MIKES
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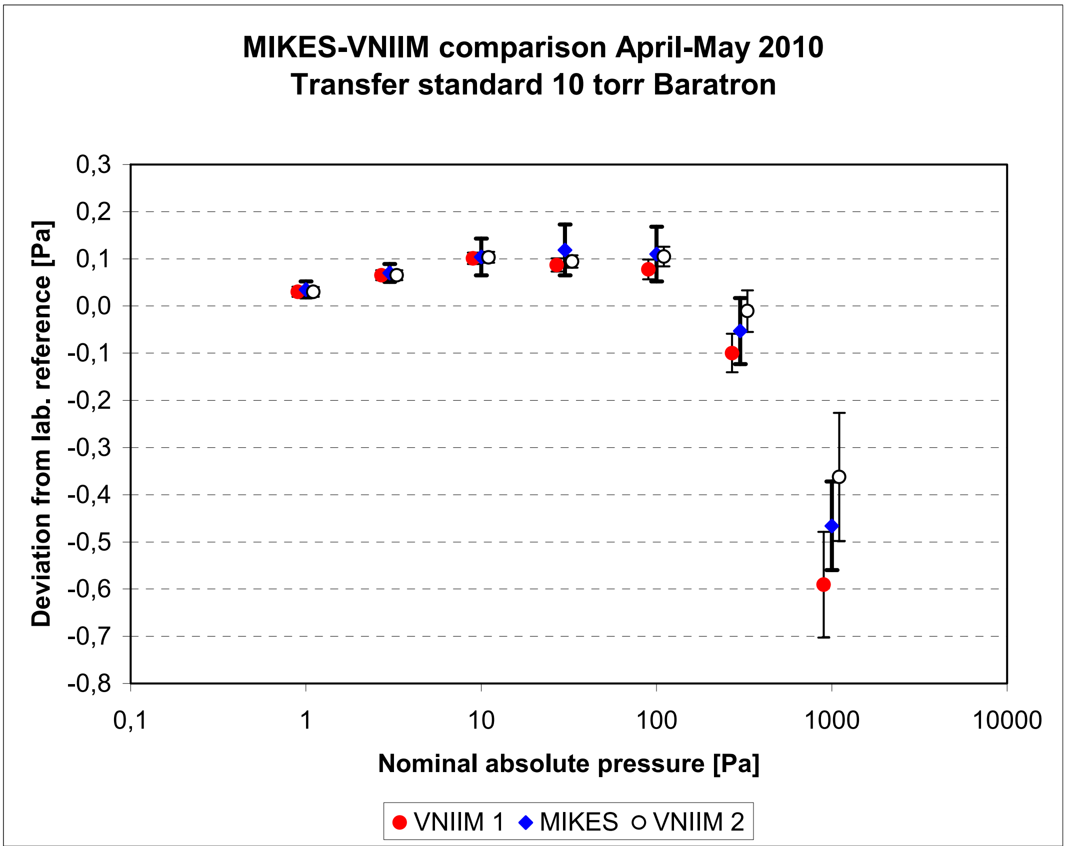

Low pressure comparison between MIKES and VNIIM

Range 1 Pa to 1000 Pa absolute

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Centre for Metrology and Accreditation (MIKES)

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Range 1 Pa to 1000 Pa absolute

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Mittatekniikan keskus
Centre for Metrology and Accreditation

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Abstract

The pressure laboratories of Centre for Metrology and Accreditation (MIKES) and D. I. Mendeleyev All-Russian Institute for Metrology (VNIIM) compared their low absolute pressures in the range 1 Pa to 1000 Pa in April and May 2010.

VNIIM has during the recent years developed a new primary standard for low pressures, a laser interferometric oil manometer (LIOM) which is based on the U-tube principle. At MIKES the standards in the compared pressure range are commercial secondary standards, a 130 Pa capacitance diaphragm gauge traceable to PTB, Berlin, and a piston manometer FPG8601. The standards of MIKES are capable of relatively low uncertainties even though they are secondary ones.

The transfer standard was a capacitance diaphragm gauge MKS Baratron 698A for the range 0 – 1000 Pa differential, equipped with a signal conditioner MKS 670B.

The agreement of the results from MIKES and VNIIM was good.

Contents

1 Introduction

D. I. Mendeleyev All-Russian Institute for Metrology (VNIIM) in St. Petersburg has during the recent years developed a new primary standard for low pressures, a laser interferometric oil manometer (LIOM) which is based on the principle of a U-tube manometer.

In spring 2010 a bilateral comparison between VNIIM and the Centre for Metrology and Accreditation (MIKES) was arranged to test the performance of the LIOM. The pressure range of the comparison was 1 Pa to 1000 Pa. At MIKES the low pressure standards are commercial secondary standards, traceable to PTB, Germany, and LNE, France. The standards of MIKES are capable to relatively low uncertainties even though they are secondary ones. A comparison between different types of standards was found to be very interesting.

2 VNIIM standard

The LIOM standard of VNIIM is based on the principle of a U-tube manometer. The height difference of the oil levels in the two U-tube branches is measured with a laser interferometer. The liquid used is a mineral oil. The pressure range of the instrument is 1 Pa to 1000 Pa and the estimated uncertainty is 0,01 Pa + $1 \cdot 10^{-4} \cdot p$ (k=2). The LIOM can be used also for low gauge pressure measurements.

The principle of LIOM had been described in Reference [1]. Figure 1 illustrates the principle as well as some of its new features. The body of LIOM is now made from stainless steel block with two honed holes. The interferometer is mounted on its lid.

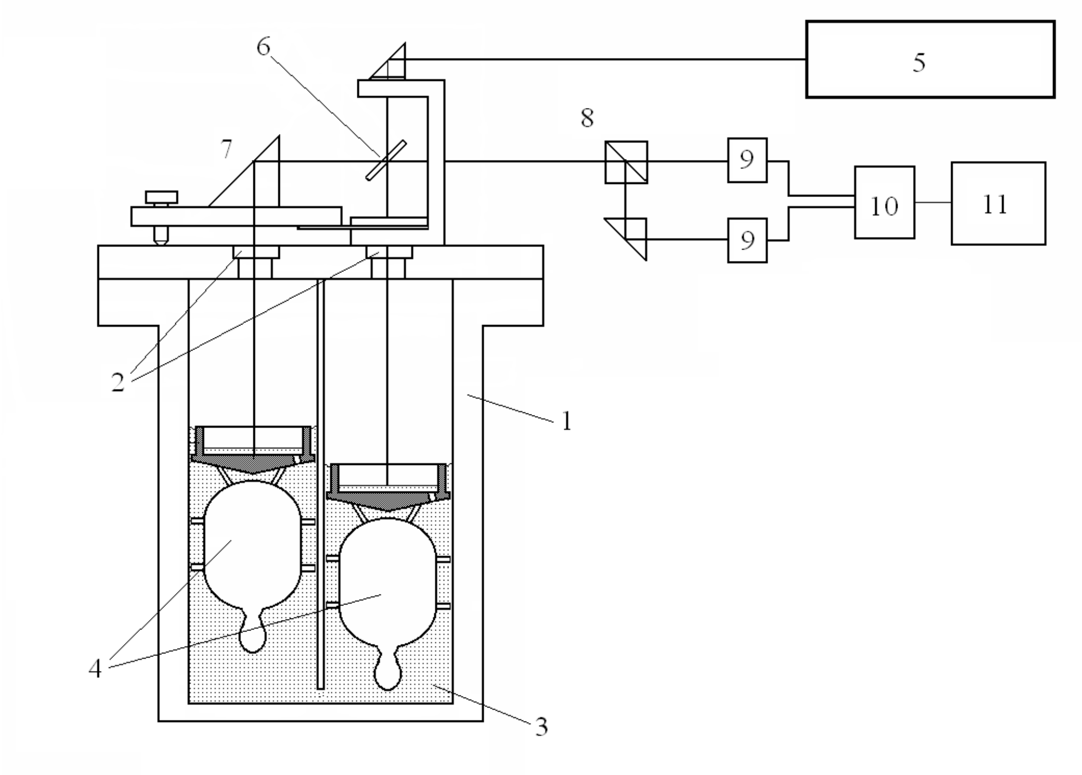

Figure 1. The principle of LIOM: 1 – U-tube; 2 – optical windows; 3 – oil; 4 – floats; 5 – He-Ne laser; 6 – half mirror; 7 – prism; 8 – polarization beam splitter; 9 – photo-diodes; 10 – interface scheme; 11 – computer.

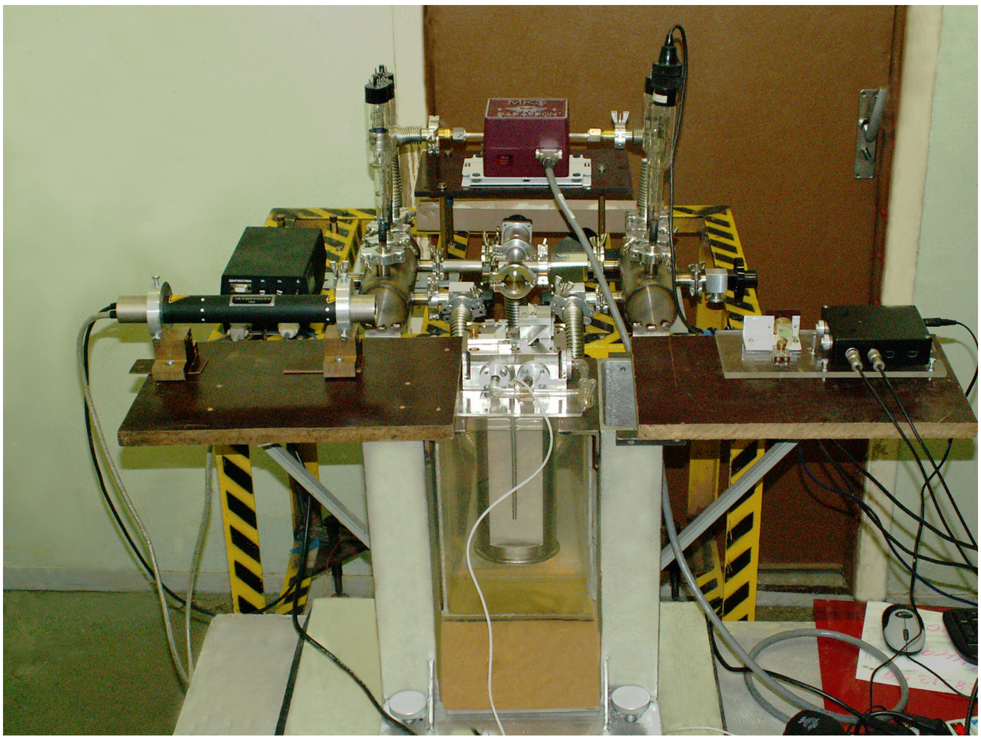

Figure 2. LIOM and the transfer standard.

3 MIKES standards

MIKES uses three different reference standards in the absolute pressure range from 0,5 mPa to 15 kPa. For the range 0,5 mPa to 0,5 Pa the standard is a spinning rotor gauge (SRG), traceable to Physikalisch-Technische Bundesanstalt (PTB), Germany. The uncertainty in the CMC tables is at present (June 2010) $3 \cdot 10^{-5}$ Pa +$\cdot 3 \cdot 10^{-2} \cdot p$.

1 Torr CDG

For the range 0,5 Pa to 20 Pa the best standard at MIKES is a capacitance diaphragm gauge (CDG) MKS Baratron 690 s/n 96018200A for the nominal range 133 Pa (1 torr) with a display unit MKS 270 s/n 932326214A. The instrument is calibrated once a year at PTB, Berlin, against their static expansion system. The CMC uncertainty is 0,013 Pa + $3 \cdot 10^{-3} \cdot p$.

The latest re-calibration on the MIKES CDG standard was carried out on January 28th 2010 [2]. Figures 3 and 4 illustrate the stability of the instrument at nominal pressures 5 Pa and 9 Pa since 2002. The results show a slow and predictable drift.

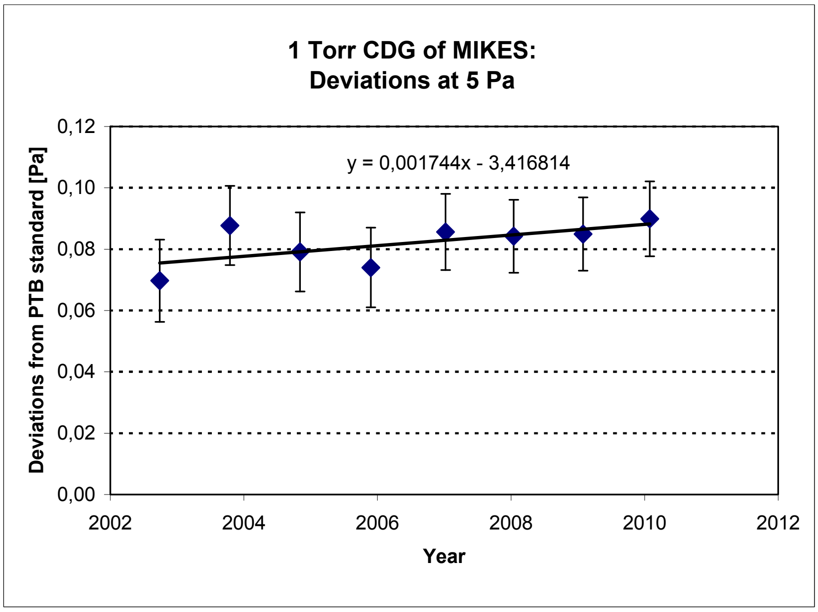

Figure 3. The stability of MIKES 1 Torr CDG at 9 Pa. Calibrations at PTB, Berlin.

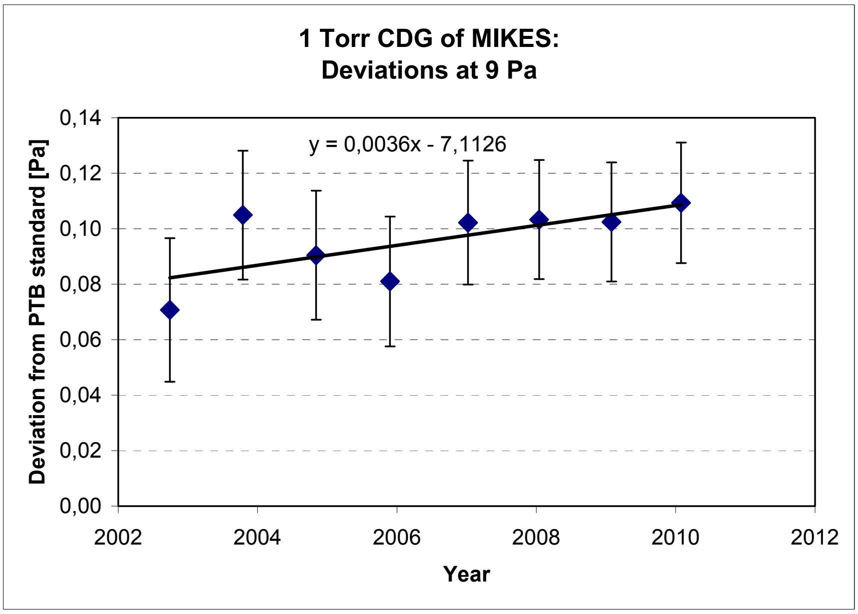

Figure 4. The stability of MIKES 1 Torr CDG at 5 Pa. Calibrations at PTB, Berlin.

FPG type piston manometer

For the range 20 Pa to 15 kPa the reference standard of MIKES is a DHI FPG 8601 type piston manometer s/n 105. The effective area of its piston-cylinder unit s/n 106 is traceable to Laboratoire national de métrologie et d'essais (LNE), France, and to the length laboratory of MIKES. The uncertainty for absolute pressure measurements in the CMC tables is $0{,}07\ \text{Pa} + 4 \cdot 10^{-5} \cdot p$.

The FPG piston manometer has been used at MIKES since 2001. The effective area has been determined once a year and the changes have been negligible. The latest calibration was carried out at MIKES on August 17th 2010 [3].

Comparison of 1 Torr CDG and FPG

The operating ranges of the 1 Torr CDG and FPG overlap which allows a cross-check on the instruments. The FPG is used to calibrate 1 Torr CDG always after receiving it back from the recalibration at PTB: The latest calibration was made on March 19th 2010 [4]. Figure 5 illustrates the results compared to those of PTB. The improvement in the zeroing method allow slightly lower uncertainties for the MIKES results than the figures in the CMC tables in June 2010 (about 0,05 Pa compared to 0,07 Pa).

The uncertainty of the PTB results is proportional to the pressure and the MIKES uncertainties in the range 5 Pa to 100 Pa are practically constant. Around 20 Pa the uncertainties from PTB and MIKES are equal.

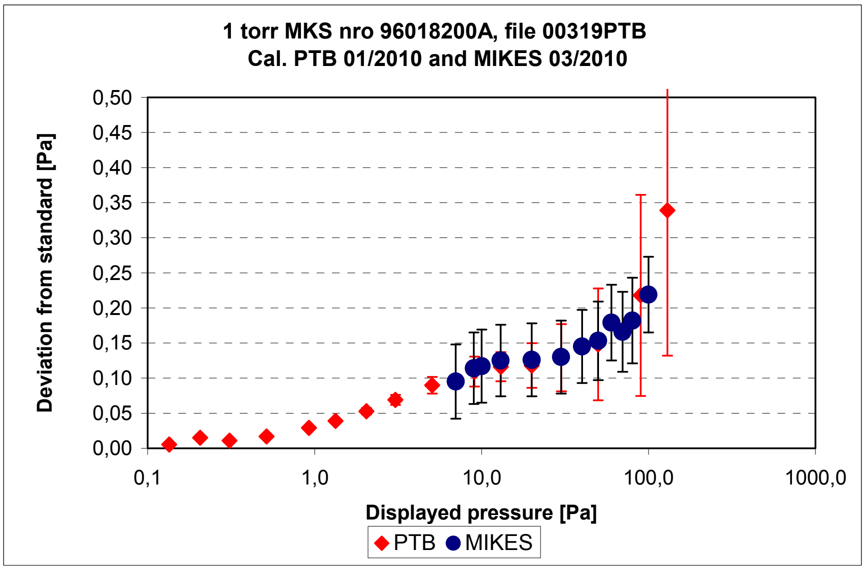

Figure 5. The MIKES and PTB results on the 1 Torr CDG. [2, 4]

4 Uncertainties of LIOM / VNIIM, MIKES and some other laboratories

The estimated uncertainty of the LIOM of VNIIM and the CMC uncertainties of MIKES and some other EURAMET laboratories are illustrated in Figure 6.

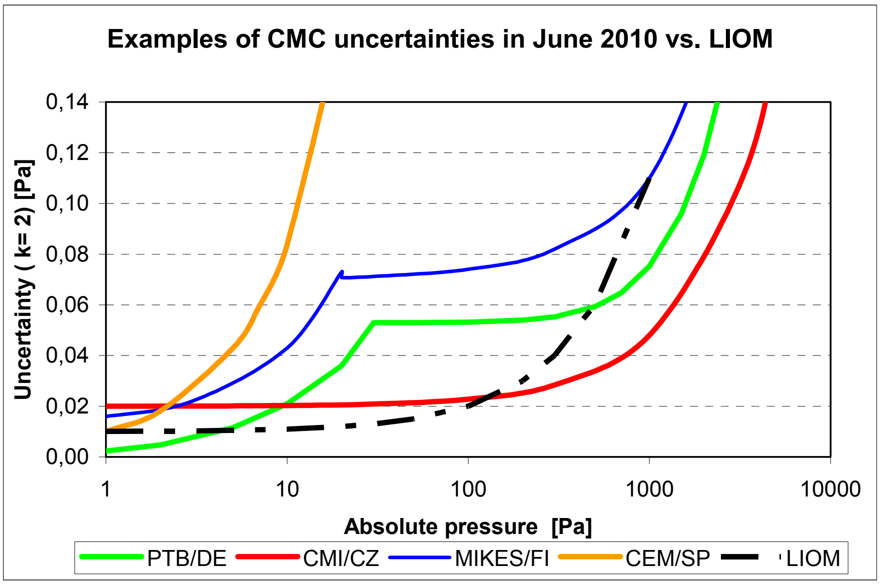

Figure 6. The estimated uncertainty (k= 2) of LIOM compared to the CMC uncertainties of some EURAMET laboratories (Source: www.bipm.org, June 2010).

The estimated uncertainty of the LIOM is low, indicating the potential of instruments based on the oil U-tube principle.

5 Transfer standard

The transfer standard in the comparison was a capacitance diaphragm gauge MKS Baratron 698A11TRA s/n 016511603 for the range 0 – 1000 Pa differential, equipped with a display unit MKS 670B s/n 016510819. The pressure readings can be recorded from the display or via serial interface RS-232.

The transfer standard was provided by VNIIM. The instrument was purchased in August 2009. Its stability was not investigated systematically. For the period from September 2009 to May 2010 its repeatability was better than 0,03% at the pressure 1000 Pa.

6 Measurement instructions

The measurement instructions prepared for the comparison are attached as Appendix 1.

For measurements in absolute mode the reference port of the transfer standard was pumped to a pressure lower than 0,005 Pa.

The task of the participants was to determine the deviation of the transfer standard reading at seven nominal pressures, 1 Pa, 3 Pa, 10 Pa, 30 Pa, 100 Pa, 300 Pa and 1000 Pa. The deviation was determined as

Deviation = Pressure displayed by the transfer standard –
Applied pressure from the laboratory standard

The transmitting gas was specified as dry air.

Five series of measurements were specified, in only the increasing direction of pressure. The nominal pressures 1 Pa, 3 Pa, 10 Pa, 30 Pa, 100 Pa were to be measured with the multiplication setting X=0,1 on the display unit of the transfer standard, and nominal pressures 100 Pa, 300 Pa and 1000 Pa with the setting X1.

The ambient temperature was specified to (21 ± 1)°C to avoid the need of thermal transpiration corrections.

7 Measurements and handling of results

The measurements were carried out twice at VNIIM and once at MIKES according to the following schedule:

VNIIM 1	24.04.2010
MIKES	19.-20.05.2010
VNIIM 2	27.05.2010

The measurements were carried out following the instructions with minor modifications only. At VNIIM the specified two measurement runs, 1 Pa to 100 Pa and 100 Pa to 1000 Pa, were combined by changing the display unit setting manually from X0,1 to X1 at 100 Pa and recording the readings with both settings.

At MIKES, the two specified measurement runs had to be divided into four because of the use of two reference standards as well as the need to use two different settings in the display unit of the MIKES reference CDG. The combinations and pressure ranges are shown in Table 1.

Table 1. MIKES standards, transfer standard settings and pressure ranges.

Transfer standard setting	MIKES standard and setting	Pressure range
X0,1	MKS X0,1	(0,5 Pa) … 1 Pa - 10 Pa
X0,1	MKS X1	10 Pa - 30 Pa
X0,1	FPG	(2,5 Pa) … 30 Pa - 100 Pa
X1	FPG	(2,5 Pa) … 100 Pa - 1000 Pa

Only the result with lowest uncertainty was selected for the comparison when there was more than one result at the same nominal pressure.

Further, nitrogen instead of air was used as the pressurised medium in MIKES measurements. The results of MIKES were presented in the calibration certificate M-10P068. [5]. Calculated uncertainties at some nominal pressures were lower than the values in the CMC tables of MIKES, due to an improved method for zeroing the reference standards.

Both laboratories sent their results to Fredrik Arrhén, SP, Sweden. The laboratories did not show their results to each other before all results of the three measurement sets were delivered.

8 Results

The results obtained at VNIIM and MIKES are presented in Table 2. Further, a summary of the results is in Figure 7.

Table 2. Results from VNIIM and MIKES and the normalised error values.

Transf. std. setting	Nom. p Pa	VNIIM 1 Deviation Pa	Unc. of dev. Pa	MIKES Std. & setting	Deviation Pa	Unc. of dev. Pa	VNIIM 1 - MIKES Pa	Normalised error E(n)
X0,1	1	0,030	0,011	MKS X0,1	0,035	0,017	-0,005	-0,24
X0,1	3	0,065	0,011	MKS X0,1	0,070	0,019	-0,005	-0,21
X0,1	10	0,101	0,012	MKS X0,1	0,104	0,039	-0,003	-0,07
X0,1	10	0,101	0,012	*MKS X1*	*0,096*	*0,039*	*0,005*	*0,12*
X0,1	30	0,087	0,013	*MKS X1*	*0,098*	*0,074*	*-0,011*	*-0,15*
X0,1	30	0,087	0,013	FPG	0,119	0,054	-0,032	-0,58
X0,1	100	0,078	0,021	FPG	0,110	0,058	-0,032	-0,53
X1	100	0,004	0,026	FPG	0,019	0,062	-0,015	-0,23
X1	300	-0,100	0,043	FPG	-0,053	0,070	-0,047	-0,57
X1	1000	-0,591	0,112	FPG	-0,466	0,094	-0,125	-0,85

Transf. std. setting	Nom. p Pa	VNIIM 2 Deviation Pa	Unc. of dev. Pa	MIKES Std. & setting	Deviation Pa	Unc. of dev. Pa	VNIIM 2 - MIKES Pa	Normalised error E(n)
X0,1	1	0,030	0,011	MKS X0,1	0,035	0,017	-0,005	-0,25
X0,1	3	0,065	0,011	MKS X0,1	0,070	0,019	-0,005	-0,22
X0,1	10	0,103	0,012	MKS X0,1	0,104	0,039	-0,001	-0,02
X0,1	10	0,103	0,012	*MKS X1*	*0,096*	*0,039*	*0,007*	*0,18*
X0,1	30	0,087	0,013	*MKS X1*	*0,098*	*0,074*	*-0,011*	*-0,15*
X0,1	30	0,094	0,013	FPG	0,119	0,054	-0,025	-0,44
X0,1	100	0,105	0,021	FPG	0,110	0,058	-0,005	-0,08
X1	100	0,040	0,026	FPG	0,019	0,062	0,021	0,32
X1	300	-0,011	0,046	FPG	-0,053	0,070	0,042	0,50
X1	1000	-0,362	0,136	FPG	-0,466	0,094	0,104	0,63

Transf. std. setting	Nom. p Pa	Average VNIIM 1&2 Deviation Pa	Unc. of dev. Pa	MIKES Std. & setting	Deviation Pa	Unc. of dev. Pa	VNIIM 1&2 - MIKES Pa	Normalised error E(n)
X0,1	1	0,030	0,011	MKS X0,1	0,035	0,017	-0,005	-0,24
X0,1	3	0,065	0,011	MKS X0,1	0,070	0,019	-0,005	-0,22
X0,1	10	0,102	0,012	MKS X0,1	0,104	0,039	-0,002	-0,05
X0,1	10	0,102	0,012	*MKS X1*	*0,096*	*0,039*	*0,006*	*0,15*
X0,1	30	0,087	0,013	*MKS X1*	*0,098*	*0,074*	*-0,011*	*-0,15*
X0,1	30	0,091	0,014	FPG	0,119	0,054	-0,028	-0,51
X0,1	100	0,091	0,024	FPG	0,110	0,058	-0,019	-0,30
X1	100	0,022	0,030	FPG	0,019	0,062	0,003	0,05
X1	300	-0,055	0,058	FPG	-0,053	0,070	-0,002	-0,02
X1	1000	-0,477	0,165	FPG	-0,466	0,094	-0,011	-0,06

A tool often used in analysing results from inter-laboratory comparisons is the normalised error E_n, which takes into account both the result and its uncertainty. The normalised error E_n for a pressure comparison 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 the 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.

The results are regarded to be in agreement within the limits of uncertainty if the absolute value of the normalised error E_n is smaller than 1.

The average of the two results from VNIIM was compared to the result from MIKES at each nominal pressure. The differences between the two VNIIM result sets were included in the uncertainties of the average values.

The normalised error values E_n for the results are shown in Table 2. For all E_n values

$$|E_n| < 1.$$

Even for separate comparisons VNIIM 1 vs. MIKES and VNIIM 2 vs. MIKES all normalised error E_n values are within the limits $-1 < E_n < +1$ indicating a good agreement.

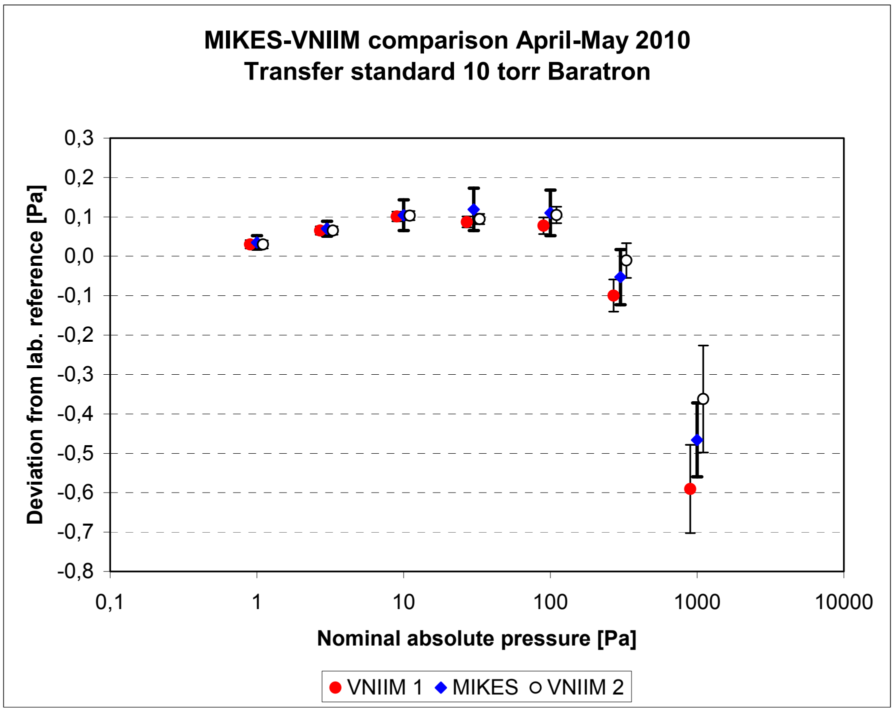

Figure 7. Summary of the results.

9 Conclusions

The agreement of the results from VNIIM and MIKES is good.

It is also worth noting that this comparison involved pressures derived with three different primary methods. The pressures realised by the static expansion system of PTB, by the U – tube manometer of VNIIM and by the pressure balance of MIKES were in a good agreement.

The transfer standard was not as stable as the reference standard of MIKES, made by the same manufacturer and based on the same technology.

10 Acknowledgement

The authors wish to thank Mr. Fredrik Arrhén of SP, Sweden, for his advice on drafting the measurement instructions and for collecting the results.

11 References

[1] Sadkovskaya I.V., Eichwald A.I. J.Phys: Conference Series 100 (2008) 092006.

[2] Kalibrierschein (Calibration Certificate) 70133 PTB 10. Physikalisch-Tech-nische Bundesanstalt, Berlin 2010.

[3] Certificate of Calibration M-09P096. Centre for Metrology and Accreditation, Espoo 2009.

[4] Certificate of Calibration M-10P033. Centre for Metrology and Accreditation, Espoo 2010.

[5] Certificate of Calibration M-10P068. Centre for Metrology and Accreditation, Espoo 2010.

APPENDIX 1

Measurement protocol for a bilateral low pressure comparison
between MIKES and VNIIM
Draft 4, 14.4.2010 MR

1. Introduction

MIKES and VNIIM will compare their low absolute pressure standards. VNIIM has developed a new primary standard for low pressures, a laser interferometric oil manometer (LIOM). The low pressure standards at MIKES are commercial secondary standards, traceable to PTB, Germany, and LNE, France. A comparison between different types of standards will be very interesting.

2. VNIIM standard

The LIOM standard of VNIIM is based on the principle of a U-tube manometer. The height difference of the oil levels in the two U-tube branches is measured with a laser interferometer. The liquid used is mineral oil. The pressure range of the instrument is 1 Pa to 1000 Pa and the estimated uncertainty is $0{,}01 \text{ Pa} + 1 \cdot 10^{-4} \cdot p$ (k=2). The LIOM can be used also for low gauge pressure measurements.

3. MIKES standards

The MIKES standards for absolute pressures below 20 Pa are a spinning rotor gauge (SRG) and a capacitance diaphragm gauge (CDG), traceable to PTB, Germany. The uncertainty for the SRG range 0,5 mPa to 0,5 Pa is $3 \cdot 10^{-5}$ Pa +$\cdot 3 \cdot 10^{-2} \cdot p$ in the BIPM CMC tables at present (February 2010). For the CDG range 0,5 Pa to 20 Pa the uncertainty is $0{,}013 \text{ Pa} + 3 \cdot 10^{-3} \cdot p$.

For the range 20 Pa to 15 kPa the standard of MIKES is a FPG type piston manometer. The effective area of the piston-cylinder unit is traceable to LNE, France. The uncertainty in the CMC tables is $0{,}07 \text{ Pa} + 4 \cdot 10^{-5} \cdot p$.

4. Transfer standard

The transfer standard is a capacitance diaphragm gauge MKS Baratron 698A11TRA s/n 016511603 for the range 0 – 1000 Pa differential, equipped with a display unit MKS 670B s/n 016510819. The pressure readings can be recorded from the display or via serial interface RS-232.

The transfer standard is made available by VNIIM. Measurements on the stability of the transfer standard have been carried out since August 2009.

The transfer standard will be hand-carried from VNIIM for the measurements at MIKES.

5. Measurements

The first set of measurements on the transfer standard will be made at VNIIM in April 2010, then MIKES will carry out their measurements in May and finally the comparison will be completed by another set of measurements at VNIIM in June 2010.

Measurement conditions

The measurements will be made in a laboratory room where the ambient temperature is 21°C ± 0,5°C.

Preparation of the transfer standard

The pressurised medium in the measurements is air.

Allow the transfer standard stabilise in the laboratory conditions at least overnight with mains and the heater on, and both pressure ports pumped down to a pressure below 0,005 Pa. Keep the heater on during measurements.

Set “Response” to “400 mSec” and “Averaging” to “50” on the display unit. Check the NUL and FS readings on the display unit and make adjustments if necessary before the actual measurements. NUL must be 0,0000 V ± 0,0002 V and FS 10,0000 V ± 0,0002 V.

Keep the reference port of the transfer standard pumped down and pre-pressurise the pressure port to 1000 Pa two times.

Connect the pressure and reference ports at pressure below 0,005 Pa and maintain the pressure for about 10 – 15 minutes. Adjust the ZERO on the display unit (Do not touch the adjustments of the CDG.) Record pressure and SYSCK reading.

Measurements

Record the transfer standard reading and the reading of the reference standard at nominal pressures 1 Pa, 3 Pa, 10 Pa, 30 Pa, 100 Pa, 300 Pa and 1000 Pa (nominal pressures of your reference standard). Use display unit setting X0.1 at nominal pressures 1 Pa, 3 Pa, 10 Pa, 30 Pa and 100 Pa (the first run) and X1 at pressures 100 Pa, 300 Pa and 1000 Pa (the second run). Record also the pressure at the reference port of the transfer standard At each nominal pressure.

Repeat both runs of the measurements five times (in increasing direction of pressure only).

Pump the transfer standard down to a pressure below 0,005 Pa for about 10 – 15 minutes and record the pressure reading of the transfer standard, and NUL and FS readings.

There is no need for thermal expiration corrections for the transfer standard readings as the heater is on, and the ambient temperature is in specified limits.

6. Results and report

The results of each measurement set (as deviations of the transfer standard reading from the laboratory standard and estimated uncertainties) will be sent within two weeks to Mr. Fredrik Arrhén, SP/Sweden. Only after receiving the results from the three measurement sets he will send them to VNIIM and MIKES. The two participants will not exchange the results with each other earlier.

The results of the comparison will be presented in a joint report.

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