Final Report

on EURAMET key comparison (**EURAMET.M.M-K2.5**) of 10 kg mass standards in stainless steel
(EURAMET Project code: EURAMET 1222)

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1 Introduction

This report describes a European regional key comparison of a stainless steel 10 kg standard as a multiple of the kilogram carried out under the auspices of EURAMET and designated Project 1222. This comparison is also a [KCDB Regional Key Comparison, registered as EURAMET.M.M-K2.5.

The objectives of this comparison are to check the measurement capabilities in the field of mass of the participating national laboratories, to facilitate the demonstration of metrological equivalence between the laboratories in Europe, and to check or support the validity of quoted calibration measurement capabilities (CMC).

This comparison provides link to CCM.M-K2 ([2]).

BEV (Austria) was the pilot laboratory and the provider of the transfer standard.

Table 1: List of Participating Laboratories

Laboratory		**Country**
Bundesamt für Eich- und Vermessungswesen	BEV	Austria
Magyar Kereskedelmi Engedélyezési Hivatal	MKEH	Hungary
Institute of Metrology of Bosnia and Herzegovina	IMBiH	Bosnia and Herzegovina
Montenegrin Bureau of Metrology	MBM	Montenegro
General Directorate of Metrology, Tirana, Albania	DPM	Albania
Physikalisch-Technische Bundesanstalt	PTB	Germany

The Institute of Kosovo Dept. of Metrology (MTI), Kosovo UNSCR 1244/99 participated in the comparison but their result does not appear in this report (see EURAMET 1222 report).
The final time schedule is listed in Table 2.

[1] Magyar Kereskedelmi Engedélyezési Hivatal, 1124 Budapest, Németvölgyi út 37-39.
[2] General Directorate of Metrology, “Sami Frasheri” Str. No. 33 Tirana, Albania
[3] Physikalisch-Technische Bundesanstalt (PTB) Darstellung Masse, Bundesallee 100 38116 Braunschweig Germany
[4] Institute of Metrology of Bosnia and Herzegovina, Dolina 6, 71000 Sarajevo
[5] Montenegrin Bureau of Metrology, Montenegro, Kralja Nikole 2, XM-81 000 Podgorica MONTENEGRO
[6] Bundesamt für Eich und Vermessungswesen, Arltgasse 35, 1160 Wien

Originally all measurements were scheduled to be finalised in four month, but due to the difficulties outlined below, it lasted more than 16 month including all the necessary measurements to determine the drift of the transfer standards.
According to the original plan, MKEH (Hungary) and BEV were to act as link laboratories. After finishing the measurements (dated 18.02.2013) the first calculation of the reference value was made. There was no discrepancy between the link laboratories (BEV, MKEH) but the results were not in as good agreement as expected. The value of MKEH after the correction applied based on the previous key-comparison (EUROMET.M.M-K2) was unexpected. After an investigation by MKEH the cause has been identified. MKEH (former OMH) had slightly magnetic standards at the time of the previous key comparison, so the value of MKEH became unsuitable for this comparison. BEV participated in the same previous key comparison, but the uncertainty declared at that time was significantly overestimated therefore the uncertainty of the reference value would have been too large. To address these difficulties with the reference value it was logical to involve a new link laboratory. PTB was very helpful and performed the measurements and provided the link.

Table 2: Time schedule of the comparison

Nominal date of the measurement	Country	Comments
10.03.2012	Austria**	Calibration against 1 kg standards
05.04.2012	Austria	First measurement.
01.08.2012	Hungary	
18.09.2012	Austria	Stability measurement
11.10.2012	BIH	
15.10.2012	Montenegro	
15.01.2013	Albania	Measurements were delayed*
18.02.2013	Austria	Stability measurement
05.04.2013	Germany	Link laboratory
19.04.2013	Austria	Stability measurement
24.07.2013	Austria**	Calibration against 1 kg standards

* After the measurements were completed in Kosovo instead of the traveling standard another weight was delivered to Albania. Unfortunately Albania completed the measurements with the wrong standard, so they had to repeat the measurements with the correct travelling standard.
** Austria also calibrated before and after the scheduled measurements the transfer standard and the E_1 10 kg standards used for monitoring against 1 kg standards.

List of contact persons for the comparison of the different participants

Table 3: List of contact persons

DPM	Defrim	**Bulku**	**Albania**	General Directorate of Metrology "Sami Frasheri" Str. No. 33 Tirana, Albania	**Phone: +355 42 233 174**	defrim.bulku@dpmk.gov.al
MBM	Tamara	**Boskovic**	**Montenegro**	Montenegrin Bureau of Metrology, Montenegro, Kralja Nikole 2, XM-81 000 Podgorica	**Phone: +382 20 643 345**	tamara.boskovic@metrologija.gov.me
IMBiH	Sejla	**Alisic**	**Bosnia and Herzegovina**	Institute of Metrology of Bosnia and Herzegovina, Dolina 6, 71000 Sarajevo	**Phone: +387 (0) 33 565-682**	sejla.alisic@met.gov.ba
BEV	Zoltan	**Zelenka**	**Austria**	Bundesamt für Eich und Vermessungswesen, Arltgasse 35, 1160 Wien	**Phone: +43 1 21110 6637**	zoltan.zelenka@bev.gv.at
MKEH	Csilla	**Vámossy**	**Hungary**	Magyar Kereskedelmi Engedélyezési Hivatal, 1124 Budapest, Németvölgyi út 37-39.	**Phone: + 36 1 4585 947**	vamossycs@mkeh.hu
PTB	Michael	**Borys**	**Germany**	Physikalisch-Technische Bundesanstalt (PTB) Darstellung Masse Bundesallee 100 38116 Braunschweig Germany	**Phone: +49 (0)531 592 1110**	Michael.Borys@ptb.de

Description of the transfer standard

A 10 kg stainless steel mass standard of the OIML design has been circulated among the participants.

The density and the magnetic susceptibilities of the mass standard have been determined by the pilot laboratory. The density of the mass standard was measured by hydrostatic weighing. The reference standard for the density measurements was a silicon sphere traceable to PTB (Germany).

The transportation case was a plastic case with the content described in the Contents List. It was the responsibility of each laboratory to organize the transport to the next participant - by hand-carrying and ensuring that all necessary customs and importation documents (ATA Carnet, where needed) were in order.

Data for mass standards determined at the pilot laboratory

The BEV determined the density of the weight by hydrostatic weighing, corresponding to method A (hydrostatic comparison, described in B 7.4 OIML R 111-1_2004).

Table 4: Data for mass standard (density and volume)

nominal value	density ρ	uncertainty u_ρ	Volume V	uncertainty u_v
10 kg	7958,46 kg m^{-3}	0,32 kg m^{-3}	1256,52 cm^3	0,05 cm^3

The BEV also determined the magnetic susceptibility of the weight. There were no values determined for the permanent magnetic polarisation.

Table 5: Data for mass standards (magnetic susceptibility)

nominal value	magnetic susceptibility χ	uncertainty $u\chi$
10 kg	$\leq$0,004	0,001

3 Summary of results reported by the participants

Stability of the transfer standards

The transfer standard was measured in four periods (out of a total of 12 measurements – see Annex: table 6 and fig. 1) by the pilot laboratory to check the stability. The expanded uncertainty of these measurements was less than 0,06 mg. Initially an increase of the mass was detected. The basis of the change was probably contamination on the bottom of the weight. The exact cause and the time cannot be identified.

The bottom of the weight was cleaned with a soft dry cloth at PTB before their measurements were performed. BEV measured -0,27 mg change before and after the measurements at PTB. This instability is negligible in relation with the measurements uncertainty of most of the participants.

As agreed among the participants, no corrections have been applied to compensate for this contamination. The standard uncertainty from this change (modelled as a rectangular distribution) is 0,075 mg. The standard uncertainty of a stability measurement performed by the pilot is 0,03 mg.

The pilot laboratory has calibrated the transfer standard and the three 10 kg E_1 standards against 1 kg standards before (10/3/2012) and after (14/7/2013) this comparison. All the three 10 kg E_1 standards gained weight during this time. The average of this change was +0,35 mg. This change was bigger than expected. The cause of it could be that the volume calibrations of these weights were completed on 16/01/2012 and the weights had not fully stabilised after the immersion in water.

This drift of the travelling standard was modelled by a linear equation in time based on the stability measurements by BEV.

The summary of the stability measurements of the travelling standards at BEV is in table 6 and can be seen in figure 1.

Measured values of mass and uncertainties

For each participant the results have been expressed as the reported mass value, (m_P) and the associated expanded uncertainty. For the pilot the value of the very first measurement was included in the evaluation. The results are shown in Table 7 alongside their corresponding expanded uncertainty (k=2).

Calculation of reference value and uncertainty

The reference value of this comparison is the measured value of the link laboratory (PTB) corrected with the result of the degree of equivalence according to the results given in the report of CCM.M-K2. The difference between the result of PTB and the reference value was $(m\text{-}m_{ref})_{\mathrm{PTB,CCM.M\text{-}K2}}$= -0,03 mg with the assigned expanded uncertainties of 0,34 mg with a level of confidence of 95 %. PTB measured 10 kg + 3,46 mg in this comparison with the expanded uncertainty of 0,30 mg. This value was corrected with the drift of the travelling standard.

Date	Measured mass
03.10.12	10 kg + 2,87 mg
07.24.13	10 kg + 3,17 mg

$$m_{Drift}=0{,}000584 \cdot \Delta t \text{ [mg]},$$

where Δt is the time interval in days between the reference measurement (PTB) and the measurement performed by the participating laboratory.

The reference value in this comparison is

$$m_{ref}=m_{PTB}-(m-m_{ref})_{PTB,CCM.M\text{-}K2}+m_{Drift}$$

With this definition, for each participant, m_{ref} is equivalent to the KCRV of the CCM.M-K2.

The uncertainty in the reference value has been calculated with the following assumptions:

- The correlation coefficient associated with a measured value provided by the linking laboratory in this comparison and the CCM.M-K2 is 0,1. This value is the estimation of PTB based on the relative long time between the CCM.M-K2 comparison and this comparison. Both measurements were performed under different conditions regarding the measuring instruments, the reference standards and the traceability chain.
- The correlation associated with measured values provided by the participant and the linking laboratory is negligible.
- Uncertainty of the instability of the travelling standard ($u(m_{inst})=0{,}08$ mg) including the drift, the contamination and the uncertainty of the monitoring.

$$u^2(m_{ref}) = u^2(m_{PTB}) + u^2[(m-m_{ref})_{PTB,CCM.M\text{-}K2}] - 2\cdot cov\,[m_{PTB}, (m-m_{ref})_{PTB,CCM.M\text{-}K2}] + u^2(m_{inst})$$

The calculated reference value is: 10 kg + 3,49 mg with the expanded uncertainty of 0,46 mg at the time of the measurement by PTB.

Mass differences

In order to compare the values of the participants it is necessary to link them to the reference value of the comparison CCM.M-K2 through the link laboratory (PTB).
The mass difference between a participant and the key comparison reference value (KCRV) of the CCM.M-K2 value is calculated from:

$$\Delta m_P = m_P - m_{ref}$$

The uncertainties have been calculated in accordance with the GUM [1]. The uncertainty of the difference between a participant's measurement and the reference value is made up of two components:

- the uncertainty in the reference value $u(m_{ref})$ *(including the instability of the travelling standard)*
- the uncertainty in the participant's measurement $u(m_P)$

$$u^2(\Delta m_P) = u^2(m_{ref}) + u^2(m_P)$$

The mass difference between each participant's measurement m_p and the KCRV of CCM.M-K2 along with the associated uncertainty can be found in Table 8 and seen in figure 3.

4 Degree of equivalence, mass difference and uncertainty between participants and reference value

The degree of equivalence is defined [3] by the two quantities: first the difference between the value obtained by the laboratory and the reference value and second the uncertainty of this difference at a level of 95 % confidence. These values are given in Table 8 for each participating laboratory.

6. Discussion

The objectives of the present comparison were to check the measurement capabilities in the field of mass of the participating national laboratories, especially the South East European countries, to facilitate the demonstration of metrological equivalence between the laboratories in Europe, and to check or support the validity of quoted calibration measurement capabilities (CMC).

The results of this comparison are linked to the Key Comparison CCM.M-K2.

For an acceptable measurement result in a comparison with a known reference value, the condition $|m_p - m_{ref}| < U(m_p - m_{ref})$ should be fulfilled. For all participant, the difference from the reference value is less than the expanded uncertainty in this difference.
See Table 8 for E_n values. $E_n = (m_p - m_{ref})/U(m_p - m_{ref})$.
Figure 4 provides an overview of the measurements related to this comparison.

8 References

[1] Guide to the Expression of Uncertainty in Measurement, BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, 1993

[2] Final Report on CIPM key comparison of multiples and submultiples of the kilogram (CCM.M-K2), Metrologia 40 (2003)

[3] Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes, BIPM, Paris, 14 October 1999

Annex:

Table 6: Stability measurements of the travelling standards at BEV

Date of the measurement	Mass of the transfer standard	Air density [kg/m^3]
10.03.2012*	10 kg + 2,87 mg	1,17
05.04.2012	10 kg + 2,95 mg	1,15
14.04.2012	10 kg + 2,91 mg	1,15
21.04.2012	10 kg + 2,89 mg	1,15
26.04.2012	10 kg + 2,89 mg	1,17
30.04.2012	10 kg + 2,92 mg	1,17
24.05.2012	10 kg + 2,94 mg	1,17
09.08.2012	10 kg + 3,08 mg	1,18
18.09.2012	10 kg + 3,09 mg	1,16
15.02.2013	10 kg + 3,37 mg	1,16
21.02.2013	10 kg + 3,39 mg	1,17
19.04.2013	10 kg + 3,12 mg	1,17
20.04.2013	10 kg + 3,11 mg	1,17
24.07.2013*	10 kg + 3,17 mg	1,17

*These measurements were performed against 1 kg standards.

Figure 1: Stability of the transfer standard

Values by BEV calibrated against 1 kg standards. (Blue).
Mass values by monitoring against the three E_1 standards corrected for the drift of the standards.
The error bars are the expanded uncertainties of the monitoring of the transfer standard.
Note: since the trend line crosses the value measured after some contamination was removed ("cleaning") it can be assumed that most of the contamination has been removed.

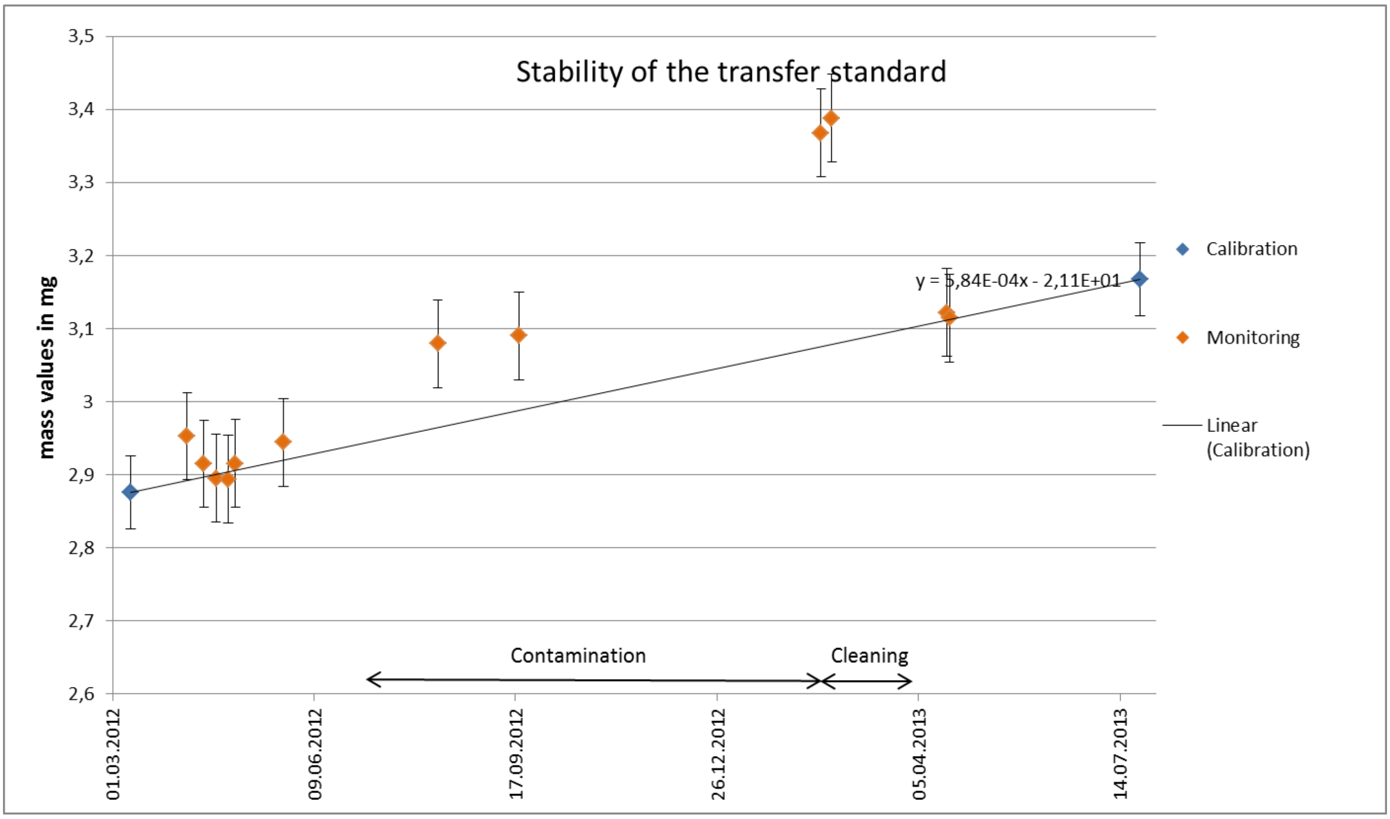


Table 7: Reported true mass value (m_P) and the associated expanded uncertainty.

Laboratory	Reported true mass	U_P (k=2)
BEV	10 kg + 2,95 mg	0,46 mg
MKEH	10 kg + 2,94 mg	0,70 mg
IMBIH	10 kg + 2,80 mg	1,6 mg
MBM	10 kg + 3,85 mg	1,7 mg
DPM	10 kg + 4,0 mg	1,9 mg
PTB	10 kg + 3,46 mg	0,30 mg

Figure 2: Mass differences from the 10 kg nominal value (m_P) and the associated expanded uncertainty.

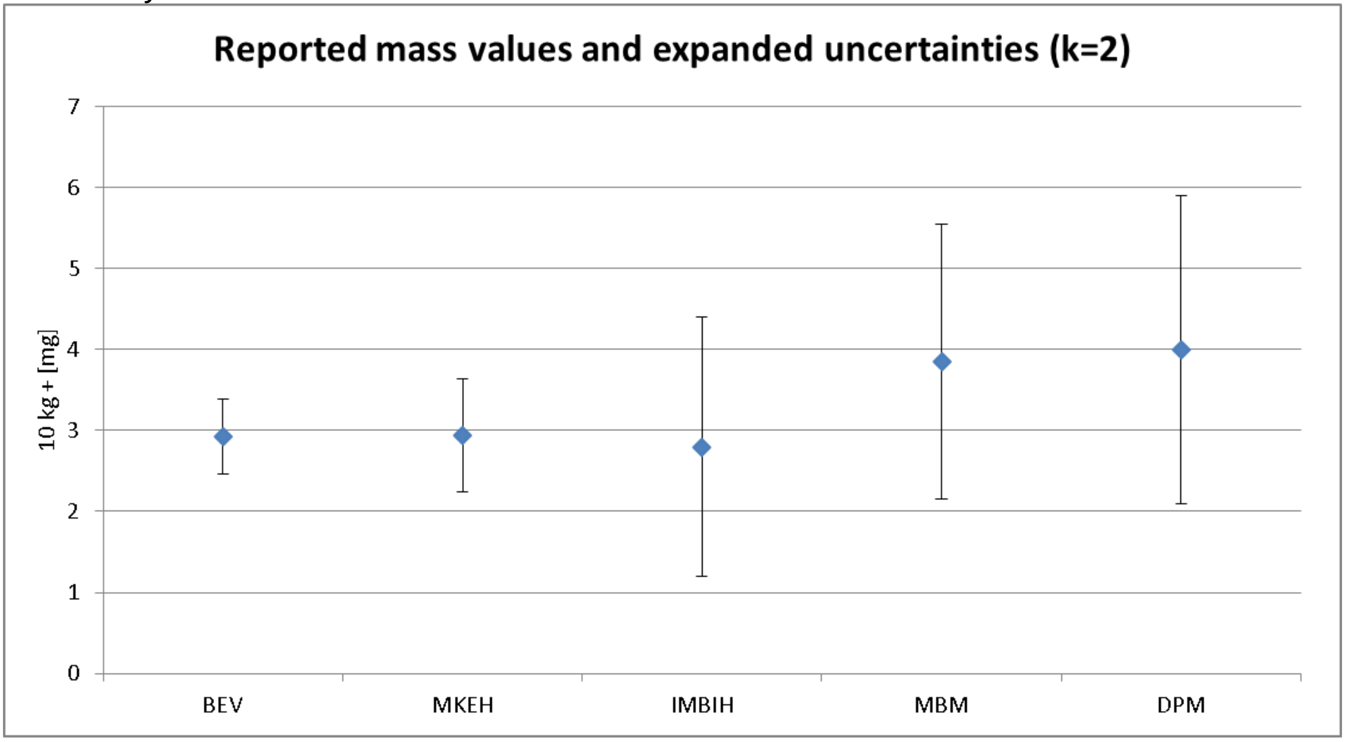


Table 8: Reference value, differences between each participant and the reference value, together with their associated uncertainties (k=2) and the E_n values.

Laboratory	m_{ref}	$m_P - m_{ref}$ [mg]	$U(m_P - m_{ref})$ (k=2) [mg]	E_n
BEV	10 kg + 3,28 mg	-0,33	0,65	-0,50
MKEH	10 kg + 3,35 mg	-0,41	0,84	-0,48
IMBIH	10 kg + 3,39 mg	-0,59	1,7	-0,35
MBM	10 kg + 3,39 mg	0,46	1,8	-0,26
DPM	10 kg + 3,44 mg	0,56	2,0	0,28

Figure 3: Differences between each participant and the reference value, together with their associated uncertainties (k=2).

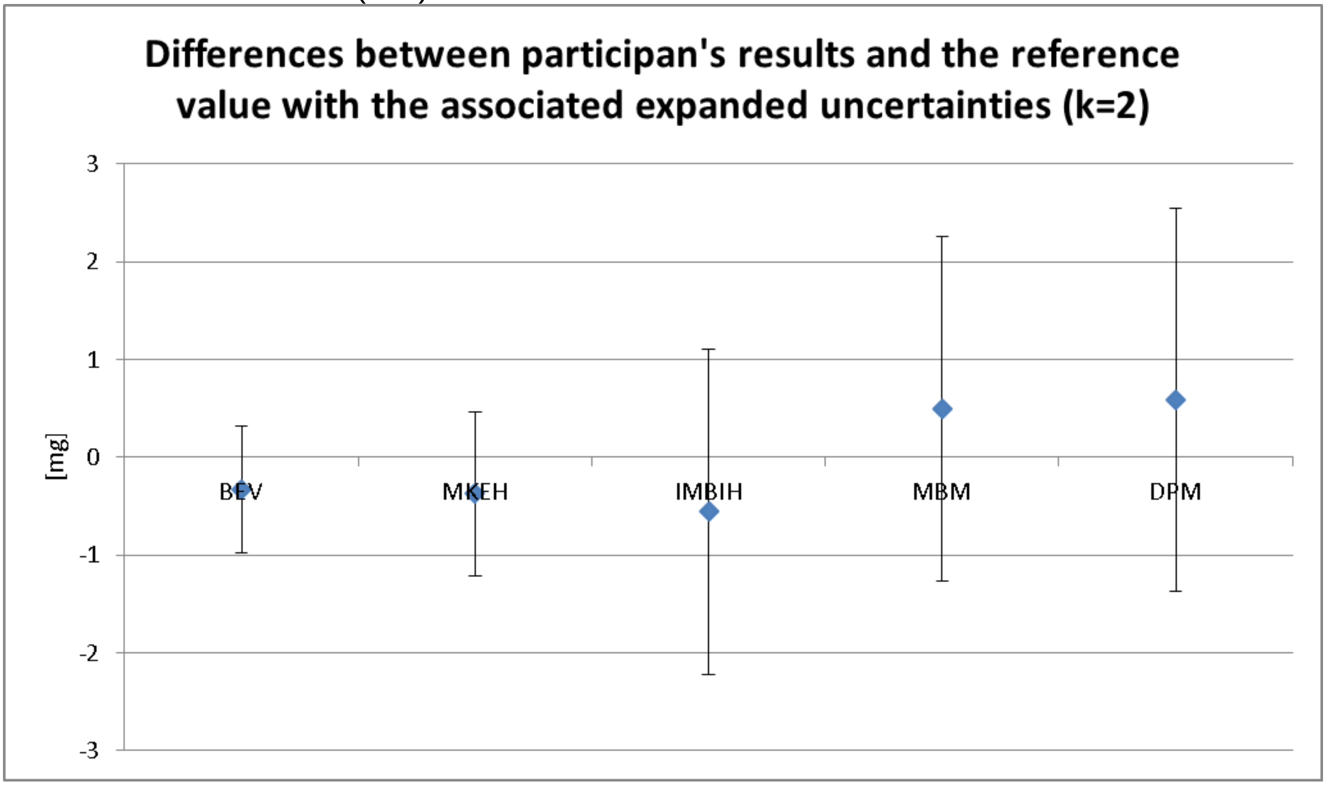
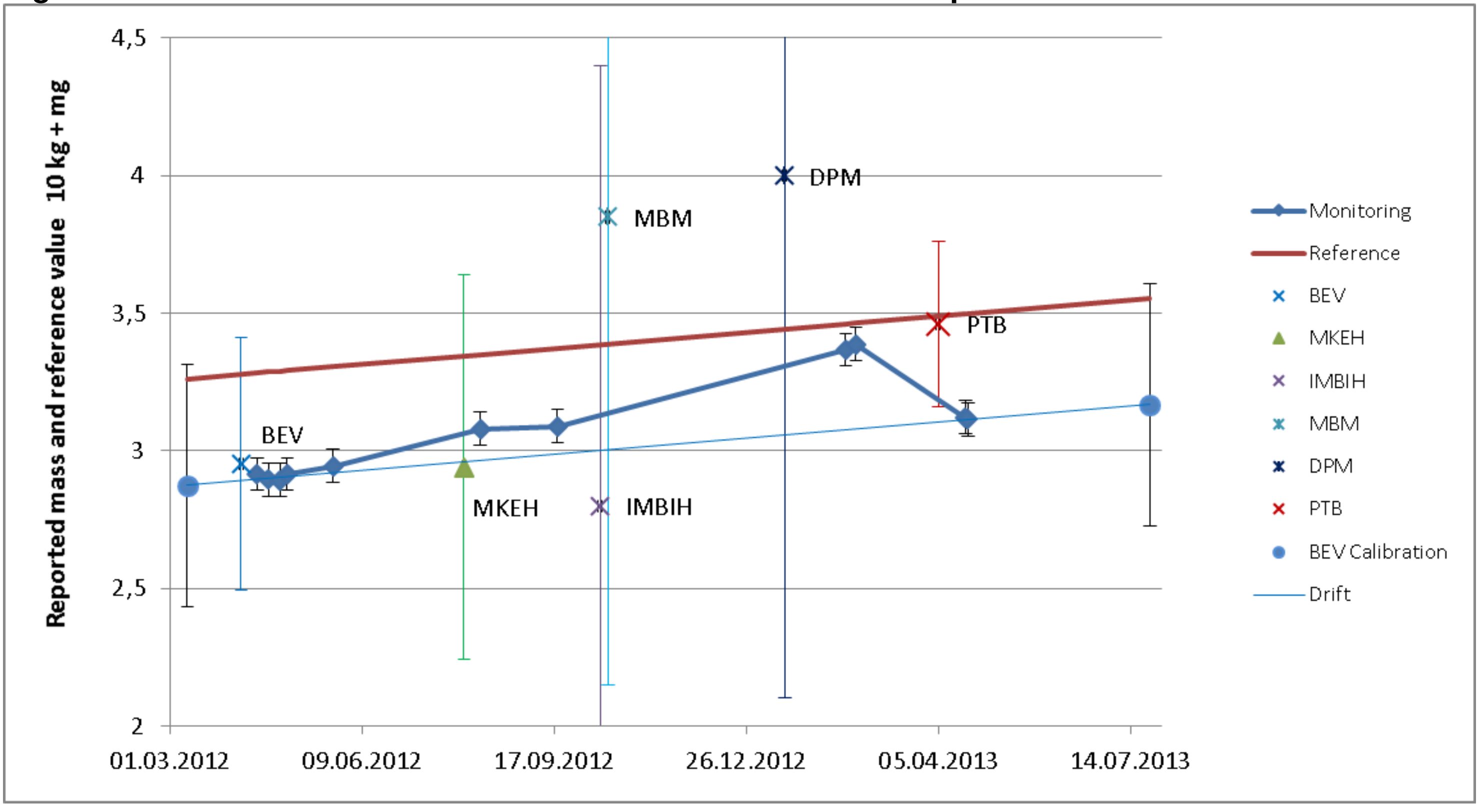


Figure 4: Overview of all the measurements related to the comparison



Blue dots: Values by BEV calibrated against 1 kg standards.
Thick blue line represents the mass values by monitoring against the three 10 kg E_1 standards. The error bars are the expanded uncertainties of the monitoring of the transfer standard. (They do not include the uncertainty of the three 10 kg E_1 standards).
X represents the measured values of the participants.
Red X is the measurements of PTB as reference laboratory.
Red line represents the reference value of the comparison.
All uncertainties are the expanded (k=2) standard uncertainties.