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Implementation of dissemination of unit of mass in emerging mass laboratories

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ABSTRACT

As industrial and technological requirements have increased in recent years, the reliable and effective dissemination of the unit of mass became more critical for laboratories where these techniques are not applied. The EMPIR project "Improvements of the realisation of the mass scale" addressed needs of the emerging mass laboratories. As part of the implementation of the dissemination techniques, two case studies were conducted. Initial case study shows the initial implementation was successful in terms of agreement of the measured values with already known information. Follow-up case study was aimed at the level of achievable uncertainties. Participating laboratories proved their capabilities in the field of dissemination of the unit of mass with uncertainties below requirements for weights of class E1 as defined by document OIML R111:2004.

1. Introduction

In recent years, industrial and technological requirements for mass metrology have increased with uncertainties at, or beyond, the current state of the art. The reliable and effective realisation of the mass scale, with appropriate uncertainties, is critical to meeting these requirements. However, this capability does not yet exist in some small and developing NMIs. The EMPIR project "Improvement of the realisation of the mass scale" [1,2] addressed the need for developing such capabilities by drafting a calibration guideline, software for evaluation of results [3] and uncertainties and through interlaboratory comparison to reach uncertainties of 0,001 mg–3,0 mg in the range 1 mg–20 kg which corresponds to uncertainty requirements for weights of class E1 according to OIML R111 [4].

Two case studies were organised during the project. The first one started at the beginning of the project, aiming to guide emerging laboratories regarding the dissemination process itself and the evaluation of results. Laboratories used their own weight sets with already known values, so it was possible to evaluate agreement with values measured by the dissemination process. The agreement was evaluated using normalised error E_n. All laboratories reported results with E_n smaller than one with minor exceptions.

A follow-up case study was organised parallel to the Pilot study comparison (EURAMET project 1556). The case study aimed to compare the uncertainties determined by the dissemination process with requirements for weights of class E1 according to OIML R111 in the ranges not covered by the pilot comparison. All laboratories used the software solution developed within the project.

All laboratories achieved the required level of uncertainties with minor exceptions. Especially in the range of 100 mg–500 mg the uncertainties were small compared to the requirements for class E1. It can be explained by the usual use of mass comparators with the resolution of 0.0001 mg and the fact that the uncertainty of the reference weight scales with the nominal mass of the weights so in the ranges below 1 g the uncertainty of reference weight is negligible compared to the standard deviation of the measurements. The significance of the uncertainty of the reference weight was most evident in the 1 kg–20 kg range, where some laboratories declared uncertainties close to the requirement of class E1.

Two laboratories evaluated the differences in use between the two types of mass comparators, the automatic and the robotic system, concerning the other factors, such as time needed to complete the measurements or the critical points of measurements. The comparison shows that both systems can achieve uncertainties of class E1, however, differences in measurement preparation, weights handling and measurement evaluation have been identified.

The case studies proved that the emerging laboratories have successfully applied the process of disseminating the unit of mass to meet the required uncertainties for weights of class E1, as stated by OIML R111. The respective CMC values are expected to be updated after successful participation in key comparisons.

2. Initial case studies

The first training focused on basic summary of dissemination procedures and evaluation of results [5]. It supplied vital input for developing laboratories to set up their own procedures to be evaluated by initial case study. Using their own mass standards, the laboratories were able to compare results of their measurements with values from calibration certificates.

Laboratories of SMD (Belgium), IMBiH (Bosnia and Herzegovina), BIM (Bulgaria), ME-BoM (North Macedonia) and DMDM (Serbia), were instructed to make a dissemination in at least one decade of weights, e.g., in range 100 g–1 kg. The system of measurements was to be chosen by the laboratory however they could ask for any recommendations. Most used range was 100 g–1 kg with two weights in each nominal mass. Two laboratories used dissemination procedure in range 100 mg–1 g and one laboratory in range 1 kg–5 kg.

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Laboratories should choose between different methods of evaluation, namely Gauss-Markoff or Lagrange approach. They calculated the results by their own means and recorded which uncertainty sources were considered when evaluation the uncertainties.

All laboratories taking part in initial case study provided values of mass standards for direct comparison with their own results as well as more information on which components of uncertainty were included in the uncertainty evaluation.

Uncertainty components included uncertainty of the reference weight, volume of the weights, standard deviation of the measurements and uncertainty of density of air. Generally, the calculated uncertainties were smaller than the uncertainties from calibration certificates depending on whether the laboratory already used the dissemination procedures before or if the weight set was calibrated by another metrology institute.

The results are evaluated in terms of normalised error

$$E_n = \frac{m_{meas} - m_{Cal}}{\sqrt{U(m_{meas})^2 + U(m_{Cal})^2}} \tag{1}$$

where the difference between measured mass m_{meas} and value from the calibration certificate m_{Cal} are normalised by the sum of the associated expanded uncertainties. Values of $|E_n| < 1$ are generally considered as satisfactory.

2.1. General considerations

As most of the laboratories were not familiar with dissemination procedure the most common issue was safe manipulation with weights. Especially when working with automatic mass comparators it is necessary to stack the weights which might cause fall of the weight and potential damage on the surface of the weight when handling or stacking improperly.

The problem of handling of weights is also visible when working with weights in milligram range. These weights are small and not quite distinguishable so the risk of mixing the weights is increased when working with weights from different sets. Another issue when working with small weights is when loading them to small weighing pan the weights can be lost due to improper manipulation with the tweezers.

Most laboratories are equipped with automatic or robotic mass comparators where the measurements run without intervention of the laboratory person. However, this is not case for all laboratories or all ranges of measurements. Such case is usual for milligram range where one laboratory pointed out the length of manual measurements.

Overall, the agreement between measured values and values from calibration certificates was great with just minor exceptions as presented in Fig. 1 which presents E_n values for all reported weights. We can see that $|E_n| < 1$ almost in all cases. In some cases, the values are very close to 0 which means the measured value was close to the known value compared to measurement uncertainties. There were even cases when the measured value was exactly same as values from calibration certificates which leads to $E_n = 0$.

We can assume the dissemination procedures were applied correctly in all laboratories.

3. Follow-up case studies

The follow-up case study aimed to evaluate dissemination process by comparison of measurement uncertainty with current CMC values or limits defined by OIML R111 for class E1. The agreement of results was already evaluated during initial case study.

The RealMass software [6] which was developed within the RealMass project and is available to download enables to evaluate components such as centre of gravity of weights or correlations between mass standards used for dissemination of the mass scale. These values were not usually used in developing laboratories as well as in some laboratories where the dissemination procedure was already applied.

All laboratories applied corrections and uncertainty analysis of volume or density of weights, air density, balance, measurement process. Some laboratories also applied corrections to centre of gravity which might influence measurements usually with weights above 100 g when not applied correctly.

All laboratories shown exceptional performance in terms of uncertainty. All laboratories were able to achieve uncertainties below limits of class E1 and in most cases also below current CMC values.

3.1. Evaluation of results

The Fig. 2 summarise all results for weights in range 1 kg–50 kg. This range was covered by laboratories of IMBiH (Bosnia and Herzegovina), BIM (Bulgaria), CMI (Czechia), ME-BoM (North Macedonia), INM (Romania) and DMDM (Serbia). The graphs show uncertainties of weights normalised to respective limits of uncertainties as defined by OIML R111. The results of other ranges are not presented here since all uncertainties of weights below 1 g were within the limits of class E1.

As the graphs show, when calibrating weights with nominal values up to 10 kg, the uncertainty usually scales with the nominal value so if the uncertainty of reference 1 kg weight is close to $U_{1kg_{E1}} = 0.17\ mg$, uncertainties of weights of higher nominal mass will be close to limit of class E1 as well. This is the case of laboratory of IMBiH which also proved that to achieve uncertainty needed for class E1 the reference standard of so-called class E0 is not needed.

Due to readability and sometimes high standard deviation the ca-

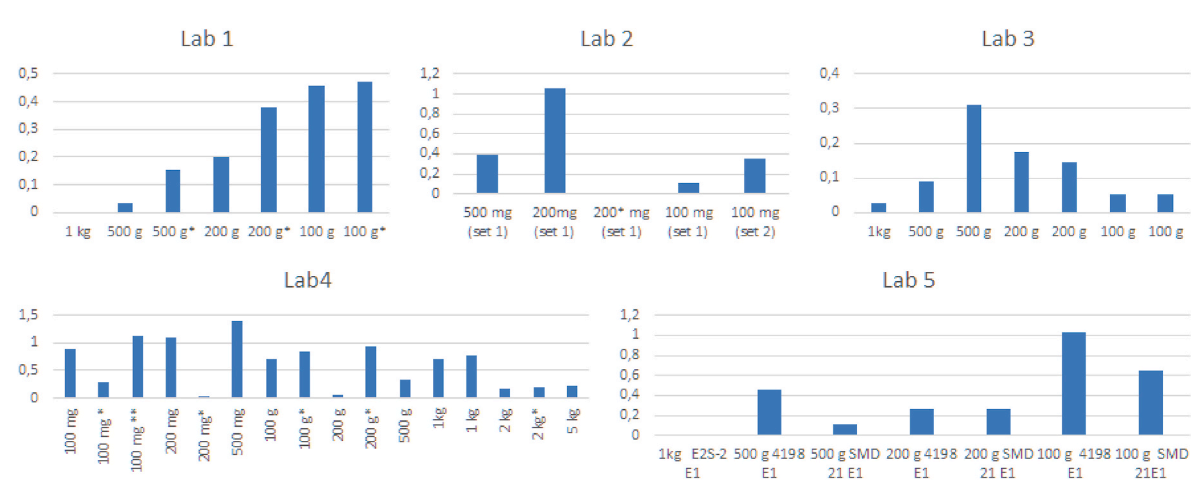

Fig. 1. Evaluation of Initial case studies by means of E_n – goal is to have $E_n < 1$. Weights identification as provided by participating laboratories.

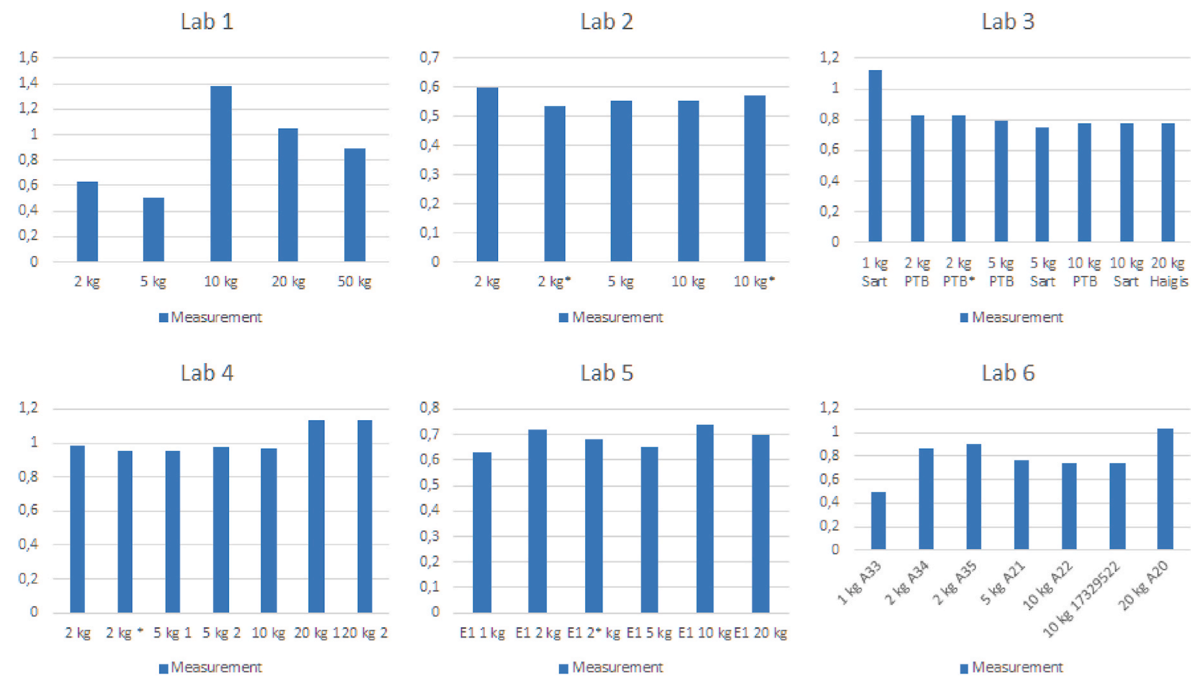

Fig. 2. Evaluation of follow-up case studies in range 1 kg–50 kg by means of comparing to uncertainties relevant to class E_1. Goal is to have values less than 1. Weights identification as provided by participating laboratories.

1 kg – 50 kg		BIM	CMI		DMDM	
m_{ref_N}		1 kg	1 kg		1 kg	
U_{ref}		0.042 mg	0.042 mg		0.05 mg	
Number of equations		13	36		12	
Type of comparator		Automatic	Automatic		Automatic	Manual
Range		1 kg – 50 kg	1 kg – 10 kg		1 kg – 10 kg	20 kg
d		0.1 mg	0.01 mg		0.1 mg	1 mg
1 kg	U				0.2 mg	
	U_{E1}				1.2	
2 kg	U	0.21 mg	0.1 mg	0.089 mg	0.32 mg	0.32 mg
	U_{E1}	0.63	0.6	0.534	0.96	0.96
5 kg	U	0.42 mg	0.23 mg		0.76 mg	0.8 mg
	U_{E1}	0.5	0.552		0.91	0.96
10 kg	U	2.3 mg	0.462 mg	0.476 mg	1.54 mg	1.54 mg
	U_{E1}	1.38	0.5544	0.5712	0.92	0.92
20 kg	U	3.5 mg			3.2 mg	
	U_{E1}	1.05			0.96	
50 kg	U	7.4 mg				
	U_{E1}	0.89				

1 kg – 50 kg		IMBiH		INM		ME-BoM	
m_{ref_N}		1 kg		1 kg		1 kg	
U_{ref}		0.15 mg		0.10 mg		0.08 mg	
Number of equations		17		11		12	
Type of comparator		Automatic	Automatic	Automatic	Manual	Automatic	Automatic
Range		1 kg – 10 kg	10 kg – 50 kg	1 kg – 10 kg	20 kg	1 kg – 10 kg	20 kg
d		0.01 mg	1 mg	0.01 mg	0.2 mg	0.01 mg	1 mg
1 kg	U			0.11 mg		0.08 mg	
	U_{E1}			0.63		0.49	
2 kg	U	0.33 mg	0.32 mg	0.24 mg	0.23 mg	0.29 mg	0.3 mg
	U_{E1}	0.99	0.96	0.72	0.68	0.87	0.9
5 kg	U	0.79 mg	0.81 mg	0.54 mg		0.64 mg	
	U_{E1}	0.95	0.97	0.65		0.77	
10 kg	U	1.61 mg		1.23 mg		1.23 mg	1.23 mg
	U_{E1}	0.97		0.74		0.74	0.74
20 kg	U	3.77 mg	3.77 mg	2.33 mg		3.44 mg	
	U_{E1}	1.13	1.13	0.7		1.03	

Fig. 3. Detailed results of laboratories participating at follow-up case study in range 1 kg–50 kg.

pabilities for calibration of 20kg weights are limited. The goal uncertainty in this case is $U_{20kg_{E1}} = 3.3\ mg$ which was achieved by two laboratories (DMDM and INM). Details of comparators and achieved uncertainties are collected in Fig. 3.

Laboratories were asked to collect notes from measurements about manipulation of weights, environmental conditions or calculating results with RealMass software.

One major issue is when working with weights in range 1 mg–500 mg especially on manual mass comparators. The milligram weights are small so risk of losing such weights is increased. Another risk arises when working with weights above 100 g which must be stacked. Many laboratories use weights with knob which are harder to stack to complete required equation.

Many of these risks can be avoided by use of robotic mass comparators but these are expensive although as shown in reports the stability and uncertainties of measurements on robotic comparators are great.

Some laboratories calibrate weights of all classes or at least up to class F2. This reduces time which can be given to perform proper dissemination procedure. Even in this case laboratories proved their capabilities in E1 class.

RealMass software developed within the project was used to evaluate results and uncertainties in all participating laboratories. Although it can manage large dissemination schemes (evaluated with up to 250 equations and 50 wt), many laboratories prefer to evaluate mass of weights in terms of decade. Number of measurements in one decade is generally small enough to have proper control during preparation of input files for the software.

Although there were some discrepancies which are yet to be solved, all laboratories proved their capabilities for reaching uncertainty levels of class E1. It is believed all laboratories will be able to prove the capabilities during key comparisons as well.

4. Comparison of robotic and automatic mass comparator for dissemination of unit of mass

Laboratories of CMI and ME-BoM compared dissemination techniques used for automatic (CMI) and robotic (ME-BoM) mass comparators. The comparison focused on manipulation of weights, duration of dissemination process and time used to set up all data needed to run the measurements.

4.1. Measurement procedure of CMI

Laboratory of primary mass metrology of Czech metrology institute is equipped with manual and automatic mass comparators to perform calibration of mass unit in range 1 mg–10 kg on the level of E1. Within this study only limited range 100 g–1 kg is used. The measurements were done on vacuum mass comparator Mettler Toledo M-One.

Mettler Toledo M-One is mass comparator with maximum capacity 1 kg and resolution 0.1 μg. It has six positions for weights. The weights can be loaded through front window or through load-lock system.

Laboratory used 1 kg stainless steel weight marked 51701 as the reference weight for this comparison. The weight was calibrated by BIPM in 2021.

Other weights are from primary weight set 15936 and special cylindrical weights. Since it is not possible to load weights next to each other laboratory must use cylindrical weights for safer stacking of the selected weight composition. Only one weight of the standard OIML shape is used at any combination of the weights. The weight set in question is 15936 which is used for dissemination of the unit of mass for customers of the laboratory.

When calibrating E1 weights laboratory uses all possible combinations of measurement positions in the automatic mass comparators. M-One has six positions for weights so in total there are fifteen possible combinations of 2 positions to compare. Other mass comparators in the laboratory such as Mettler Toledo AT1006 have four positions which enables to use up to six combinations.

Within this comparison all positions were always used. After placement of the weights onto the load alternator the centring process started. It consisted of five times loading of each position to the weighing pan. Reason of this process is to align centre of gravity of the weight or weight combination with the weighing pan, so the pan does not move during the measurement.

Laboratory usually uses 10*ABBA procedure which is repeated three times for each combination. The duration of the measurement in this case is about 82 hours. For this study laboratory used only 6*ABBA repeated only one time for measurements with loads 500 g, 200 g, and 100 g. In this case the measurement time is 18 hours.

The complete dissemination procedure took 1 week to complete. New measurement was prepared each day. Setting up and centring process took about 1–2 hours. If another mass comparator with a smaller number of positions was used the length of the process would be longer by about 1 week. If standard procedure were used the length of the calibration would be longer by about 1 week as well.

4.2. Measurement procedure of ME-BoM

Mass calibration laboratory at Bureau of metrology from North Macedonia (BoM) is equipped with Robotic system to perform calibration of weights in range 1 mg–1 kg on the level of E1. The range between 100 g–1 kg was measured on Sartorius CCR10-1000.

Sartorius CCR10-1000 is fully automatic mass comparator (Robotic system) with maximum capacity of 1002 g, resolution 0.1 μg/1 μg and sixty-two magazine positions for the weights. The measurements are controlled and calculated the obtained results to the calibration certificate by ScalesNet-M PC software, provided with the CCR10-1000.

Laboratory from BoM used 1 kg stainless steel weight, E0 class of accuracy, OIML shape, serial number 25329512, manufacturer Sartorius. The weight was calibrated by CMI in 2020.

Eight weights were used for this study. 1 kg and 100 g were from the pilot study comparison for realisation of the mass scale (Euramet No. 1556). The other six were reference standards from the laboratory, E1 class of accuracy (500 g, two pieces, cylindrical, 200 g, 200 g and 100 g, discs). The disc-shaped weights were used because of the possibility to perform measurements on an automatic comparator for safer stacking of the selected weight composition.

BoM used CCR10-1000 with a repetition of three times for each combination. A design with twelve combinations was used for the measurements. For this study, mass laboratory used 6*ABBA cycles, with one pre-cycle for centring process. Reason of this pre-cycle is to align centre of gravity of the weight or weight combination with the weighing pan. Setting up and centring process took around 1 hour.

The total measurement time and calculation of uncertainty budget took 17 hours.

4.3. Results and discussion

The dissemination of the unit of mass with automatic mass comparators is time consuming process which takes up to 3 weeks for the range 100 g–1 kg depending on the required uncertainties of the measurements. Due to necessity of stacking the weights the operator must be experienced and careful when setting the weights. It is almost impossible to rely only on the standard OIML weights without use of disc or cylindrical weights. These weights add more complexity to the measurement system. On the other hand, if some measurement fails for any reason, it is easier to repeat just the wrong measurement.

Most of the issues described above should be solved with robotic mass comparators which can set up needed weight compositions without more weights.

5. Conclusions

One of the objectives of EMPIR project 19RPT02 RealMass is to develop and implement calibration methods to realise, improve and maintain the mass scale in countries where mass scale measurement capabilities are less developed. The development and implementation of the calibration methods for range 1 mg–20 kg was one of the key objectives of the project. This report studies development of laboratories which were to build capabilities for dissemination of mass scale.

The initial case study aimed to show overall agreement of initial dissemination procedure applied in developing laboratories to values of mass calibrated in different laboratory. The comparison of values from dissemination and calibration certificates was made by E_n number. Almost in all cases the E_n was smaller than 1 which indicates the dissemination procedure was applied correctly even after initial training only.

The follow-up case study was organized about 2 years after the initial case study. With knowledge gathered during the project the laboratories were asked to make dissemination process in two ranges and evaluate the results with the software developed as one part of the project. The aim was to achieve expanded uncertainties in range 0.001 mg–3 mg which corresponds to limit for uncertainty of class E1 according to OIML R111. All reported uncertainties were normalised to respective limit of class E1, so goal was to achieve values less than 1. The goal was achieved almost in all cases with minor exceptions for weights above 1 kg. This range is usually covered by automatic comparators, but some laboratories use manual comparators for weights above 10 kg where the standard deviation of the measurements could be larger than needed for class E1.

All laboratories proved their capabilities for dissemination of unit of mass in range 1 mg–20 kg with uncertainties 0.001 mg–3.0 mg. With such findings the objective related to implementation of the dissemination techniques for traceable calibration of the unit of mass in the range 1 mg–20 kg was successfully achieved.

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