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Mass Comparison: 6 g microbalance

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Abstract

A comparison of a 6 g microbalance was carried out in June 2008 at the Centre for Metrology and Accreditation (MIKES). Five participants from three accredited calibration laboratories from Finland participated in the comparison. The reference laboratory was MIKES.

The comparison was made with a high-resolution (d = 0,001 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 good agreement with the results of MIKES.

Tiivistelmä

Mittatekniikan keskus (MIKES) järjesti kesäkuussa 2008 massan vertailumittauksen. Vertailu tehtiin 6 g:n tarkkuusvaa'alla. Vertailumittaukseen osallistui viisi kalibroijaa kolmesta akkreditoidusta kalibrointilaboratoriosta Suomesta. Vertailun referenssilaboratoriona oli MIKES.

Vertailuun käytetyn vaa'an resoluutio oli 0,001 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 June 2008 at the Centre for Metrology and Accreditation (MIKES). The comparison was made with a 6 g microbalance.

The aim of the comparison was not only to compare measurement results but also to some extent compare uncertainty components and contents of calibration certificates. No detailed calibration instructions were given to the laboratories.

Five persons from three accredited mass calibration laboratories from Finland participated in the comparison.

2 Balance

The comparison was made with a high-resolution laboratory balance: Sartorius CCE6 n/o 21609683. The capacity of the balance is 6,1 g and its resolution is 0,0001 mg. The weighing range is 0-6,1 g. During the calibration the resolution of the display was set to 0,001 mg. The balance has internal adjusting weights. The balance was adjusted in the morning and at noon during the calibration period. 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 0,1 mg – 5 g	E_2	0,0015 – 0,005 mg	2008

4 Participants

The following laboratories participated in the comparison:

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

The measurements were made on 10-11.6.2008.

5 Measurements by MIKES

5.1 Measurement methods

The balance was on a stone table in a temperature and humidity controlled laboratory. (Temperature was 21,5±0,5 °C and relative humidity was 44±1 %RH). MIKES calibrated the balance with methods which are included in the EURAMET/cg-18 (guidelines on the calibration of non-automatic weighing instruments).

The balance was loaded two times in the following way:

- The balance reading with zero load was set to zero
- The first load was applied and the reading was recorded
- The first load was removed
- The zero was checked and re-zeroed if necessary
- The second load was applied and the reading was recorded
- The second load was removed
- The zero was checked and re-zeroed if necessary
- This procedure was applied up to the largest load.
- After that the loading sequence was reversed starting from the largest load.

The loads were 0,1 g, 0,2 g, 0,5 g, 1 g, 1,5 g, 2 g, 2,5 g, 3 g, 3,5 g, 4 g, 4,5 g, 5 g, 5,5 g and 6 g. The loading was as centric as possible. Eccentric errors at 2 g and repeatability at 5 g were measured. The balance was calibrated with this method each day during the comparison. The calibration was performed before each participating laboratory performed calibration.

The weights were stored on the balance table during the calibration period.

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. Measurements made before (2-9.6.2008) and during the comparison days (10-11.6.2008) 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 = 0,0007 – 0,004 mg; 0-6 g)
- repeatability (u = 0,000 5 mg)
- resolution (u = 0,000 4 mg)
- eccentricity (u = 0 ,001 mg)
- fitting uncertainty (includes day to day variation) (u = 0,000 5 - 0,001 5 mg)
- convection (u = 0,000 - 0,002 mg)
- air buoyancy correction (u = 0,000 - 0,002 mg)

The calibration of the weights was performed before the comparison. The uncertainties of weights are equal to the uncertainties in the CMC tables of MIKES.

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 0,002 mg (5 g) if the air pressure changes 2 hPa or the temperature changes 0,5 °C. Only changes after previous adjustment were included.

The average values of different days were taken as reference values. Hysteresis was also measured in some calibrations. Its effect revealed to be small (less than 0,001 mg).

6 Measurement instructions

The following information was given to the participants in advance:
The model of the balance, Sartorius CCE6, MAX 6,1 g, d = 0,001 mg and No 21609683. The range to be calibrated 0-6 g. 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 E. In most cases they are called laboratories. Figure 2 shows the measurements results of the participants.

Figure 2 shows results with increasing load from all laboratories including MIKES. Expanded measurement uncertainties are also presented.

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

In addition to Figure 2 the results are also given Table 2. The raw results are also given in Annex 1. The expanded uncertainties correspond 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 Load (g)	A		B		C		D		E		MIKES	
	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	0,0000	0,0017	0,0000	0,0017	0,000	0,020	0,000	0,003	0,000	0,001	0,000	0,003
0,001	-0,0010	0,0035	-0,0010	0,0035	0,001	0,020	-0,001	0,003	-0,001	0,002	0,000	0,003
0,005					0,001	0,020					0,000	0,003
0,01					0,000	0,020	0,000	0,003	0	0,002	0,000	0,003
0,05	-0,0030	0,0092	-0,0025	0,0095	0,001	0,020					0,000	0,003
0,1					0,001	0,020	0,000	0,006	0,001	0,002	0,000	0,003
0,2							0,000	0,006	0	0,002	0,000	0,003
0,251	-0,0095	0,0181	-0,0090	0,0181							0,000	0,003
0,5					0,003	0,021	0,001	0,020	0,001	0,003	0,001	0,004
0,751	-0,0165	0,0301	-0,0145	0,0301							0,001	0,004
1					0,005	0,021	0,003	0,020	0,003	0,003	0,001	0,004
1,751	-0,0060	0,0451	-0,0040	0,0451							0,002	0,005
2							0,002	0,020	0,003	0,004	0,002	0,006
3					0,007	0,023					0,003	0,007
3,751	-0,0075	0,0646	-0,0045	0,0646							0,003	0,008
4							0,006	0,020	0,008	0,008	0,004	0,008
5					0,013	0,025	0,009	0,020	0,012	0,006	0,006	0,010
5,951	0,0010	0,0931	0,0040	0,0931							0,010	0,011
6					0,015	0,026	0,014	0,020	0,017	0,008	0,010	0,011

Table 3, E_n values for the comparison

LAB Load (g)	A	B	C	D	E
0,001	-0,25	-0,25	0,04	-0,27	-0,32
0,005			0,04		
0,01			-0,01	-0,03	-0,03
0,05	-0,33	-0,27	0,04		
0,1			0,04	-0,04	0,21
0,2				-0,06	-0,10
0,251	-0,54	-0,51			
0,5			0,11	0,01	0,06
0,751	-0,58	-0,51			
1			0,18	0,09	0,35
1,751	-0,17	-0,12			
2				0,01	0,17
3			0,19		
3,751	-0,17	-0,12			
4				0,10	0,35
5			0,25	0,12	0,50
5,951	-0,10	-0,06			
6			0,17	0,16	0,49

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	-0,58 … -0,1	0,002 – 0,093 mg
B	-0,51 ... -0,06	0,002 – 0,093 mg
C	-0,01 …+0,25	0,020 – 0,026 mg
D	-0,27 … +0,16	0,003 – 0,020 mg
E	-0,32 … +0,50	0,001 – 0,008 mg

The result in an interlaboratory comparison is considered acceptable if the absolute values of the normalised errors E_n are less than 1. In this comparison all E_n values are below 1.

8 Measurement procedures and contents of certificates

All laboratories determined the loading curve up to the maximum load. Some laboratories did not remove weights during loading. All laboratories determined repeatability, eccentric loading error and hysteresis.

Laboratories A and B made two loading and unloading sequences. The weights were not removed during loading. Following uncertainty components are given in the certificate: hysteresis, eccentric loading, repeatability and resolution. OIML class E_2 weights were used, which gives relatively high uncertainties. The certificates also give calculated minimum load (values 0,06 mg and 0,22 mg) corresponding to 99 % reliability with 95 % uncertainty. Also the short time stability is given.

Laboratory C has measured the range 0-0,5 g with individual weights and the range 1-6 g by adding/removing weights continuously. The following uncertainty components are given in the certificate: hysteresis, eccentric loading and repeatability. Other uncertainty components are not specified. Also the minimum load of 2 mg corresponding to 99,95 % reliability with 95% uncertainty is given.

Laboratories D and E measured both loading and unloading sequences. The certificate states that the following uncertainty components have been included: hysteresis, weights, resolution, repeatability and eccentric loading. In addition to the certificate the laboratory D also sent a list of all uncertainty components for each load. The calculated expanded uncertainties 0,0022 – 0,0086 mg were smaller than in the certificate. The E_n values calculated with these uncertainties were less than 0,26.

Repeatability was determined at 5 g by most laboratories. Eccentric loading error was determined at 2 g. Laboratories D and E did not give the load they used to measure repeatability or eccentric loading error. The methods used by the laboratories are in accordance with publication J6/1998 or EURAMET/cg-18.

Table 5, Uncertainty components included in uncertainty calculations.

LAB	weights	Hysteresis	resolution	repeatability	eccentric loading
A	X	X	X	X	X
B	X	X	X	X	X
C	X	X	X	X	X
D	X	X	X	X	X
E	X	X	X	X	X

In Table 5 uncertainty components included in the uncertainty calculations are given. Table 6 gives the magnitudes of those components whose values are given in the certificates. For laboratory E the values in Table 6 have been calculated from relative values.

Table 6. Uncertainty components given as standard uncertainties. The uncertainties for the weights are the largest uncertainties that the laboratory has announced. Those cells whose magnitudes could not be obtained from the certificates are left empty.

LAB	Weights (mg)	Hysteresis (mg)	Resolution (mg)	Repeatability (mg)	Eccentric loading (mg)	Weight class
A		0,0003	0,0004	0,0003;0,0014	0,0006	E_2
B		0,0003	0,0004	0,0003;0,0011	0,0006	E_2
C		0		0,0005;0.0008	0,0006	E_1
D	0,004	0,0003	0,0004	0,001	0-0,0011	E_1
E	0,004	0,0012		0,0012	0-0,0017	E_1
MIKES	0,004	-	0,0004	0,0004	0,0005	E_2 (E_1)

The OIML classes of weights used in the calibration are also given in Table 6. At MIKES the E_2 weights have been calibrated with the same uncertainty as would be given to E_1 weights. In the calibration certificates of laboratories A, B and C the number of the calibration certificate of the weights is given.

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

9 Conclusions

Three accredited mass calibration laboratories participated in a comparison of 6 g laboratory balance. All results from the participating laboratories were in agreement with the reference values of MIKES.

The balance, Sartorius CCE6, was found to be sufficiently stable. No improvement in uncertainty would have been gained by increasing the resolution from 0,001 mg to

0,0001 mg. The uncertainty is mainly determined from the uncertainty of the used weights. If weights of OIML class E_2 are used they should be calibrated as E_1 weights.

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 (www.mikes.fi)

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)

"Guidelines on the calibration of non-automatic weighing instruments", EURAMET/cg-18/v.01, 2007, (www.euramet.org)

Annex 1: Results given by the laboratories

E = error of indication, U = expanded uncertainty
up = increasing load, down = decreasing load
All values in mg

Lab A	*E*	*E*	*U*
NIM (g)	up	down	.
0	0,000	0,000	0,002
0,001	-0,001	0,000	0,004
0,051	-0,003	-0,002	0,010
0,251	-0,009	-0,008	0,018
0,751	-0,016	-0,016	0,030
1,751	-0,005	-0,006	0,045
3,751	-0,007	-0,007	0,065
5,951	0,002	0,002	0,093

Lab B	*E*	*E*	*U*
NIM (g)	up	down	
0	0,000	0,000	0,002
0,001	-0,001	-0,001	0,004
0,051	-0,003	-0,002	0,010
0,251	-0,009	-0,008	0,018
0,751	-0,014	-0,015	0,030
1,751	-0,004	-0,005	0,045
3,751	-0,005	-0,005	0,065
5,951	0,004	0,004	0,093

Lab C	*E*	*E*	*U*
NIM (g)	up	down	
0	0,000	0,000	0,020
0,001	0,002	0,002	0,020
0,005	0,001	0,001	0,020
0,01	0,000	0,000	0,020
0,05	0,001	0,001	0,020
0,1	0,001	0,001	0,020
0,5	0,003	0,003	0,021
1	0,005	0,005	0,021
3	0,007	0,007	0,023
5	0,013	0,013	0,025
6	0,015	0,015	0,026

Lab D	*E*	*U*
NIM (g)	up	
0	0,000	0,003
0,001	-0,001	0,003
0,01	0,000	0,003
0,1	0,000	0,006
0,2	0,000	0,006
0,5	0,001	0,020
1	0,003	0,020
2	0,002	0,020
4	0,006	0,020
5	0,009	0,020
6	0,014	0,020

LAB E	E	*U*
NIM (g)	up	
0	0	0,001
0,001	-0,001	0,002
0,01	0	0,002
0,1	0,001	0,002
0,2	0	0,002
0,5	0,001	0,003
1	0,003	0,003
2	0,003	0,004
4	0,008	0,008
5	0,012	0,006
6	0,017	0,008

Figure 1: MIKES results. The solid curve is a polynomial fit to the results. The uncertainties are expanded uncertainties of one calibration.

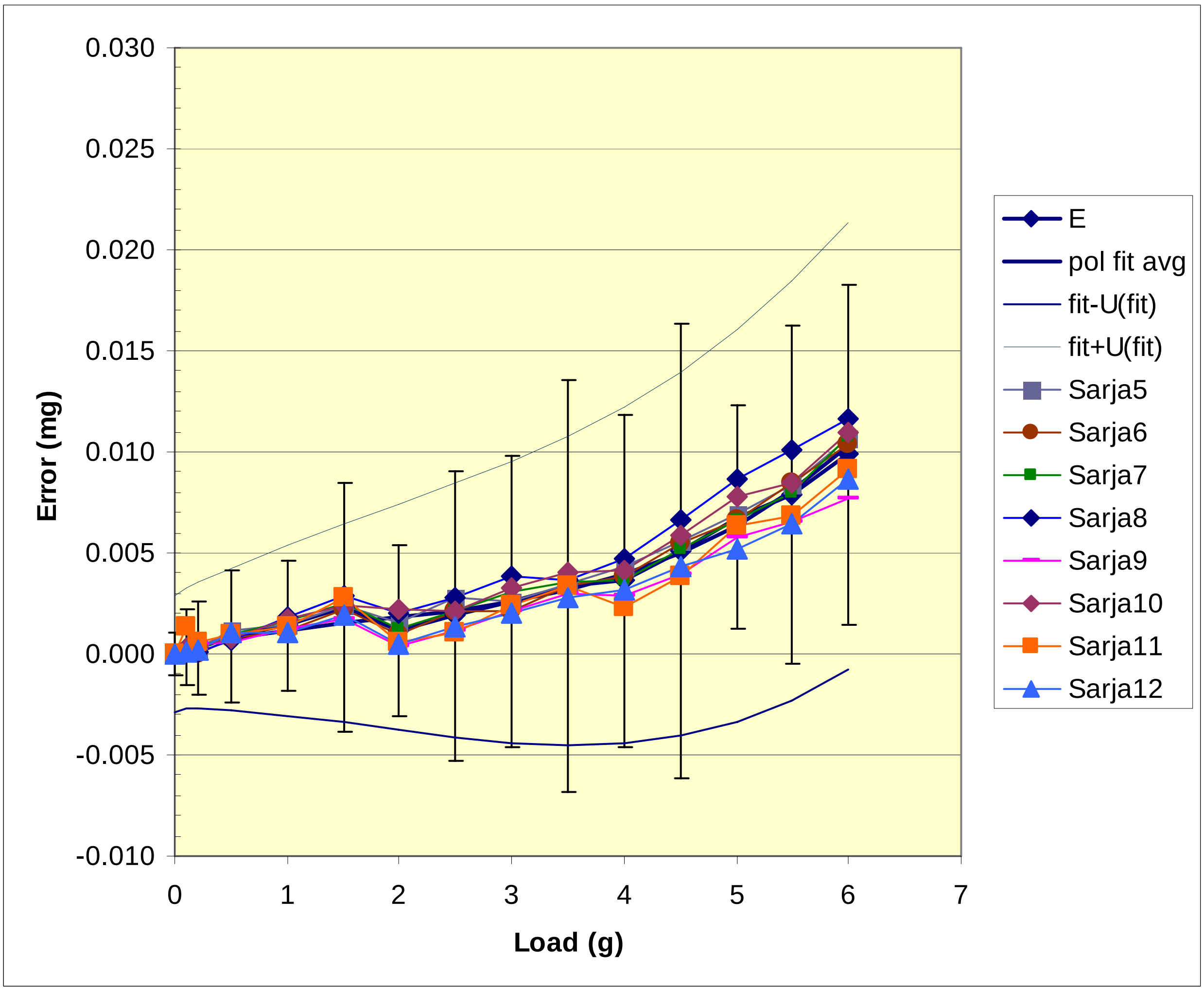
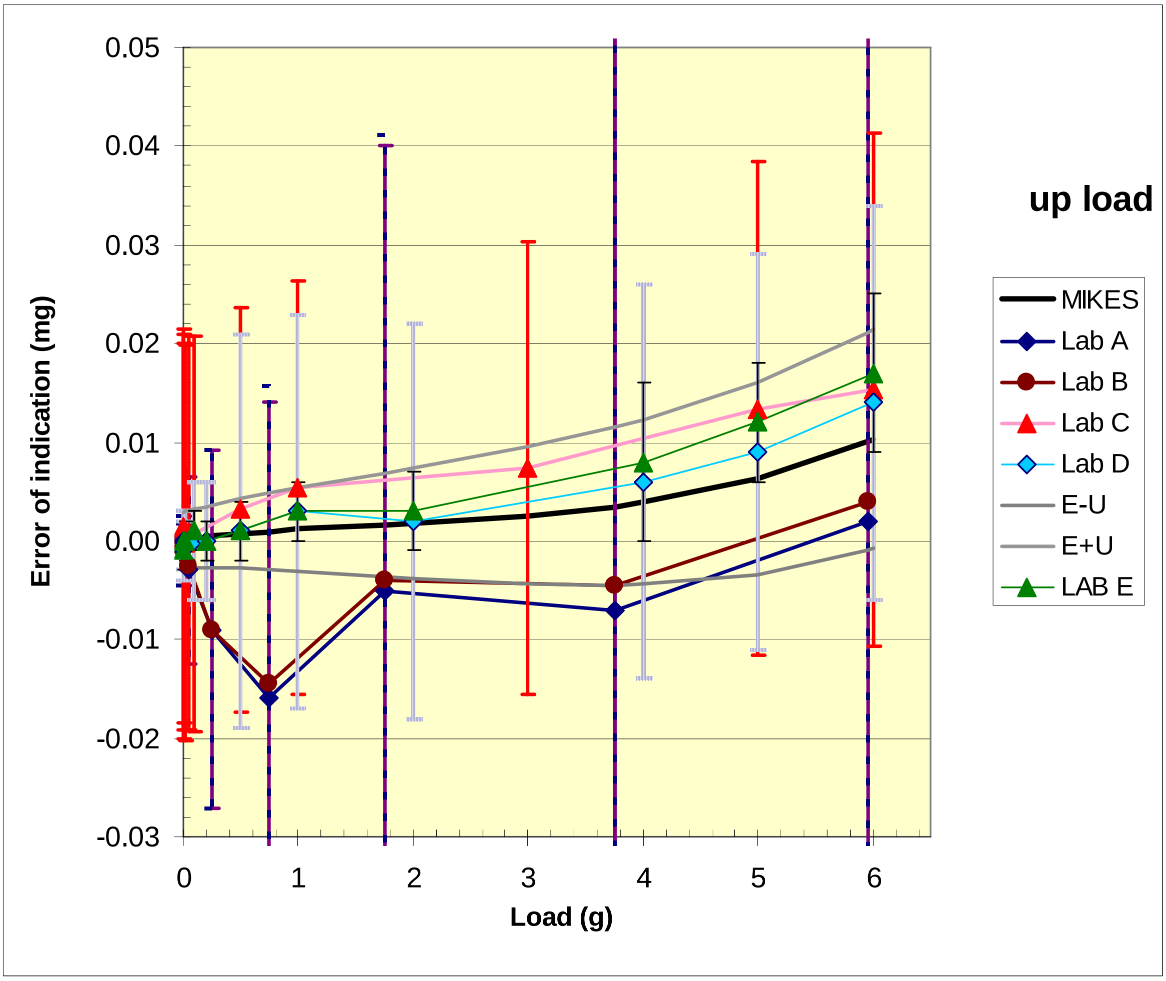
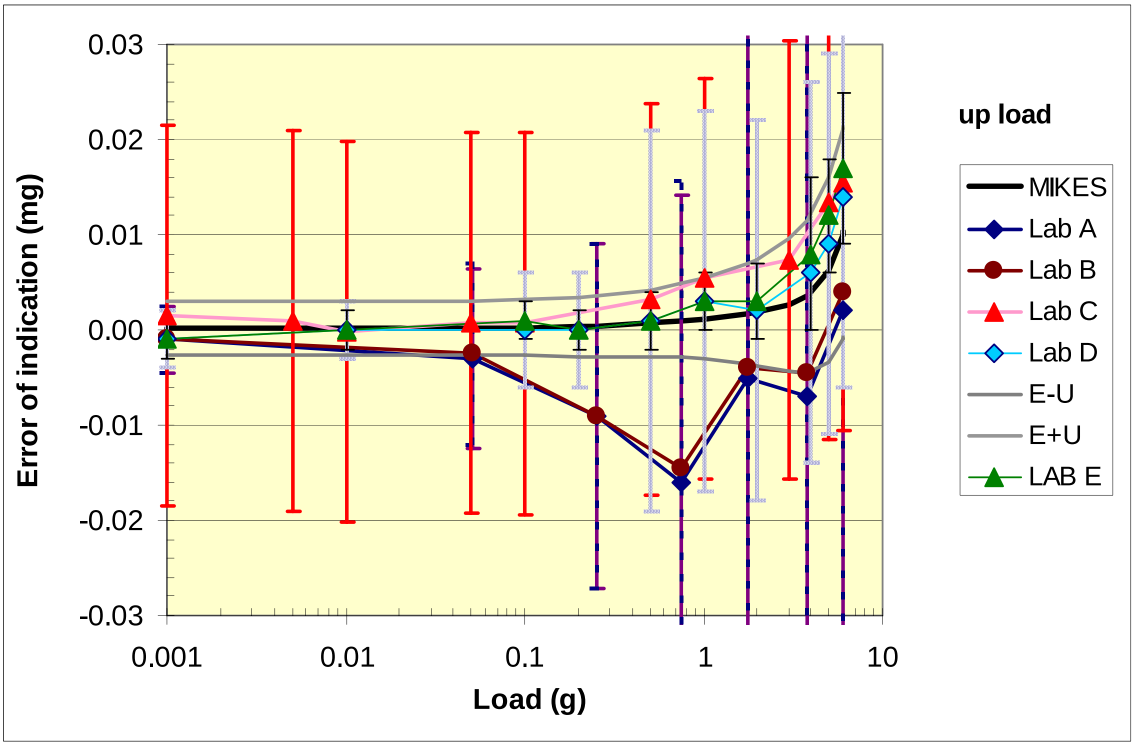


Figure 2. Results of the comparison, a) linear load scale, b) logaritmic load scale



a)



b)

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