^{-7} < p < 1.3 \times 10^{-4}$	0.0024	3.5×10^{-10}
$1.3 \times 10^{-4} < p < 2.6 \times 10^{-2}$	0.00175	0
$2.6 \times 10^{-2} < p < 5.1$	0.0010	0
$5.1 < p < 1000$	0.0002	4×10^{-3}

[11] In excel workbook "*sea5 uncertainty in reference pressure.xls*"

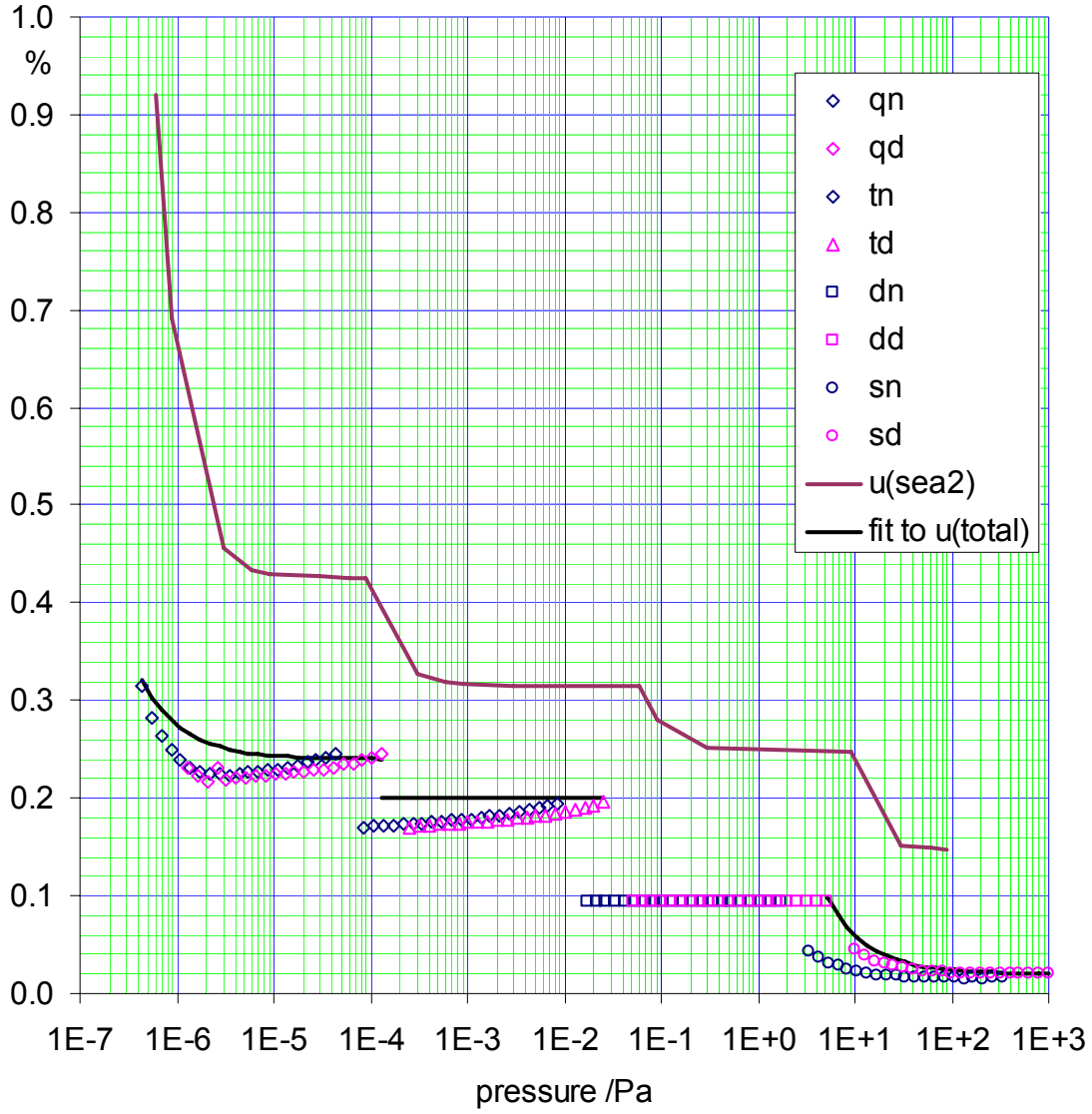

Figure 15. Estimated overall uncertainty (*k*=1) for the various expansion regimes {qn,qd,…,sd} and fit to the envelope of uncertainties. Also shown is uncertainty for sea2.

END