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CENTRE FOR METROLOGY AND ACCREDITATION

Julkaisu J5/2002

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1 INTRODUCTION

In 2001 the Centre for Metrology and Accreditation (MIKES) started a project for expanding its absolute pressure range downwards from 0,2 Pa, using two spinning rotor type vacuum gauges (SRGs) as reference standards.

The Swedish National Testing and Research Institute (SP) has since many years performed calibrations of low vacuum gauges in the range from 0,0005 Pa to 5 Pa with SRGs.

Vaisala Oyj, a well-known Finnish manufacturer of barometers, has a long experience of using SRGs for measuring the reference vacuum of absolute pressure balances in their measurement standards laboratory.

The contacts between the three laboratories are traditionally close. Their co-operation consists of the exchange of information, laboratory visits and joint comparisons. A comparison of spinning rotor gauges in 2001 – 2002 was the latest phase in the co-operation.

2 TRANSFER STANDARD

The transfer standard was a MKS spinning rotor control unit type SRG-2-RS-232, a sensing head type SRG-SH 700 and a metal finger containing a steel bearing ball.

Control unit serial number: 20750G
Sensing head serial number: 921676
Finger serial number: 191241
The ball was not identifiable.

MIKES has been using the instrument since 1997, not as a calibration standard but for controlling the "zero" of capacitance diaphragm vacuum gauges. After the initial calibration by the manufacturer in 1997, the instrument has been calibrated twice at the Swedish national laboratory for pressure, FFA in 1997 [1] and SP in 2000 [2].

Based on the manufacturer's information and the FFA and SP calibrations the following data inputs in the control unit were used in the comparison (nitrogen was used as a medium):

	Data set "A"	**Data set "D"**
Pressure range	$p < 1$ Pa	1 Pa< p < 10 Pa
Ball diameter:	4,50 mm	4,50 mm
Ball density:	7,70 gcm^{-3}	7,70 gcm^{-3}
Molecular weight:	28,013 g mol^{-1}	28,013 g mol^{-1}
Viscosity:	0	175,69 μpois
Accommodation factor:	0,9835	0,99

For each measurement the Offset was determined based on the residual drag and the temperature of the vessel was entered into the control unit.

3 MEASUREMENTS WITH THE VAISALA STANDARD

3.1 Equipment and traceability

The Vaisala standard used in the comparison was a MKS spinning rotor control unit type SRG-2-RS-232, a sensing head type SRG-SH 700 and a metal finger containing a steel bearing ball.

Control unit serial number: 20491G
Sensing head serial number: 91794G
Finger serial number: 90768
The ball was not identifiable.

The traceability for this working standard is obtained by an annual calibration against another MKS SRG-2 Spinning Rotor Gage (the Primary Vacuum Standard) of the Vaisala Measurement Standards laboratory. The latest calibration was made on 31.8.2001 [3].

The estimated uncertainty of the Vaisala working standard is between 0,005 Pa to 0,12 Pa (3,5 to 2,8 % of reading) in range from 0,3 Pa to 8,5 Pa. The uncertainty can be presented as 0,0016 Pa + 0,028 x $\boldsymbol{p}$ (nitrogen N_2 as a medium, coverage factor k=2)

The dominating uncertainty components are the instability of the accommodation coefficient, the uncertainty of the gas viscosity due to the impurities of the gas and the non-linearity in the upper part of the spinning rotor operating range.

The Vaisala Primary Vacuum Standard is traceable to the National Institute of Standards and Technology (NIST, USA). The calibration interval is 36 months. The latest calibration was made on December 27, 1999 using the NIST Primary Vacuum Standard. The medium was nitrogen. The uncertainty given for the Vaisala unit in the NIST certificate was between 0,3 to 0,1 % of reading in the range from 0,3 Pa to 8,5 Pa (k=2).

3.2 Measurements

The measurements were made at the pressure laboratory of MIKES on 12.9.2001. For both SRGs, the fingers containing the ball were mounted horizontally on the calibration vessel and the sensing heads were mounted onto the finger assembly in a vertical position. The systems were left operating overnight before the calibration with the vessel pumped at a very low pressure.

The measurements were made in the pressure range from 0,5 Pa to 5 Pa by Sari Semenoja. The calibration was undertaken using nitrogen gas. The temperature of the calibration vessel was 21,6 °C ± 1,0 °C.

The following data set were used for the Vaisala SRG:

Ball diameter:	4,50 mm	4,493 mm
Ball density:	7,70 gcm^{-3}	7,78 gcm^{-3}
Molecular weight:	28,013 g mol^{-1}	28,013 g mol^{-1}
Viscosity:	178,6 μpois	178,6 μpois
Accommodation factor:	0,99	0,965

The measurement time for the transfer standard was 10 seconds, and 3 seconds for the Vaisala standard.

At each nominal pressure six readings were recorded, three in ascending and three in descending pressure order. The results are given as averages in Tables 1 and 2 with the code V.

4 MEASUREMENTS WITH THE SP STANDARD

4.1 Equipment and traceability

The SP standard used in this comparison consists of a MKS spinning rotor control unit type SRG-2, a sensing head type SRG-SH 700 and a metal finger containing one bearing ball.

Control unit serial number: 20513G
Sensing head serial number: 91815G
Finger serial number: 90739
The ball was not identifiable.

The traceability of the SP standard is maintained by regular calibrations at the National Physical Laboratory (NPL), Teddington. The latest calibration was performed on the 19th of April in 2001 [4]

The best measurement capability of the SP instrument is estimated as 3 x 10^{-5} Pa + 0,027 x ***p*** in the range from 5 x 10^{-4} Pa to 5 Pa.

4.2 Measurements

The measurements in the range from 5 x 10^{-4} Pa to 5 Pa were made at the pressure laboratory of SP on 22.10.2001 by Viktoria König.

The arrangements were similar to those used in the measurements with the Vaisala standard. The systems were left operating overnight before the calibration at a very low pressure. The calibration was undertaken using nitrogen gas. The temperature of the calibration vessel was 21,4 °C ± 1,0 °C. Reference pressures were generated three times in succession, in ascending pressure order.

The settings of SP's SRG were:

Data set:	"A"
Ball diameter:	4,50 mm
Ball density:	7,7 gcm^{-3}
Molecular weight:	28,013 g mol^{-1}
Viscosity:	175,69 μpois
Accommodation factor:	0,985

The settings of MIKES transfer SRG are given at point 2.

The measurements were taken with following sampling intervals:

Pressure (mbar):	Sampling interval (s):
$< 5 * 10^{-3}$	30
$5 * 10^{-3} – 5 * 10^{-2}$	20

At each pressure the gauge reading was printed five times. The results, as mean values are shown in Tables 1 and 2 with the code SP.

5 MEASUREMENTS WITH THE MIKES STANDARD

5.1 Equipment and traceability

The MIKES standard used in the measurements consists of the following items: a MKS spinning rotor control unit type SRG-2, a sensing head type SRG-SH 700 and a metal finger containing one bearing ball.

Control unit serial number: 05000821
Sensing head serial number: 94097G
Finger serial number: 19143
The ball was not identifiable.

The MIKES standard was calibrated at the National Physical Laboratory (NPL), Teddington, on 19th of December 2001 in the range from 3 x 10^{-4} Pa to 3 Pa using nitrogen as medium [5].

The measurement uncertainty given in the NPL certificate was 6 x 10^{-5} Pa + 0,006 x $\boldsymbol{p}$. This was the first calibration for this spinning rotor gauge of MIKES.

The best measurement capability of the MIKES instrument is estimated as 3 x 10^{-5} Pa + 0,03 x $\boldsymbol{p}$ in the range from 5 x 10^{-4} Pa to 5 Pa.

5.2 First measurements M1 in January 2002

The first measurements with the MIKES standard were made in the range 1 x 10^{-2} Pa to 5 Pa in the pressure laboratory of MIKES on 23.1.2002 by Sari Semenoja.

The arrangements were again similar to those used in the measurements with the Vaisala standard. The systems were left operating overnight before the calibration at a very low pressure. The calibration was undertaken using nitrogen gas. The temperature of the calibration vessel was 21,5 °C ± 1,0 °C. Reference pressures were generated three times, two in ascending and one in descending pressure order.

The settings used for the MIKES standard were the following:

Ball diameter:	4,50 mm
Ball density:	7,7 gcm^{-3}
Molecular weight:	28,016 g mol^{-1}
Viscosity:	17,63 μPa s
Accommodation factor:	0,993

The settings for the transfer standard are given at point 2.

At each pressure the gauge reading was recorded two or three times. The results, as mean values, are shown in Tables 1 and 2 with the code M1.

5.3 Second measurements M2 in April 2002

The second measurements with the MIKES standard were made on 30.4.2002 after getting more experience in the measurements in vacuum range, and after some modifications in the pressure control system. The measurements were made in the range 5 x 10^{-4} Pa to 5 Pa by Markku Rantanen and Sari Semenoja.

The arrangements and the settings were the same as in the measurement M1. The temperature of the calibration vessel was 22,2 °C ± 1,0 °C. The reference pressures were generated three times in succession in ascending pressure order. At each pressure the gauge reading was recorded four or five times. The results, as mean values, are shown in Tables 1 and 2 with the code M2.

6 RESULTS

A summary of results is shown in Tables 1 and 2 and in Figure 1. In Table 1 the results, as deviations from the transfer standard, are given in pascals as well as the uncertainties. In Table 2 and Figure 1 the results are given in percentages of the reading.

A tool often used in analysing results from interlaboratory comparisons is the normalised error E_n, which takes into account both the result and its uncertainty. The normalised error E_n is calculated as

$$E_n = \frac{(p_{transfer} - p_{std})_{Lab} - (p_{transfer} - p_{std})_{Ref}}{\sqrt{(U_{Lab}^2 + U_{Ref}^2)}}$$

where

$p_{transfer}$ is pressure indicated by the transfer standard,

p_{std} is the pressure of the laboratory standard,

U_{Lab}	is the uncertainty of the laboratory result, and
U_{Ref}	is the uncertainty of the reference value.

Table 1. Summary of results. The deviations and uncertainties are given in pascals.
V = Vaisala 12.9.01, SP = SP 22.10.01, M1 = MIKES 23.1.02, M2 = MIKES 30.4.02

Nominal pressure		Deviation from transfer standard	Deviation from ref.	Unc. k=2
Pa		Pa	Pa	Pa
	ref	0,000008	0,000000	0,000057
0,0005	SP	0,000024	0,000015	0,000074
	M2	-0,000007	-0,000015	0,000058
0,001	ref	0,000006	0,000000	0,00011
	SP	0,000035	0,000294	0,00006
	M2	-0,000023	-0,000294	0,00006
0,005	ref	-0,000019	0,000000	0,00027
	SP	0,000052	0,000071	0,00017
	M2	-0,000090	-0,000071	0,00019
0,01	ref	-0,000051	0,000000	0,00044
	SP	0,000066	0,000117	0,00030
	M1	0,000463	0,000514	0,00038
	M2	-0,000168	-0,000117	0,00033
0,05	ref	-0,00027	0,00000	0,00184
	SP	0,00022	0,00049	0,00138
	M1	0,00012	0,00039	0,00173
	M2	-0,00075	-0,00049	0,00153
0,1	ref	-0,00047	0,00000	0,0037
	SP	0,00051	0,00098	0,0027
	M1	-0,00070	-0,00024	0,0030
	M2	-0,00144	-0,00098	0,0030
0,5	ref	-0,0001	0,0000	0,0142
	V	-0,0001	0,0000	0,0156
	SP	0,0052	0,0053	0,0136
	M1	-0,0082	-0,0081	0,0151
	M2	-0,0085	-0,0084	0,0151
1	ref	-0,0133	0,0000	0,0070
	V	-0,0133	0,0000	0,0296
	SP	0,0018	0,0151	0,0270
	M1	-0,0168	-0,0035	0,0302
	M2	-0,0183	-0,0050	0,0300
5	ref	-0,074	0,000	0,062
	V	-0,074	0,000	0,142
	SP	0,021	0,095	0,135
	M1	-0,077	-0,003	0,150

M2	-0,055	0,019	0,150

Table 2. Summary of results in percentages of reading and normalised errors.
V = Vaisala 12.9.01, SP = SP 22.10.01,M1 = MIKES 23.1.02, M2 = MIKES 30.4.02

Nominal pressure Pa		Deviation from tr. %	Deviation from ref. %	Unc. k=2 %	**Median as ref. e(n)**	SP result as ref. e(n)
	ref	1,64	0,00	11,45	**0,00**	
0,0005	SP	4,71	3,08	14,81	**0,16**	
	M2	-1,44	-3,08	11,50	**-0,19**	-0,33
0,001	ref	0,60	0,00	10,95	**0,00**	
	SP	3,54	2,94	5,90	**0,24**	
	M2	-2,34	-2,94	6,32	**0,23**	-0,68
0,005	ref	-0,38	0,00	5,32	**0,00**	
	SP	1,04	1,42	3,32	**0,23**	
	M2	-1,80	-1,42	3,73	**-0,22**	-0,56
0,01	ref	-0,51	0,00	4,40	**0,00**	
	SP	0,66	1,17	2,99	**0,22**	
	M1	4,63	5,14	3,77	**0,89**	0,82
	M2	-1,68	-1,17	3,32	**-0,21**	-0,52
0,05	ref	-0,53	0,00	3,67	**0,00**	
	SP	0,44	0,97	2,76	**0,21**	
	M1	0,24	0,77	3,45	**0,15**	-0,04
	M2	-1,50	-0,97	3,06	**-0,20**	-0,46
0,1	ref	-0,47	0,00	3,69	**0,00**	
	SP	0,51	0,98	2,73	**0,21**	
	M1	-0,70	-0,24	3,02	**-0,05**	-0,29
	M2	-1,44	-0,98	3,03	**-0,20**	-0,48
0,5	ref	-0,02	0,00	2,84	**0,00**	
	V	-0,02	0,00	3,12	**0,00**	-0,25
	SP	1,03	1,05	2,71	**0,27**	
	M1	-1,64	-1,62	3,01	**-0,39**	-0,65
	M2	-1,70	-1,68	3,01	**-0,41**	-0,66
1	ref	-1,33	0,00	0,70	**0,00**	
	V	-1,33	0,00	2,96	**0,00**	-0,37
	SP	0,18	1,51	2,70	**0,54**	
	M1	-1,68	-0,35	3,02	**-0,11**	-0,45
	M2	-1,83	-0,50	3,00	**-0,16**	-0,49
5	ref	-1,48	0,00	1,24	**0,00**	
	V	-1,48	0,00	2,83	**0,00**	-0,48
	SP	0,41	1,89	2,70	**0,64**	
	M1	-1,54	-0,06	3,00	**-0,02**	-0,48
	M2	-1,09	0,39	3,00	**0,12**	-0,37

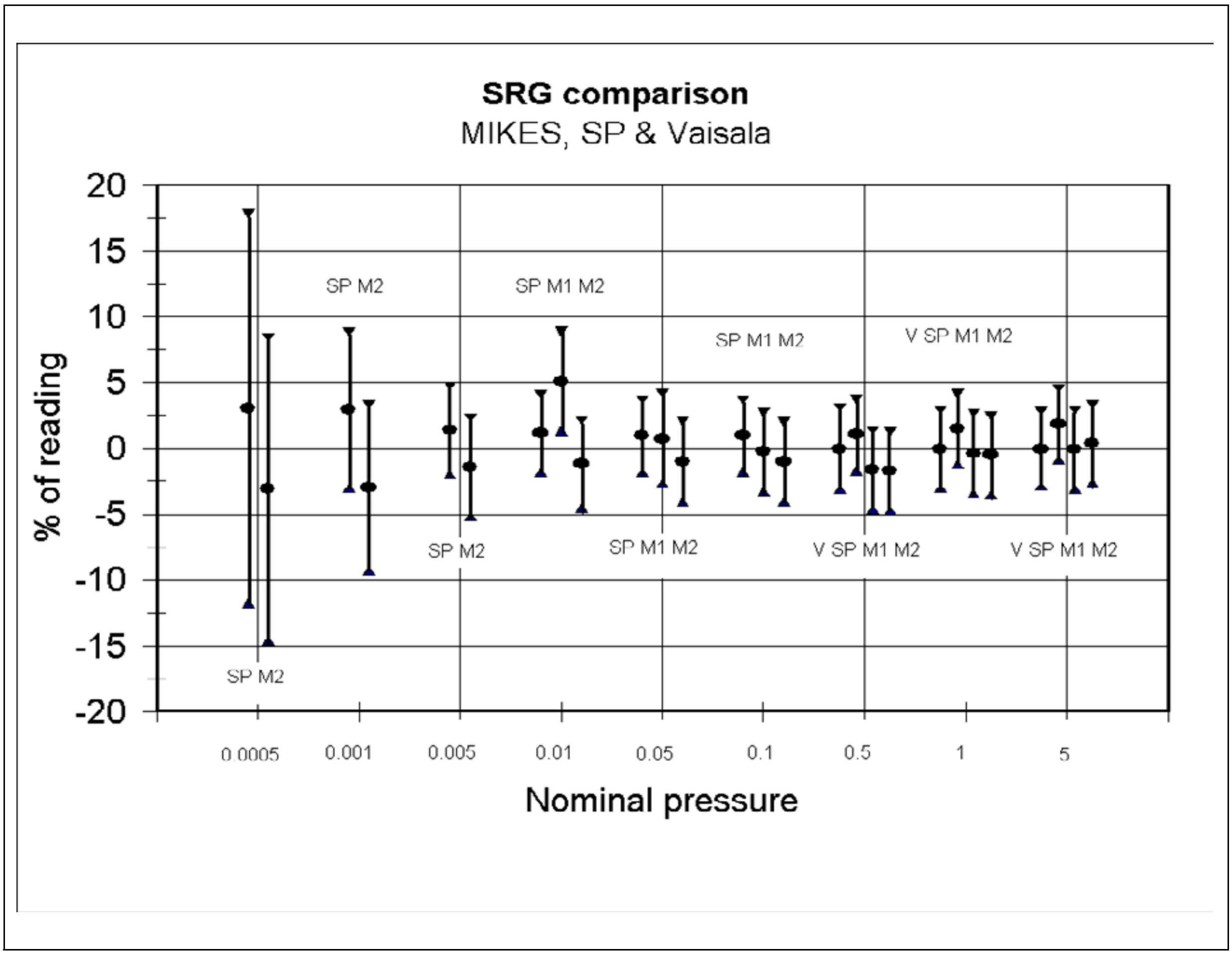

Figure 1. Summary of results in percentages of reading and normalised errors.
V = Vaisala 12.9.01, SP = SP 22.10.01,M1 = MIKES 23.1.02, M2 = MIKES 30.4.02

The E_n-values of all the results are shown in Table 2.

The primary reference value for the comparison was taken as the median from the results of Vaisala, SP and the second measurement of MIKES. The uncertainty of the median has been calculated using the method of Müller as described in the final report on the EUROMET comparison #389 [6]:

$$s = \frac{1{,}858}{\sqrt{(n-1)}} \; x \; MAD$$

where *s* is the uncertainty
n is the number of participants contributing to the reference value
MAD is the median of absolute deviations from the median.

The uncertainty of the median is relatively large when calculated for such a small population as n = 2 or 3. The uncertainty of the reference value was still increased as an allowance of 0,7% (k=2) for the instability of the transfer standard was included. The value 0,7% is based on the duration of the comparison and the manufacturer's specification stating that the stability is better than 1% within one year.

Table 2 shows the deviation from the reference value and the normalised error value E_n for each result. All the absolute values of E_n are well below 1.

As an alternative way of calculating the data the SP results were taken as reference values. Even now all the absolute values of E_n are below 1. Here, too, an allowance of 0,7% (k=2) for the instability of the transfer standard was included in the uncertainty of the reference value.

7 CONCLUSIONS

The results obtained by MIKES, SP and Vaisala are in a good agreement in the range from 0,1 Pa to 5 Pa. Only MIKES and SP made measurements below this range, down to 0,0005 Pa. Their results are equal within the claimed uncertainties. However, both MIKES and SP need another comparison in the EUROMET level to get linked with a key comparison in this pressure range.

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