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Mass Comparison: 610 g laboratory balance

Kari Riski
Leena Stenlund
Mittatekniikan keskus

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MIKES
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Abstract

A comparison of a 610 g balance was carried out in December 2005 and January 2006 by the Centre for Metrology and Accreditation (MIKES). Six participants from four accredited calibration laboratories from Finland participated in the comparison. The reference laboratory was MIKES.

The comparison was made with a high resolution (d = 0,1 mg) laboratory balance. The measurements were made at MIKES. The participants calibrated the balance according to their own measurement procedures using their own weights. Measurement results of the laboratories were taken from calibration certificates.

All results were in reasonable agreement with the results of MIKES.

Tiivistelmä

Mittatekniikan keskus (MIKES) järjesti joulukuussa 2005 ja tammikuussa 2006 massan vertailumittauksen. Vertailu tehtiin 610 g:n tarkkuusvaa'alla. Vertailumittaukseen osallistui kuusi kalibroijaa neljästä akkreditoidusta kalibrointilaboratoriosta Suomesta. Vertailun referenssilaboratoriona oli MIKES.

Vertailuun käytetyn vaa'an resoluutio oli 0,1 mg. Mittaukset tehtiin MIKESissä. Vertailuun osallistuneet laboratoriot tekivät mittaukset omien mittausmenetelmiensä mukaisesti käyttäen omia punnuksiaan. Mittaustulokset on otettu laboratorioiden antamista kalibrointitodistuksista.

Kaikki mittaustulokset olivat mittausepävarmuuksien sisällä samoja kuin MIKESin tulokset.

Contents

1 Introduction

A mass comparison was arranged in December 2005 and in January 2006 by the Centre for Metrology and Accreditation (MIKES). The comparison was made with a 610 g laboratory balance.

The aim of the comparison was not only to compare measurement results but also to compare measurement methods, uncertainties analysis and contents of calibration certificates. No detailed calibration instructions were given to the laboratories.

Six persons from four accredited mass calibration laboratories from Finland participated in the comparison.

2 Balance

The comparison was made with a high resolution laboratory balance: Sartorius ME614S n/o 17010129. The capacity of the balance is 610 g and its resolution is 0,1 mg. The weighing range is 0-610 g. The balance has internal adjusting weights. The balance is adjusted automatically at 8.00 in the morning and at 12.00 at noon. At MIKES the balance is used as a mass comparator.

3 Reference laboratory

The mass laboratory of MIKES is the national standard laboratory for mass in FINLAND. The traceability of mass comes from BIPM. In the calibration of the balance the following weights set was used:

Table 1, Reference weights used by MIKES

SET	OIML Class	Uncertainty (k=2)	Calibrated
P13 10g, 20g, 20g*, 50g, 100g, 200g, 200g*, 500g	E_2	0,007 - 0,05 mg	2004

4 Participants

The following laboratories participated in the comparison:

Oy G.W. Berg & Co Ab, K029, Vantaa,
Inspecta Oy, K004, Helsinki,
Raute Precision OY, K019, Lahti,
Teopal Oy, K037, Espoo,

The measurements were made on 20.12.2005, 21.12.2005, 22.12.2005, 9.1.2006 and 13.1.2006.

5 Measurements by MIKES

5.1 Measurement methods

The balance was on a stone table in a temperature controlled laboratory. The temperature and humidity were constant (about 21 °C and 45 %RH) during the calibrations. MIKES calibrated the balance with methods which are included in the EA Guidelines 10/18 on the calibration of nonautomatic weighing instruments.

The balance was loaded continuously in the following way:

- zero load reading was recorded
- first load was applied and the reading was recorded
- next load was added with or without removing the previous load and the reading was recorded

This procedure was applied up to the largest load. After that the loads were removed in the reverse order. The loads were 0 g, 20 g, 50 g, 100 g, 200 g, 300 g, 400 g, 450 g, 500 g, 550 g and 600 g. The loading was as centric as possible. The measurements with this method took about 1/2 hour. Eccentric errors at 200 g and repeatability at 500 g were measured. The balance was calibrated with this method each day during the comparison. The calibration was performed either before or after the participating laboratory.

Normally the unused weights were on the balance table before and during the calibration. One set of measurements was made with the weights inside the balance. This is shown in Fig 1 as a light blue line. The results were slightly different from the results of the normal procedure. At 100 g the E_n value for this test was -1,0. Only contribution from weights, resolution, repeatability and eccentricity were included in the uncertainty budget of MIKES.

5.2 Measurement results

The measurement results were calculated by the formula:

$$E = I - m,$$

where E is the error of indication of the balance, I is the indication of the balance and m is the conventional mass of the weights.

Figure 1 shows measurement results of MIKES for increasing load (up load). The results for degreasing load were similar. Only measurements made during the comparison days (20.12.2005 – 13.1.2006) were used in the analysis of the results. A third order polynomial fit to the measurement results was calculated and it was used as a reference value.

5.3 Measurement uncertainty

The measurement uncertainties in Fig. 1 are expanded uncertainties. In Fig. 1 the average curve has the following uncertainty components:

- weights (u = 0,007 – 0,05 mg; 0-600 g)
- repeatability (u = 0,05 mg)
- resolution (u = 0,041 mg)
- eccentricity (u = 0 - 0,2 mg; 0 – 600 g)
- fitting uncertainty (includes day to day variation) (u = 0,0 – 0,16 mg; 0-600 g)
- convection (u = 0,00 – 0,12 mg; 0-600 g)
- air buoyancy correction (u = 0,00 – 0,17 mg; 0-600 g)

The effect of heat convection was estimated from conditions where the temperature inside the balance is 1 ˚C different from the temperature of the weights. In MIKES the weighs were left outside the balance between loadings. This may produce convection forces and therefore it is included in the measurement uncertainty.

The air buoyancy was not corrected but it was included in the uncertainty. The air buoyancy was estimated to change 0,2 mg (600 g) if the air pressure changes 2 hPa or the temperature changes 0,5 °C. Only changes after previous adjustment are important.

The results of average values of different days were taken as reference values. Hysteresis was also calculated in most calibrations (u = 0,003-0,09 mg). Its effect revealed to be quite small. The hysteresis was not taken as a part of uncertainty measurements.

6 Measurement instructions

The following information was given to the participants in advance:
Balance model Sartorius ME614S, MAX 610 g, d = 0,1 mg, Nro17010129. The range to be calibrated: 0-600 g and if possible at least loads 100 g, 200 g and 500 g with no

adjustment. It was allowed to bring weights to MIKES in advance. For each laboratory the measurement time was four hours. No further instructions for the measurement method or loading points were given. The participants were asked to send their results as calibration certificates to MIKES after the measurements.

7 Results

The persons who made the calibrations are identified with randomly selected letters from A to F. In most cases they are called laboratories. Figures 2 - 8 shows the measurements results of the participants.

Figure 2 shows up loading from all laboratories including the MIKES reference value. Figures 3 - 8 show the MIKES reference value and each laboratory's up and down loading results perceptively. Expanded measurement uncertainties are also presented for both the reference value and for each laboratory.

All laboratories presented their results as calibration certificates. According to the certificates the uncertainties were estimated using the document EA-4/02.

Main results are given in Figure 2 and in Table 2. The raw results are also given in Annex 1. The error of indication is denoted by *E*. The uncertainties *U* are expanded uncertainties corresponding to a coverage probability of 95 %. In all cases the coverage factor is two (k=2).

A tool often used in analysing the 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 given as:

$$E_n = \frac{E_{lab} - E_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}} \quad .$$

here the subscript *lab* refers to the calibration laboratory and *ref* refers to MIKES. For the reference values the results of MIKES were used.

Table 2, Results of the comparison, *E* = error of indication of the balance, *U* = expanded uncertainty

LAB	A		B		C		D		E		F		MIKES	
Load (g)	*E* mg	*U* mg	*E* mg	*U* mg	*E* mg	*U* mg	*E* mg	*U* mg	*E* mg	*U* mg	*E* mg	*U* mg	*E* mg	*U* mg
0	0	0,3	0	0,41	0	0,12	0	0,1	0	0,3	0	0,2	0,00	0,13
0,001							-0,1	0,1	-0,1	0,2			0,00	0,13
0,1							0	0,1	-0,1	0,2			0,00	0,14
0,5							0	0,1	-0,1	0,2			0,00	0,14
1	0	0,3	-0,1	0,41	0	0,13	-0,1	0,1	-0,1	0,2	0,01	0,2	0,00	0,14
3					0	0,14							-0,01	0,14
5					0	0,17							-0,01	0,14
6			-0,1	0,41									-0,01	0,14
10					0	0,2	-0,1	0,1	-0,1	0,2			-0,02	0,14
20											0,01	0,2	-0,05	0,14
30					-0,1	0,27							-0,07	0,14
50					-0,3	0,35	-0,1	0,3	-0,1	0,2	-0,02	0,2	-0,12	0,16
51	-0,2	0,3											-0,12	0,16
56			-0,3	0,42									-0,14	0,16
100	-0,3	0,3			-0,5	0,45	-0,2	0,5	-0,2	0,3	-0,03	0,2	-0,24	0,18
106			-0,5	0,43									-0,25	0,19
150	-0,4	0,3											-0,34	0,22
200	-0,5	0,4					-0,1	1	-0,3	0,3	-0,2	0,2	-0,41	0,26
206			-0,8	0,46									-0,42	0,26
300	-0,6	0,4			-0,9	0,79					-0,23	0,5	-0,43	0,34
306			-0,9	0,5									-0,42	0,35
400											-0,02	0,5	-0,19	0,43
450	-0,3	0,5											0,05	0,48
500	0,2	0,5					0,6	1,9	0,6	0,5	0,42	0,5	0,39	0,52
506			-0,2	0,59									0,44	0,52
550													0,85	0,57
600	1,2	0,6			0,1	1,29	1,8	1,9	1,8	0,5	1,59	1	1,42	0,64
606			0,8	0,65									1,50	0,66

Table 3, E_n values for the comparison

Load (g)	A	B	C	D	E	F
0	0,00	0,00	0,00	0,00	0,00	0,00
0,001				-0,60	-0,41	
0,1				0,00	-0,41	
0,5				0,01	-0,41	
1	0,01	-0,23	0,01	-0,58	-0,40	0,05
3			0,04			
5			0,05			
6		-0,20				
10			0,10	-0,45	-0,31	
20			0,34			0,24
30			-0,09			
50			-0,47	0,06	0,08	0,40
51	-0,23					
56		-0,37				
100	-0,18		-0,54	0,07	0,11	0,76
106		-0,53				
150	-0,17					
200	-0,18			0,30	0,29	0,65
206		-0,72				
300	-0,32		-0,55			0,33
306		-0,78				
400						0,26
450	-0,51					
500	-0,27			0,10	0,29	0,04
506		-0,81				
550						
600	-0,25		-0,92	0,19	0,46	0,14
606		-0,76				

Calculated E_n-values of all the results are shown in Table 3. A summary of the E_n-values and expanded uncertainties are given in Table 4.

Table 4, Summary of E_n values and expanded uncertainties

Laboratory	E_n-values	Uncertainties
A	-0,5 … 0,01	0,30 - 0,60 mg
B	-0,81 ... -0,23	0,41 - 0,65 mg
C	-0,92 …0,01	0,12 - 1,29 mg
D	-0,60 … 0,30	0,10 - 1,90 mg
E	-0,41 … 0,46	0,20 - 0,50 mg
F	0,04 … 0,76	0,20 - 1,00 mg
MIKES		0,13 - 0,66 mg

The result in an interlaboratory comparison is regarded acceptable if the absolute value of the normalised error E_n is less than 1. In this comparison all the results of the laboratories are acceptable. The laboratories (B and C) have largest negative E_n values. They did not remove the weights from the balance during loading. This may be a reason for the large E_n values.

8 Measurement procedures and contents of certificates

All laboratories calibrated the balance by first loading it to the largest load and then unloading it. Some laboratories did not remove weights during loading (at least B and C). All laboratories determined eccentric loading error and hysteresis.

Lab A gave a measurement uncertainty budget where all components were presented and the combined uncertainty could be recalculated. No complaints or remarks could be made about the calculations or presentation of the results. The calibration certificate was sufficient.

The combined measurement uncertainty of Lab B did not quite correspond to the components presented in the calibration certificate. Otherwise the calibration certificate was sufficient.

The eccentric loading error and hysteresis determined by Lab C were not taken as a part of the measurement uncertainty. The magnitudes of these components were given in the certificate and an overall uncertainty including these components is given. The contents of the calibration certificate were sufficient.

Lab D had measurement uncertainty components which were not quite consistent. The measurement uncertainties were confusingly introduced. The calibration certificate was otherwise sufficient.

Lab E presented an accurate measurement uncertainty analysis but it was confusingly introduced. The calibration certificate was otherwise sufficient.

Lab F did not include hysteresis or eccentric loading to the measurement uncertainty. The magnitude of these components was given in the certificate. Laboratory also gave calculated measurement uncertainties which were very low and unrealistic.

Repeatability was determined at 500 g by labs A and B, at 600 g by Lab F and at 200 g and 500 g by Lab C. Eccentric loading error was determined at 200 g by Labs A, B, C and F. Labs D and E did not announce the loads they used to measure repeatability or eccentric loading error. Two labs had also determined short time stability, Lab A with at 500 g and Lab B at 600 g. The methods used by the laboratories are in accordance with publication J6/1998 or EA 10/18. The publications are not however very specific and leaves much freedom for the calibration procedure.

Table 5, Uncertainty components included in uncertainty calculations. The meaning of the symbols is following: *x* = the component is announced to be part of the uncertainty calculation, *o* = the component is measured but not included in the uncertainty calculations,

LAB	weights	Hysteresis	resolution	repeatability	eccentric loading
A	X	X	X	X	X
B	X	X	X	X	X
C	X	X[1]	X	X	X[1]
D	X	X	X	X	X
E	X	X	X	X	X
F	X	O	X	X	O

[1] only in the overall uncertainty (a single value for the whole weighing range)

In Table 6 magnitudes of uncertainty components for different laboratories are given.

Table 6, Uncertainty components in standard uncertainties. The uncertainties are the largest uncertainty that the lab had announced. The ones marked with () have been calculated from the data of the laboratory and may be incorrect. A question mark is given for components whose magnitudes could not be revealed or predicted.

LAB	Weights (mg)	Hysteresis (mg)	Resolution (mg)	Repeatability (mg)	Eccentric loading (mg)	Weight class
A	0,25	0,03	0,04	0,05	0,12	E_2 /F_1
B	0,3	0,09	0,04	0,05	0,17	E_2 /F_1
C	0,3	0,06	0,04	0,05	0,12	E_2
D	(0,3)	(0,21)	(0,18)	0	(0,88)	E_1 /E_2
E	(0,12)	(0,09)	(0,07)	(0,12)	(0,17)	E_1 /E_2
F	?	0,3	0,04	0,1	0,2	E_2
MIKES	0,05	0,08	0,04	0,05	0,12	E_2

The OIML classes of weights used in the calibration are given in Table 6. All laboratories except Lab D and E included the number of the calibration certificate and the place of calibration for the weights in their own calibration certificate.

Four labs (B, D, E and F) presented loading graph attached to calibration certificate. Most of these graphs were not visual enough to offer any added information. Mostly the problem was on the graphs scale that was used.

As a rule the certificates of all participating laboratories were in accordance with the requirements of ISO/IEC 17025.

After receiving a draft of this report laboratories D and F made corrections to the uncertainties of their results. The changes are given in Annex 2.

9 Conclusions

Four accredited mass calibration laboratories participated in a comparison of 610 g laboratory balance. The calibrations were made at MIKES in December 2005 and in January 2006.

All results from the participating laboratories were in agreement with the reference values of MIKES.

The balance Sartorius ME614S was found to be relatively stable and its resolution was sufficient for this comparison. As with most balances with more than 1 million scale division the result of the calibration depends somewhat on the method of calibration.

10 References

EA-4/02: “Expression of the Uncertainty of Measurement in Calibration”, (www.european-accreditation.org)

SFS-ISO/IEC 17025: “General requirements for the testing and calibration laboratories”, ISO, 2005

“Vaakojen kalibrointiohje”, Julkaisu J6/1998, MIKES 1998

Weights of Classes E_1, E_2, F_1, F_2, M_1, M_{1-2}, M_2, M_{2-3} and M_3 , OIML R111, 2004 (www.oiml.org)

“EA guidelines on the calibration of non-automatic weighing instruments”, EA-10/18, 2004, (www.european-accreditation.org)

KR 13.6.2006

Annex 1: Results given by the laboratories

E = error of indication, U = expanded uncertainty

All values in mg — up = increasing load — down = decreasing load

Lab A	*E*	*E*	*U*
LOAD (g)	**up**	**down**	
0	0,00	0,00	0,3
1	0,00	0,10	0,3
51	-0,20	-0,20	0,3
100	-0,30	-0,30	0,3
150	-0,40	-0,40	0,3
200	-0,50	-0,50	0,4
300	-0,60	-0,50	0,4
450	-0,30	-0,20	0,5
500	0,20	0,30	0,5
600	1,20	1,20	0,6

Lab D	*E*	*U*
LOAD (g)	**up**	
0	0	0,1
0,001	-0,1	0,1
0,1	0	0,1
0,5	0	0,1
1	-0,1	0,1
10	-0,1	0,1
50	-0,1	0,3
100	-0,2	0,5
200	-0,1	1
500	0,6	1,9
600	1,8	1,9

Lab B	*E*	*E*	*U*
LOAD (g)	**up**	**down**	
0	0	0	0,41
1	-0,1	0	0,41
6	-0,1	0	0,41
56	-0,3	-0,2	0,42
106	-0,5	-0,5	0,43
206	-0,8	-0,8	0,46
306	-0,9	-0,7	0,5
506	-0,2	0,1	0,59
606	0,8	0,8	0,65

Lab E	*E*	*U*
LOAD (g)	**up**	
0	0	0,3
0,001	-0,1	0,2
0,1	-0,1	0,2
0,5	-0,1	0,2
1	-0,1	0,2
10	-0,1	0,2
50	-0,1	0,2
100	-0,2	0,3
200	-0,3	0,3
500	0,6	0,5
600	1,8	0,5

Lab C	*E*	*E*	*U*[1]
LOAD (g)	**up**	**down**	
0	0	0	0,12
1	0	0	0,13
3	0	0	0,14
5	0	0	0,17
10	0	0	0,2
30	-0,1	-0,1	0,27
50	-0,3	-0,2	0,35
100	-0,5	-0,3	0,45
300	-0,9	-0,9	0,79
600	0,1	0,1	1,29
	Combined uncertainty[1]		**1,32**

Lab F	*E*	*E*	*U*[1,2]
LOAD (g)	**up**	**down**	
0	0	0,1	0,2
1	0,01	0,11	0,2
20	0,01	0,11	0,2
50	-0,02	0,18	0,2
100	-0,03	-0,13	0,2
200	-0,2	-0,2	0,2
300	-0,23	-0,13	0,5
400	-0,02	0,08	0,5
500	0,42	0,72	0,5
600	1,59	1,59	1
calculated uncertainty[2]			**0,2**

[1] uncertainties due to hysteresis and eccentric loading are not included
[2] *best measurement capability*

Annex 2: Comments from the laboratories

Laboratory D:

A recording error was found in the original measurement results. After correcting the results the uncertainties were reduced significantly. A new certificate was given. The results are given in the table below. Also calculated E_n values are given. All E_n values are below 1.

Load (g)	*E* (mg)	*U* (mg)	E_n
0	0	0,1	0,00
0,001	-0,1	0,1	-0,60
0,1	0	0,1	0.00
0,5	0	0,1	0.01
1	-0,1	0,1	-0,58
10	-0,1	0,1	-0,45
50	-0,1	0,1	0,11
100	-0,2	0,1	0,18
200	-0,1	0,2	0,96
500	0,6	0,4	0,32
600	1,8	0,3	0,53

Laboratory F:

The uncertainties of the reference weights were incorrect in the original calculations. After correcting the results the maximum uncertainty of the weights is 0,3 mg. At 600 g the uncertainty increased from 0,2 mg to 0,4 mg. The calculated uncertainties and E_n values are given in the table below. All E_n values are below 1.

Load (g)	*E* (mg)	*U* (mg)	E_n
0	0,0	0,2	0,00
1	0,0	0,2	0,05
20	0,0	0,2	0,24
50	0,0	0,2	0,40
100	0,0	0,2	0,76
200	-0,2	0,3	0,54
300	-0,2	0,3	0,44
400	0,0	0,3	0,33
500	0,4	0,3	0,04
600	1,6	0,4	0,22

Fig. 1, MIKES, up loading

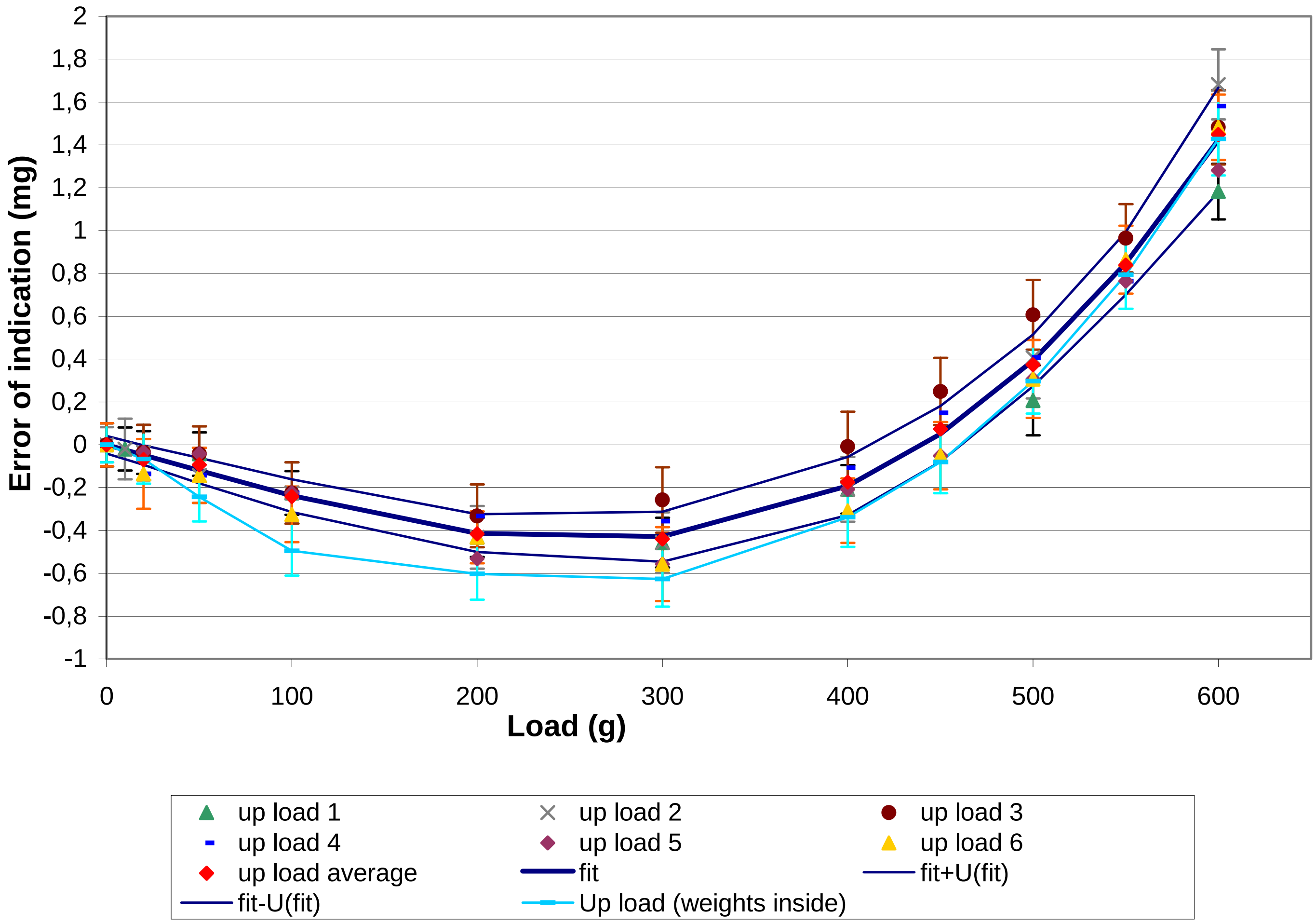


Fig. 2
MIKES and laboratories A-F, up loading

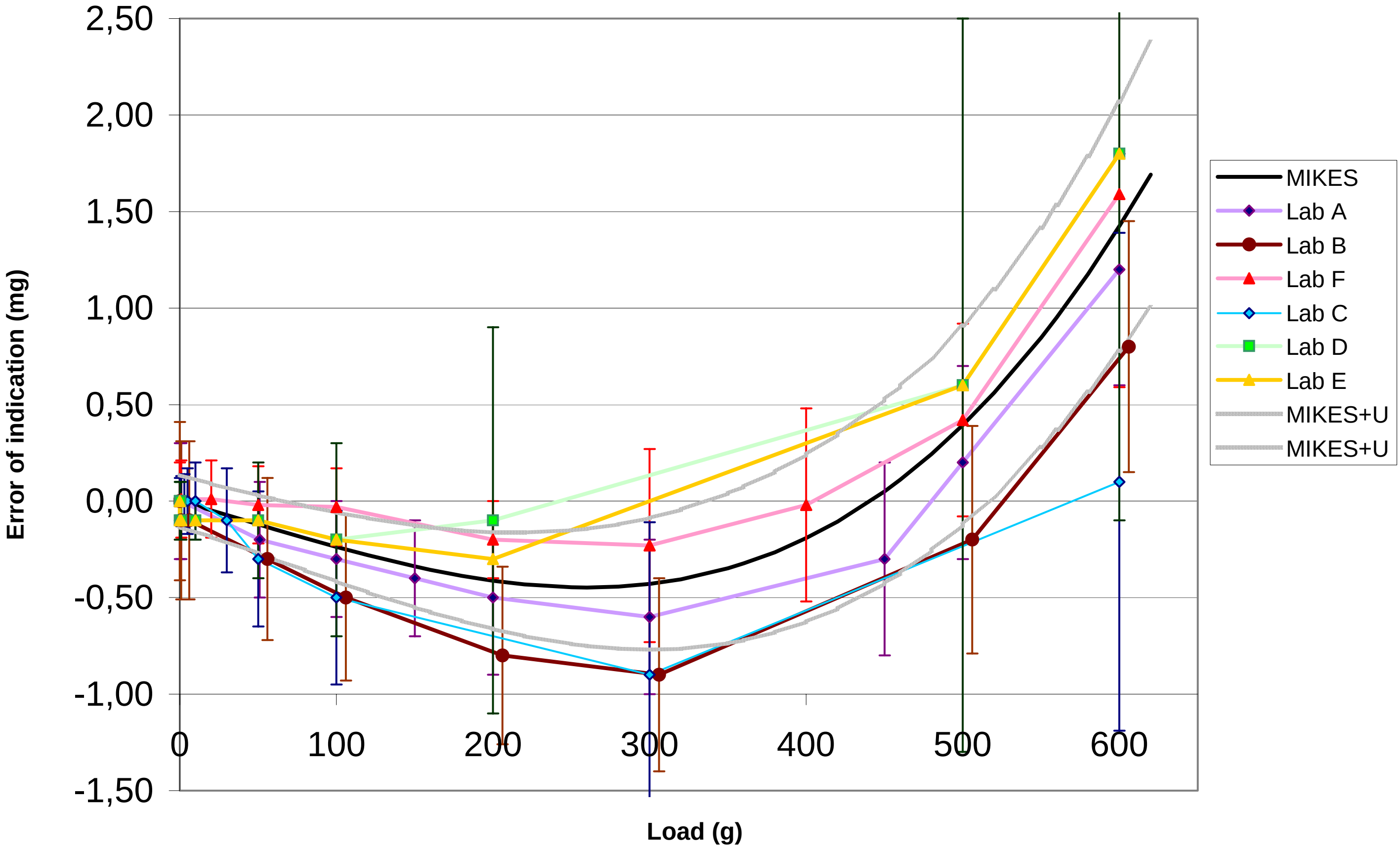


Fig. 3
MIKES and LAB A

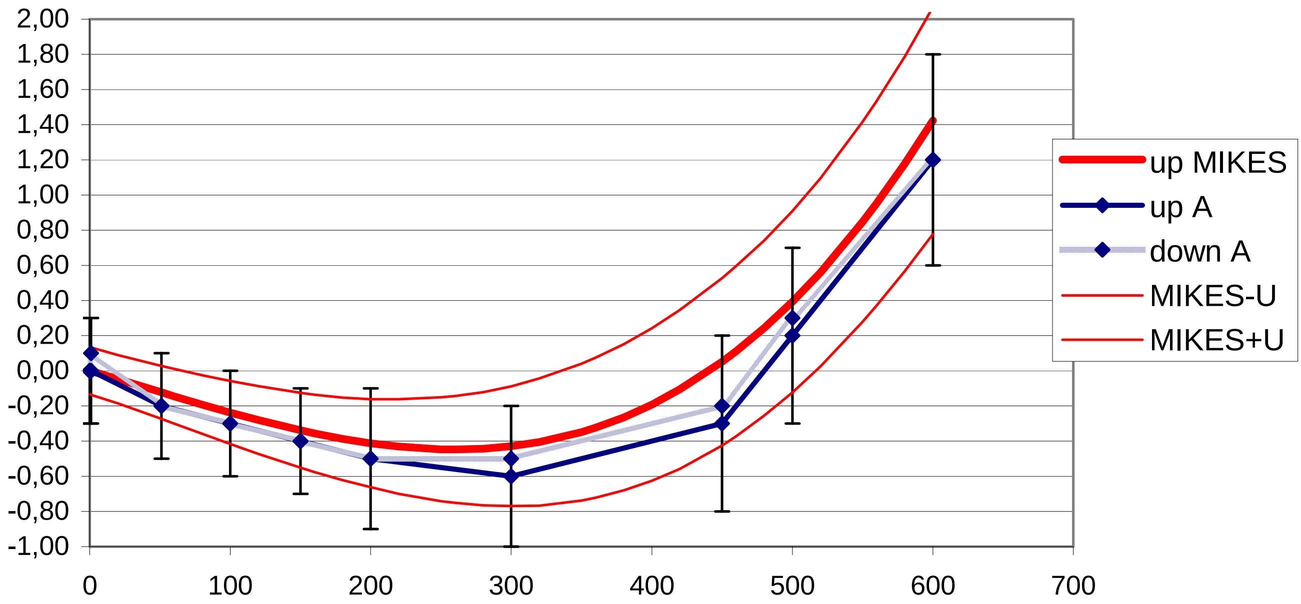


Fig. 4
MIKES and LAB B

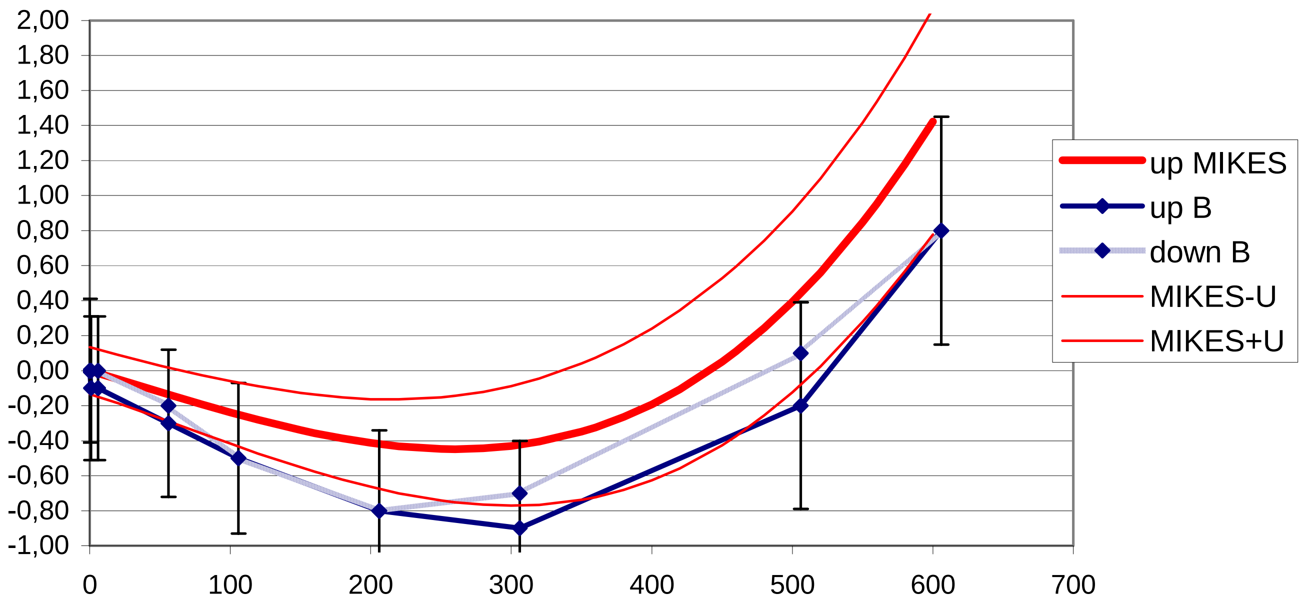


Fig. 5
MIKES and LAB C

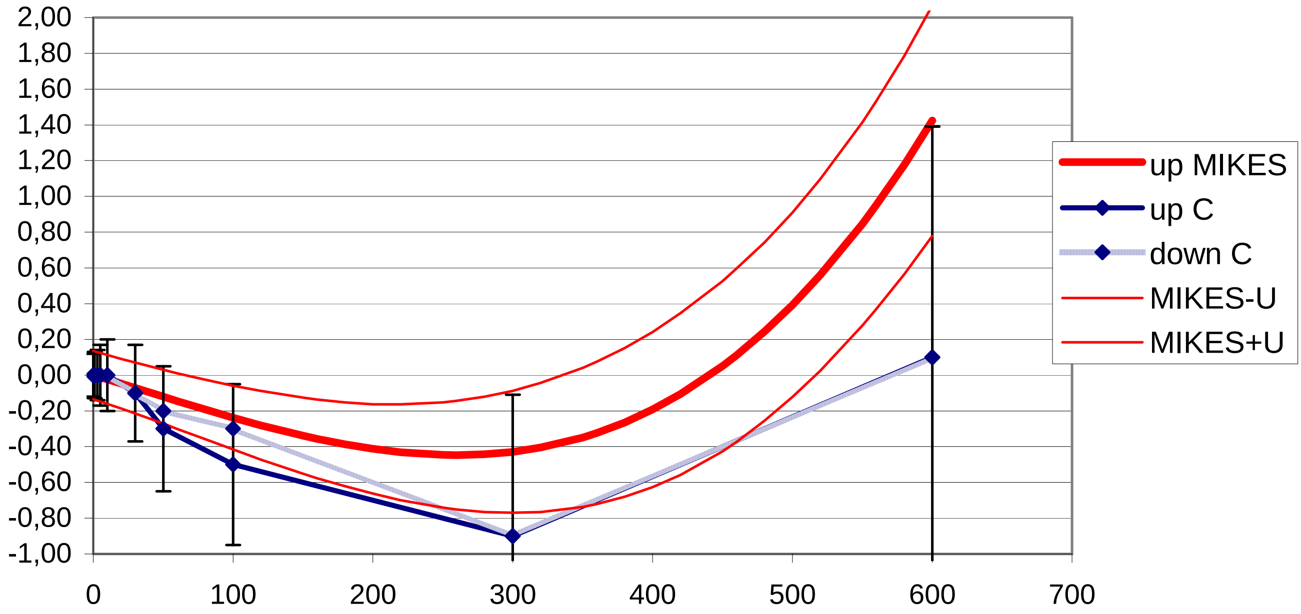


Fig. 6
MIKES and LAB D

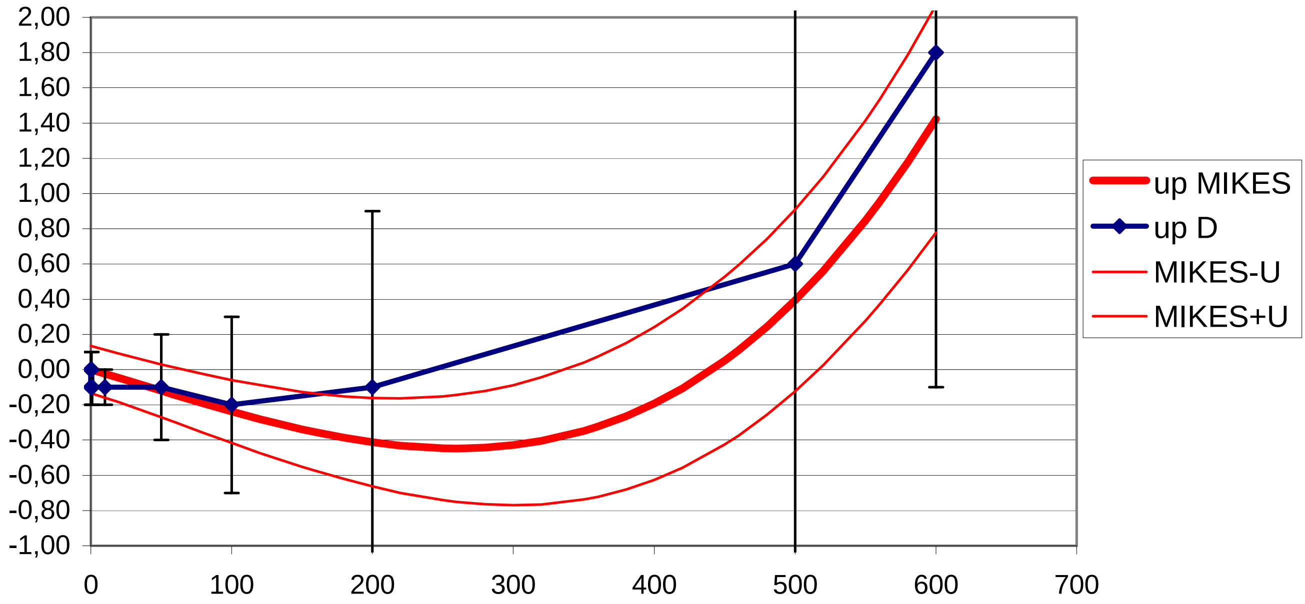


Fig. 7
MIKES and LAB E

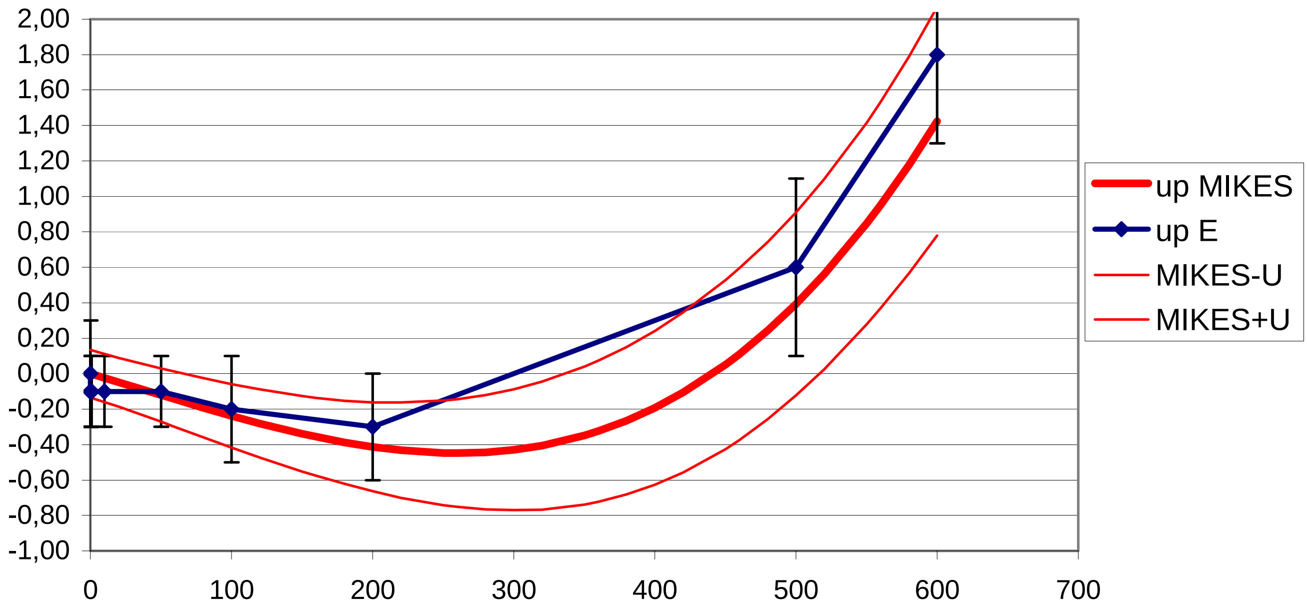
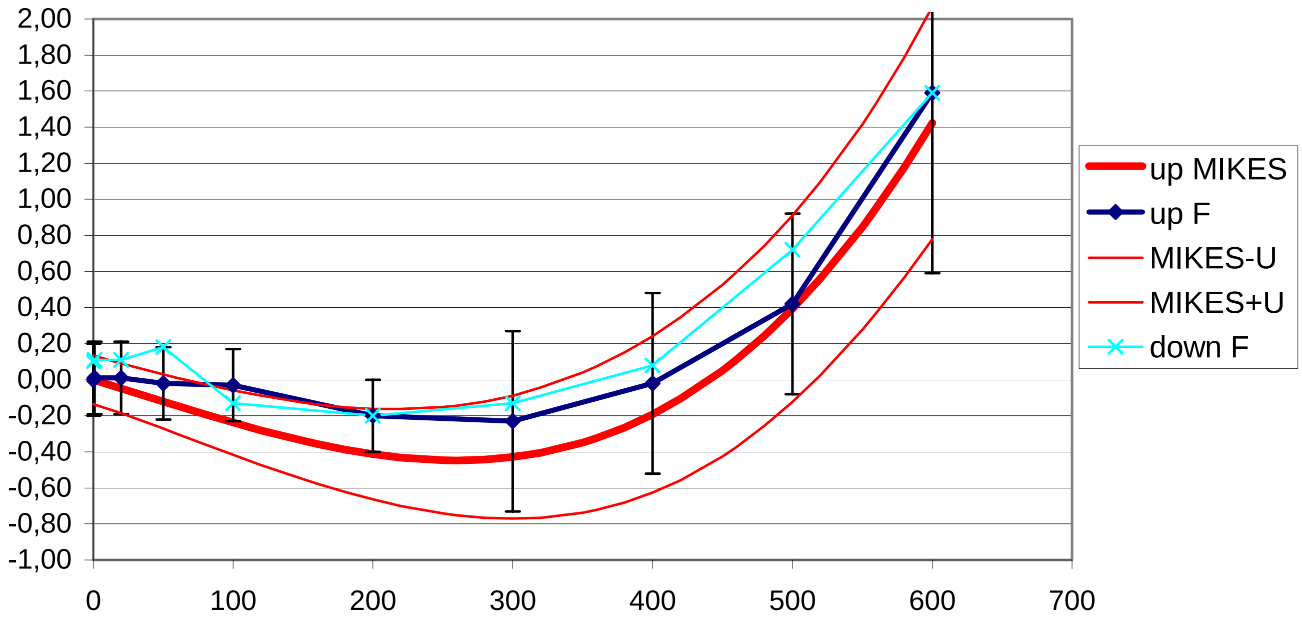


Fig. 8
MIKES and LAB F



Recent publications

J8/2003	J. Järvinen (Toim*.), Kansallinen mittanormaalitoiminta ja sen kehittäminen 2003 - 2007*
J1/2004	J. Järvinen, M. Heinonen, A. Lassila, R. Rajala (Eds.) *Finnish National standards Laboratories Annual Report 2003*
J2/2004	S. Semenoja, M. Rantanen, J. Leskinen and A. Pitkäkoski, *Comparison in the absolute pressure range 100 kPa to 2100 kPa between MIKES and Vaisala Oyj*
J3/2004	V. Esala, *Pituuden vertailumittaus D6, loppuraportti*
J4/2004	J. Halttunen, *Coriolis-mittarin vertailumittaus, syksy 2002. Interlaboratory comparison of a Coriolis flowmeter, Autumn 2002*
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Tilaukset: Kirsi Tuomisto, puh. 010 6054 436, e-mail tilaukset@mikes.fi.

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MIKES
Mittatekniikan keskus
Centre for metrology
and accreditation

• P.O.Box 9, Tekniikantie 1, FI-02151 ESPOO, Finland
• Tel. +358 10 6054 000 • Fax +358 10 6054 299
• www.mikes.fi