FINAL REPORT on the

BILATERAL KEY COMPARISON EURAMET.M.M-K2.3

«Comparison of 10 kg, 500 g and 20 g Mass Standards» (EURAMET 1198)

by

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Abstract:

This report summarizes the results of a bilateral comparison of mass standards with nominal values of 10 kg, 500 g and 20 g between EIM (Greece) and BoM (FYR Macedonia). The objectives of the bilateral comparison were to facilitate the demonstration of metrological equivalence between the two participating national laboratories and to verify the measurement capabilities of BoM in support of its CMC claims in the field of mass standard calibration.
Results are consistent with the key comparison reference values of CCM.M-K2 to which this comparison was linked via the regional key comparison EURAMET.M.M-K2.

JULY 2012

1. Introduction

This report describes a bilateral regional key comparison in the calibration of stainless steel mass standards with nominal values of 10 kg, 500 g and 20 g registered as EURAMET project 1198. It has also been registered in the KCDB as EURAMET.M.M-K2.3. The objectives of this comparison can be summarized as follows: a) the evaluation of the participating laboratories' capability to reliably determine the "true" mass of mass standards with nominal values of 10 kg, 500 g and 20 g, b) the provision of objective evidence demonstrating the metrological equivalence between the participating laboratories in the field of mass and c) the provision of support to the CMC claims of the participating laboratories.

The execution of this comparison was necessitated by the performance of one of the laboratories (BoM) in the regional key comparison EURAMET.M.M-K2.2 (EURAMET project 1120) for the nominal mass values of 500 g and 20 g. Additionally, the need to provide support for its CMC claims up to 10 kg was recognized by the same laboratory, since this measurand was not included in the above mentioned regional key comparison.

The intention of this comparison is to provide a link to CCM M.M.-K2 through EURAMET.M.M-K2 to which the pilot laboratory (EIM) has participated, in order to demonstrate equivalence to the corresponding KCRV's.

2. Participating Laboratories

For this bilateral comparison EIM undertook the role of the coordinating and pilot laboratory

The details of the pilot and participating laboratory are shown below:

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3. Circulation Scheme and Time Schedule

The travelling standards were hand-carried between the participating laboratories. It was the responsibility of the participating laboratory to safely transport the standards from and to the pilot laboratory, ensuring that all necessary customs and importation procedures (ATA Carnet) were followed. The participating laboratory was also responsible to document and report to the pilot any incident, where the artefacts may have been polluted or damaged.

The transfer standards were measured by the pilot laboratory three times within a short period of time before their circulation and one more time after returning at the end of the comparison. The participating laboratory had approximately 2 weeks to perform its measurements and another two weeks to prepare and send its report to the pilot laboratory.

The transfer standards arrived at the participating laboratory on 30.09.11 and were returned to the pilot laboratory on 03.11.11.

4. Description of Transfer Standards

One set, of three mass standards, with material properties (including magnetic susceptibility and polarization) and shape commensurate with OIML class E1, was provided by the pilot laboratory to be used as transfer standards with the following nominal mass values:

nominal value
10 kg
500 g
20 g

The weights were housed in individual wooden boxes and designated as 013505/00 (500 g and 20 g) and 013500/00 (10 kg). The density and volume of the mass standards was provided by the pilot laboratory (Annex 1). The stability of the standards was monitored by the pilot laboratory.

5. Technical Protocol

The agreed technical protocol is included in Annex 2.

6. Results

6.1. Transfer Standard Stability

Results obtained by EIM for each of the transfer standards are shown in table 1 as the differences, (Δm_p), between the true mass (m_p) and the nominal mass (m_o) values, along with the associated uncertainties $U(\Delta m_p)$ at k=2.

From these data, significant instabilities of the transfer standards could not be detected and thus no attempt was made to perform any corrections due to changes in the mass standards. Any existing instability was modelled as a step change and was included as an additional component in the uncertainty of the reference values. This was estimated as a rectangular distribution by considering the pre- (Δm_{-P1}) and post- (Δm_{-P2}) circulation measurements of the pilot laboratory as shown below

$$u(\Delta m_{instability}) = \frac{\Delta m_{-P1} - \Delta m_{-P2}}{2\sqrt{3}} \qquad (1)$$

Table 1. Results of the pilot laboratory for each mass standard, as differences Δm_p in mg, between the true and nominal mass value and the associated uncertainty (k=2).

m_n / DATE	10 kg		500 g		20 g	
	Δm_p	$U(\Delta m_p)$	Δm_p	$U(\Delta m_p)$	Δm_p	$U(\Delta m_p)$
23.09.11	-4,2	1,9	-0,24	0,10	0,013	0,010
28.09.11	-3,9	1,9	-0,23	0,10	0,013	0,010
29.09.11	-3,9	1,9	-0,23	0,10	0,013	0,010
04.11.11	-4,0	1,9	-0,22	0,10	0,017	0,010

6.2. Participant Results

Due to the fact that the individual pilot measurements are correlated, the pre-circulation pilot laboratory measurement, Δm_{-P1}, was calculated through a weighted least squares procedure [1]. The covariance was estimated, as shown in Appendix C, by the methods described in the ISO “GUM” [2] and was included in the estimation as the non-diagonal elements of the error matrix **Φ**. The measurement model describing the above is, $\mathbf{y} = \mathbf{X}\mathbf{a} + \boldsymbol{\varepsilon}$, where **y** is the vector of the pilot laboratory's pre-circulation measurements, **X** is the design matrix, **a** the vector of unknown parameters and **ε** the error of the measurements y_i with $E(\boldsymbol{\varepsilon}) = 0$ and $\mathrm{cov}(\boldsymbol{\varepsilon}) = \boldsymbol{\Phi}$. In this case, **a** is a single parameter, Δm_{-p1}, representing the generalized weighted mean [3] of the pilot pre-circulation measurements, given by

$$\Delta m_{-P1} = \left(\mathbf{X}^T \boldsymbol{\Phi}^{-1} \mathbf{X}\right)^{-1} \mathbf{X}^T \boldsymbol{\Phi}^{-1} \mathbf{y} \qquad (2)$$

with an associated standard uncertainty obtained from

$$\mathrm{cov}(\Delta m_{-P1}) = \left(\mathbf{X}^T \boldsymbol{\Phi}^{-1} \mathbf{X}\right)^{-1} \qquad (3)$$

The pre- circulation results obtained this way as well as the post- circulation result for each transfer standard are designated in table 2 as EIM-1 and EIM-2 respectively. The results of the participating laboratory (BoM) for each transfer standard also appear in the same table 2. All results are shown as differences, (Δm), between the true mass (m) and the nominal mass (m_o) value respectively along with the associated uncertainties U(Δm) for k=2.

Table 2. Results of the pilot (pre- and post- circulation measurements) and participating laboratories for each mass standard, as differences Δm in mg, between the true and nominal mass value and the associated uncertainty (k=2).

m_n / Lab	10 kg		500 g		20 g	
	Δm	U(Δm)	Δm	U(Δm)	Δm	U(Δm)
EIM-1	-4,0	1,7	-0,237	0,086	0,0130	0,0089
BoM	-3,2	3,2	-0,244	0,11	0,0180	0,017
EIM-2	-4,0	1,9	-0,222	0,10	0,0171	0,010

6.3. Reference Values and Uncertainty

For the purpose of this comparison it was decided that reference values and their uncertainty be formed by the results of both participants. This is usually done by applying the procedure described by Cox [4]. In this particular circumstance though, the measurements of the participating laboratory (BoM) are partially traceable, through the mass reference standards, to the pilot laboratory (EIM) and as such are correlated. Thus, in order to account for all correlations present the reference values were estimated through the application of generalized least squares procedure with a non-diagonal error matrix, the off-diagonal elements again being the necessary covariance terms [1]. Correlation coefficients were assigned while respecting the "1/a" limit as suggested by Cox et. al. [3]. This ensures consistency of results when additional information in the form of covariance is included in the model. Results were checked for discrepancies from the reference values following the procedure described by Cox [4]. Consistency of the measurements used to form the reference values was checked by performing a test of the form $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0{,}05$.

Hence, the reference values Δm_{ref} for true mass and their associated uncertainties were obtained from the model of §6.2 in which $\mathrm{cov}(\boldsymbol{\varepsilon}) = \boldsymbol{\Sigma}$, which includes the uncertainty due to traveling standard instability $u(\Delta m_{instability})$ as estimated from (1). The reference values and their associated standard uncertainties were estimated as

$$\boldsymbol{\Delta m}_{ref} = \left(\mathbf{X}^T \boldsymbol{\Sigma}^{-1} \mathbf{X}\right)^{-1} \mathbf{X}^T \boldsymbol{\Sigma}^{-1} \boldsymbol{\Delta m} \qquad (4)$$

$$\mathrm{cov}(\boldsymbol{\Delta m}_{ref}) = \left(\mathbf{X}^T \boldsymbol{\Sigma}^{-1} \mathbf{X}\right)^{-1} \qquad (5)$$

These reference values in addition to their expanded uncertainties are shown in table 3. The participants' results along with the reference values and their uncertainties are depicted in figure 1.

Table 3. Reference values and associated uncertainties (k=2) for EURAMET.M.M-K2.3

Nominal Value	Reference Value Δm_{ref} (mg)	Uncertainty (mg)
10 kg	-4,0	1,7
500 g	-0,234	0,082
20 g	0,0146	0,0088

6.4. Degrees of Equivalence and Uncertainty

The degree of equivalence is expressed as the difference between the true mass as determined by each participant from the reference value

$$D_{i,k} = \Delta m_{i,k} - \Delta m_{ref,k} \qquad (6)$$

In the above, indices i and k correspond to the laboratory and the standards nominal mass value, respectively. The associated expanded uncertainty of the degree of equivalence, $U(D_{i,k})$, at a 95% confidence level, taking into account the correlation between the participants' results with the reference value is calculated as

$$U(D_{i,k}) = 2\sqrt{u(\Delta m_{i,k})^2 - u(\Delta m_{ref,k})^2} \qquad (7)$$

Table 4 and figure 2 show the degree of equivalence and the associated expanded uncertainty of the participant as well as of the pilot laboratory whose measurements are shown separately for before and after circulation, and designated as EIM-1 and EIM-2 respectively.

Table 4. Participants' degrees of equivalence, D, and associated expanded uncertainties, U(D) in mg.

m_n / LAB	10 kg		500 g		20 g	
	D	U(D)	D	U(D)	D	U(D)
EIM-1	-0,063	0,47	-0,0024	0,025	-0,0016	0,0017
BoM	0,78	2,7	-0,0098	0,069	0,0034	0,015
EIM-2	-0,023	0,99	0,012	0,050	0,0025	0,0048

7. Link to CCM.M-K2 Key Comparison

The pilot laboratory (EIM) has participated in both EURAMET.M.M-K2 and EURAMET.M.M-K2.1. It was decided to use the results of the former in order to provide a basis for a link to CCM.M-K2 keeping along the same line that was adopted in the evaluation of results of EURAMET.M.M-K2.2 [5]. In the present context, the application of the linking method presented in Appendix A of [6], used to link EURAMET.M.M-K2 to CCM.M-K2, results in a simple exercise of implementing constrained GLS in the analysis of comparison measurements along the lines discussed in [7, 8]. Nevertheless, for the sake of uniformity, this method was used to analyze the results of this key comparison and to provide the link to CCM.M-K2.

Thus, the model used in accordance with [8] is written as $Y_i = D_i + \Delta m_{ref}^{link} + \varepsilon_i$, subject to the constraint that the KCRV, K, determined in CCM.M-K2 is equal to the true mass of the corresponding traveling standard, m_c, i.e., $K - m_c = 0$. In this model, Y_i is the determined mass difference from the nominal value, D_i is the degree of equivalence to the KCRV of laboratory i, Δm_{ref}^{link} is the difference of the value of the traveling standard at the time of this comparison from the nominal value and ε_i is the random error of laboratory i. The estimation of the parameters D_i and Δm_{ref}^{link}, is effected through the use of Gauss-Markov least squares in which the constraint is included in the design matrix, **X**, and its uncertainty, as obtained in the CCM key comparison, is included in the error matrix, **Φ**.

Additionally, correlations between the results of the participants as well as correlation effects between the linking laboratory's (EIM) results and its degree of equivalence obtained in EURAMET.M.M-K2 were taken into account in the error matrix for which a correlation coefficient of 0,4 was assumed.

Application of the method results in a vector of parameters, **β**, including the participant degrees of equivalence of their linked results as well as the linked reference value and the corresponding uncertainties as

$$\boldsymbol{\beta} = \left(\mathbf{X}^{T}\boldsymbol{\Phi}^{-1}\mathbf{X}\right)^{-1}\mathbf{X}^{T}\boldsymbol{\Phi}^{-1}\mathbf{Y} \qquad (8)$$

$$\mathrm{cov}(\boldsymbol{\beta}) = \left(\mathbf{X}^{T}\boldsymbol{\Phi}^{-1}\mathbf{X}\right)^{-1} \qquad (9)$$

The degrees of equivalence for EIM as presented in table 12 of NPL Report ENG 13 [6], March 2009 and used in the above analysis, are as follows:

Nominal mass of 10 kg

D_{EIM} = 0,06 mg with expanded uncertainty $U(D_{EIM})$ = 1,91 mg

Nominal mass of 500 g

D_{EIM} = 0,017 mg with expanded uncertainty $U(D_{EIM})$ = 0,087 mg

Nominal mass of 20 g

D_{EIM} = -0,001 mg with expanded uncertainty $U(D_{EIM})$ = 0,011 mg

The degrees of equivalence of the linked results, D_i^{link} ,are shown in table 5 and figure 3. The linked reference values and the associated expanded uncertainties are also shown in the same table. Finally, the mutual degree of equivalence between the laboratories, $D_{i,j}^{link}$, is also shown in the same table.

Table 5. Linked results of the comparison for each mass standard, showing the degrees of equivalence D_i^{link}, the mutual degrees of equivalence, $D_{i,j}^{link}$, and the reference value $\Delta m_{ref}{}^{link}$ along with the corresponding expanded uncertainty values in mg.

m_n / LAB	10 kg		500 g		20 g	
	D_i^{link}	$U(D_i^{link})$	D_i^{link}	$U(D_i^{link})$	D_i^{link}	$U(D_i^{link})$
EIM	0,06	1,9	0,017	0,087	-0,0015	0,011
BoM	0,88	3,1	0,0028	0,12	0,0017	0,017

	10 kg	500 g	20 g
$D_{i,j}^{link}$	0,82	-0,014	0,0033
$U(D_{i,j}^{link})$	2,4	0,063	0,013
$\Delta m_{ref}{}^{link}$	-4,1	-0,248	0,016
$U(\Delta m_{ref}{}^{link})$	1,9	0,089	0,011

8. Conclusions

A bilateral key comparison between EIM and BoM was conducted in the period from September to November 2011 and registered as EURAMET project 1198. It has also been registered as EURAMET.M.M-K2.3 in the BIPM KCDB database. The results reported by both participants are consistent with the key comparison reference values as well as with each other. These values have been linked to the CCM.M-K2 through the results obtained by the pilot and linking laboratory (EIM) in the key comparison EURAMET.M.M-K2.

9. References

1. L. Nielsen, "*Evaluation of measurement intercomparisons by the method of least squares*", DFM Report DFM-99-R39 (2000).

2. International Organization for Standardization (ISO), "*Guide to the Expression of Uncertainty in Measurements*", Geneva, 1st edition (1995).

3. M.G. Cox et. al., *"The generalized weighted mean of correlated quantities",* Metrologia **43** (2006), S268.

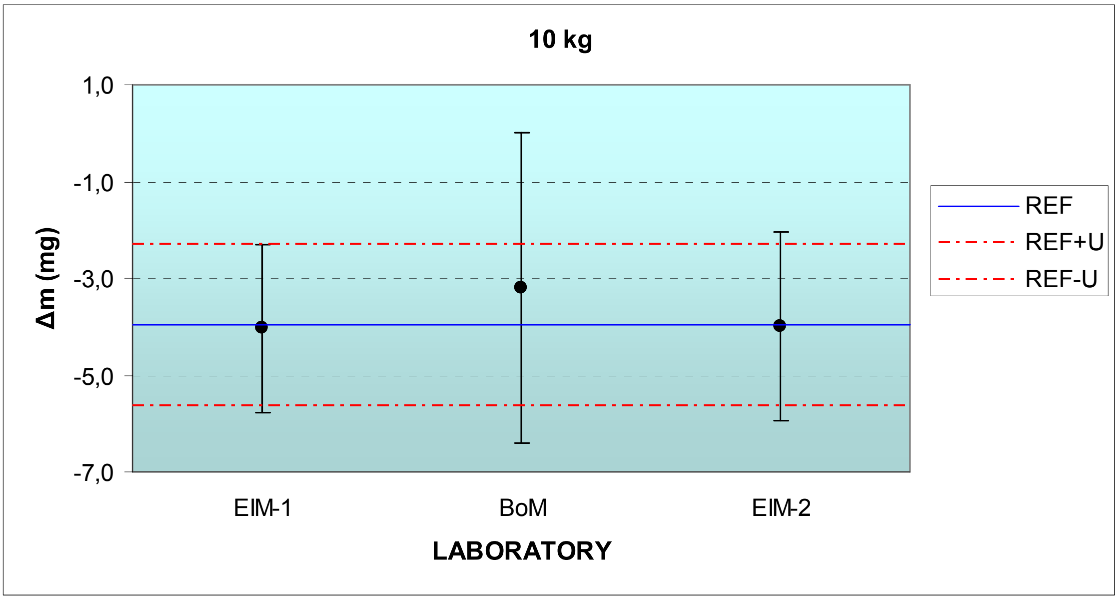
4. M.G. Cox, "*The evaluation of key comparison data",* Metrologia **39** (2000), 589.

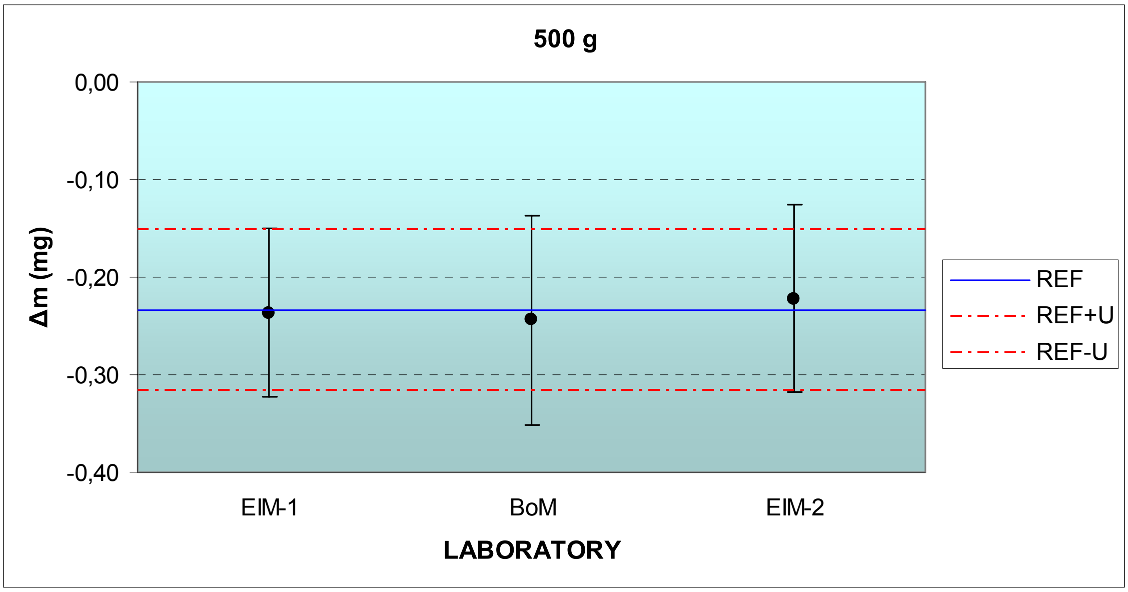
5. D. Steindl et. al., "*Final report on EURAMET key comparison (EURAMET.M.M-K4.2 and EURAMET.M.M-K2.2) of 1 kg and submultiples of the kilogram standards in stainless steel (project code: EURAMET 1120)*", Metrologia **49**, Tech. Supp. 1A, (2012).

6. M. Perkin, *"Report on EURAMET key comparison of multiples and submultiples of the kilogram (EURAMET.M.M-K2)",* NPL Report ENG13 (2009).

7. D.R. White, *"On the analysis of measurement comparisons"* Metrologia, **41** (2004), 122.

8. C.M. Sutton, *"Analysis and linking of international measurement comparisons",* Metrologia **41** (2004), 272.





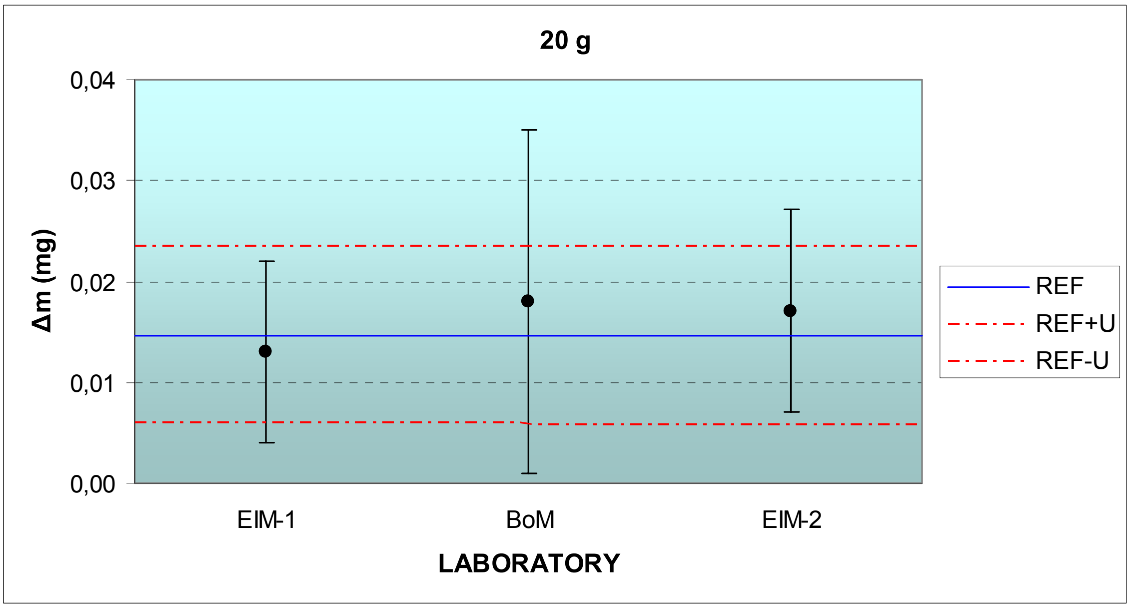
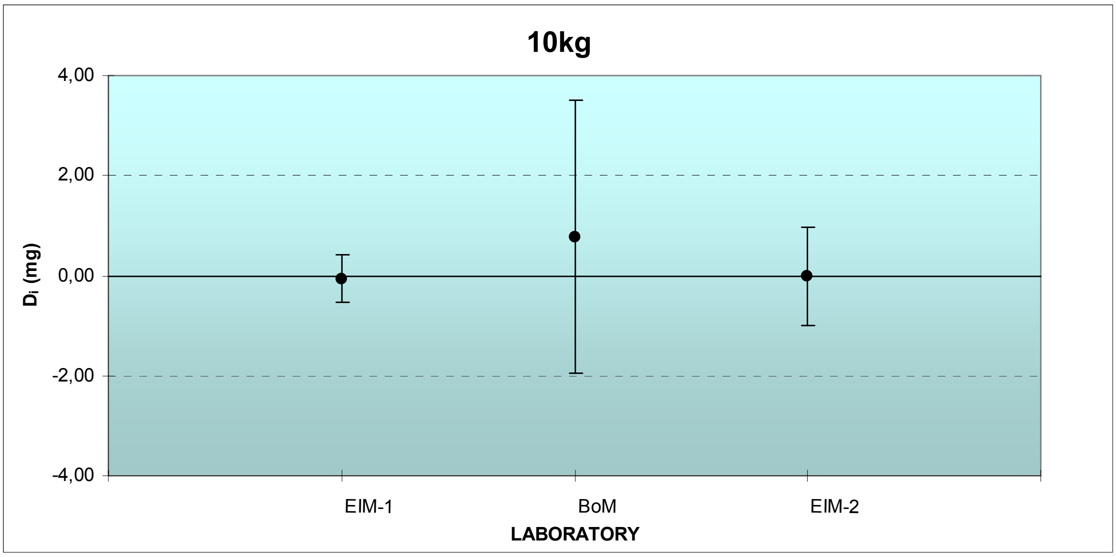
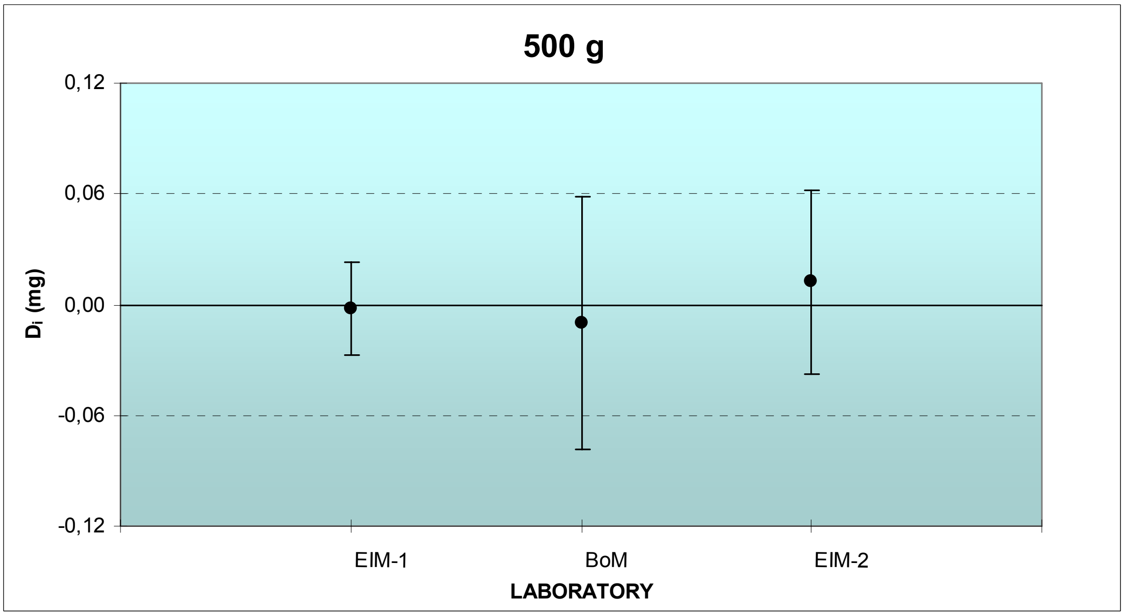


Figure 1. Mass differences obtained by the participating laboratories. The solid line represents the reference value and the dashed lines its uncertainty. Uncertainty shown is for a 95% confidence level (k=2).





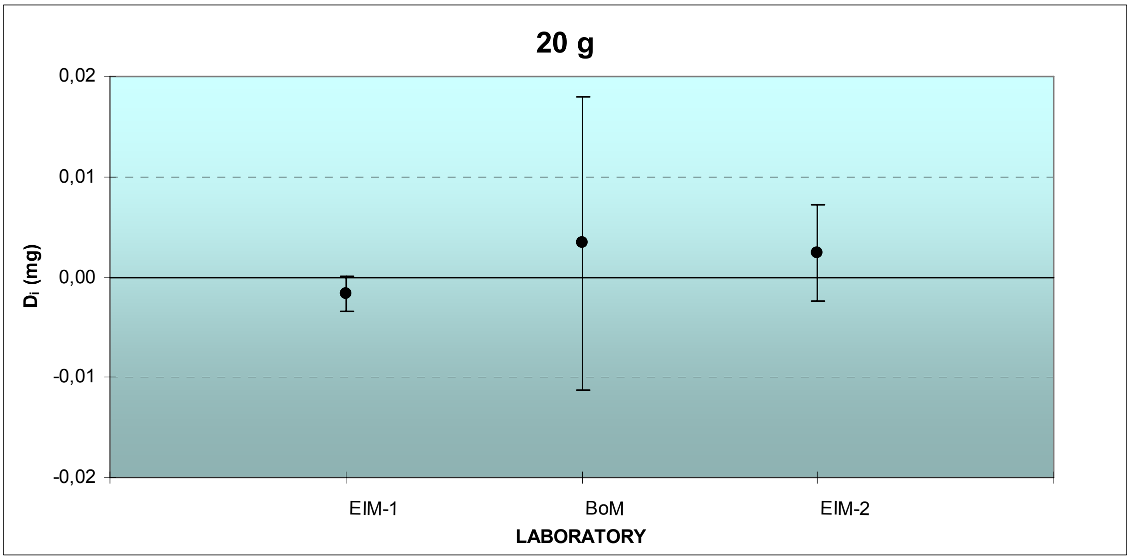


Figure 2. Degrees of equivalence, D_i, of the participant laboratories and corresponding uncertainty $U(D_i)$ (k=2).

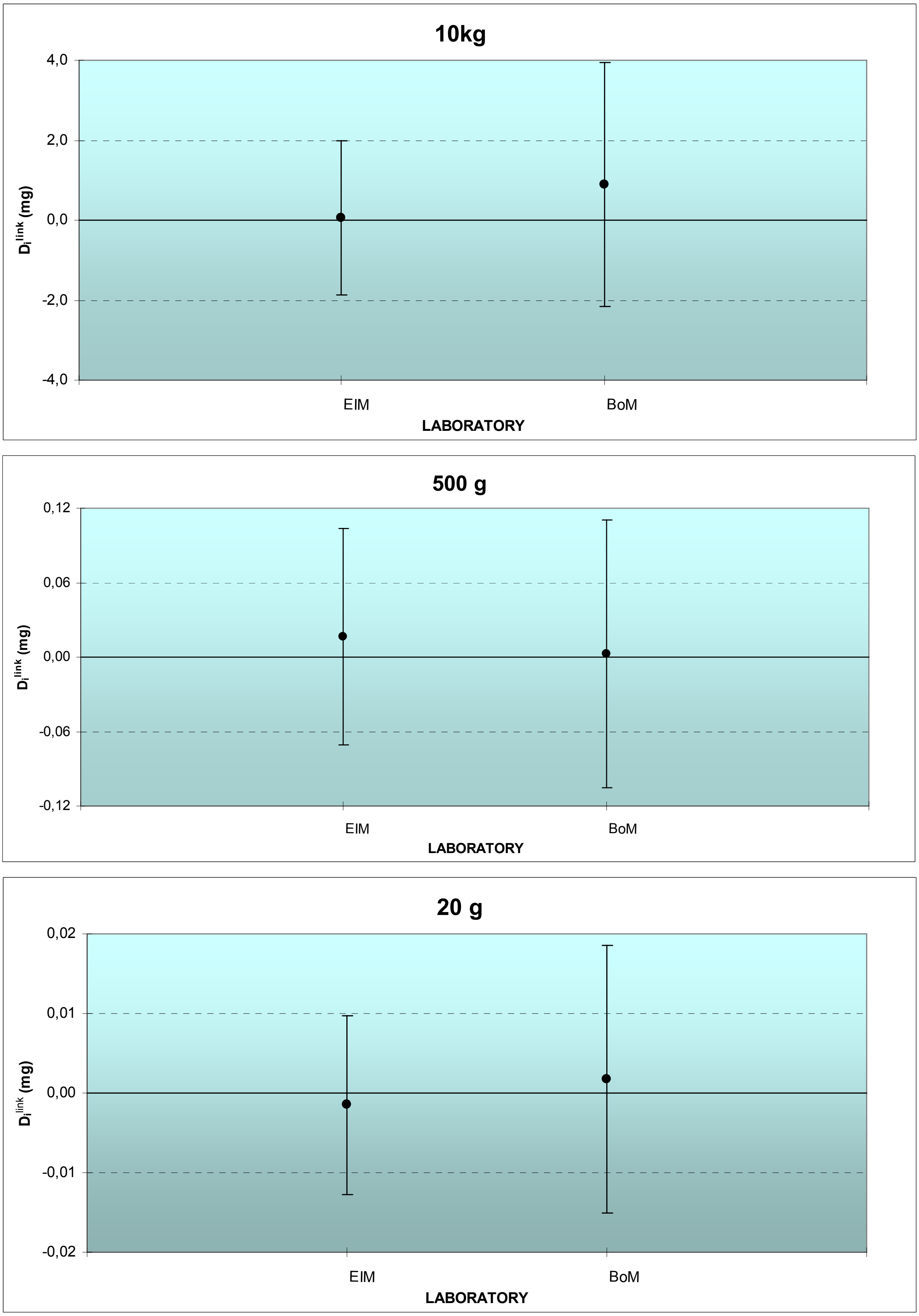


Figure 3. Degrees of equivalence, D_i^{link}, of the linked results of the participant laboratories and the corresponding uncertainty $U(D_i^{link})$ (k=2).

ANNEX A

3.11 Data for mass standards provided by the pilot laboratory

Nominal value	Density ρ (kg/m^3)	Uncertainty u_ρ (1σ)	Volume V @ 20°C (cm^3)	Uncertainty u_V (1σ)
10 kg	8012,1	1,25	1248,1	0,2
500 g	8010,1	1,25	62,421	0,004
20 g	8015	3	2,495 4	0,001

ANNEX B
Technical Protocol for Bilateral Comparison of Mass Standards of Nominal Mass 10 kg, 500 g and 20 g

1 Introduction

1.1 Aim
The objective of the present bilateral comparison is to assist the Bureau of Metrology (BoM) in its effort to provide evidence of its technical competence in the field of mass metrology. In addition through the intercomparison it will have the opportunity to validate the calibration measurement capabilities of the Mass Laboratory.

1.2 Organisation
The bilateral comparison, EIM-MAS-05, of mass standards of 10 kg, 500g and 20g will be organized by the Hellenic Institute of Metrology (EIM) which will also undertake the role of the pilot laboratory, which includes the determination of reference values and the evaluation of performance of the participating laboratory.

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1.3 Mass Standards
One set, of three mass standards, with properties commensurate with OIML class E2, will be used as the transfer standards with the following nominal mass values:

nominal value
10 kg
500 g
20 g

The density of the mass standards will be provided by the pilot laboratory. Stability of the standards has also been monitored by the pilot laboratory.

2 General Information and Procedure

2.1 Unpacking, handling and care of the standards

Read these instructions first!

The mass standards should be manipulated only with appropriate tools.

They should *never* be touched with bare hands!

A visual inspection of the surfaces should be made and the results noted on the corresponding paper sheet. The pilot laboratory should be informed about the result of the visual inspection as soon as possible by fax (see page 1). The standards should be stored in a place where they are protected from dust, aerosols and vapours all the time they are not in use.

2.2 Measurements to be performed
The participating laboratories shall determine the conventional mass and the "true" mass of every standard in the set. Before the mass measurements, dust particles should be removed from the surfaces of the standards by a soft brush. No further washing should be performed.
All weighings should be performed in air. For the buoyancy correction, the air density should be determined using the laboratory's standard procedure, specified on the annexed form.
Measurements should be done after an appropriate acclimatization time (as specified in OIML R111 for class E2). The results and filled forms should be sent by email to the coordinator and by conventional post to the address specified in section 1.2.

2.3 Paperwork
The forms (enclosed) for the measurement results, data of the ambient conditions, instruments used and traceability of the participant's reference standards should be copied, filled in and returned to EIM within 2 weeks after the measurements.

2.4 Transportation
The transportation case is an aluminium suitcase with contents as described in the Contents List. It is the responsibility of the laboratory when it has the travelling standards to safely transport the standards by hand-carrying and ensuring that all necessary customs and importation documents (Carnet, where needed) are in order.
Every incident, where the artefacts may have been polluted or damaged, should be documented and communicated to the pilot laboratory as soon as possible. Also, the pilot laboratory should be informed about any delay or required change of the time schedule.

3 Annexed papers and forms

3.2. Transportation protocol (arrival), contact person of the laboratory
3.3. Transportation protocol (departure), contact person of the laboratory
3.4. Calibration results
3.5. Environmental data during calibration
3.6. Uncertainty budget
3.7. Details of the balances, the instruments for air density determination
3.8. Description of the measuring room
3.9. Traceability description
3.10. Record of the surfaces of the transfer standards
3.11 Data provided by pilot lab
Packing List

4 Time schedule

The participating laboratory will have a period of approx. 2 weeks to perform and complete their measurements according to the following time schedule:
Beginning: week 39 (starting on September 26th 2011)

ANNEX C

Since the measurement results of the participating laboratories exhibit correlations the following calculations are performed in order to exemplify the estimation of covariance and correlation coefficients which were used in the analysis of the mass comparison results.

Correlations resulted from:
a) a single reference standard used for repeated measurements of the traveling standard by a single laboratory, or
b) measurements of the traveling standard by two laboratories one of which is traceable to the other.

In both these situations let mass determination i, be represented by the equation

$$\Delta m_{T,i} = (m_R - m_o) + \rho_{a,i}(V_T - V_R) + \Delta W_1 \qquad \text{(C.1)}$$

and, similarly, mass determination j represented by

$$\Delta m_{T,j} = (m_R - m_o) + \rho_{a,j}(V_T - V_R) + \Delta W_2 \qquad \text{(C.2)}$$

Then, the covariance $u(\Delta m_{T,i}, \Delta m_{T,j})$ of the two measurements is given by

$$u(\Delta m_{T,i}, \Delta m_{T,j}) = \frac{\partial \Delta m_{T,i}}{\partial m_R}\frac{\partial \Delta m_{T,j}}{\partial m_R}u^2(m_R) + \frac{\partial \Delta m_{T,i}}{\partial V_R}\frac{\partial \Delta m_{T,j}}{\partial V_R}u^2(V_R) + \frac{\partial \Delta m_{T,i}}{\partial V_T}\frac{\partial \Delta m_{T,j}}{\partial V_T}u^2(V_T)$$

in which R and T signify the reference and traveling standards respectively, resulting in the expression

$$u(\Delta m_{T,i}, \Delta m_{T,j}) = u^2(m_R) + \rho_{a,i}\rho_{a,j}[u^2(V_R) + u^2(V_T)] \qquad \text{(C.3)}$$

Correspondingly, the correlation coefficient is obtained by the expression

$$r_{i,j} = \frac{u(\Delta m_{T,i}, \Delta m_{T,j})}{u(\Delta m_{T,i})u(\Delta m_{T,j})} \qquad \text{(C.4)}$$

Based on the assumed values for the parameters appearing in the above relation, as shown in the following table for the different nominal mass, calculated values for the covariance and correlation coefficient are presented which were used in the various calculations of reference values for the comparison.

	10 kg	**500 g**	**20 g**
$u(m_R)$ (mg)	0,75	0,038	0,004
$u(V_R) = u(V_T)$ (cm^3)	0,2	0,004	0,001
$\rho_{a,i} \approx \rho_{a,j}$ (mg/cm^3)	1,18	1,18	1,18
$u(\Delta m_{T,i}, \Delta m_{T,j})$ (mg^2)	0,67	0,0014	1,9E-05
$r_{EIM-1,\ EIM-2}$	0,7	0,6	0,8
$r_{EIM-i,BoM}$	0,4	0,6	0,4