Final Report on EURAMET comparison of 500 kg mass standard EURAMET.M.M-S7

EURAMET project No. 1300

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Abstract

In order to demonstrate the equivalence in calibration of 500 kg mass standard among National Metrology Institutes a supplementary comparison has been carried out by 20 members of EURAMET. The overall result shows a good consistency among the participants.

July 2021

1. Objectives

The objectives of the comparison were to facilitate the demonstration of metrological equivalence between the participating national laboratories and to check, confirm or improve the capabilities of quoted calibration measurement capabilities (CMC) at 500 kg. Details relevant for the comparison were specified in the technical protocol.

2. Participants

The pilot laboratory for the comparison was Metrology Institute of the Republic of Slovenia (MIRS) represented by Matej Grum. The list of participants with their contact details is given in the Table 1.

Laboratory	Address	Contact Person
	(Meassurements)	
Czech Metrology	Romana Havelky 17,	Jaroslav Zůda
Institute (CMI)	CZ-58601	+420 602 551 921
		jzuda@cmi.cz
Metrology Institute of	Tkalska 15, SI-3000	Goran Grgić
the Republic of	Celje, Slovenia	+ 386 1 24 42 732
Slovenia (MIRS)		goran.grgic@gov.si
Central Office of	2 Elektoralna Str., 00-	Michał Nawotka
Measures (GUM)	139 Warsaw, Poland	(+48) 22 581 9335
		m.nawotka@gum.gov.pl
Latvian National	K. Valdemara, 157,	Tatjana Žandarova
Metrology Centre,	Riga, LV-1013, Latvia	+371 67 517 728; +371 67 517 726
Metrology Bureau		tatjana.zandarova@latmb.lv
(LATMB)		
AS Metrosert -	Teaduspargi 8, 12618	Allar Pärn
Estonian Central	Tallinn, Estonia	+37253889335
Office of Metrology		allar.parn@metrosert.ee
VTT, MIKES	Tehdaskatu 15, Fl-	Sauli Kilponen
Metrology	87100 Kajaani, Finland	+358 50 4434178
		Sauli.kilponen@vtt.fi
Research Institutes of	Brinellgatan 4, SE-502	Bengt Gutfelt
Sweden (RISE)	64 Borås, Sweden	+46 10 516 54 76
		bengt.gutfelt@ri.se
Justervesenet – The	Fetveien 99, NO-2007	Pekka T. Neuvonen
Norwegian Metrology	Kjeller, Norway	+ 47 64 84 84 70
Service (JV)		ptn@justervesenet.no
Bundesamt für Eich-	Arltgasse 35, 1160	Zoltan Zelenka
und	Vienna	+ 43 1 211 10 6607
Vermessungswesen		zoltan.zelenka@bev.gv.at
(BEV)		
Eidgenössisches	Lindenweg 50, 3003	Christian Wüthrich
Institut für Metrologie	Bern-Wabern,	+41 58 387 04 23
(METAS)	Switzerland	christian.wuethrich@metas.ch
Physikalisch-	Bundesallee 100,	Michael Borys
Technische	38116 Braunschweig,	+ 49 531 592 1110
Bundesanstalt (PTB)	Germany	michael.borys@ptb.de
National	Stanton Avenue,	Mannie Panesar
Measurement and	leddington,	+44 (0)20 8943 7246
Regulation Office	Middlesex, United	mannie.panesar@nmo.beis.gov.uk
	Kingdom, TW11 0JZ	

Table 1: Participants in the comparison.

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Instituto Portugues da	Rua Antonio Glao, 2;	Pedro Conceição
Qualidade (IPQ	Almada, 2829-513	+351212948170
	Caparica, PORTUGAL	pconceicao@ipq.pt
FPS Economy,	Haachtsesteenweg	Hugo Pirée
Metrology Division	1795, 1130 Brussel,	+32 2 277 76 10
(SMD-ENS)	Belgium	hugo.piree@economie.fgov.be
Budapest Főváros	Nagytétényi út 15,	Csilla Vámossy
Kormányhivatala	1222 Budapest,	+36 1 4585 947
(BFKH)	Hungary	vamossycs@bfkh.gov.hu
Institute for Metrology	Čatići bb, 72240	Šejla Ališić
of Bosnia and	Kakanj, Bosna i	+387 33 568 920; +387 33 568 948
Herzegovina (IMBIH)	Hercegovina	sejla.alisic@met.gov.ba;
Bureau of Metrology	Arsenija Boljeviča bb,	Goran Vukoslavović
(MBM)	81000 Podgorica,	+382 20 601 360; +382 67 596 125
	Montenegro	goran.vukoslavovic@metrologija.gov.me
Directorate for	Mike Alasa 14,	Dragan Pantić
measures and	Belgrade, Serbia	+ 381 11 2024 417
precious metals		pantic@dmdm.rs
Ministry of economy -	Blvd.Jane Sandanski	Bianka Mangutova-Stoilkovska
Bureau of metrology	109a, 1000 Skopje, R.	+389 2 24 03 676, ext.029
(BoM)	Macedonia	bianka.stoilkovska@bom.gov.mk
Bulgarian Institute of	52 B »G. M. Dimitrov«	Mariana Miteva
Metrology (DG NCM)	Blvd., Sofia 1040,	+ 3592 9702 759, +359 885 747 411
(BIM)	Bulgaria	m.miteva@bim.government.bg
National Institute of	Sos. Vitan-Barzesti no.	George Florian Popa
Metrology (INM)	11, sector 4, 042122	+40754824217
	Bucharest, Romania	george.popa@inm.ro

3. Transfer standard

The transfer standard for the comparison was a stainless steel cylindrical weight with the nominal mass 500 kg provided by CMI. The density of the standard was 7888,2 kg/m³, associated uncertainty (k=2) was 1,5 kg/m³. Producer: ZDAS. Serial number: 1. Dimensions: diameter 60 cm, height 25 cm.



Figure 1: Transfer standard - side view

In the technical protocol values of the magnetic properties, i.e. the magnetic polarisation and magnetic susceptibility of the standard were not reported. However, during the comparison PTB reported that the susceptibility seems to be increased in some areas at the bottom of the weight. They observed values up to the limit (and at one position slightly above) of the OIML class F1. The polarisation of the weight was found suitable. Later the susceptibility was measured also by MIRS ($0,1 < \mu \le 1,5$; measured by the attracting method, with a limited choice of permanent magnets) and at the end by CMI ($\mu = 0,06$; measured by the attracting method).

Stability check of the transfer standard was performed by CMI at the beginning, three times during the comparison and at the end of the circulation schedule (see Section 5 for details).

The transfer standard was housed in a wooden container on a wooden pallet.

4. Circulation schedule (actual)

The transfer standard circulated between participants according to the list in Table 2. Due to difficulties related to the custom clearance and Covid-19 pandemic the actual circulation time exceeded the planned one.

NMI	Country	Date	Remarks
СМІ	Czech Republic	June 2017	also used for stability evaluation
GUM	Poland	July 2017	
LATMB	Latvia	September 2017	
Metrosert	Estonia	September 2017	
MIKES	Finland	October 2017	
RISE	Sweden	November 2017	
JV	Norway	December 2017	
СМІ	Czech Republic	February 2018	stability measurement only
BEV	Austria	April 2018	
METAS	Switzerland	June 2018	
IPQ	Portugal	August 2018	
СМІ	Czech Republic	January 2019	stability measurement only
PTB	Germany	February 2019	
NMO	United Kingdom	March 2019	
SMD	Belgium	May 2019	
MIRS	Slovenia	July 2019	
СМІ	Czech Republic	August 2019	stability measurement only
BFKH	Hungary	October 2019	
IMBIH	Bosnia and Herzegovina	December 2019	
MBM	Montenegro	January 2020	
DMDM	Serbia	March 2020	
BOM	Macedonia	June 2020	
BIM	Bulgaria	July 2020	
INM	Romania	September 2020	
СМІ	Czech Republic	December 2020	stability measurements only
BEV	Austria	February 2021	repeated measurement

Table 2: Actual circulation sched	ule of the transfer standard.
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It was the responsibility of the participating laboratories to organize the transport to the next participant. When necessary, the standard was accompanied by an ATA Carnet, which was provided

by CMI. The transfer standard was transported between the participating laboratories mostly by courier companies.

When the standard arrived at the participating laboratory, the transportation container and its contents were checked for damage. A visual inspection of the surfaces of the standard was made and the results noted on the measurement report. No significant accidents occurred during the circulation.

5. Calculation of reference value and degrees of equivalence

The participating laboratories determined the conventional mass value of the transfer standard according their normal calibration procedure. Each participating laboratory reported its measured conventional mass difference from the nominal value of the transfer standard together with the expanded uncertainty. Reporting the conventional mass instead of the mass for calibration of 500 kg standards is most common practice of the participants.

The stability of the transfer standards was monitored by CMI. The transfer standards were measured five times, measurements at the beginning were taken also as a part of the comparison and the other four measurements only as additional stability measurements. The results of the stability measurements are given in the Table 3. m_s represents measured deviation of the conventional mass from the nominal mass of the transfer standard during the stability measurement and U the corresponding expanded uncertainty.

		lanuaru
Date	m_s	U
06/2017	1,31 g	0,19 g
02/2018	1,29 g	0,19 g
01/2019	1,24 g	0,21 g
09/2019	1,20 g	0,23 g
12/2020	1,39 g	0,23 g

Table 3: Stability of the transfer standard

The standard uncertainty of the mass stability measurements by CMI $u(m_s)$, which only includes the contributions relevant for the relative mass measurements was estimated to be 0,07 g.

According to Clause 9.3 OIML R 111 [1], the limit values of permanent magnetization and magnetic susceptibilities are defined in such a way that, at magnetic fields and magnetic field gradients possibly present on balance pans, they produce a change of the conventional mass of less than 1/10 of the maximum permissible error of the test weight. However, it is not easy (and probably not even possible) to quantify the effect of a magnetised/susceptible weight on the mass determination results since it depends so much on the equipment used. For the purpose of this report the influence of magnetic properties to instability of the transfer standard, which was determined to be approximately on the level of requirements for class F1, was taken into account as

$$u(m_{magnetic}) = \frac{1}{\sqrt{3}} \frac{MPE(F1 \text{ for } 500 \text{ kg})}{10}$$

The analysis of the stability shows that it is not necessary to consider the drift of the transfer standard. However, the uncertainty of the instability $u(m_{inst})$ of the standard due to:

- the maximum mass change Δm_s of all five stability measurements of the transfer standard obtained from Table 2 (rectangular distribution),
- the uncertainty of the stability measurements and

- the uncertainty due to influence of magnetic properties of the transfer standard was taken into account

$$u(m_{inst}) = \sqrt{\frac{\Delta m_s^2}{12} + u^2(m_s) + u^2(m_{magnetic})} = 0,17 \text{ g}$$

The reference value for the transfer standards was calculated by weighted least squares analysis of the measurement results taking into account the uncertainties of the measured values [2,3]. It was assumed that there was no correlation between the results of the participants.

The uncertainty component due to instability of the transfer standard was included in the calculation of the weighted average and the chi-square test by combining it with the uncertainty given by the laboratory $u(m_i)$

$$u'^{2}(m_{i}) = u^{2}(m_{i}) + u^{2}(m_{inst})$$

A chi-squared test (with 19 degrees of freedom and at the 0,05 level of significance) was applied [2] to carry out an overall consistency check of the results obtained.

The degree of equivalence d_i was calculated as a difference between the participant's measured value m_i and the reference value m_{ref} , both given as the difference from nominal mass 500 kg of the transfer standard

$$d_i = m_i - m_{ref}$$

The standard uncertainty of the degree of equivalence $u(d_i)$ for measurement results which were used in the calculation of the reference value was calculated by

$$u(d_i) = \sqrt{u'^2(m_i) - u^2(m_{ref})}$$

where $u'(m_i)$ is the uncertainty given by the laboratory combined with the uncertainty component due to instability of the transfer standard and $u(m_{ref})$ uncertainty of the reference value.

In a case where the consistency check had failed, the criterion $|d_i| > 2u(d_i)$ was used to identify results which were discrepant compared with the reference value. If the absolute value of degree of equivalence was larger than the expanded uncertainty (*k*=2) of the degree of equivalence, then the result was considered as discrepant at a 5 % level of significance.

The result with the highest discrepancy was excluded from the next round of calculation of the new reference value and reference standard uncertainty. When none of the results which were used in the calculation of the reference value were discrepant then the last calculated reference value is considered as final reference value.

For the measurements which were considered discrepant and were not used in the calculation of the reference value the standard uncertainty $u(d_i)$ was calculated by

$$u(d_i) = \sqrt{u'^2(m_i) + u^2(m_{ref})}$$

After the consistency check was applied in the first calculation of the comparison reference value, discrepant results were found for 3 laboratories. As per CIPM MRA protocol [4] these participants were contacted to check that there is no arithmetic, typographical or transcription errors without providing them with initial comparison reference value.

IMBiH and BoM had an arithmetic error and sent the corrected results.

Also BEV 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 BEV identified the cause and asked to repeat the measurements. BEV stated that: "It looks like the balance had more serious problems after the EURAMET measurements, and it was repaired several times. It

could have already affected the EURAMET measurements." BEV repeated measurements and reported new values. This is the reason that both BEV initial and repeated measurement results are included in the comparison report.

After taking the corrected results mentioned above into account, the consistency check didn't fail any more.

6. Summary of the results

Each participating laboratory was requested to report the following information:

- The conventional mass value of the transfer standard and associated expanded measurement uncertainty.
- Uncertainty budget.
- Details on the used reference mass standards including their traceability.
- Details of the balance used in the comparison.
- Description of the measurement procedure.
- Instruments used for measurement of ambient conditions.
- Laboratory environmental conditions during the measurements.

Table 4 provides information on how 500 kg reference mass was composed and which balances were used by the participants. In order to indirectly define possible magnetic effect of the standard on the balances the participating laboratories were subsequently asked to provide an estimate of the distance of the closest surface of the transfer standard from the balance force transducer.

Laboratorv	Composition	Balance	Resolution	Estimated distance
CMI	10 x 50 kg	KC 500-1	0,1 g	150 mm
GUM	10 x 50 kg	HRP.500.4Y.KO	0,1 g	220-250 mm
LATMB	500 kg	MC 0,5 t	1 g	(35 ÷ 38) mm
Metrosert	9 x 50 kg + 50 kg platform	KC 500-1	0,1 g	50 mm
MIKES				Not applicable.
	10 x 50 kg	Equal arm beam	0,02 g	Mechanical balance.
RISE	8 x 50 + 100 kg	KC500	0,1 g	130 0m
JV	500 kg	XP604KM	0,1 g	~ 128 0m
BEV	24 x 20 kg + 20 kg basket	XP604KMC	0,1 g	(150 ± 25) mm
METAS	stack of 25 x 20 kg or 500	XP604	0,1 g	
	kg monolithic (2 pieces)			100 mm
IPQ	500 kg (3 pieces)	XP1003K	0,5 g	55 mm
NMO	500 kg (2 pieces)	KC500-1	0,01 g	No estimate provided.
SMD	500 kg	XPE604KMC	0,1 g	No estimate provided.
MIRS	24 x 20 kg + 20 kg basket	XP604KM	0,1 g	120 mm
BFKH	500 kg	KA-500	0,1 g	100-120 0m
IMBIH	500 kg	PTF 600	1 g	No estimate provided.
MBM	22 x 20 kg + 60 kg basket	XP604KM	0,1 g	30 mm
DMDM	22 x 20 kg + 60 kg basket	KC500-1	0,1 g	(100 ± 10) mm
BOM	500 kg	CCS600K	1 g	(70 ± 4) mm
BIM	500 kg	KCC1000	1 g	180 mm
INM		XP604KM,		
	23 x 20 kg + 40 kg rack	KC1000	0,1 g	at least 200 mm

Table 4: Equipment used

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

The participants' results for comparison of the 500 kg transfer standard (the measured conventional mass difference from the nominal value of the transfer standard m_i with the corresponding standard uncertainty $u(m_i)$), the degrees of equivalence d_i with the corresponding standard uncertainty $u(d_i)$ and the ratio $d_i/u(d_i)$ are reported in Table 5 together with the calculated reference value m_{ref} with its corresponding standard uncertainty $u(m_{ref})$. The results are plotted on Figures 2 and 3.

Based on its observations concerning the magnetic properties of the transfer standard as mentioned in Section 3, PTB didn't report its results. A confirmation of magnetic properties of the transfer standard on the level of OIML R 111 [1] class F1 would be a limiting factor for their measurements and would not allow them to support their CMCs.

BEV results are reported twice. The initial discrepant result is reported under BEV (1) and the repeated result is reported under BEV (2). The result BEV (1) was not included in calculation of the reference value.

Laboratory	m_i	$u(m_i)$	d_i	$u(d_i)$	$d_i/u(d_i)$
	g	g	g	g	
CMI	1,31	0,10	0,13	0,17	0,76
GUM	1,90	1,00	0,72	1,01	0,71
LATMB	0,73	2,20	-0,45	2,20	-0,21
Metrosert	3,10	1,00	1,92	1,01	1,90
MIKES	1,37	0,38	0,19	0,40	0,47
RISE	1,40	0,75	0,22	0,76	0,29
JV	-0,40	0,75	-1,58	0,76	-2,07
BEV (1)	-0,30	0,40	-1,48	0,41	-3,58
METAS	0,61	0,25	-0,57	0,29	-1,99
IPQ	-0,25	0,80	-1,43	0,81	-1,76
NRO	1,12	0,13	-0,06	0,19	-0,34
SMD	2,22	0,62	1,03	0,63	1,63
MIRS	1,13	0,37	-0,05	0,39	-0,13
BFKH	1,99	0,52	0,81	0,53	1,52
IMBIH	-2,56	4,00	-3,74	4,00	-0,93
MBM	0,23	1,16	-0,95	1,17	-0,81
DMDM	1,50	1,30	0,32	1,31	0,24
BOM	1,47	1,20	0,29	1,21	0,24
BIM	1,40	1,45	0,22	1,46	0,15
INM	0,70	0,75	-0,48	0,76	-0,63
BEV (2)	1,40	0,35	0,22	0,38	0,58

Table 5: Participants' results, reference value and degrees of equivalence for the 500 kg standard comparison.

 $m_{ref} =$ **1,18 g** $u(m_{ref}) =$ **0,10 g**

Due to the magnetic properties of the transfer standard (see Chapter 3 for details), in general the results of the comparison cannot support the calibration capabilities of the laboratories with the expanded measurement uncertainty of less than 0,80 g. However, it needs to be taken into account that the magnetic impact depends on the equipment used, where in particular the distance between the centre of gravity of the transfer standard and the load cell is important in the case of the load cell with electromagnetic compensation. No such influence is relevant if a mechanical balance is used. The "limit" value of the expanded measurement uncertainty of 0,80 g was determined based on the provisions of OIML R 111 for a 500 kg weight of accuracy class F1, because values of susceptibility up to the limit (and at one position slightly above) of the OIML class F1 were observed during the comparison.



Figure 2: Results for the 500 kg standard with expanded uncertainties (k=2) as reported by participants. Red data point represents the discrepant result, which was not included in the calculation of the reference value. Black data points represent the stability measurements.





7. Conclusion

The comparison EURAMET.M.M-S7 was piloted by MIRS, twenty laboratories calibrated the conventional mass of the 500 kg transfer standard. The standard and its stability measurements were provided by CMI.

The comparison was conducted between June 2017 and February 2021.

The uncertainty component due to instability of the transfer standard was considered in the calculation of the weighted average and in the chi-square test.

BEV results were reported twice. The initial result was discrepant and therefore not included in calculation of the reference value.

With the exception mentioned above, the chi-square test consistency check did not fail and all results were included in the calculation of the reference value.

The degrees of equivalence of the results mostly show a good agreement of participants' results with the reference value, however one laboratory shows the absolute value of the ratio $d_i / u(d_i)$ slightly above 2.

Due to the magnetic properties of the transfer standard, in general the results of the comparison cannot support the calibration capabilities of the laboratories with the expanded measurement uncertainty of less than 0,80 g.

EURAMET Guide on Comparisons [5] suggests that the participants should give a written statement indicating if their results are consistent with the CMC claims or not. The participants' statements regarding the CMCs are listed below:

Laboratory	Written statement indicating if the result is consistent with the CMC claims or not. If
	not, corrective actions should be described.
CMI	The result is consistent with the CMC claims.
GUM	The result is consistent with the CMC claims.
LATMB	No CMC published for 500 kg yet.
Metrosert	No CMC published for 500 kg yet.
MIKES	The result is consistent with the CMC claims.
RISE	The result is consistent with the CMC claims.
JV	We discovered a technical problem (corner load) with the balance during the measurements for this comparison. This has been communicated to the pilot laboratory throughout the whole comparison. According to the manufacturer of the balance, this balance type is known to have this type of technical issues. The balance was repaired and we have implemented better routines, and shortened the testing interval, to discover technical issues earlier in the future.
	An extra uncertainty contribution is added due to the corner load in accordance with EURAMET G18, however this does not account for a possible systematic error in the measurement due to faulty balance. Since the balance is repaired now (2018), uncertainty contributions due to corner load and repeatability can be reduced. Thus our uncertainty falls close to 1,1 g, which is our CMC. Due to the possilibity of systematic error, it is difficult to state from these results if our CMC is confirmed. However, after the technical problem was solved, calibration
	results for our internal reference weight yields results consistent with historical data. This was not the case just before the comparison.

BEV	The result is consistent with the CMC claims.
METAS	The offset respective to the reference value (0,57 g) as well as the claimed
	uncertainty (0,5 g, k=2) are within our CMC for 500 kg which is 1 g.
IPQ	No CMC published for 500 kg yet.
NMRO	No CMC published for 500 kg yet.
SMD	The result is consistent with the CMC claims.
MIRS	No CMC published for 500 kg yet.
BFKH	BFKH do not want to change the measurement uncertainty in the CMC table because a new balance and standards weights are under procurement. (BFKH reported higher uncertainty (U = 1,04 g) than published CMC at 500 kg (U = 0,80 g).)
IMBIH	No CMC published for 500 kg yet.
MBM	No CMC published for 500 kg yet.
DMDM	The result is consistent with the CMC claims.
BOM	No CMC published for 500 kg yet.
BIM	No CMC published for 500 kg yet.
INM	The result is consistent with the CMC claims.

8. References

[1] Weights of classes E_1 , E_2 , F_1 , F_2 , M_1 , M_{1-2} , M_2 , M_{2-3} , M_3 , International recommendation OIML R111-1, OIML, 2004.

[2] M.G. Cox, The evaluation of key comparison data, Metrologia, 2002, Vol. 39, 589-595.

[3] Nielsen, L. "Evaluation of measurement intercomparisons by the method of least squares", DFM-99-R39 (2000).

[4] Measurement comparisons in the CIPM MRA, CIPM MRA-D-05 Version 1.6

[5] EURAMET Guide on Comparisons, EURAMET Guide No. 4, Version 1.0 (05/2016)