MITTATEKNIIKAN KESKUS

CENTRE FOR METROLOGY AND ACCREDITATION

Julkaisu J6/2002

CALIBRATION OF A 130 Pa CDG: COMPARISON OF THE RESULTS FROM MIKES, PTB AND MKS DEUTSCHLAND

Markku Rantanen
Sari Semenoja

Helsinki 2002

MITTATEKNIIKAN KESKUS
CENTRE FOR METROLOGY AND ACCREDITATION

Julkaisu J6/2002

CALIBRATION OF A 130 Pa CDG: COMPARISON OF THE RESULTS FROM MIKES, PTB AND MKS DEUTSCHLAND

Markku Rantanen
Sari Semenoja

Helsinki 2002

CONTENTS

CALIBRATION OF A 130 Pa CDG: COMPARISON OF THE RESULTS FROM MIKES, PTB AND MKS DEUTSCHLAND

1 INTRODUCTION

A 130 Pa capacitance diaphragm vacuum gauge was calibrated in three pressure laboratories during September and October of 2002: at first in the MKS Instruments Deutschland GmbH, then in Physikalisch-Technische Bundesanstalt (PTB) and finally in the Centre for Metrology and Accreditation (MIKES). This offers an excellent opportunity to compare the results from the three laboratories.

2 BACKGROUND

Since 1997 the Centre for Metrology and Accreditation (MIKES) was using a set of two capacitance diaphragm vacuum gauges (CDGs) as reference standards for the absolute pressure range 0,2 Pa to 1000 Pa, backed with a spinning rotor gauge for the zero control. The CDGs were in the beginning traceable to SP (Swedish National Testing and Research Institute) and later to the accredited pressure calibration laboratory of MKS Instruments Deutschland GmbH, Munich.

In 2001 the Centre for Metrology and Accreditation (MIKES) started a project for improving the measurement capability in the vacuum range. The absolute pressure range was expanded downwards from 0,2 Pa using two spinning rotor type vacuum gauges (SRGs) as reference standards. Further, a new piston manometer was purchased to be a reference standard for the absolute pressure range from about 2 Pa to 15 kPa. This instrument is of a novel type; a force balanced piston gauge (FPG), developed by the DH Instruments, Inc., USA.

The validation process of the new reference standards consisted of several pressure comparisons. In the SRG range (from 0,005 Pa to 5 Pa) a comparison was arranged between MIKES, SP Swedish National Testing and Research Institute and the measurement standards laboratory of Vaisala Oyj [1].

The effective area of the FPG was at first determined at MIKES by a comparison to a conventional pressure balance with the effective area of 980 mm^2 in the gauge pressure range from 5 kPa to 15 kPa. The result was then confirmed in the comparisons in the same range with SP and Nederlands Meetinstuut (NMi).

An important part of the validation was a direct comparison of the FPG with the mercury column manometer of PTB Braunschweig in the absolute pressure range from 1 kPa to 15 kPa.

The present paper covers measurements in the absolute pressure range from 0,1 Pa to 120 Pa.

3 TRANSFER STANDARD

The transfer standard was a MKS capacitance diaphragm gauge type Baratron 690A01TRA s/n 96018200A with a control unit type 270C s/n 93236214 and a channel selector type 274 s/n 94164100A.

If ***mbar*** is selected for the unit in the pressure display and the range multiplier is in the normal (1) position, the resolution is 0,00001 mbar. With the range multiplier in position 0,1 the resolution 0,000001 is obtained.

The equipment was earlier used in the pressure laboratory of MIKES as a reference standards for the absolute pressure range 0,2 Pa to 100 Pa. Unfortunately the previous calibration history was lost in Summer 2002. The control unit suffered from a transport failure and some of the essential electronics parts had to be repaired or replaced.

4 MEASUREMENTS IN MKS

The pressure calibration laboratory of MKS Instruments Deutschland is accredited by Deutscher Kalibrierdienst DKD (laboratory code DKD-K-04601).

The measurements on the transfer standard were made in the range from 0,1 Pa to 133 Pa on the 18th of September 2002. The results were given in a calibration certificate [2]. The laboratory standard used in the calibration was another MKS capacitance diaphragm gauge, type Baratron 698A11TRA. The measurements are traceable to PTB. A summary of the results is given in Tables 1 and 2 with the code MKS.

5 MEASUREMENTS IN PTB

Physikalisch-Technishe Bundeanstalt (PTB) is the national metrology institute of Germany. The vacuum laboratory is located in Berlin.

The measurements on the transfer standard in the pressure range from 0,1 Pa to 123 Pa were made on the 27th of September 2002 using a primary standard applying the static expansion method. The results were presented in a calibration certificate dated 10th of October 2002 [3]. A summary of the results is given in Table 1 with the code PTB.

6 MEASUREMENTS IN MIKES

6.1 Measurements with the FPG

The standard used in the first measurement in MIKES, the force-balanced piston manometer FPG8601 s/n 105 was delivered by the DH Instruments, Inc. The serial number of the piston-cylinder assembly is 106.

The pressure in the FPG is defined by means of the force measured using a high precision load cell and the effective area of the piston-cylinder assembly. The piston is not rotating and it is maintained in the centered position by a constant gas flow through the annular gap. For operation in the absolute mode a capacitance diaphragm gauge (CDG) is used for the reference pressure, which typically is about 0,2 Pa.

The nominal value of the effective area is 980 mm^2, and the latest calibration for the effective area was made on the 27th of August 2002 [4]. The latest check of the reference pressure CDG was made on the 6th of September 2002.

The estimated uncertainty for the FPG of MIKES is 0,04 Pa + 6 x 10^{-5} x $\boldsymbol{p}$. The constant part is mainly due to the uncertainty in the reference pressure measurement, and the pressure dependent part is in fact defined by the uncertainty in determining the effective area. According to the manufacturer it is possible to obtain uncertainties below 0,025 Pa + 3 x 10^{-5} x $\boldsymbol{p}$.

The measurements using the FPG were made in the pressure range from 0,9 Pa to 120 Pa on the 8th of October 2002 by Sari Semenoja and Markku Rantanen. The results were given in the certificate of calibration No. M-02P083 [5]. Two up-and-down measurement cycles were performed. The results, as mean values, are shown in Table 1 with the code M-FPG.

6.2 Measurements with the SRG

The spinning rotor gauge (SRG) 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 SRG 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 [6].

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

The measurements on the transfer standard were made in the pressure range from 0,1 Pa to 3 Pa on the 22nd of October 2002 by Sari Semenoja and Markku Rantanen. The results were given in the certificate of calibration No. M-02P086 [7]. Two up-and-down measurement cycles were performed. The results, as mean values, are shown in Table 1 with the code M-SRG.

Table 1. Calibration of 130 Pa CDG. Summary of results.
Result [Pa] = $p_{transfer} - p_{lab}$
Result [%] = $100*(p_{transfer} - p_{lab})/p_{nominal}$

Nominal pressure Pa	Lab.	Result Pa	Result %	Deviation from PTB result in Pa	Deviation from PTB result in %	Unc. k=2 Pa	Unc. k=2 %	PTB result as reference E(n)
0.1	PTB	0.0036	3.60	0.0000	0.00	0.0006	0.55	0.00
	MKS	0.0040	4.00	0.0004	0.40	0.0026	2.60	0.15
	M-SRG	0.0059	5.88	0.0023	2.28	0.0031	3.10	0.72
0.2	PTB	0.0068	3.40	0.0000	0.00	0.0008	0.39	0.00
	MKS	0.0070	3.50	0.0002	0.10	0.0046	2.30	0.04
	M-SRG	0.0096	4.79	0.0028	1.39	0.0063	3.13	0.44
0.5	PTB	0.0152	3.04	0.0000	0.00	0.0015	0.29	0.00
	MKS	0.0160	3.20	0.0008	0.16	0.0030	0.60	0.24
	M-SRG	0.0246	4.91	0.0094	1.87	0.0163	3.26	0.57
0.7	PTB	0.0197	2.81	0.0000	0.00	0.0019	0.27	0.00
	M-SRG	0.0318	4.54	0.0121	1.73	0.0231	3.30	0.52
1	PTB	0.0268	2.68	0.0000	0.00	0.0028	0.28	0.00
	MKS	0.0290	2.90	0.0022	0.22	0.0046	0.46	0.41
	M-SRG	0.0382	3.82	0.0114	1.14	0.0311	3.11	0.37
	M-FPG*	0.0000	0.00	-0.0268	-2.68	0.0420	4.20	-0.64
2	PTB	0.0449	2.25	0.0000	0.00	0.0055	0.28	0.00
	MKS	0.0490	2.45	0.0041	0.21	0.0088	0.44	0.40
	M-SRG	0.0679	3.40	0.0230	1.15	0.0606	3.03	0.38
3	PTB	0.0558	1.86	0.0000	0.00	0.0078	0.26	0.00
	M-SRG	0.0851	2.84	0.0293	0.98	0.0889	2.96	0.33
	M-FPG*	0.0350	1.17	-0.0208	-0.69	0.0420	1.40	-0.49
5	PTB	0.0706	1.41	0.0000	0.00	0.0136	0.27	0.00
	MKS	0.0770	1.54	0.0064	0.13	0.0170	0.34	0.29
	M-FPG	0.0570	1.14	-0.0136	-0.27	0.0430	0.86	-0.30
7	PTB	0.0735	1.05	0.0000	0.00	0.0184	0.26	0.00
	M-FPG	0.0650	0.93	-0.0056	-0.12	0.0420	0.60	-0.13
10	PTB	0.071	0.71	0.000	0.00	0.026	0.26	0.00
	MKS	0.084	0.84	0.013	0.13	0.033	0.33	0.31
	M-FPG	0.066	0.66	-0.005	-0.05	0.043	0.43	-0.10
20	PTB	0.055	0.27	0.000	0.00	0.030	0.15	0.00
	MKS	0.074	0.37	0.019	0.10	0.066	0.33	0.27
	M-FPG	0.055	0.28	0.001	0.00	0.042	0.21	0.01
30	PTB	0.009	0.03	0.000	0.00	0.045	0.15	0.00
	MKS	0.070	0.23	0.061	0.20	0.100	0.33	0.56
	M-FPG	0.031	0.10	0.022	0.07	0.042	0.14	0.36
50	PTB	-0.040	-0.08	0.000	0.00	0.075	0.15	0.00
	MKS	0.049	0.10	0.089	0.18	0.170	0.34	0.48
	M-FPG	-0.007	-0.01	0.033	0.07	0.044	0.09	0.38
70	PTB	-0.080	-0.11	0.000	0.00	0.110	0.16	0.00
	MKS	0.029	0.04	0.109	0.16	0.230	0.33	0.43
	M-FPG	-0.036	-0.05	0.044	0.06	0.049	0.07	0.37
100	PTB	-0.131	-0.13	0.000	0.00	0.151	0.15	0.00
	MKS	-0.001	-0.00	0.130	0.13	0.330	0.33	0.36
	M-FPG	-0.059	-0.06	0.072	0.07	0.046	0.05	0.45
120	PTB	-0.136	-0.11	0.000	0.00	0.151	0.13	0.00
	MKS*	0.055	0.05	0.191	0.16	0.396	0.33	0.45
	M-FPG	-0.015	-0.01	0.121	0.10	0.048	0.04	0.76

* interpolated result and uncertainty

PTB: PTB, static expansion system
MKS: MKS, reference CDG
M-SRG: MIKES, spinning rotor gauge
M-FPG: MIKES, force balanced piston gauge

7 DISCUSSION OF THE RESULTS

A summary of results is shown in Table 1, and graphically in Fig. 1.

In Table 1 the results (as deviations from the PTB values) and the uncertainties are given in pascals, and also as percentages of the readings respectively. The uncertainties are given with the coverage factor k = 2.

Some results and uncertainties were adjusted to common nominal pressures using the linear interpolation method. These results are marked with a star (*).

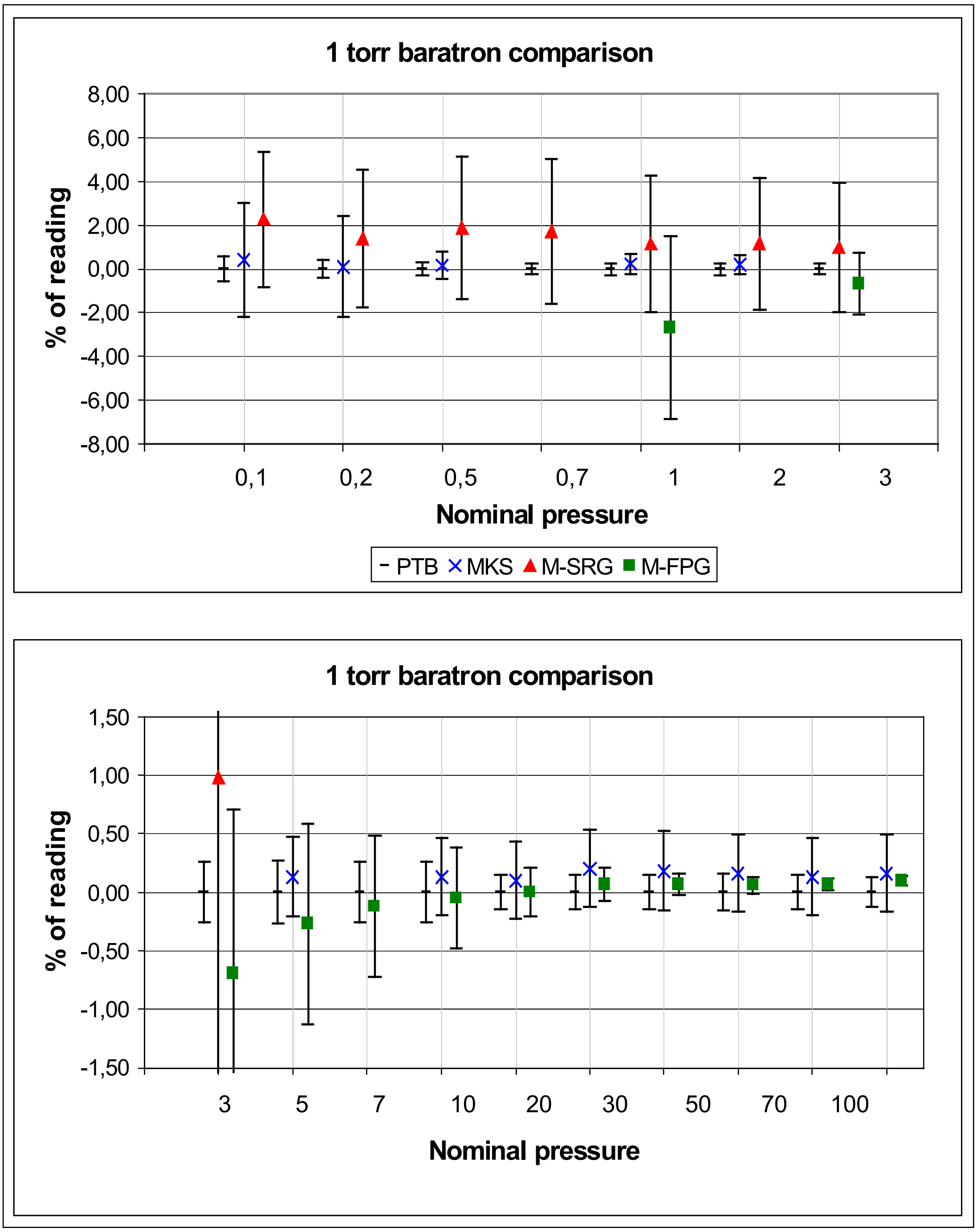

Figure 1. Summary of results.

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.

The results and uncertainties of PTB were taken as reference values for obvious reasons. The minor uncertainty components due to the zero setting and the instability of the transfer standard were ignored for simplicity. All the measurements were carried out during a period of one month.

Table 1 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.

In the pressure range below 10 Pa the uncertainties of the results of both MIKES standards are clearly larger than those of PTB and MKS. However, even these relatively large uncertainties are smaller than the uncertainties of MIKES in the CMC-tables at present [8], see Fig. 2.

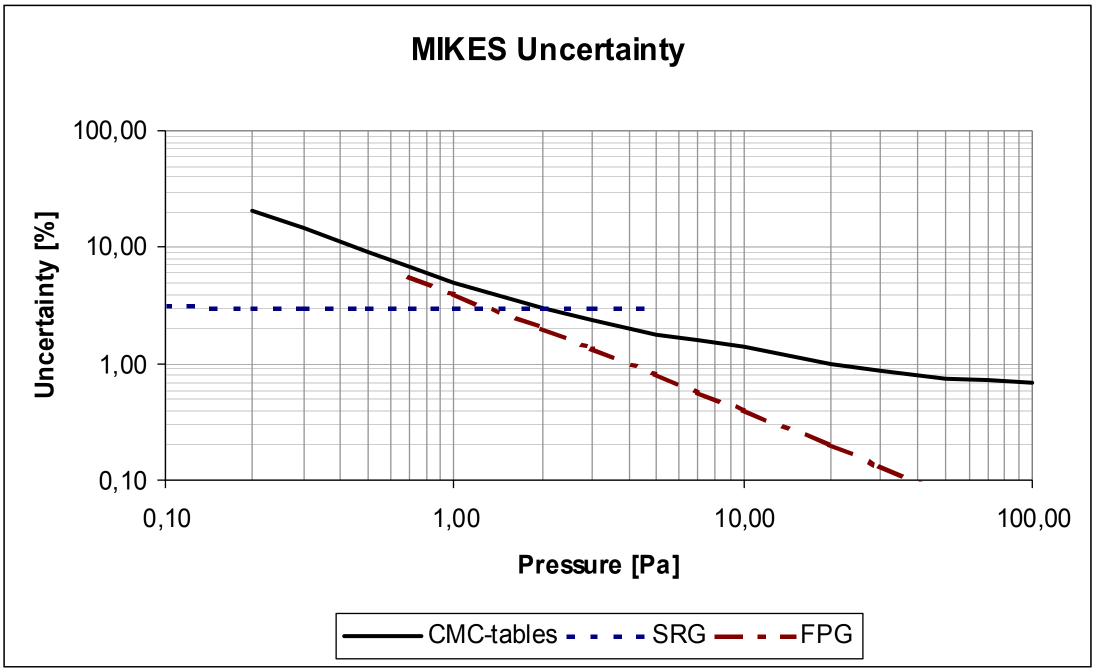

Figure 2. MIKES uncertainty.

At nominal pressures 1 Pa and 3 Pa the results of both MIKES standards differ from each other, but the difference is within the claimed uncertainties. The normalised errors E_n for the difference of the two MIKES standards are 0,73 and 0,51 for 1 Pa and 3 Pa respectively. The overlapping operation range of the two MIKES standards is not the best for either of them.

In the pressure range above 30 Pa the results of MIKES, obtained with the FPG and a direct pressure measurement, have smaller uncertainties than the results of PTB. This is due to the intrinsic uncertainty of the static expansion process.

8 CONCLUSIONS

The results obtained by PTB, MKS Deutschland and MIKES are in a good agreement in the pressure range from 0,1 Pa to 120 Pa.

The uncertainty of MIKES results is large compared to the results of the two other laboratories in the range below10 Pa.

9 REFERENCES

[1] König, V., Pitkäkoski, A., Rantanen, M. & Semenoja, S.: Comparison of spinning rotor vacuum gauges between MIKES, SP, and Vaisala Oyj. Julkaisu J5/2002, MIKES

[2] Calibration Certificate 2451 DKD-K-04601, MKS Instruments Deutschland GmbH, 2002

[3] Kalibrierschein vom 10.10.2002, Kalibrierzeichen: 2122 PTB 02

[4] Certificate of calibration M-02P082 (on FPG using PG7601). MIKES 2002

[5] Certificate of calibration M-02P083 (on Btron using FPG). MIKES 2002

[6] Certificate of calibration MP01/01V1289. National Physical Laboratory (NPL), 2001.

[7] Certificate of calibration M-02P086 (on Btron using SRG). MIKES 2002

[8] CMC-tables for pressure in BIPM database. www.bipm.fr: Continue in English: MRA, JCRB and key comparison database: MRA: Outcome: Database: Appendix C (Calibration and measurement capabilities CMCs): Mass and related quantities (M): Country: Finland, Go To. Address: http://kcdb.bipm.fr/BIPM-KCDB/AppendixC/M/FI/M_FI.pdf. www.bipm.fr 2002.

MR & SS 19.12.2002

J1/1999 Nordic Intercomparison in Barometric Pressure
J2/1999 Automaattisten punnustenvaihtimien suunnittelu, toteutus ja käyttö
J3/1999 Intercomparison of Gauge Pressure Measurements between SP/FFA and MIKES in the Range 32 kPa … 132 kPa
J4/1999 Ainemäärän kansallisen mittanormaalijärjestelmän toteuttamista ja organisaatiota koskeva selvitys
J5/1999 Mikrobiologisen metrologian tilanneselvitys ja kehittämissuunnitelma
J6/1999 Finnish National Standards Laboratories FINMET. Annual Report 1998
J7/1999 Lämpötilan vertailumittaus L10, S-tyypin termoelementin kalibrointi
J8/1999 Mekaanisten värähtelyiden mittausten kartoitus
J9/1999 Intercomparison of the Hydrometer Calibration Systems at the IMGC and the MIKES
J10/1999 National Basis for Traceability in Humidity Measurements
J1/2000 Intercomparison of Temperature Standards of Lithuania and Finland
J2/2000 Finnish National Standards Laboratories FINMET. Annual Report 1999
J3/2000 Mass Comparison M3
J4/2000 Mass and Volume Comparisons at MIKES
J5/2000 Nanometritason mittaukset, kartoitus
J6/2000 Nordic Intercomparison in Gauge Pressure Range 0 … 2 MPa
J1/2001 Mikrobiologian kvantitatiivisten viljelymääritysten mittausepävarmuus
J2/2001 Finnish National Standards Laboratories. Annual Report 2000
J3/2001 Lämpötilan vertailumittaus L11, PT100-anturin sovitusmenetelmän kehittäminen
J4/2001 High Precision Roundness. Euromet Project 533. Final Report
J5/2001 Kaasun kosteuden mittaaminen
J6/2001 Intercomparison of Humidity Standards
J7/2001 Comparisons in the Pressure Range from 50 kPa to 350 kPa
J1/2002 Lämpötilan mittaus
J2/2002 Annual Report 2001
J3/2002 Uncertainty of quantitative Determinations derived by Cultivation of Microorganisms
J4/2002 Calibration of gauge blocks by mechanical comparison. Final Report
J5/2002 Comparison of spinning rotor vacuum gauges between MIKES, SP and Vaisala Oyj
J6/2002 Calibration of a 130 Pa CDG: Comparison of the results from MIKES, PTB and MKS Deutschland

Tilaukset toimistosihteeri Kirsi Tuomisto, puh. (09) 6167 457,
e-mail kirsi.tuomisto@mikes.fi.

MITTATEKNIIKAN KESKUS
CENTRE FOR METROLOGY AND ACCREDITATION
P.O. Box 239
FIN- 00181 HELSINKI
Tel. +358 9 616 761
Telefax +358 9 616 7467

ISBN 952-5209-72-5
ISSN 1235-5704