

Final report on EURAMET comparison on 1 kg stainless steel mass standards

EURAMET.M.M-K4.2015

(Project number: EURAMET 1346)

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Abstract

In order to demonstrate the equivalence in calibration of mass standards among National Metrology Institutes (NMIs) of EURAMET this key comparison (KC) on 1 kg stainless steel mass standards has been carried out under the auspices of EURAMET. The comparison was undertaken with reference to the International Prototype Kilogram (IPK) as the definition of the unit of mass. The overall result shows good consistency among the participants.

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1 Introduction

This regional key comparison, EURAMET.M.M-K4.2015 was based on the decision of EURAMET technical committee (TC-Mass) meeting held in 2014. The scope was calibration of 1 kg stainless steel mass standards against the secondary stainless steel mass standards of the participants.

The comparison was carried out using a protocol similar to the Technical Protocol of CCM.M-K4 [1] with respect to the rules for measurement comparisons in the CIPM MRA [2] and EURAMET [3].

The comparison was piloted by BEV - Bundesamt für Eich- und Vermessungswesen. A support group was composed of the EURAMET TC-Mass contact persons at DFM, METAS, NPL and PTB. The role of the support group was to assist the pilot laboratory in decision making, problem solving encountered during the process of the comparison and for compiling the draft reports. The comparison was planned in four petals.

2 Participants

Table 1. Information on the participants

National Institute of Metrology	Acronym	Country
Bureau International des Poids et Mesures	BIPM	-
Bundesamt für Eich- und Vermessungswesen	BEV	Austria
Government Office of the Capital City Budapest	BFKH	Hungary
Bulgarian Institute of Metrology	BIM	Bulgaria
Bureau of metrology	BoM	R. Macedonia
Czech Metrology Institute	CMI	Czech Republic
Danish Fundamental Metrology	DFM	Denmark
Direktorate of Measures and Precious Metals	DMDM	Serbia
Hellenic Institute of Metrology	EIM	Greece
State Office for Metrology	DZM	Croatia
Institute of Metrology of B&H	IMBiH	Bosnia and Herzegovina
Institutul National de Metrologie	INM	Romania
Instituto Português da Qualidade	IPQ	Portugal
Justervesenet	JV	Norway
Laboratoire national de métrologie et d'essais	LNE	France
Latvian National Metrology Centre Ltd.	LNMC	Latvia
AS Metrosert	AS Metrosert	Estonia
VTT Technical Research Centre of Finland	MIKES	Finland
Metrology Institute of the Republic of Slovenia	MIRS	Slovenia
National Physical Laboratory	NPL	United Kingdom
National Metrology Laboratory	NSAI	Ireland
Research Institutes of Sweden	RISE	Sweden
FPS Economy, DG Quality and Safety, Metrology Division	SMD	Belgium
Slovenský Metrologický Ústav ¹	SMU	Slovakia
TUBITAK-UME	TUBITAK-UME	Turkey
Eidgenössisches Institut für Metrologie	METAS	Switzerland
Dutch Metrology Institute	VSL	Netherlands
National Measurements Calibration Center	SASO	Saudi Arabia

¹ SMU cancelled its participation without submitting any results

3 Travelling standards

The travelling standards for each petal were pairs of stainless steel mass standards with 1 kg nominal value of different density. Twelve 1 kg stainless steel travelling standards were used, 8 of them were provided by BEV and were cylindrical and 4 were provided by Häfner Gewichte GmbH and had OIML shape.

The stability of the travelling standards had been regularly monitored by the pilot laboratory by comparisons against a group of 12 BEV stainless steel standards. Measurements were made between August 2015 and June 2018 (some additional measurements were performed in December 2018). No cleaning of the travelling standards was carried out during this time.

The mass of the standards was not adjusted. The data determined by the pilot laboratory for each mass standard is listed in table 2.

Table 2. Information on the travelling standards

Identification	Volume at 20 °C cm ³	Uncertainty (k=2) cm ³	Coefficient of cubical expansion 10 ⁻⁶ °C ⁻¹	Height of centre of mass mm	Volume magnetic susceptibility	Magnetic polarization smaller than µT
B1	124,814 82	0,000 60	45,0	27,5	0,0042	0,21
B2	124,815 22	0,000 60	45,0	27,5	0,0042	0,08
B3	124,814 21	0,000 60	45,0	27,4	0,0041	0,06
B4	124,814 91	0,000 60	45,0	27,5	0,0041	0,09
B5	125,646 40	0,000 60	47,8	27,7	0,0090	0,32
B6	125,649 41	0,000 60	47,8	27,5	0,0034	0,04
B7	127,003 80	0,000 60	47,8	27,5	0,0032	0,30
B8	125,584 94	0,000 60	47,8	27,5	0,0055	0,17
ANU	125,496 13	0,000 60	49,1	36,2	0,0035	0,13
ANT	125,498 59	0,000 60	49,1	36,2	0,0032	0,04
12H	124,857 30	0,000 60	45,9	36,3	0,0041	0,04
12G	124,859 67	0,000 60	45,9	36,2	0,0041	0,10

Uncertainty of the centre of mass was less than 2 mm (k=2).

4 Circulation of the travelling standards

Two mass standards were sent to each participating institute. The comparison was carried out in four simultaneous petals. In each petal there was an extra stability measurement carried out at the pilot laboratory, which divided the petal into a and b parts. Each participant within a petal received the same travelling standards.

- | | | |
|----------|----------|--------------|
| Petal 1: | B1, B5 | (Cylinder) |
| Petal 2: | B2, B6 | (Cylinder) |
| Petal 3: | ANU, 12H | (OIML shape) |
| Petal 4: | ANT, 12G | (OIML shape) |

The mass standards B4, B8, B3 and B7 served as reserve and for control purposes.

The transportation case was a plastic case with separate holes for holding each wooden box. The wooden boxes were placed in plastic bags. The travelling standards were wrapped in clean and dust free lens paper.

Lens papers, gloves, brush and forceps were included for keeping the weights as clean as possible.

In principle, the travelling standards were to be hand-carried from one participant to the next. However, transportation by courier was allowed if it was rational.

Each laboratory had three weeks to carry out the measurements and one week to transport them to the next laboratory.

The pilot laboratory calibrated these standards against the group of 12 BEV reference weights before the circulation; in between the circulations and after all the measurements had been completed.

The laboratories BEV, BIPM, LNE, METAS and NPL served as linking laboratories to key comparison CCM.M-K4. NPL volunteered to measure two pairs of travelling standards (petals 1a and 4a). It was agreed that a better overlap between the CCM and EURAMET key comparisons would be achieved if the linking laboratories carried out their measurements sequentially. With this exception the circulation was arranged so that the transportation distance between any two successive laboratories was minimized.

In Table 3 there is an overview of the sequence of circulation of the travelling standards. The empty cells show that during that timeframe no measurements were performed. The causes of gaps are mainly customs delays and other practical reasons.

SMU (Slovenský Metrologický Ústav) cancelled its participation after carrying out their measurements without submitting any results, but did provide a short description about the technical problem they experienced with their balance.

CMI submitted an obviously discrepant measurement result. The pilot let the laboratory know that the result was discrepant without any further information. After a careful investigation CMI identified the cause and asked to repeat the measurements. CMI stated that: "The measurements had to be repeated due to the problems found on mass comparator Mettler Toledo M-One. The hook holding special wire for adjustment was touching the weighing pan not only during adjustment but also during regular measurement. The weight with nominal mass 100 mg was usually used. The measured difference with this weight was small so it was almost impossible to see any changes." CMI finally used a different balance, the Mettler Toledo AT1006 for the second measurement.

The other laboratories, whose measurements were finally identified as discrepant for the calculation, were not contacted, since their minor discrepant results were not considered as being significant by the support group.

Table 3. The sequence of the circulation and the nominal dates of measurements

Approximate date of measurements	NMIs in petals			
	Petal 1	Petal 2	Petal 3	Petal 4
20.06.2017 - 20.06.2017	BEV			
05.08.2017 - 12.08.2017	NPL			NPL
03.09.2017 - 14.09.2017	DFM	METAS	CMI*	BEV
10.10.2017 - 10.10.2017	RISE			
25.10.2017 - 22.11.2017	MIKES		MIRS	JV
13.11.2017 - 14.12.2017		BIPM	DZM	AS Metrosert
12.12.2017 - 04.01.2018	VSL	LNE		LATMB
13.01.2018 - 13.01.2018	BEV			
04.03.2018 - 06.03.2018	INM		IMBih	SMD - ENS
27.03.2018 - 30.03.2018	BIM		BoM	NSAI NML
24.04.2018 - 30.04.2018	EIM		DMDM	IPQ
19.05.2018 - 29.05.2018	SMU**			BFKH
25.06.2018 - 25.06.2018	BEV			
05.07.2018 - 05.07.2018			SASO	
11.07.2018 - 13.08.2018		TUBITAK-UME	BEV	
29.11.2018 - 29.11.2018		BEV		
20.05.2019 - 20.05.2019		CMI***		
24.05.2019 - 24.05.2019		BEV		

Notes:

- NMIs name written in bold indicate the linking laboratories. Where BEV is not written in bold indicates that the measurements carried out were served only for controlling the mass change of the travelling standards.
- * First measurements in CMI.
- ** SMU measurement. No results were submitted.
- *** Second measurements in CMI.

5 Stability of the travelling standards

The stability of the travelling standards was monitored at the BEV prior to the comparison between August 2015 and June 2017, as well as during the comparison from June 2017 to June 2018, altogether measured eight times. The masses were compared against the stainless steel standard group of BEV. After an initial period, the mass evolution of the standards became linear with the elapsed time and small enough to start the circulation among the laboratories.

The relevant mass changes are not only from the “natural” mass evolution, but from the circulation and the handling of the travelling standards.

The drift was assumed as linear during the time of the circulation and was included in the calculations (see sections 7 and 8).

6 Summary of results received from participants

Each participating laboratory was requested to provide the following information to the pilot laboratory:

- The mass value of the two travelling standards and their associated uncertainty.
- The conventional mass value of the two travelling standards and their associated uncertainty.
- The mass difference between the two travelling standards and its associated uncertainty.
- Details of the balance used in the comparison.
- Details on the mass standards used, including their traceability.
- Laboratory conditions during the measurements.
- Method of the air density calculation.
- Uncertainty calculation of the mass value.
- For linking laboratories: The values of the BIPM amendments.
- Correlation between the submitted results of the linking laboratories in this and CCM comparisons.

The uncertainties claimed by each participant had to be supported by the relevant uncertainty budgets, which followed the templates provided in the technical protocol.

The results obtained by the participants for the travelling standards are given in Section 7 (table 5). As mentioned in section 5, the stabilities of the travelling standards were satisfactory during the comparison and were included in the data analysis. The most important data regarding to the traceability of the laboratories is compiled in Table 4.

Table 4. Estimated correlations (standard uncertainties; $k=1$) due to the traceability to other laboratories

Laboratory	Traceable to	uncertainty (mg)	Standard type
BEV	BIPM	0,003	Prototype
BFKH	BEV	0,010	Stainless Steel
BIM	BIPM	0,003	Stainless Steel
BIPM	BIPM	0,003	Stainless Steel
BOM	CMI	0,010	Stainless Steel
DMDM	BIPM	0,003	Stainless Steel
CMI	BIPM	0,003	Prototype
DFM	BIPM	0,003	Prototype
EIM	BIPM	0,003	Stainless Steel
DZM	BIPM	0,003	Stainless Steel
IMBIH	BEV	0,010	Stainless Steel
INM	BIPM	0,003	Stainless Steel
IPQ	BIPM	0,003	Stainless Steel
Justervesenet	BIPM	0,003	Prototype
LATMB	DFM	0,010	Stainless Steel
LNE	BIPM	0,003	Prototype
METAS	BIPM	0,003	Prototype
Metrosert	PTB	0,003	Stainless Steel
MIKES	BIPM	0,003	Prototype
MIRS	BIPM	0,003	Stainless Steel
NPL	BIPM	0,003	Prototype
NSAI NML	BEV	0,010	Stainless Steel
RISE	BIPM	0,003	Prototype
SASO	BIPM	0,003	Prototype
SMD	BIPM	0,003	Stainless Steel
UME	BIPM	0,003	Prototype
VSL	BIPM	0,003	Stainless Steel

7 Data analysis using the results of the linking laboratories participated in CCM.M-K4 to calculate the degrees of equivalence

The mass corrections $y_i = m_i - 1 \text{ kg}$ and associated standard uncertainties $u_{\text{lab}}(y_i)$, $i = 1, \dots, 82$ reported by the 27 participants (including pilot and linking laboratories) are listed in Table 5 under the heading ‘Results’. The column headed ‘Standard’ identifies the measured mass standard, and the column headed ‘Time’ indicates the time t_i of the measurement relative to the first measurements of the comparison.

The results were analysed using the method of least squares as described in two DFM reports [6][7]. The measured quantities \mathbf{y} are assumed to be realisations of a multivariate random variable with expectation

$$E(\mathbf{y}) = \mathbf{X}\boldsymbol{\alpha},$$

where \mathbf{X} is a known design matrix characterising the measurements performed and $\boldsymbol{\alpha}$ is an unknown vector characterising the circulated travelling standards.

In the present case, $y_i = m_i - 1$ kg is a measured mass correction of a mass standard having a (small) linear drift in time t_i plus a (small) random change with expectation 0 and standard deviation $u_{\text{obj}}(y_i)$. The expectation values of the $n = 82$ mass corrections measured for the 8 circulated mass standards as listed in Table 5 are therefore modelled by the matrix below;

$$\left(\begin{array}{c} E(y_1) \\ \vdots \\ E(y_{12}) \\ E(y_{13}) \\ \vdots \\ E(y_{21}) \\ E(y_{22}) \\ \vdots \\ E(y_{33}) \\ E(y_{34}) \\ \vdots \\ E(y_{42}) \\ E(y_{43}) \\ \vdots \\ E(y_{53}) \\ E(y_{54}) \\ \vdots \\ E(y_{62}) \\ E(y_{63}) \\ \vdots \\ E(y_{73}) \\ E(y_{74}) \\ \vdots \\ E(y_{82}) \end{array} \right) = \left(\begin{array}{cccccccccccccccc} 1 & t_1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ \vdots & \vdots \\ 1 & t_{12} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & t_{13} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ \vdots & \vdots \\ 0 & 0 & 1 & t_{21} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & t_{22} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ \vdots & \vdots \\ 0 & 0 & 0 & 0 & 1 & t_{33} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{34} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ \vdots & \vdots \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{42} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{43} & 0 & 0 & 0 & 0 & 0 & 0 \\ \vdots & \vdots \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{53} & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{54} & 0 & 0 & 0 & 0 & 0 \\ \vdots & \vdots \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{62} & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{63} & 0 & 0 \\ \vdots & \vdots \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{73} & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{74} \\ \vdots & \vdots \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & t_{82} \end{array} \right) \left(\begin{array}{c} a_1 \\ a_2 \\ a_3 \\ a_4 \\ a_5 \\ a_6 \\ a_7 \\ a_8 \\ a_9 \\ a_{10} \\ a_{11} \\ a_{12} \\ a_{13} \\ a_{14} \\ a_{15} \\ a_{16} \end{array} \right)$$

In this model, the quantity t_i is the time for the measurement of the mass correction y_i , whereas the parameters a_{2j-1} and a_{2j} are intercept and slope for the assumed linear drift in mass of travelling standard no. $j = 1, \dots, 8$.

Note that the model does not include any data from the CCM.M-K4 comparison. The reason is that the results submitted to CCM.M-K4 were traceable to the mass unit “as maintained by the BIPM”, which later proved to have had a significant time dependent offset from the mass of the International Prototype of the Kilogram (IPK) [8], whereas the results submitted to EURAMET.M.M-K4.2015 are traceable to the IPK through amended calibration results of national prototypes provided by BIPM. Since the CCM decided that results of key comparisons carried out by the CCM will not be corrected [5], any correlation between results submitted to EURAMET.M.M-K4.2015 and results submitted to CCM.M-K4 has been eliminated. That is, there is no reason to believe that an offset from the KCRV observed for a linking laboratory in CCM.M-K4 should be preserved in EURAMET.M.M-K4.2015. The fact that the circulated objects are different in the two comparisons and the fact that there are no correlations between the results in the two comparisons implies that the two comparisons can be analysed independently of each other. As a consequence, EURAMET.M.M-K4.2015 was linked as requested in ref. [3] to the KCRV of CCM.M-K4 in the following way: The predicted mass values \hat{y}_i and associated standard uncertainties $u(\hat{y}_i)$, $i=1, \dots, 82$, were determined from a least squares adjustment as described in reference [7] by including only the EURAMET.M.M-K4.2015 results provided by those laboratories (including the pilot lab) which participated in the CCM.M-K4 and the EURAMET.M.M-K4.2015 comparisons (linking laboratories). This exceptional procedure was discussed and endorsed by the CCM WGD-kg and the EURAMET TC-M in 2019. The result of a full adjustment, using the results of all participants, is shown in section 8.

The measurement results included are identified in Table 5 under the heading ‘Included?’ (y = yes, n = no). The degrees of freedom of this adjustment are $\nu = 22$.

The covariance matrix Σ associated with the observed values \mathbf{y} consists of two parts: $\Sigma = \Sigma_{\text{lab}} + \Sigma_{\text{obj}}$. The first part, Σ_{lab} , is the covariance matrix contribution from the participating laboratories with elements $u_{\text{lab}}(y_i, y_j)$, whereas the second part, Σ_{obj} , is the covariance matrix contribution from the measurement object with diagonal elements $u_{\text{obj}}^2(y_i)$ and off-diagonal elements equal to 0.

The square root of the diagonal elements of Σ_{lab} are the standard uncertainties $u_{\text{lab}}(y_i)$ given in Table 5. The off-diagonal elements were calculated based on the following assumptions:

1. The correlation coefficient between measurements performed at the same laboratory at the same time t_i is $r = 0.95$.
2. The correlation coefficient between measurements performed at the same laboratory but at different times t_i and t_j is $r = 0.90$.
3. The covariances between BEV and laboratories traceable to BEV are $(0.010 \text{ mg})^2$.
4. The covariances between DFM and laboratories traceable to DFM are $(0.010 \text{ mg})^2$.
5. The covariances between CMI and laboratories traceable to CMI are $(0.010 \text{ mg})^2$.
6. The covariances between BIPM and laboratories traceable to BIPM are $(0.003 \text{ mg})^2$.

If no random change in the mass values of the circulated objects is assumed, that is $u_{\text{obj}}(y_i) = 0 \text{ mg}$, the observed chi-square value of the adjustment becomes $\chi^2_{\text{obs}} = 36$ with corresponding probability $\Pr\{\chi^2(\nu) > \chi^2_{\text{obs}}\} = 3.3\%$. This indicates that non-zero uncertainty contributions $u_{\text{obj}}(y_i)$ due to random variation in the mass of the circulated standards have to be included in the adjustment. This has been done by assuming a common uncertainty contribution $u_{\text{obj}}(y_i) = \sigma$, $i = 1, \dots, 82$ independent of the measurement object, and by adjusting σ to give an observed chi-square value χ^2_{obs} equal to the expectation value $E(\chi^2(\nu)) = \nu$ of the adjustment, i.e. $\chi^2_{\text{obs}} = 22$. This leads to the common uncertainty contribution $u_{\text{obj}}(y_i) = 0.0027 \text{ mg}$ given in Table 5.

The resulting reference values \hat{y}_i and associated standard uncertainties $u(\hat{y}_i)$, $i = 1, \dots, 82$, are listed in Table 5. For each result of the comparison a normalised deviation $d_i = (y_i - \hat{y}_i)/u(y_i - \hat{y}_i)$ has been calculated. As discussed in ref [3], results for which $|d_i| > 2$ are considered as discrepant results at a 5 % level of significance. There are four such results in Table 5, but they are only marginally discrepant.

The degrees of equivalence for the 27 participants have been calculated as the average

$$D_j = \frac{1}{n_j} \sum_{i \in L_j} y_i - \hat{y}_i, \quad j = 1, \dots, 27$$

where n_j is the number of results provided by laboratory j and L_j is the set of indices of those results. When calculating the associated standard uncertainties $u(D_j)$, the covariances between the differences $y_i - \hat{y}_i$ being averaged have been taken into account. The calculated degrees of equivalence (Table 6) are shown in Figure 1, where the error bars represent the expanded uncertainties $2u(D_i)$.

Table 5. Results reported by the participants of EURAMET.M.M-K4.2015 and reference values linked to CCM.M-K4

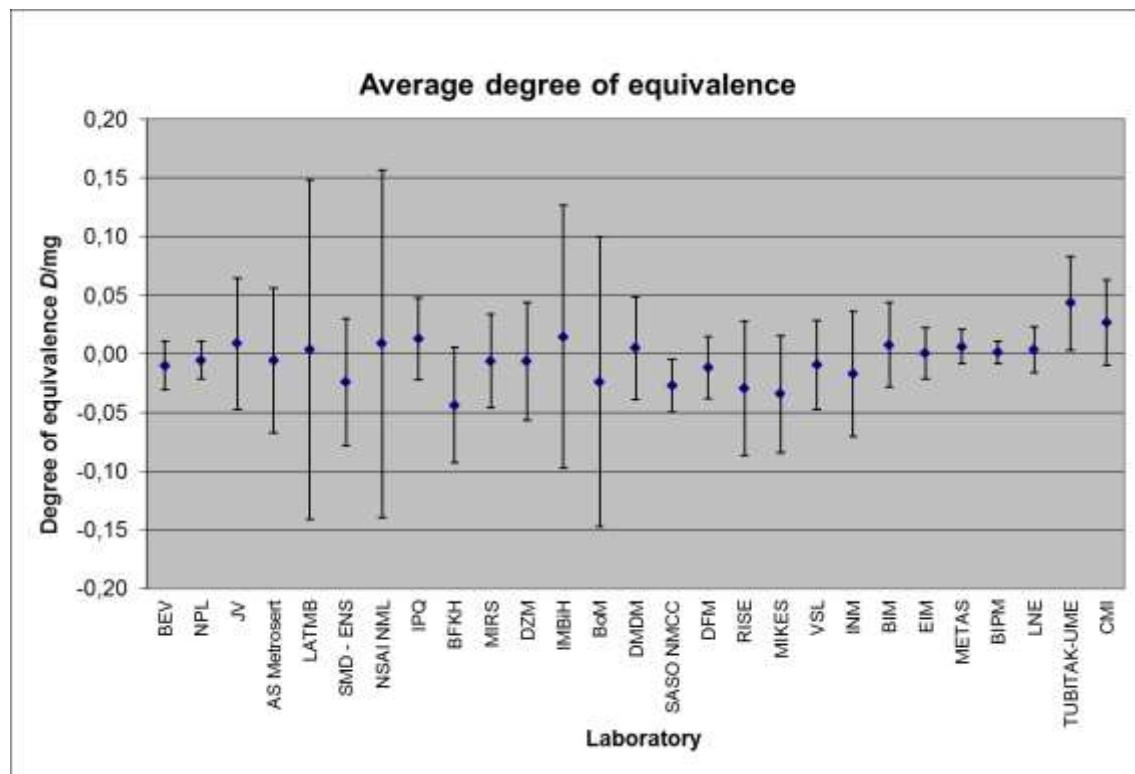
NMI	Time day	Standard ID	Results		Stability $u_{\text{obj}}(y_i)$ mg	Reference value \hat{y}_i mg	Norm. deviation d_i	Inclu- ded? (y/n)	
			y_i mg	$u_{\text{lab}}(y_i)$ mg					
BEV	0	12G	-0,1046	0,0120	0,0027	-0,0993	0,0056	-0,49	y
NPL	53	12G	-0,1071	0,0097	0,0027	-0,0953	0,0054	-1,39	y
BEV	82	12G	-0,1040	0,0122	0,0027	-0,0932	0,0053	-0,96	y
JV	155	12G	-0,0817	0,0280	0,0027	-0,0877	0,0053	0,21	n
AS Metrosert	177	12G	-0,0950	0,0310	0,0027	-0,0861	0,0053	-0,29	n
LATMB	198	12G	-0,0840	0,0731	0,0027	-0,0845	0,0054	0,01	n
BEV	207	12G	-0,0945	0,0120	0,0027	-0,0838	0,0054	-0,97	y
SMD - ENS	259	12G	-0,1140	0,0271	0,0027	-0,0799	0,0057	-1,24	n
NSAI NML	282	12G	-0,0765	0,0750	0,0027	-0,0782	0,0058	0,02	n
IPQ	314	12G	-0,0640	0,0172	0,0027	-0,0758	0,0060	0,66	n
BFKH	343	12G	-0,1240	0,0250	0,0027	-0,0736	0,0063	-2,01	n
BEV	370	12G	-0,0802	0,0120	0,0027	-0,0716	0,0065	-0,83	y
BEV	0	12H	-0,0869	0,0120	0,0027	-0,0764	0,0062	-0,99	y
MIRS	131	12H	-0,0811	0,0198	0,0027	-0,0748	0,0055	-0,31	n
DZM	148	12H	-0,0810	0,0251	0,0027	-0,0746	0,0055	-0,25	n
BEV	207	12H	-0,0854	0,0120	0,0027	-0,0739	0,0055	-1,04	y
IMBiH	259	12H	-0,0751	0,0567	0,0027	-0,0733	0,0057	-0,03	n
BoM	280	12H	-0,1000	0,0617	0,0027	-0,0731	0,0058	-0,44	n
DMDM	308	12H	-0,0480	0,0217	0,0027	-0,0727	0,0060	1,11	n
BEV	370	12H	-0,0813	0,0120	0,0027	-0,0720	0,0065	-0,89	y
SASO	380	12H	-0,0986	0,0105	0,0027	-0,0719	0,0066	-2,23	n
BEV	0	ANT	0,7988	0,0120	0,0027	0,8079	0,0056	-0,83	y
NPL	53	ANT	0,7971	0,0097	0,0027	0,8097	0,0054	-1,49	y
BEV	82	ANT	0,8071	0,0122	0,0027	0,8107	0,0053	-0,32	y
JV	155	ANT	0,8243	0,0282	0,0027	0,8132	0,0053	0,39	n
AS Metrosert	177	ANT	0,8110	0,0310	0,0027	0,8140	0,0053	-0,10	n
LATMB	198	ANT	0,8210	0,0733	0,0027	0,8147	0,0054	0,09	n
BEV	207	ANT	0,8024	0,0120	0,0027	0,8150	0,0054	-1,15	y
SMD - ENS	259	ANT	0,8022	0,0271	0,0027	0,8168	0,0057	-0,53	n
NSAI NML	282	ANT	0,8332	0,0750	0,0027	0,8176	0,0058	0,21	n
IPQ	314	ANT	0,8323	0,0172	0,0027	0,8187	0,0060	0,76	n
BFKH	343	ANT	0,7820	0,0250	0,0027	0,8197	0,0063	-1,50	n
BEV	370	ANT	0,8117	0,0120	0,0027	0,8206	0,0065	-0,86	y
BEV	0	ANU	0,6838	0,0120	0,0027	0,6941	0,0062	-0,97	y
MIRS	131	ANU	0,6881	0,0198	0,0027	0,6945	0,0055	-0,32	n
DZM	148	ANU	0,6884	0,0252	0,0027	0,6946	0,0055	-0,24	n
BEV	207	ANU	0,6829	0,0120	0,0027	0,6948	0,0055	-1,08	y
IMBiH	259	ANU	0,7257	0,0567	0,0027	0,6950	0,0057	0,54	n
BoM	280	ANU	0,6740	0,0632	0,0027	0,6951	0,0058	-0,33	n
DMDM	308	ANU	0,6800	0,0217	0,0027	0,6952	0,0060	-0,68	n
BEV	370	ANU	0,6863	0,0120	0,0027	0,6954	0,0065	-0,87	y
SASO	380	ANU	0,6675	0,0105	0,0027	0,6955	0,0066	-2,33	n
BEV	0	B1	-0,0793	0,0120	0,0027	-0,0662	0,0058	-1,20	y
NPL	46	B1	-0,0676	0,0097	0,0027	-0,0696	0,0055	0,24	y
DFM	75	B1	-0,0818	0,0130	0,0027	-0,0717	0,0054	-0,74	n
RISE	112	B1	-0,1020	0,0286	0,0027	-0,0744	0,0053	-0,95	n
MIKES	127	B1	-0,1090	0,0250	0,0027	-0,0755	0,0053	-1,32	n
VSL	175	B1	-0,0870	0,0190	0,0027	-0,0790	0,0054	-0,41	n

NMI	Time day	Standard ID	Results		Stability $u_{\text{lab}}(y_i)$ mg	Reference value		Norm. deviation d_i	Inclu- ded? (y/n)
			y_i mg	$u_{\text{obj}}(y_i)$ mg		\hat{y}_i mg	$u(\hat{y}_i)$ mg		
BEV	207	B1	-0,0972	0,0120	0,0027	-0,0813	0,0054	-1,44	y
INM	257	B1	-0,1040	0,0267	0,0027	-0,0849	0,0057	-0,70	n
BIM	283	B1	-0,0853	0,0179	0,0027	-0,0868	0,0058	0,08	n
EIM	311	B1	-0,0890	0,0099	0,0027	-0,0888	0,0060	-0,01	n
BEV	370	B1	-0,1006	0,0120	0,0027	-0,0931	0,0065	-0,72	y
BEV	0	B2	-0,0355	0,0120	0,0027	-0,0228	0,0052	-1,14	y
METAS	86	B2	-0,0159	0,0087	0,0027	-0,0234	0,0050	0,99	y
BIPM	146	B2	-0,0228	0,0066	0,0027	-0,0238	0,0049	0,21	y
LNE	178	B2	-0,0192	0,0110	0,0027	-0,0241	0,0049	0,48	y
BEV	207	B2	-0,0384	0,0120	0,0027	-0,0243	0,0049	-1,25	y
TUBITAK-UME	419	B2	0,0150	0,0200	0,0027	-0,0258	0,0052	2,00	n
BEV	527	B2	-0,0347	0,0120	0,0027	-0,0265	0,0056	-0,75	y
CMI	699	B2	0,0050	0,0173	0,0027	-0,0278	0,0063	1,81	n
BEV	703	B2	-0,0388	0,0120	0,0027	-0,0278	0,0064	-1,05	y
BEV	0	B5	1,4755	0,0120	0,0027	1,4868	0,0058	-1,04	y
NPL	46	B5	1,4862	0,0097	0,0027	1,4854	0,0055	0,09	y
DFM	75	B5	1,4712	0,0130	0,0027	1,4845	0,0054	-0,97	n
RISE	112	B5	1,4520	0,0286	0,0027	1,4834	0,0053	-1,08	n
MIKES	127	B5	1,4480	0,0250	0,0027	1,4829	0,0053	-1,38	n
VSL	175	B5	1,4700	0,0190	0,0027	1,4815	0,0054	-0,59	n
BEV	207	B5	1,4638	0,0120	0,0027	1,4805	0,0054	-1,52	y
INM	257	B5	1,4640	0,0267	0,0027	1,4790	0,0057	-0,55	n
BIM	283	B5	1,4920	0,0179	0,0027	1,4782	0,0058	0,74	n
EIM	311	B5	1,4780	0,0099	0,0027	1,4773	0,0060	0,06	n
BEV	370	B5	1,4687	0,0120	0,0027	1,4755	0,0065	-0,65	y
BEV	0	B6	1,2051	0,0120	0,0027	1,2135	0,0052	-0,76	y
METAS	86	B6	1,2175	0,0087	0,0027	1,2131	0,0050	0,58	y
BIPM	146	B6	1,2143	0,0066	0,0027	1,2128	0,0049	0,29	y
LNE	178	B6	1,2145	0,0110	0,0027	1,2127	0,0049	0,18	y
BEV	207	B6	1,1995	0,0120	0,0027	1,2125	0,0049	-1,15	y
TUBITAK-UME	419	B6	1,2570	0,0200	0,0027	1,2115	0,0052	2,23	n
BEV	527	B6	1,2052	0,0120	0,0027	1,2110	0,0056	-0,52	y
CMI	699	B6	1,2310	0,0184	0,0027	1,2101	0,0063	1,09	n
BEV	703	B6	1,1981	0,0120	0,0027	1,2101	0,0064	-1,14	y

Table 6. (Average) Degree of equivalence

NMI	D_i mg	$U(D_i)$ mg	$D_i/U(D_i)$
BEV	-0,010	0,021	-0,50
NPL	-0,005	0,016	-0,34
JV	0,009	0,056	0,15
AS Metrosoft	-0,006	0,062	-0,10
LATMB	0,003	0,145	0,02
SMD - ENS	-0,024	0,054	-0,45
NSAI NML	0,009	0,148	0,06
IPQ	0,013	0,035	0,36
BFKH	-0,044	0,049	-0,90
MIRS	-0,006	0,040	-0,16
DZM	-0,006	0,050	-0,13
IMBiH	0,014	0,112	0,13
BoM	-0,024	0,124	-0,19
DMDM	0,005	0,044	0,11
SASO	-0,027	0,023	-1,19
DFM	-0,012	0,027	-0,44
RISE	-0,030	0,057	-0,52
MIKES	-0,034	0,050	-0,69
VSL	-0,010	0,038	-0,26
INM	-0,017	0,053	-0,32
BIM	0,008	0,036	0,21
EIM	0,000	0,022	0,01
METAS	0,006	0,015	0,41
BIPM	0,001	0,009	0,13
LNE	0,003	0,020	0,17
TUBITAK-UME	0,043	0,040	1,08
CMI	0,027	0,036	0,74

Figure 1. (Average) degrees of equivalence for the 27 participants of key comparison EURAMET.M.M-K4.2015. The error bars indicate the expanded uncertainties $U(D_i)$.



8 Data analysis including all the laboratories to calculate the reference value

A data analysis has been performed by including all laboratories, except those classified as discrepant, in the calculation of the reference values, keeping all input data unchanged. The result of this analysis is shown in Table 7. Three results have been classified as discrepant at 5 % level of significance. The measured mass corrections and the calculated reference values are shown graphically in Figure 2-9.

In this analysis, the degrees of freedom of the least squares adjustment are $v = 63$. The observed chi-square value is $\chi^2_{\text{obs}} = 70$ with associated probability $\Pr\{\chi^2(v) > \chi^2_{\text{obs}}\} = 24\%$.

The calculated degrees of equivalence (Table 8) are shown in Figure 10, where the error bars represent the expanded uncertainties $2u(D_i)$.

Table 7. Results reported by the participants and regional reference values of EURAMET.M.M-K4.2015.

NMI	Time day	Standard ID	Results		Stability $u_{\text{obj}}(y_i)$ mg	\hat{y}_i mg	$u(\hat{y}_i)$ mg	Norm. deviation d_i	Inclu- ded (y/n)
			y_i mg	$u_{\text{lab}}(y_i)$ mg					
BEV	0	12G	-0,1046	0,0120	0,0027	-0,0985	0,0051	-0,55	y
NPL	53	12G	-0,1071	0,0097	0,0027	-0,0954	0,0048	-1,33	y
BEV	82	12G	-0,1040	0,0122	0,0027	-0,0936	0,0047	-0,90	y
JV	155	12G	-0,0817	0,0280	0,0027	-0,0893	0,0045	0,27	y
AS Metrosert	177	12G	-0,0950	0,0310	0,0027	-0,0880	0,0045	-0,23	y
LATMB	198	12G	-0,0840	0,0731	0,0027	-0,0867	0,0045	0,04	y
BEV	207	12G	-0,0945	0,0120	0,0027	-0,0862	0,0045	-0,73	y
SMD - ENS	259	12G	-0,1140	0,0271	0,0027	-0,0831	0,0047	-1,15	y
NSAI NML	282	12G	-0,0765	0,0750	0,0027	-0,0817	0,0047	0,07	y
IPQ	314	12G	-0,0640	0,0172	0,0027	-0,0798	0,0049	0,94	y
BFKH	343	12G	-0,1240	0,0250	0,0027	-0,0780	0,0051	-1,87	y
BEV	370	12G	-0,0802	0,0120	0,0027	-0,0764	0,0052	-0,34	y
BEV	0	12H	-0,0869	0,0120	0,0027	-0,0775	0,0056	-0,86	y
MIRS	131	12H	-0,0811	0,0198	0,0027	-0,0738	0,0048	-0,37	y
DZM	148	12H	-0,0810	0,0251	0,0027	-0,0734	0,0048	-0,31	y
BEV	207	12H	-0,0854	0,0120	0,0027	-0,0717	0,0047	-1,21	y
IMBiH	259	12H	-0,0751	0,0567	0,0027	-0,0703	0,0049	-0,08	y
BoM	280	12H	-0,1000	0,0617	0,0027	-0,0697	0,0050	-0,49	y
DMDM	308	12H	-0,0480	0,0217	0,0027	-0,0689	0,0052	0,98	y
BEV	370	12H	-0,0813	0,0120	0,0027	-0,0672	0,0057	-1,29	y
SASO	380	12H	-0,0986	0,0105	0,0027	-0,0669	0,0058	-2,74	n
BEV	0	ANT	0,7988	0,0120	0,0027	0,8055	0,0053	-0,61	y
NPL	53	ANT	0,7971	0,0097	0,0027	0,8080	0,0049	-1,25	y
BEV	82	ANT	0,8071	0,0122	0,0027	0,8094	0,0048	-0,20	y
JV	155	ANT	0,8243	0,0282	0,0027	0,8128	0,0046	0,41	y
AS Metrosert	177	ANT	0,8110	0,0310	0,0027	0,8138	0,0046	-0,09	y
LATMB	198	ANT	0,8210	0,0733	0,0027	0,8148	0,0046	0,08	y
BEV	207	ANT	0,8024	0,0120	0,0027	0,8153	0,0046	-1,13	y
SMD - ENS	259	ANT	0,8022	0,0271	0,0027	0,8177	0,0048	-0,58	y
NSAI NML	282	ANT	0,8332	0,0750	0,0027	0,8188	0,0049	0,19	y
IPQ	314	ANT	0,8323	0,0172	0,0027	0,8203	0,0051	0,72	y
BFKH	343	ANT	0,7820	0,0250	0,0027	0,8216	0,0053	-1,61	y

NMI	Time day	Standard ID	Results		Stability $u_{\text{lab}}(y_i)$ mg	Reference value		Norm. deviation d_i	Inclu- ded (y/n)
			y_i mg	$u_{\text{obj}}(y_i)$ mg		\hat{y}_i mg	$u(\hat{y}_i)$ mg		
BEV	370	ANT	0,8117	0,0120	0,0027	0,8229	0,0055	-1,02	y
BEV	0	ANU	0,6838	0,0120	0,0027	0,6942	0,0056	-0,95	y
MIRS	131	ANU	0,6881	0,0198	0,0027	0,6936	0,0048	-0,28	y
DZM	148	ANU	0,6884	0,0252	0,0027	0,6936	0,0047	-0,21	y
BEV	207	ANU	0,6829	0,0120	0,0027	0,6933	0,0047	-0,91	y
IMBiH	259	ANU	0,7257	0,0567	0,0027	0,6930	0,0049	0,58	y
BoM	280	ANU	0,6740	0,0632	0,0027	0,6930	0,0050	-0,30	y
DMDM	308	ANU	0,6800	0,0217	0,0027	0,6928	0,0052	-0,60	y
BEV	370	ANU	0,6863	0,0120	0,0027	0,6925	0,0057	-0,57	y
SASO	380	ANU	0,6675	0,0105	0,0027	0,6925	0,0057	-2,17	n
BEV	0	B1	-0,0793	0,0120	0,0027	-0,0666	0,0052	-1,14	y
NPL	46	B1	-0,0676	0,0097	0,0027	-0,0700	0,0049	0,28	y
DFM	75	B1	-0,0818	0,0130	0,0027	-0,0722	0,0047	-0,77	y
RISE	112	B1	-0,1020	0,0286	0,0027	-0,0750	0,0046	-0,95	y
MIKES	127	B1	-0,1090	0,0250	0,0027	-0,0761	0,0045	-1,33	y
VSL	175	B1	-0,0870	0,0190	0,0027	-0,0797	0,0045	-0,39	y
BEV	207	B1	-0,0972	0,0120	0,0027	-0,0821	0,0045	-1,32	y
INM	257	B1	-0,1040	0,0267	0,0027	-0,0858	0,0047	-0,69	y
BIM	283	B1	-0,0853	0,0179	0,0027	-0,0878	0,0048	0,14	y
EIM	311	B1	-0,0890	0,0099	0,0027	-0,0899	0,0049	0,10	y
BEV	370	B1	-0,1006	0,0120	0,0027	-0,0943	0,0054	-0,57	y
BEV	0	B2	-0,0355	0,0120	0,0027	-0,0236	0,0048	-1,05	y
METAS	86	B2	-0,0159	0,0087	0,0027	-0,0236	0,0045	0,98	y
BIPM	146	B2	-0,0228	0,0066	0,0027	-0,0235	0,0044	0,13	y
LNE	178	B2	-0,0192	0,0110	0,0027	-0,0235	0,0044	0,41	y
BEV	207	B2	-0,0384	0,0120	0,0027	-0,0235	0,0044	-1,30	y
TUBITAK-UME	419	B2	0,0150	0,0200	0,0027	-0,0233	0,0046	1,95	y
BEV	527	B2	-0,0347	0,0120	0,0027	-0,0232	0,0049	-1,02	y
CMI	699	B2	0,0050	0,0173	0,0027	-0,0231	0,0056	1,69	y
BEV	703	B2	-0,0388	0,0120	0,0027	-0,0231	0,0056	-1,43	y
BEV	0	B5	1,4755	0,0120	0,0027	1,4843	0,0059	-0,82	y
NPL	46	B5	1,4862	0,0097	0,0027	1,4836	0,0054	0,30	y
DFM	75	B5	1,4712	0,0130	0,0027	1,4832	0,0051	-0,97	y
RISE	112	B5	1,4520	0,0286	0,0027	1,4826	0,0048	-1,08	y
MIKES	127	B5	1,4480	0,0250	0,0027	1,4824	0,0048	-1,39	y
VSL	175	B5	1,4700	0,0190	0,0027	1,4816	0,0046	-0,62	y
BEV	207	B5	1,4638	0,0120	0,0027	1,4811	0,0046	-1,52	y
INM	257	B5	1,4640	0,0267	0,0027	1,4804	0,0048	-0,62	y
BIM	283	B5	1,4920	0,0179	0,0027	1,4800	0,0049	0,69	y
EIM	311	B5	1,4780	0,0099	0,0027	1,4795	0,0052	-0,17	y
BEV	370	B5	1,4687	0,0120	0,0027	1,4786	0,0058	-0,91	y
BEV	0	B6	1,2051	0,0120	0,0027	1,2136	0,0048	-0,75	y
METAS	86	B6	1,2175	0,0087	0,0027	1,2132	0,0045	0,54	y
BIPM	146	B6	1,2143	0,0066	0,0027	1,2130	0,0045	0,24	y
LNE	178	B6	1,2145	0,0110	0,0027	1,2128	0,0044	0,16	y
BEV	207	B6	1,1995	0,0120	0,0027	1,2127	0,0044	-1,15	y
TUBITAK-UME	419	B6	1,2570	0,0200	0,0027	1,2118	0,0046	2,28	n
BEV	527	B6	1,2052	0,0120	0,0027	1,2114	0,0049	-0,55	y
CMI	699	B6	1,2310	0,0184	0,0027	1,2107	0,0056	1,15	y
BEV	703	B6	1,1981	0,0120	0,0027	1,2107	0,0057	-1,15	y

Figure 2-9. Measured mass corrections $y_i = m_i - 1 \text{ kg}$ for the 8 travelling standards and calculated reference values. Error bars reflect the combined standard uncertainties $u(y_i)$ that include the standard uncertainties $u_{lab}(y_i)$ reported by the laboratories and the uncertainty contribution $u_{obj}(y_i) = 0.0027 \text{ mg}$ due to the random variation in the masses of the measurement objects.

Figure 2

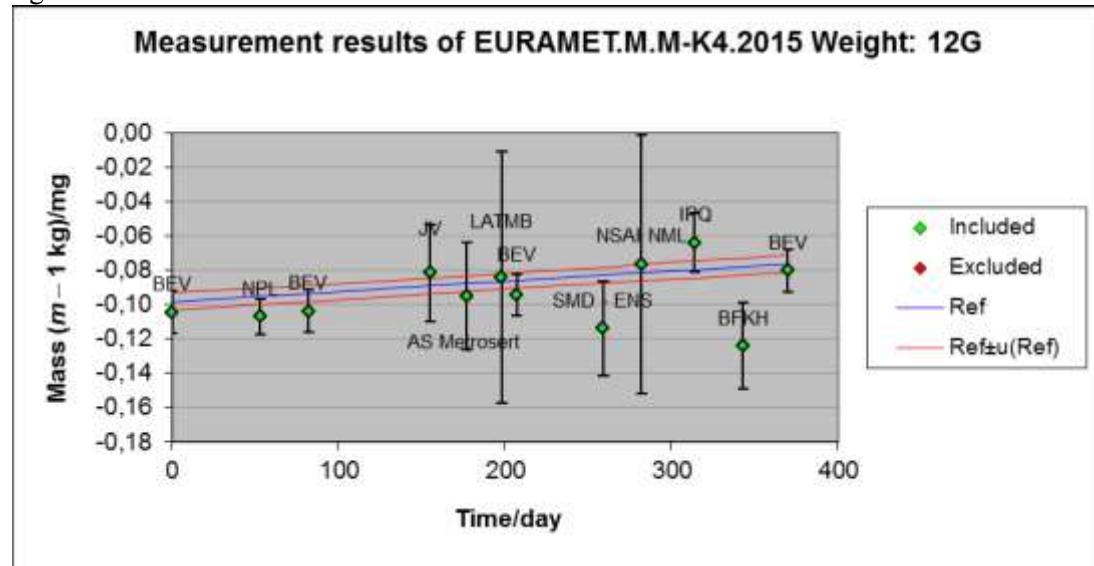


Figure 3.

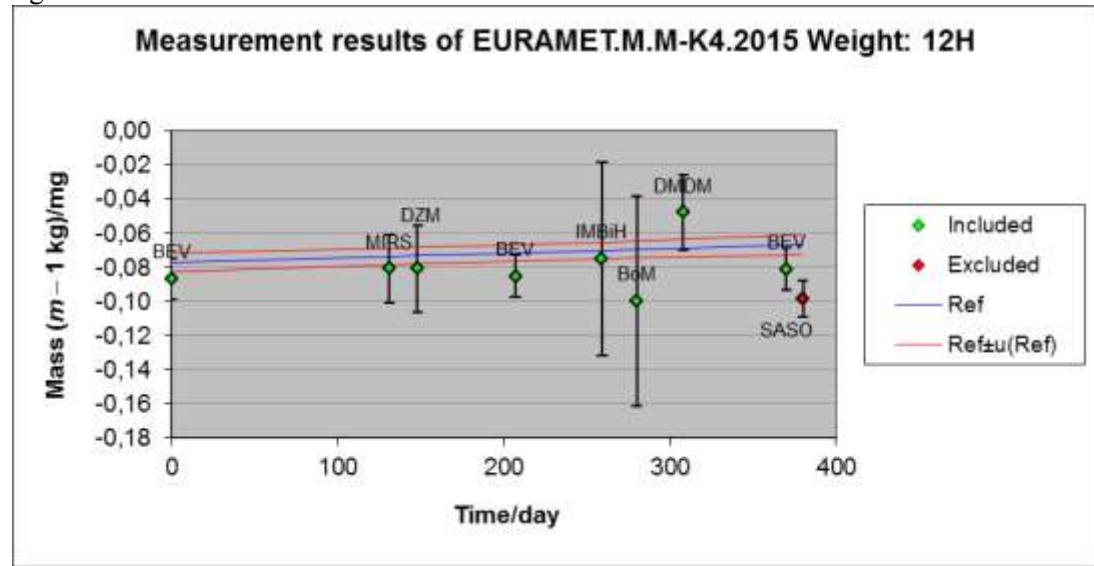


Figure 4

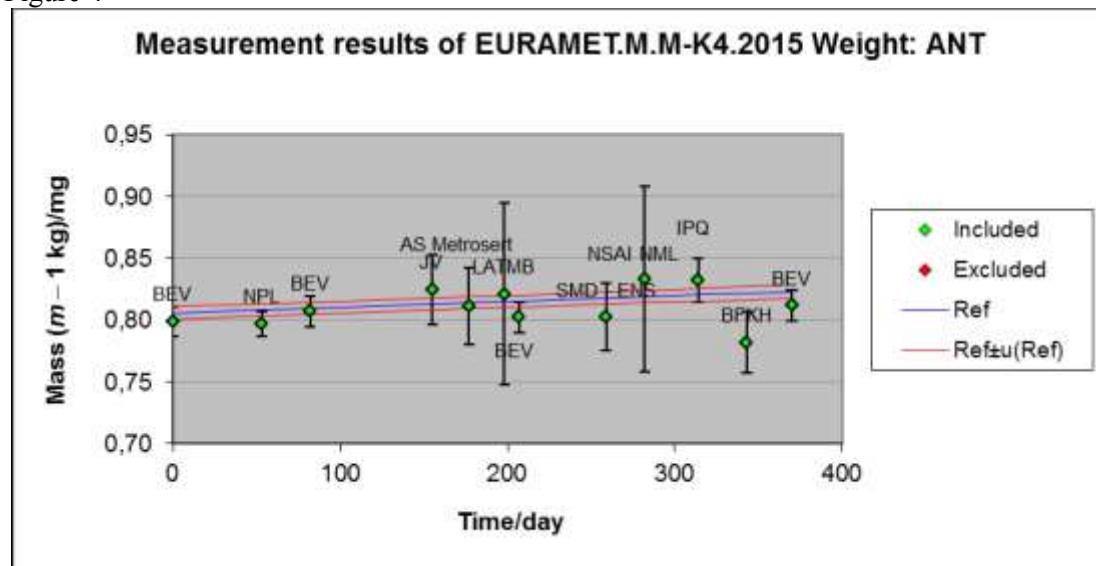


Figure 5

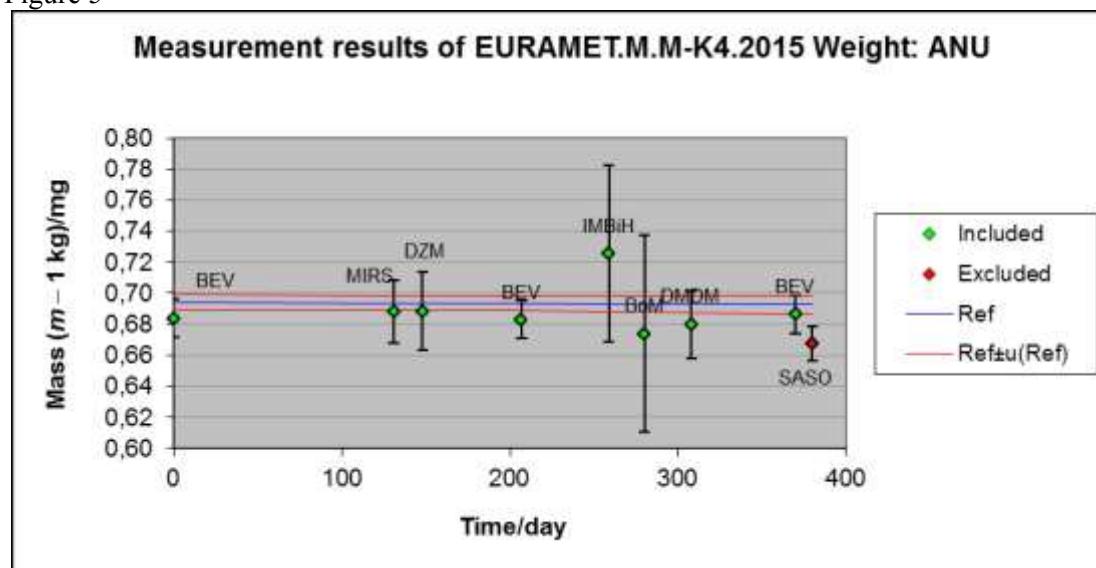


Figure 6

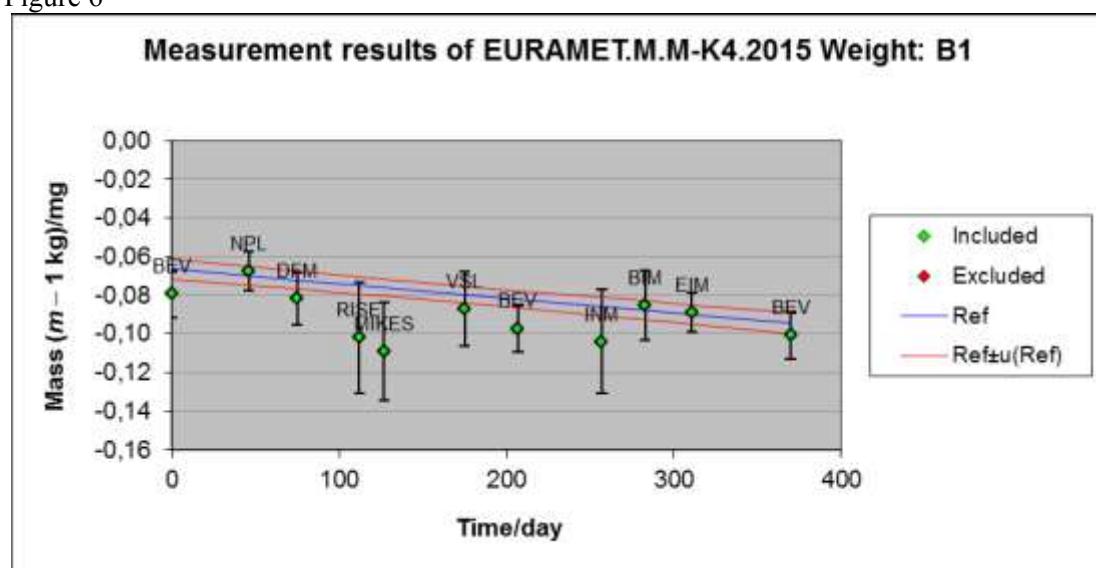


Figure 7

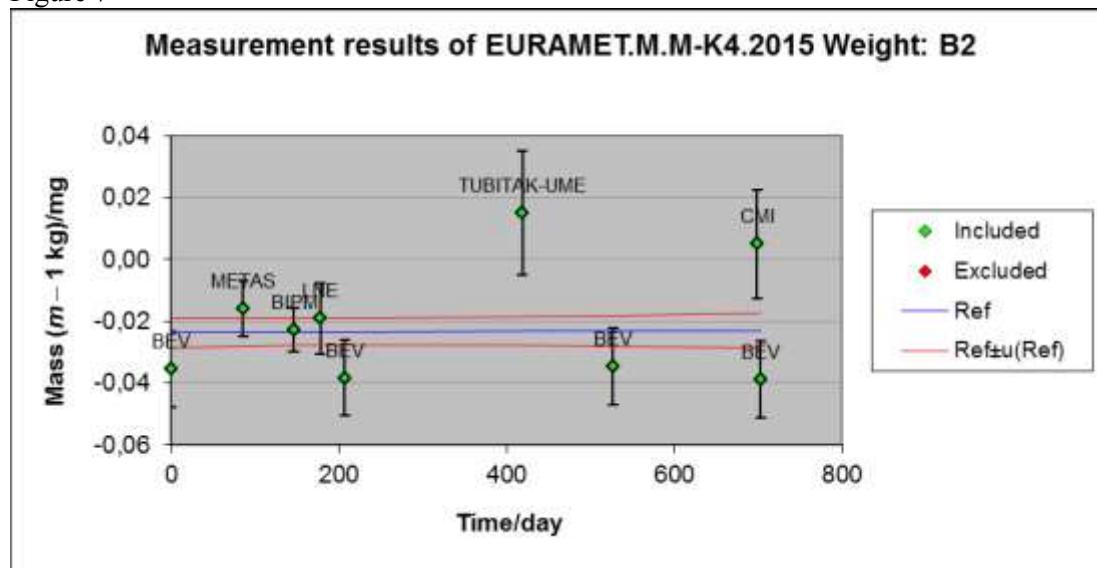


Figure 8

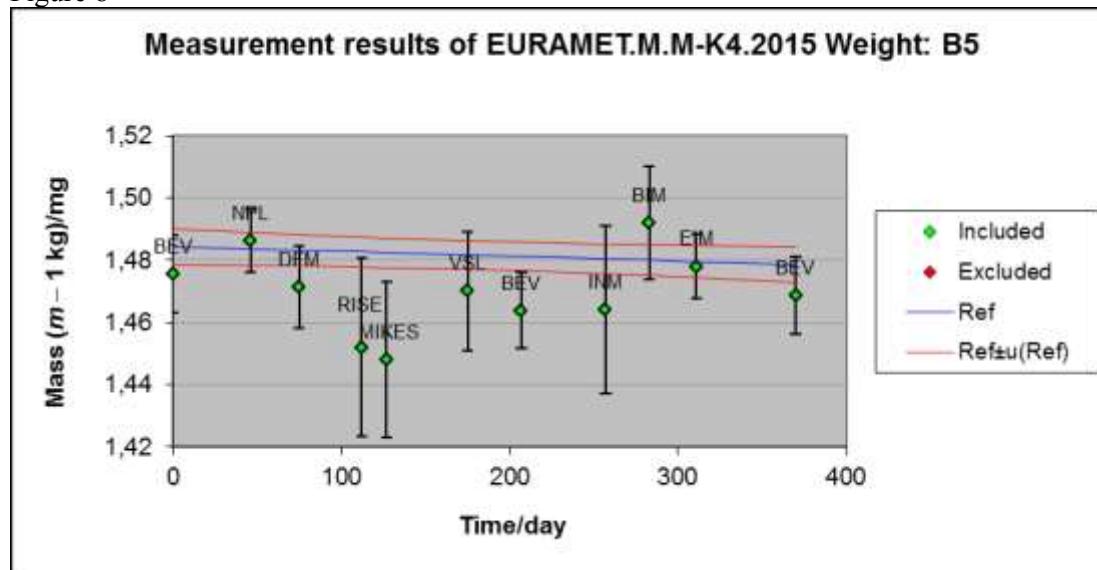


Figure 9

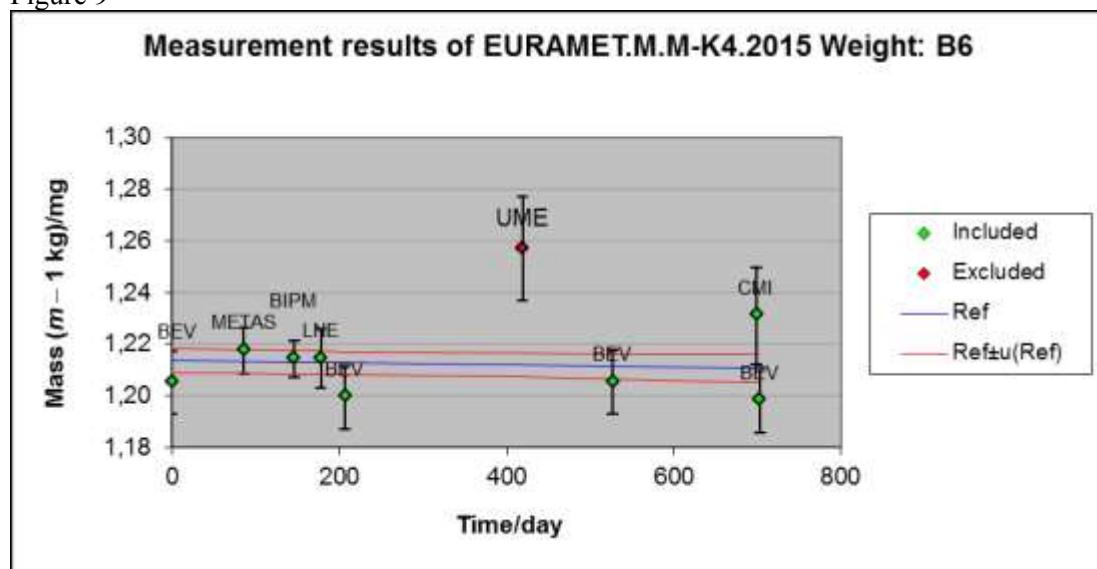
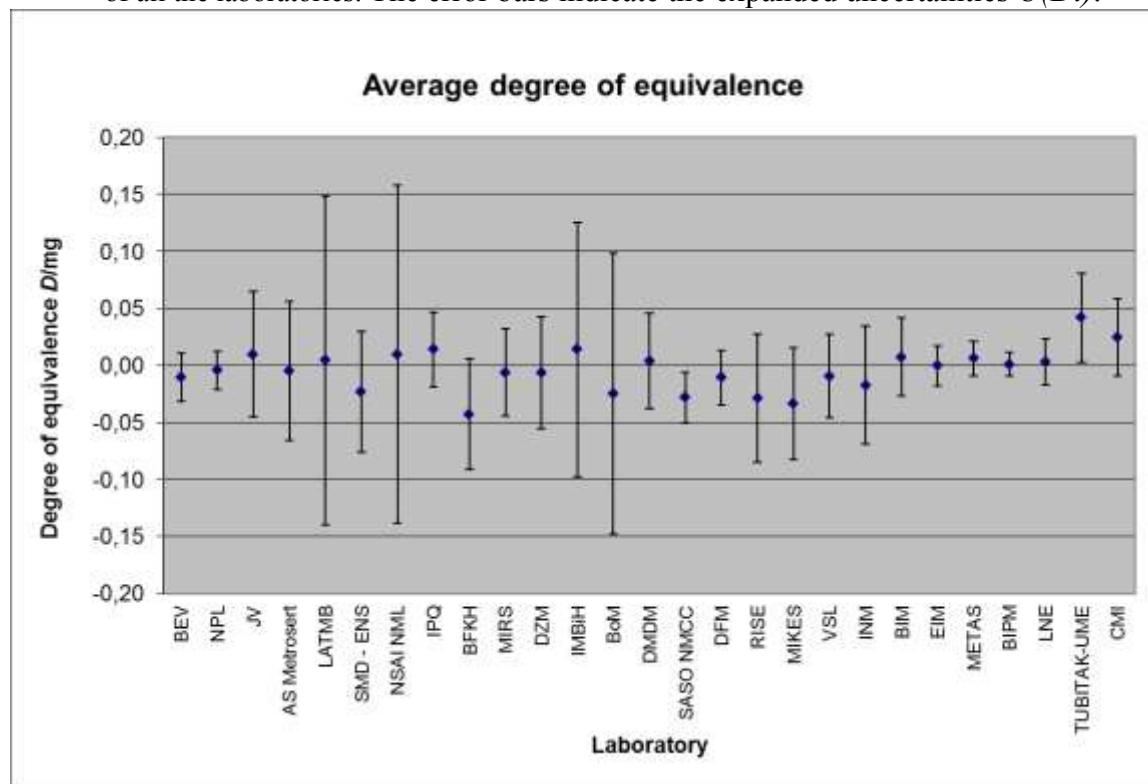


Table 8. (Average) Degree of equivalence; including the results of all the laboratories

NMI	D_i mg	$U(D_i)$ mg	$D_i/U(D_i)$
BEV	-0,010	0,021	-0,49
NPL	-0,004	0,017	-0,26
JV	0,010	0,055	0,17
AS Metrosert	-0,005	0,061	-0,08
LATMB	0,004	0,144	0,03
SMD - ENS	-0,023	0,053	-0,44
NSAI NML	0,010	0,148	0,07
IPQ	0,014	0,033	0,42
BFKH	-0,043	0,049	-0,88
MIRS	-0,006	0,038	-0,17
DZM	-0,006	0,049	-0,13
IMBiH	0,014	0,112	0,12
BoM	-0,025	0,123	-0,20
DMDM	0,004	0,042	0,10
SASO	-0,028	0,022	-1,28
DFM	-0,011	0,024	-0,45
RISE	-0,029	0,056	-0,52
MIKES	-0,034	0,049	-0,69
VSL	-0,009	0,037	-0,26
INM	-0,017	0,052	-0,33
BIM	0,007	0,034	0,21
EIM	0,000	0,017	-0,02
METAS	0,006	0,015	0,39
BIPM	0,001	0,010	0,10
LNE	0,003	0,020	0,15
TUBITAK-UME	0,042	0,039	1,08
CMI	0,024	0,034	0,72

Figure 10. Degrees of equivalence of key comparison EURAMET.M.M-K4.2015 including the results of all the laboratories. The error bars indicate the expanded uncertainties $U(D_i)$.



9 Conclusion

This comparison was undertaken with reference to the International Prototype Kilogram (IPK) as the definition of the kilogram. The results and uncertainties submitted by the participants were therefore traceable to the IPK which, at the time of the comparison, had zero uncertainty. Subsequent to the completion of the measurements for this comparison the SI unit of mass has been redefined and the value of IPK has an associated standard uncertainty of 10 µg. As a separate exercise the CMCs of a number of the participants in this comparison have been increased to take the revised uncertainty in the IPK into account but no adjustments to the results (or uncertainties) reported as part of this comparison were made.

Despite of minor discrepancies all the results of the participating laboratories were considered as consistent with the reference values.

Two laboratories were identified to be statistically discrepant. The discrepancies were considered as insignificant.

One of them (TUBITAK-UME) has currently a much larger CMC value (0,1 mg) than the expanded uncertainty reported in this comparison while the other laboratory (SASO) has no published CMC for mass [9].

The applied linking method was discussed during the TC-Mass and Related Quantities (TC-M) Annual Meeting in April 2019, Budapest and endorsed during the CCM WGD-kg meeting during the 17th CCM meeting, held in May 2019, Paris.

10 References:

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