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Comparison in absolute pressure range 0,02 hPa ... 10 hPa between MIKES and Beamex

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Helsinki 2003

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MITTATEKNIIKAN KESKUS
CENTRE FOR METROLOGY AND ACCREDITATION

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ABSTRACT

A comparison P15 in the absolute pressure range 0,02 hPa … 10 hPa was arranged in 2003 between the Centre for Metrology and Accreditation (MIKES) and the accredited pressure calibration laboratory K026 of Oy Beamex Ab. The transfer standard was a 10 torr capacitance diaphragm gauge by CCM Instruments.

All the results of the Beamex laboratory were in a good agreement with the results of MIKES.

TIIVISTELMÄ

Mittatekniikan keskus (MIKES) järjesti keväällä 2003 vertailumittauksen P15 Oy Beamex Ab:n akkreditoidun kalibrointilaboratorion K026 kanssa absoluuttipainealueella 0,02 hPa … 10 hPa . Vertailulaitteena oli CCM Instrumentsin valmistama kapasitiivinen 10 torr vakuumimittari.

Kaikki Beamexin laboratorion saamat paineen mittaustulokset olivat mittausepävarmuuksien puitteissa samoja kuin MIKESin tulokset.

CONTENTS

COMPARISON IN ABSOLUTE PRESSURE RANGE 0,02 hPa … 10 hPa BETWEEN MIKES AND OY BEAMEX AB

1 INTRODUCTION

At present, the calibration laboratory K026 of Oy Beamex Ab is the only one in Finland accredited for absolute pressures below 5 kPa. In early Summer 2003 a comparison P15 was arranged between the Centre for Metrology and Accreditation (MIKES) and the Beamex laboratory in the absolute pressure range 0,02 hPa … 10 hPa.

2 TRANSFER STANDARD

The transfer standard was a CCM Instruments 10 torr capacitance diaphragm gauge CCMH-10, serial number 4H10171, equipped with a display unit LPC-501, serial number 4S0116. Its resolution is 0,001 mbar (=hPa) when this unit is selected for the display. The instrument has been used since 1994 as a working standard in the pressure laboratory of MIKES. The stability of the gauge is good except for some tendency to the zero drift.

3 VACUUM STANDARDS OF MIKES

MIKES is a participant in the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). The recognised calibration and measurement capabilities are shown in Appendix C of the MRA (for details see http://www.bipm.org).

In 2003 the following uncertainties are given in the Appendix C for the vacuum range of MIKES:

Range	Uncertainty (k = 2)
0,2 Pa ... 10 Pa	0,04 Pa + 0,01 · p (p is pressure)
10 Pa ... 1000 Pa	0,08 Pa + 0,006 · p
1000 Pa ... 120 kPa	1 Pa + 3,5 · 10 - 5 · p

In the range from 0,2 Pa to 1000 Pa the values are based on two capacitance diaphragm gauges traceable to the accredited calibration laboratory of MKS Germany. In the range from 1000 Pa to 120 Pa a digital piston manometer is used. It is traceable to the French national standards at BNM-LNE.

During the last years MIKES was working for lower pressures and smaller uncertainties, and new standards were purchased. An updating process of the ranges and uncertainties is going on.

4
VACUUM STANDARDS OF BEAMEX

The reference standards of Beamex for low absolute pressures are two capacitance diaphragm gauges (maximum pressures 1 torr and 100 torr).They are traceable to MIKES.

The accredited uncertainties for low absolute pressures are as follows:

Range	Uncertainty (k = 2)
0,13 Pa ... 130 Pa	0,12 Pa + 0,01 · p (p is pressure)
130 Pa ... 1000 Pa	0,7 Pa + 0,007 · p
1 kPa ... 10 kPa	1,6 Pa + 0,0003 · p

5
MEASUREMENT PROCEDURES

The transfer standard was allowed to stabilise for at least 10 hours power switched on and pumped to a pressure below 1 hPa. Immediately before starting the measurements the transfer standard reading was adjusted to zero at a pressure below 0,01 Pa.

The measurements were made using nine nominal pressures: 0,02 hPa, 0,05 hPa, 0,1 hPa, 0,2 hPa, 0,5 hPa, 1 hPa, 2 hPa, 5 hPa and 10 hPa. The measurements were made using both increasing and decreasing pressures. Two up-and-down cycles were performed at MIKES and at Beamex three.

The medium was nitrogen.

Three calibrations were made at MIKES:

MIKES 1 = **M1** 9.5.2003
MIKES 2 = **M2** 3.6.2003
MIKES 3 = **M3** 10.7.2003

The calibration at Beamex laboratory was made on 27.5.2003.

6 REFERENCE VALUES

The averages of the three MIKES calibrations were selected as reference values for the comparison.

Figure1 shows all the result points of MIKES calibrations as deviations from the straight line fitted to the data of the first calibration at MIKES (= M1) using the method of least squares. There is some drift in the results but the instability of the transfer standard was negligible compared to the measurement uncertainties.

The major uncertainty components taken into account were the following:

- uncertainty in setting the zero of the transfer standard
- resolution of the transfer standard
- scatter of the results
- uncertainty of the measurement standard.

According to the manufacturer the effects of calibration temperature can be neglected in a typical laboratory environment.

7 RESULTS

Both laboratories presented their results as calibration certificates and calculated the uncertainty of calibration using the document EA-4/02 and a coverage factor k = 2.

A summary of all the result is shown in Appendix 1. The figures 2 and 3 show the results.

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 is calculated as

$$E_n = \frac{\left(p_{transfer} - p_{std}\right)_{lab} - \left(p_{transfer} - p_{std}\right)_{ref}}{\sqrt{\left(U_{lab}^{\,2} + U_{ref}^{\,2}\right)}}$$

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.

The E_n-values of all the results are shown in Appendix 1.

A summary of the E_n-values is in the following:

Laboratory	Range of E_n-values
Beamex	-0,86 … -0,02
MIKES 1	-0,05 … 0,28
MIKES 2	-0,22 … 0,00
MIKES 3	-0,19 … 0,17

The result in an inter-laboratory comparison is regarded as correct within the limits of uncertainty, if the absolute value of the normalised error E_n is less than 1.

In this case the E_n-values for all the results are between -1 and +1.

The absolute E_n-values for Beamex results are higher at low nominal pressures. There seems to be a small, practically constant offset in the Beamex results. This feature may be an indication of a zero drift.

8 CONCLUSIONS

All the results from the Beamex laboratory were in a good agreement with the results from MIKES.

The transfer standard, a CCM Instruments capacitance diaphragm gauge was found suitable for a comparison simulating a routine calibration job.

9 REFERENCES

EA-4/02: Expression of the Uncertainty of Measurement in Calibration

ISO/IEC 17025: General requirements for the testing and calibration laboratories.

Certificate of Calibration K026-03P514. Oy Beamex Ab Calibration Laboratory.

Certificates of Calibration M-03P062, M-03P071 and M-03P093. Centre for Metrology and Accreditation

MR 29.10.2003

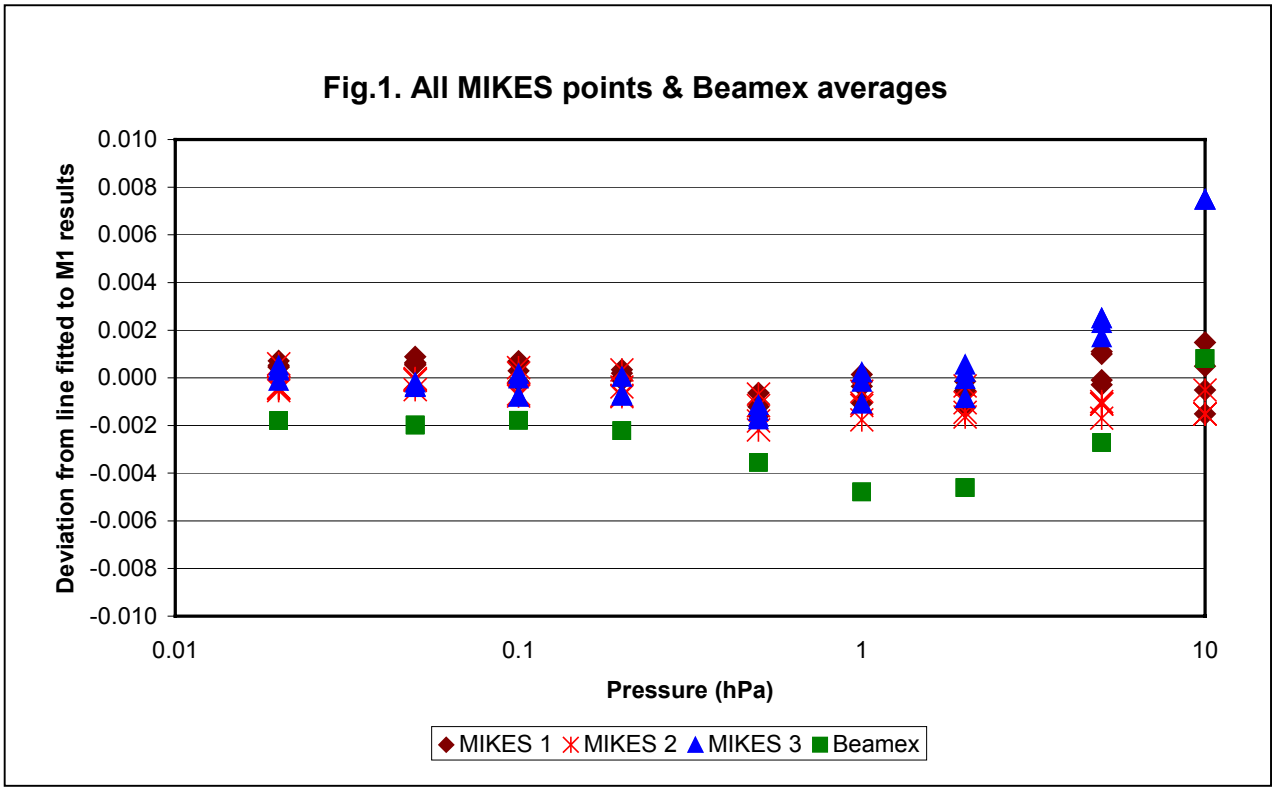
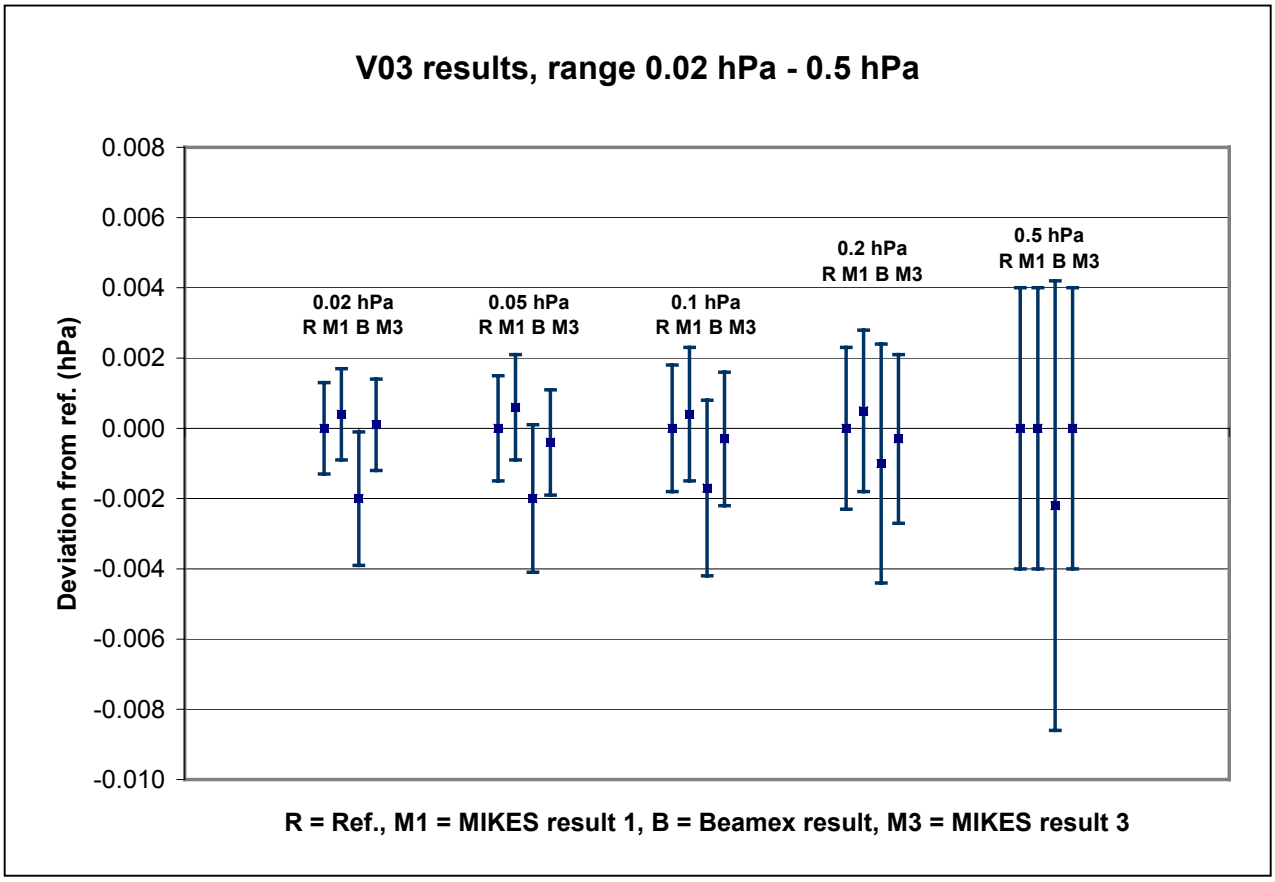
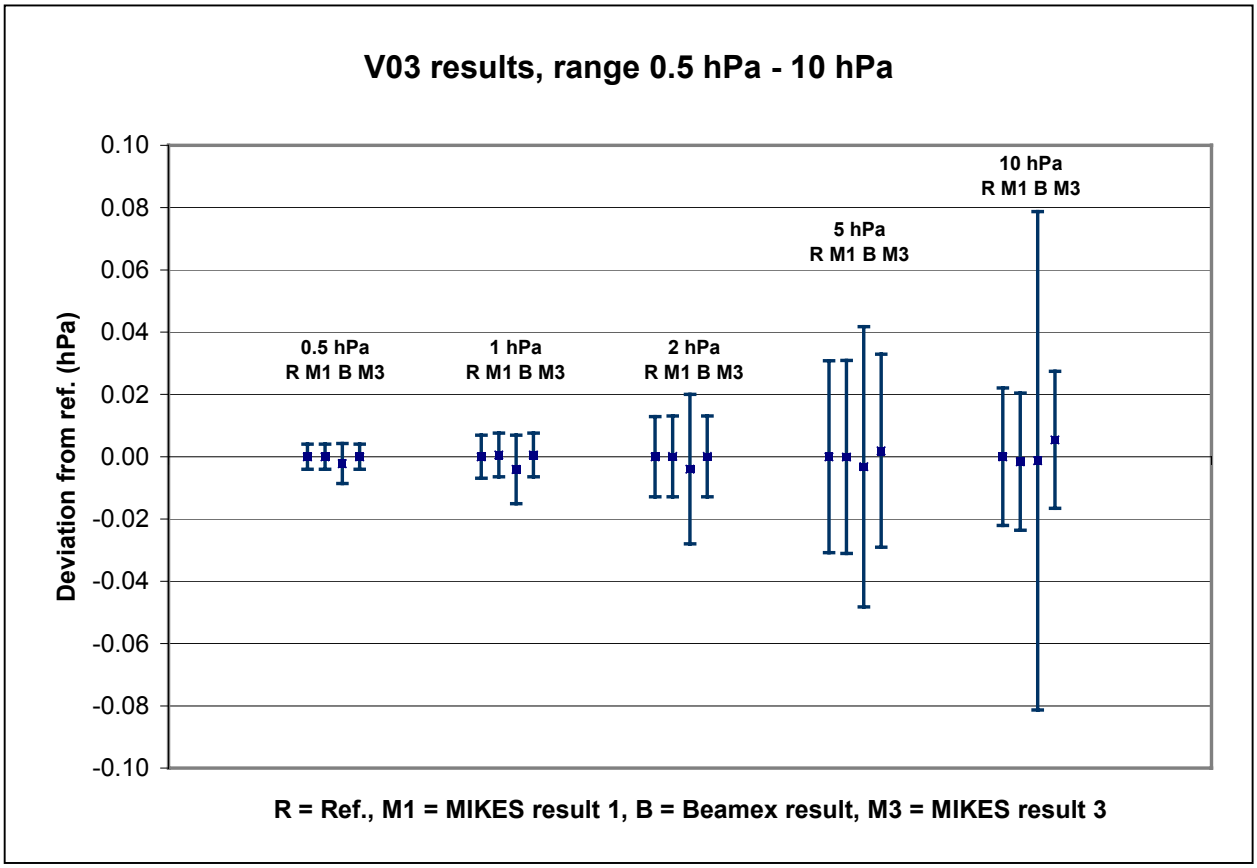


Fig.1. All MIKES points & Beamex averages
Deviation from line fitted to M1 results
0.010
0.008
0.006
0.004
0.002
0.000
-0.002
-0.004
-0.006
-0.008
-0.010
0.01
0.1
1
10
Pressure (hPa)
MIKES 1
MIKES 2
MIKES 3
Beamex


V03 results, range 0.02 hPa - 0.5 hPa
Deviation from ref. (hPa)
0.008
0.006
0.004
0.002
0.000
-0.002
-0.004
-0.006
-0.008
-0.010
0.02 hPa
R M1 B M3
0.05 hPa
R M1 B M3
0.1 hPa
R M1 B M3
0.2 hPa
R M1 B M3
0.5 hPa
R M1 B M3
R = Ref., M1 = MIKES result 1, B = Beamex result, M3 = MIKES result 3


V03 results, range 0.5 hPa - 10 hPa
Deviation from ref. (hPa)
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
0.5 hPa
R M1 B M3
1 hPa
R M1 B M3
2 hPa
R M1 B M3
5 hPa
R M1 B M3
10 hPa
R M1 B M3
R = Ref., M1 = MIKES result 1, B = Beamex result, M3 = MIKES result 3

APPENDIX 1.
Summary of results in pressure intercomparison V03

Lab code	M1					
Nominal pressure	Result	Uncert.	Ref. value	Ref. uncertainty	Deviation from ref.	E(n)
hPa	hPa	hPa	hPa	hPa	hPa	
0.02	0.0004	0.0013	0.0000	0.0013	0.0004	0.20
0.05	0.0007	0.0015	0.0001	0.0015	0.0006	0.28
0.1	0.0008	0.0019	0.0004	0.0018	0.0004	0.17
0.2	0.0014	0.0023	0.0009	0.0023	0.0005	0.16
0.5	0.0020	0.0040	0.0020	0.0040	-0.0000	-0.00
1	0.007	0.007	0.0064	0.0069	0.0006	0.06
2	0.014	0.013	0.014	0.013	0.0001	0.01
5	0.037	0.031	0.037	0.031	-0.0001	-0.00
10	0.074	0.022	0.076	0.022	-0.0016	-0.05

Lab code	Beamex					
Nominal pressure	Result	Uncert.	Ref. value	Ref. uncertainty	Deviation from ref.	E(n)
hPa	hPa	hPa	hPa	hPa	hPa	
0.02	-0.0020	0.0019	0.0000	0.0013	-0.0020	-0.86
0.05	-0.0019	0.0021	0.0001	0.0015	-0.0020	-0.79
0.1	-0.0014	0.0025	0.0004	0.0018	-0.0017	-0.55
0.2	-0.0001	0.0034	0.0009	0.0023	-0.0010	-0.24
0.5	-0.0002	0.0064	0.0020	0.0040	-0.0022	-0.29
1	0.0023	0.011	0.0064	0.0069	-0.0041	-0.32
2	0.0098	0.024	0.014	0.013	-0.0040	-0.15
5	0.0339	0.045	0.037	0.031	-0.0032	-0.06
10	0.0743	0.080	0.076	0.022	-0.0013	-0.02

Lab code	M2					
Nominal pressure	Result	Uncert.	Ref. value	Ref. uncertainty	Deviation from ref.	E(n)
hPa	hPa	hPa	hPa	hPa	hPa	
0.02	-0.0004	0.0014	0.0000	0.0013	-0.0004	-0.22
0.05	-0.0001	0.0015	0.0001	0.0015	-0.0002	-0.10
0.1	0.0002	0.0019	0.0004	0.0018	-0.0002	-0.06
0.2	0.0008	0.0024	0.0009	0.0023	-0.0001	-0.02
0.5	0.0020	0.0040	0.0020	0.0040	-0.0000	-0.00
1	0.006	0.007	0.0064	0.0069	-0.0004	-0.04
2	0.013	0.013	0.014	0.013	-0.0009	-0.05
5	0.035	0.031	0.037	0.031	-0.0021	-0.05
10	0.072	0.022	0.076	0.022	-0.0036	-0.11

Lab code	M3					
Nominal pressure	Result	Uncert.	Ref. value	Ref. uncertainty	Deviation from ref.	E(n)
MPa	MPa	MPa	MPa	MPa	MPa	
hPa	hPa	hPa	hPa	hPa	hPa	
0.02	0.0001	0.0013	0.0000	0.0013	0.0001	0.04
0.05	-0.0003	0.0015	0.0001	0.0015	-0.0004	-0.19
0.1	0.0001	0.0019	0.0004	0.0018	-0.0003	-0.10
0.2	0.0006	0.0024	0.0009	0.0023	-0.0003	-0.08
0.5	0.0020	0.004	0.0020	0.0040	-0.0000	-0.00
1	0.007	0.007	0.0064	0.0069	0.0006	0.06
2	0.014	0.013	0.014	0.013	0.0001	0.01
5	0.039	0.031	0.037	0.031	0.0019	0.04
10	0.081	0.022	0.076	0.022	0.0054	0.17

Recent publications

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