MITTATEKNIIKAN KESKUS

CENTRE FOR METROLOGY AND ACCREDITATION

Julkaisu J4/2000

MASS AND VOLUME COMPARISONS AT MIKES

Additional results to the EA intercomparison of weights 1 mg – 100 g (Ma1) and to the EUROMET intercomparison of ceramic spheres (EUROMET 339)

Kari Riski

Helsinki 2000

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SUMMARY

The masses of weights (1 mg, 10 mg, 500 mg, 1 g, 50 g and 100 g) and volumes of weights (1 g, 50 g and 100 g) of EA intercomparison Ma1 have been determined at MIKES. Also the mass and the volume of a 55 mm ceramic sphere of EUROMET intercomparison (Project 339) have been determined. The weights were measured during the EA intercomparison and the sphere was measured soon after the EUROMET intercomparison. The normalised deviations (E_n values) for the mass and the volume of the weights and for the volume of the sphere were below one. For the mass of the ceramic sphere the E_n value was above one ($E_n = 2,0$).

TIIVISTELMÄ

Mittatekniikan keskus (MIKES) on kalibroinut EA:n vertailussa Ma1 olleet punnukset (1 mg, 10 mg, 500 mg, 1 g, 50 g ja 100 g), sekä EUROMET vertailussa nro 339 olleen halkaisijaltaan 55 mm suuruisen keraamisen pallon. Punnukset mitattiin vertailun aikana ja pallo vertailun jälkeen. Vertailujen tulokset eivät olleet tiedossa, mittausten aikana. Vertailun tuloksista lasketut normalisoidut poikkeamat (E_n arvot) punnusten massoille ja tilavuuksille sekä keraamisen pallon tilavuudelle olivat pienempiä kuin 1. Keraamisen pallon massalle E_n arvo oli 2,0.

CONTENTS

1. WEIGHT INTERCOMPARISON (EA Ma1)

1.1 INTRODUCTION

The EA Interlaboratory comparison for Mass and Volume Ma1 was carried out between June 1996 and April 1999. The comparison was of the highest level and it was intended for laboratories which calibrate weights of OIML class E_1 and/or E_2 . Some of these laboratories are national laboratories which have been accredited. The draft report of the comparison /1/ has been circulated in EA. The measurements at MIKES were made at the beginning of September 1997. The results were send to FINAS shortly after the measurements. The reference laboratory was PTB (Germany). The reference laboratory measured the masses of the weights at the beginning and at the end of the intercomparison and two times during the intercomparison. The volumes of the weights were determined before the comparison. Altogether 20 accredited laboratories were included in the draft report. Six laboratories measured the volumes of the weights.

1.2 STANDARDS

The mass standards 100 g, 50 g and 1 g were standard cylindrical weights with lifting knobs. The mass standards 500 mg, 10 mg and 1 mg were wire weights. The weights are made of stainless steel. The measurement instructions are given in Appendix 1. The (true) masses of all weights and the volumes or densities of the weights 100 g, 50 g and 1 g were to be determined.

1.3 RESULTS

At MIKES the masses and volumes of the weights were measured following the standard procedures of MIKES. Because of limited measuring time the waiting time between volume and mass measurements was only about one week. The volume of the weights were measured by hydrostatic weighing. The mass of the 100 g weight was measured by direct comparison and the masses of the other weights were measured by subdivision using 100 g, 10 g and 100 mg weights as reference weights.

Results of the comparison are given in Tables 1 and 2. The masses and volumes of the weights and their expanded uncertainties for MIKES are copied from the calibration certificate in Appendix 2. The corresponding values for the reference laboratory (REF) come from the draft report /1/.

Table 1. Results for the masses of the weights, U is the expanded uncertainty of mass, E_n is the normalised deviation (see text).

NOMINAL	MIKES		REF		MIKES-REF	
VALUE	Mass (true)	U (k = 2)	Mass (true)	U (k = 2)	Difference	E_n
100 g	100,000 091 g	0,000 020 g	100,000 087 g	0,000 006 g	0,004 mg	0,19
50 g	50,000 046 g	0,000 015 g	50,000 036 g	0,000 004 g	0,010 mg	0,64
1 g	1,000 008 g	0,000 005 g	1,000 007 g	0,000 001 g	0,001 mg	0,20
500 mg	500,001 mg	0,004 mg	500,000 7 mg	0,000 8 mg	0,000 3 mg	0,08
10 mg	10,002 3 mg	0,001 1 mg	10,002 7 mg	0,000 4 mg	-0,000 4 mg	-0,34
1 mg	1,002 8 mg	0,001 1 mg	1,002 9 mg	0,000 4 mg	0,000 1 mg	-0,09

The mass of some of the weights changed during the comparison. The most probable reason for the change is the volume determination. The mass of the 100 g weight decreased 16 µg and the mass of the 50 g weight decreased 9 µg during the loop in which MIKES took part. Interpolated mass values of date 16.9.1997 given in the draft report were used as reference values. The draft report also gives an uncertainty for the drift of the masses. This uncertainty component is not included in the results of Table 1. The volumes of the weights were expected to be stable.

Table 2. Results for the volumes of the 1 g, 50 g and 100 g weights, U is the expanded uncertainty of volume.

NOMINAL	MIKES		REF		MIKES-REF	
VALUE	Volume	U (k = 2)	Volume	U (k = 2)	Difference	E_n
100 g	12,557 cm^3	0,003 cm^3	12,558 0 cm^3	0,001 0 cm^3	-0,001 0 cm^3	-0,31
50 g	6,277 2 cm^3	0,000 8 cm^3	6,277 0 cm^3	0,000 9 cm^3	0,000 2 cm^3	0,17
1 g	0,126 3 cm^3	0,000 4 cm^3	0,126 1 cm^3	0,000 5 cm^3	0,000 2 cm^3	0,31

The E_n values in tables 1 and 2 were calculated using the formula:

$$E_n = \frac{X_{MIKES} - X_{REF}}{\sqrt{U_{MIKES}^2 + U_{REF}^2}}$$

where X_{MIKES} is the measurement result of MIKES and X_{REF} is the measurement result of the reference laboratory and U_{MIKES} is the expanded uncertainty of MIKES and U_{REF} is the expanded uncertainty of the reference laboratory. The expanded uncertainties have been calculated using the coverage factor k = 2.

It has been generally agreed within EA that if the absolute value of E_n is less than unity then the measurement result can be accepted. If the E_n value is larger than one then either the measurement result is erroneous or the uncertainty has been underestimated.

All E_n values in Tables 1 and 2 fulfil the acceptance criteria. The uncertainties in Tables 1 and 2 are somewhat larger than the Calibration Measurement Capability (CMC) of MIKES. This is due to the limited measuring time.

2. VOLUME INTERCOMPARISON (EUROMET 339)

2.1 INTRODUCTION

The EUROMET project number 339 "Intercomparison of volume standards by hydrostatic weighing" was carried out between January 1996 and January 1999. Twelve European laboratories took part in the comparison. The comparison was organized and piloted by OFMET (Switzerland). When the intercomparison was started MIKES did not have enough facilities for the comparison. Due to some improvements in instrumentation it would now be possible take part to the intercomparison.

After the comparison was completed the co-ordinator of the project Dr. Philippe Richard kindly informed us that the standards of the project are available to us and other laboratories. One of the standards was sent to MIKES where measurements were made between 7 and 26 April 2000. The results were sent to OFMET in May 2000. The final report of the original comparison was published in August 2000 /2/.

2.2 INSTRUMENTATION AT MIKES

At MIKES the volume of a solid object is measured using the hydrostatic weighing method. The object is immersed in water. The instrumentation has been designed at MIKES. The volume of the water vessel is 15 litres. Temperature of water is measured with a PT100 rod sensor with a diameter of 5 mm. The resistance of the sensor is measured with a nanovoltmeter (HP 34420A). The temperature of the water is not controlled. It can be varied by changing the temperature of the laboratory. The water vessel is open to atmosphere. It is assumed that the water is saturated with air.

The mass of the object in water is measured with a 1 kg mass comparator (Mettler AT 1004). The resolution of the comparator is 0,1 mg. In all measurements the weight of the body in water is compared with reference weights. The reference weights are placed on the standard weighing pan of the comparator. An additional weighing pan in water is hanging from the bottom of the comparator. The water surface is penetrated with a 0,25 mm (diameter) Ta wire. The object in water can be inserted or removed from the weighing pan with a manually operated weight handler. The handler has two weighing positions.

At MIKES two density standards can be used as reference standards. One standard is distilled water. The conductivity of the water was measured and it was less than 1 µSi/cm at the beginning of the comparison. The other density standard is a silicon crystal with known density. The density is traceable to NBS (now NIST) through a certificate dated 1982. The density value at 20 °C is 2329,075 kg/m^3 with a standard uncertainty of 0,007 kg/m^3 . The volume of the crystal is about 88,7 cm^3. The shape of the crystal is a cleft cylinder. The diameter of the cylinder is about 75 mm.

2.3 TRANSFER STANDARDS

There were three volume standards in the original intercomparison. The standards were ceramic spheres made of Ekasin 2000 HIP (Si_3N_4/MgO). The nominal diameters of the spheres were 55 mm, 75 mm and 85 mm. The cubic thermal expansion coefficient of the material is $4{,}8 \cdot 10^{-6}$ 1/K. Because of expected difficulties in measuring the masses of the largest spheres only the smallest sphere was measured at MIKES.

2.4 RESULTS

The mass of the 55 mm sphere (CS 55) was measured with a 1 kg mass comparator (Mettler HK1000MC). It was not possible to measure the mass directly. An additional support on which the sphere lies during the weighing was needed. Because there were difficulties in finding such a support the aluminium piece circulating with the sphere was used. No efforts were made to remove static electricity from the ceramic sphere. It was assumed that static electricity would not affect the weighing results.

The volume of the sphere was measured by hydrostatic weighing using both distilled water and a silicon crystal as a density standard.

The results of this comparison are given in Tables 3 and 4. The medians given in /2/ were used as the reference values. The standard uncertainty of the median value is taken from /2/. It has been calculated by the formula:

$$u_{ref} = \frac{1{,}9}{\sqrt{n-1}} \operatorname{median}(|x_i - \operatorname{median}\{x_i\}|)$$

where x_i (i = 1…n) are the results of the n laboratories /2/. The mass and the volume of the sphere were assumed to be stable.

The expanded uncertainty U_{REF} was calculated by multiplying the standard uncertainty with a coverage factor $k = 2$.

Table 3. Results for the mass of the CS 55 sphere.

OBJECT	**MIKES**		**REF (EUROMET 339)**		**MIKES-REF**	
	Mass	U (k = 2)	Mass	U (k = 2)	Difference	E_n
CS 55	277 138,93 g	0,18 mg	277 139,32 g	0,076 mg	-0,39 mg	-2,0

The difference in mass –0,39 mg is larger than expected. Possible reasons for the difference are static electricity and lack of experience in weighing such spheres. No systematic difference in the mass standards of MIKES and the other laboratories is expected. The main uncertainty contributions are the mass of the standards and the mass of the support.

Table 4. Results for the volume of the CS 55 sphere.

STANDARD	**MIKES**		**REF (EUROMET 339)**		**MIKES-REF**	
AT MIKES	Volume	U (k = 2)	Volume	U (k = 2)	Difference	E_n
Water	87 165,23 mm^3	1,12 mm^3	87 165,45 mm^3	0,60 mm^3	-0,22 mm^3	-0,17
Si-crystal	87 165,00 mm^3	0,88 mm^3	87 165,45 mm^3	0,60 mm^3	-0,45 mm^3	-0,43

In the first row of Table 4 the reference standard is distilled water and in the second row of Table 4 it is the Si crystal. The difference between the two results is much less than the uncertainty.

Uncertainty budgets for volume determination at MIKES is given in Tables 5 and 6.

Table 5. Uncertainty of volume measurement of CS 55 when using water as density reference,

Parameter	**Standard uncertainty**
Mass standards	0,02 mg
Air buoyancy	0,024 mg
Comparator	0,3 mg
Water temperature	10 mK
Water density at 20 °C	0,005 kg/m^3
Mass difference	0,10 mg
Mass of CS55	0,09 mg
Combined standard uncertainty	0,56 mm^3

Table 6. Uncertainty of volume measurement of CS 55 when using Si-crystal as volume reference.

Parameter	Standard uncertainty
Mass standards	0,021 mg
Air buoyancy	0,015 mg
Comparator	0,3 mg
Water temperature	10 mK
Density of water at 20 °C	0,005 kg/m^3
Density of the Si-crystal	0,007 kg/m^3
Mass of the Si-crystal	0,05 mg
Mass of CS55	0,10 mg
Mass difference	0,06 mg
Combined standard uncertainty	0,44 mm^3

Ambient conditions during the volume determination are shown in Table 7.

Table 7. Ambient and liquid conditions during volume measurements

Parameter	Minimum	Maximum	Mean Value
Air density	1,17 kg/m^3	1,20 kg/m^3	1,179 kg/mg/m^3
Air temperature	21,8 °C	22,9 °C	22,2 °C
Air pressure	995 hPa	1016 hPa	1005 hPa
Relative Humidity	34 %RH	49 %RH	46 %RH
CO_2 content assumed			0,04 %
Liquid temperature	19,48 °C	20,80 °C	19,99 °C
Immersion depth			140 mm

2.5 DISCUSSION

The results for the volume of the sphere are close to the reference values. The discrepancy in the determination of the mass of the sphere is probably due to static electricity. The other possibility is the mass of the support used.

The volume results were recalculated using the reference mass values of Table 3 for the mass of CS 55. As a result all volumes increased by about 0,4 mm^3 and the E_n values changed from –0,2 (-0,4) to about +0,2 (0,0) when water (silicon) as density reference. Because the changes are less than the standard uncertainty of the volume determination, no definite conclusion can be derived. This also shows that the incorrect mass value does not invalidate the volume results.

3 CONCLUSIONS

The results of the comparisons show that there are no large deviations between MIKES and the other laboratories which participated in these mass and volume comparisons.

REFERENCES

/1/ *Ma1 Draft Report EA Interlaboratory Comparison 'Mass'*, Deutscher Kalibrierdienst (DKD), PTB Braunschweig, February 2000.

/2/ Philippe Richard, *Intercomparison of volume standards by hydrostatic weighing*, Euromet Project No 339, Final report, OFMET, Switzerland, August 2000.

EAL INTERLABORATORY COMPARISON Ma 1
(mass, buoyancy correction)

1. General Information

1.1 Accreditation body, responsible for the organization of the interlaboratory comparison:

Physikalisch-Technische Bundesanstalt (PTB)
DKD
Postfach 33 45
D - 38023 Braunschweig
Germany
Contact: Mr. E. Fay

Tel: 0049 531 592 8320
Fax: 0049 531 592 8305

1.2 Reference Laboratory

Physikalisch-Technische Bundesanstalt (PTB)
Laboratory 1.11: Unit of Mass
Postfach 33 45
D - 38023 Braunschweig
Germany
Contact: Dr. M. Gläser

Tel: 0049 531 592 1110
Fax: 0049 531 592 1105

2. Device

The device to be circulated consists of the following items, packed in a box of approx. dimensions: 14 cm x 15 cm x 6 cm and weight: 2 Kg.

Type:	3 cylindrical mass standards with knob (100g, 50 g, 1 g) and 3 polygonal wire-shaped mass standards (500 mg, 10 mg, 1 mg)
Material:	stainless steel
manufacturer:	Mettler-Toledo
Control mark:	54/94 (on the box)
Nominal values:	100g, 50 g, 1 g, 500 mg, 10 mg, 1 mg

3. Transportation

The device should be transported by car, train or plane whichever appears safest for the device. The items should be unpacked by an expert for mass calibration immediately upon receipt by the calibration laboratory and checked for damage, in particular, a visual inspection of the surfaces should be made and the results noted on the Receipt Form.

4. Handling and storing

The mass standards should be manipulated with appropriate pincers. After arrival, they shall be removed from the transportation box and stored under bell jars. The standards shall be stored for five days in the laboratory, preferably in the balance room, before starting the mass determinations. If the laboratory determines the volume or density by the hydrostatic method, this should be done first. After that, the standards shall be stored for five days in the laboratory before the mass determinations.

5. Measurements

The measurements have to be performed according to the normal procedure as agreed with the Accreditation Body.

In particular, the (true) mass of each of the six standards has to be determined. The determination of mass includes the correction for air buoyancy using the air density determined for the conditions during the calibration and using the volume or density of the standard. Laboratories accredited for volume or density determination of mass standards shall determine the volumes or densities of the 100 g- 50 g- and 1 g-standards. If the volume or density cannot be determined by the calibrating laboratory, it will be communicated by the organizing Accreditation Body on request.

6. Circulation Scheme

At the beginning and at the end of the circulation scheme, measurements will be performed by the Reference Laboratory.

If there is any delay in a country, the number of participating laboratories within that country shall be reduced, so that transport to the next country can be carried out in time according to the fixed schedule.

7. Report

Each participant shall send the completed Receipt Form to its Accreditation Body immediately upon receipt of the device. The participant shall send a formal certificate with the summary of results and the additional information sheets Nos. 1–7 within two weeks after the calibration to the office of its Accreditation Body.

The Accreditation Body will send copies of these documents, together with a summary to the organizing Accreditation Body. In case of damage to the device, the organizing Accreditation Body shall be informed as soon as possible.

8. Uncertainty

This calculation shall be carried out according to the method prescribed in WECC Doc. 19-1990.

APPENDIX 2

MITTATEKNIIKAN KESKUS
CENTRE FOR METROLOGY AND ACCREDITATION

KANSALLINEN MITTANORMAALILABORATORIO
NATIONAL STANDARDS LABORATORY

PL 239 (P.O.B), FIN-00181 Helsinki, Finland
Tel. int+358 9 616761*, Fax: int+358 9 6167467
Kotisivu: www.mikes.fi , Sähköposti: etunimi.sukunimi@mikes.fi

KALIBROINTITODISTUS
KALIBRERINGSBEVIS
Certificate of Calibration

Nro/nr/*no.*: M-M 1015

Tilaaja - Uppdragsgivare: *Customer*	EAL
Kalibroitu laite - Kalibrerat instrument: *Calibrated instrument*	Weight set: 1 mg, 10 mg, 500 mg, 1 g, 50 g and 100 g
Valmistaja - Tillverkare: *Manufactured by*	Mettler-Toledo AG, Switzerland
Tyyppi - Typ: *Model*	OIML-class: E
Sarjanumero - Serienummer: *Serial number*	BUND PTB 1.11 - 5494
Päiväys - Datum: *Date*	Helsinki 12. September 1997
Allekirjoitukset - Underskrifter: *Signatures*	Erikoistutkija Kari Riski

Liitteitä - Bilagor:
Documents attached -

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Certificate of Calibration: M-M 1015
12.9.1997
Calibrated by KR and EJ

The calibrated objects are knob weights (100 g, 50 g, 1 g) and wire weights (1 mg, 10 mg, 500 mg). The weights are made from stainless steel. The weights are in a wooden box. The volumes of the 100 g, 50 g and 1 g weights were determined by hydrostatic weighing using distilled water as the density reference.

The weights were calibrated 1-10.9.1997 under the following environmental conditions: temperature 21±1 °C, air pressure 982-1028 hPa and relative humidity about 52 %RH. The weights were calibrated against the reference standards P9 100g 6, P9 100g 7, P18 10 g, P8 10 g, P19 100 mg and P8 100 mg either by direct comparison (100 g) or by subdivision. The weighings were made with the mass comparators of MIKES.

The uncertainty of mass measurement contains the following components: reference standards, weighing instrument, repeatability of measurements and air buoyancy. The expanded uncertainty has been obtained by multiplying the combined standard uncertainty by the coverage factor k=2. For normal distribution this corresponds to a coverage probability of 95 %. If several weights are used simultaneously, their uncertainties should be added in a linear way. The mass standards used are traceable to the national kilogram Pt-Ir prototype No 23.

Measurement results:

Volume determination:

Nominal value	Volume (20 °C)	Uncertainty (k=2) ±
100 g	12,557 cm^3	0,003 cm^3
50 g	6,277 2 cm^3	0,000 8 cm^3
1 g	0,126 3 cm^3	0,000 4 cm^3

Mass determination:

Nominal value	Mass (true mass)	Uncertainty (k=2) ±
100 g	100,000 091 g	0,000 020 g
50 g	50,000 046 g	0,000 015 g
1 g	1,000 008 g	0,000 005 g
500 mg	500,001 mg	0,004 mg
10 mg	10,002 3 mg	0,001 1 mg
1 mg	1,002 8 mg	0,001 1 mg

For the 500 mg, 10 mg and 1 mg weights the density 7950±50 kg/m^3 was assumed.

KR

Julkaisut 1999 - 2000

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

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