Erratum to: Interlaboratory Comparison of Five Standard Weights of Class F₂ in Several Romanian Laboratories

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Abstract: This paper reports the results of measurements performed on five standard weights of class F₂, by several metrology laboratories throughout Romania. The comparison began in 2009 and concluded in 2010. Mass Laboratory of National Institute of Metrology (INM) acted as pilot laboratory (PL) and reference laboratory (RL) for the programme. Five standard weights of class F₂ (nominal values: 5 kg, 1 kg, 200 g, 100 g and 10 g) were sent to the participants. The laboratory's results are presented for each weight, along with the associated uncertainty and the normalized errors of each laboratory, with respect to the INM. The results obtained by the participating laboratories in this comparison can be used to validate the measurement capabilities (in the calibration of weights) claimed/approved under the quality management systems.

Keywords: Interlaboratory comparison; Normalized error; Pilot laboratory; Reference laboratory

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1. Introduction

By interlaboratory comparisons the individual performances of the participating calibration laboratories are determined and continuously monitored and thus identifying potential problems, if any, for which corrective actions are to be initiated. The results of interlaboratory comparisons are a way of monitoring the validity of the results of calibrations and provide confidence to the customer.

Initially, for this interlaboratory comparison, alongside the reference laboratory, the following 22 regional laboratories of Romanian Bureau of Legal Metrology announced their participation: Neamț, Vaslui, Suceava, Bacău, Botoșani, Iași, Craiova, Râmnicu Vâlcea, Slatina, Drobeta Turnu-Severin, Târgu Jiu, Brașov, Sibiu, București, Cluj-Napoca, Oradea, Satu Mare, Baia Mare, Zalău, Bistrița, Galați și Constanța.

Subsequently, two of them, Constanta and Suceava laboratories, announced their withdrawal from this interlaboratory comparison.

The interlaboratory comparison was performed between the remaining twenty-one laboratories during the period June 2009 to March 2010.

The transfer standards used in this comparison were carefully selected by the reference laboratory, RL, and the comparison scheme was chosen to minimize the influence of any instability in their mass.

The results were analyzed using E_n (normalized error) values, which is the most commonly used method for determining the performance of an individual calibration laboratory.

A pilot laboratory (PL) acts as coordinator for an interlaboratory comparison being responsible for preparing the transfer standards, for centralizing the results and also for preparing the final report of the intercomparison.

A reference laboratory (RL) establishes the reference values of the transfer standards at the beginning of the intercomparison, at the end, or when necessary.

For this interlaboratory comparison, Mass Laboratory of National Institute of Metrology (INM) acted as pilot (PL) and also as reference laboratory (RL).

2. Circulation Scheme

Interlaboratory comparison was organized using a circular scheme, with two petals. The first petal covered ten laboratories, while second one included eleven laboratories.

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3. Reference Values

The transfer standards (5 kg, 1 kg, 200 g, 100 g and 10 g) were stainless steel weights with a density of $(7,950 \pm 140) \text{ kg/m}^3$ for each weight (in accordance with B.7.9.3 of [1]). The reference values x_{ref} for transfer standards were determined by RL at three different times: at the beginning (June 2009), at the middle (October 2009) and at the end of interlaboratory comparison (March 2010).

It was found that the transfer standards of 200 g, 100 g and 10 g have a very good stability, with their drift being zero or very small. For the transfer standards of 5 kg and 1 kg, it was found that the stability of their mass value is satisfactory, the drift being smaller compared with the associated uncertainty. Even if the drift values were within the limits of uncertainty, the reference values were calculated taking into account of these small drift values.

4. Measurement Instructions

The following information about the transfer standards was given in advance to the participants: nominal masses and densities (with their uncertainties). The participants carried out the calibrations without re-determining the density of the weights.

In addition, instructions were given how to handle, store and transport the weights. For each laboratory the measurement time was 2 weeks.

Following the intercomparisons practice, for the confidentiality of results, each laboratory has been assigned a code (1–23), the reference laboratory (RL) having the number “1”.

No detailed calibration instructions were given to the laboratories. In accordance with measurement instructions, the participants were able to choose the method of calibration. Thus, laboratories have opted to use the Borda method, using one or more reference standards.

In addition, in the measurement instructions which accompanied the itinerant standards, was specified that before the determinations, any dust particles must be removed from the surface of a weight with brush, other methods of cleaning or washing being forbidden.

5. Participant's Tasks

It was the participant's tasks to determine the (conventional) mass values of the standards weights with the respective measurement uncertainties. The nominal values of the weights were selected such that the weighing

instruments and mass standards of the participants could be tested within a wide range. The evaluation by the participant was to furnish the following information:

- mass (conventional) and its uncertainty of the five mass standards;
- traceability of reference standards used;
- physical properties of the reference standards used;
- method used for calibration;
- specifications of the measuring instruments used (weighing instruments, barometers, hygrometers, thermometers);
- ambient atmospheric conditions at the time of each measurement.

The participants were requested to report the uncertainty budget in details.

6. Results of Laboratory Comparison

6.1. Generalities

A full calibration report with all relevant data and uncertainty estimates based on recommendation was requested to be sent to the RL.

Most laboratories have presented the results in accordance with the requirements of the technical protocol that accompanied the traveling standards.

Two of the 22 participating laboratories (3 and 23) decided to withdraw from the interlaboratory comparison while the laboratory no. 18 did not report their results for 5 kg weight, because it has no appropriate standards for this nominal value.

The results for the 200 g weight belonging to laboratory no. 16 were not considered, as they were submitted in corrected form after receiving the draft report.

6.2. Presentation of Results

6.2.1. Results in Table Form

Each laboratory reported the measured mass value that is assigned to each of the five artifacts, together with an expanded uncertainty for each weight. For all laboratories, the coverage factor was 2.

All data are reported on the sample as received. The results are presented exactly that were sent by the participants. In Table 1, the laboratory's codes were ordered in ascending way. The data obtained for the reference laboratory for all three calibrations, at beginning, middle and end, are also shown in Table 1.

A tool often used in analyzing the results from interlaboratory comparisons is the normalized error E_n , which

Table 1 Deviation (D) from nominal mass and expanded uncertainty (U) for each weight reported by the participants

Laboratory's code	5 kg		1 kg		200 g		100 g		10 g	
	D	U	D	U	D	U	D	U	D	U
	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg
1	9,9	10,1	1140,3	2,2	282,30	0,30	5620,51	0,18	-9,75	0,07
1	9,7	10,1	1140,1	2,2	282,30	0,30	5620,49	0,18	-9,74	0,07
1	9,6	10,1	1139,5	2,2	282,30	0,30	5620,45	0,18	-9,76	0,07
2	11	26	1141	5	282,3	1,0	5620,5	0,5	-9,75	0,20
3										
4	4	83	1140	16	282,3	1	5620,52	0,5	-9,75	0,20
5	5	25	1139,7	5,0	282,2	1,0	5620,5	0,5	-9,72	0,20
6	9	25	1141	5	282,3	1,0	5620,5	0,5	-9,74	0,20
7	77,00	64,88	1130,00	7,40	283,40	0,14	5620,90	0,12	-9,51	0,02
8	23,000	9,327	1129,508	4,579	282,543	0,218	5620,525	0,176	-9,735	0,121
9	12,2	8,8	1140,0	2,0	282,20	0,34	5620,51	0,34	-9,778	0,034
10	85	48	1140	10	282,4	0,1	5620,3	0,1	-9,7	0,1
11	102,0	26,3	1140,4	5,13	282,3	0,903	5620,5	0,43	-9,74	0,197
12	5	25	1139	5	282	1	5620,5	0,5	-9,8	0,2
13	23	20	1140	2	282,3	0,3	5620,5	0,3	-9,7	0,1
14	-13	26	1143,3	5	281,8	1,0	5620,4	0,5	-9,47	0,20
15	7	25	1138,5	5,0	282,4	1,0	5620,15	0,5	-9,65	0,20
16	7	25	1139,8	5,0			5620,5	0,5	-9,7	0,2
17	13,750	22,098	1134,954	3,550	282,406	0,376	5620,557	0,266	-9,739	0,210
18			1140,27	5	281,992	1	5619,507	0,5	-0,17	0,2
19	21,25	72,82	1134,25	6,26	282,84	1,22	5620,75	0,87	-9,69	0,42
20	-3	25	1139,5	5,0	282,3	1,0	5620,5	0,5	-9,75	0,20
21	9	1,504	1138	0,085	1,74	0,1	5620,47	0,07	-9,76	0,02
22	12	16	1140,5	2,4	282,1	0,6	5620,5	0,30	-9,61	0,08
23										

takes into account both the measured result and its uncertainty. The normalized error E_n is given as [2, 3]:

$$E_n = \frac{x_{\text{lab}} - x_{\text{ref}}}{\sqrt{U_{\text{lab}}^2 + U_{\text{ref}}^2}}$$

where E_n is the normalized error, x_{lab} is the result of measurement carried out by a participant laboratory, x_{ref} is the measured reference value of RL, U_{ref} is the measurement uncertainty of RL, U_{lab} is the measurement uncertainty reported by participant laboratory.

Using this formula, an acceptable measurement and reported uncertainty would result in an E_n value of between -1 and $+1$, with a desired value close to zero. The E_n data for each laboratory is presented in the Table 2.

The measurement uncertainty of RL in Table 2 is calculated taking into account the drift. The E_n calculation provides supplementary information concerning the measurement capability of the participating laboratories.

6.2.2. Results in Graphical Form

The measurement results thus compiled for participating laboratories along with reference values are graphically presented in Figs. 1a–5a and in expanded scale in Figs. 1b–5b. The horizontal red line represents the reference value while green line its measurement uncertainty. The vertical blue line depicts the measurement uncertainty of the participating laboratories. All the values of measurement uncertainty reported by the participating laboratories are at a coverage factor $k = 2$.

6.3. Analysis of Results

From the Table 2 it can be seen as following:

- for values of the normalized errors in the range $[-1...+1]$, laboratory “ i ” is compatible with RL and

Table 2 Normalized deviations E_n from reference values

Nominal value	5 kg	1 kg	200 g	100 g	10 g
U_{ref}	10,1	2,2	0,30	0,18	0,07
Laboratory's code	Normalized deviations E_n				
1	0,00	0,00	0,00	0,00	0,00
2	0,04	0,13	0,00	-0,02	0,00
3					
4	-0,07	0,02	0,00	0,10	0,01
5	-0,18	-0,09	-0,10	0,00	0,12
6	-0,03	0,13	0,00	-0,01	0,04
7	1,02	-1,32	3,32	1,88	3,40
8	0,97	-2,00	0,66	0,25	0,14
9	0,19	0,01	-0,22	0,08	-0,45
10	1,53	-0,02	0,32	-1,00	0,39
11	3,28	0,14	0,00	0,09	0,07
12	-0,17	-0,17	-0,29	0,04	-0,26
13	0,59	-0,04	0,00	0,02	0,34
14	-0,81	0,60	-0,48	-0,16	1,30
15	-0,10	-0,31	0,10	-0,66	0,45
16	-0,10	-0,06		0,01	0,20
17	0,17	-1,11	0,22	0,32	0,08
18		0,07	-0,30	-1,82	45,49
19	0,16	-0,83	0,43	0,32	0,14
20	-0,48	-0,14	0,00	-0,01	-0,01
21	-0,06	-0,69	-887,21	0,09	-0,02
22	0,12	0,14	-0,30	0,04	1,27
23					
				$E_n > 1$	
				$E_n < -1$	
				$U > 1/3 E_T$ for F_2 class

Fig. 1 The difference between participants' results and reference value and uncertainty for 5 kg weight. **b:** Details of **a** in expanded scale

is considered to be capable to perform calibration of class F_2 standard weights in the analyzed domain with corresponding measurement uncertainties;

– eight participating laboratories (2, 5, 6, 9, 12, 13, 15, 20) have obtained all the results compatible with those of the reference laboratory for class F_2 ;

Fig. 2 The difference between participants' results and reference value and uncertainty for 1 kg weight. **b:** Details of **a** in expanded scale

Fig. 3 The difference between participants' results and reference value and uncertainty for 200 g weight. **b:** Details of **a** in expanded scale

Fig. 4 The difference between participants' results and reference value and uncertainty for 100 g weight. **b:** Details of **a** in expanded scale

– results of laboratories (4, 10, 11, 17, 19), which are distinguished by yellow, are not compatible with those of the reference laboratory for class F₂, even the results are in the range $[-1...+1]$, because they reported uncertainties larger than one-third of the maximum permissible error (E_T) for class F₂;

– the results of the weights for which the normalized errors were not in the range $[-1$ to $+1]$, these were not analyzed for the measurement capability, because these results are inappropriate.

RL sent out the summary results in a draft report to all participants, with the code number in place of the laboratory

Fig. 5 The difference between participants' results and reference value and uncertainty for 10 g weight. **b:** Details of **a** in expanded scale

name. Each laboratory would see all results but would not know the owners of results other than its own.

The PL (RL) prepared the final report taking into account all comments and observations received from the participants.

Then, the final report is sent to participating laboratories and to all stakeholders.

7. Conclusions

Analyzing the results of interlaboratory comparison, it can be seen that approximately 14 % from the total results are discrepant.

Eleven laboratories (2, 4, 5, 6, 9, 12, 13, 15, 16, 19, and 20) have all the results consistent with those of the reference laboratory. Among them:

- eight laboratories (2, 5, 6, 9, 12, 13, 15, 20) have all the results compatible with those of the RL for accuracy class F_2 ;
- laboratory number 16 has four consistent results for accuracy class F_2 and a result could not be considered because it was corrected too late;
- laboratory number 4 has three compatible results for accuracy class F_2 ;
- for laboratory number 19, all the results are not compatible with those of the RL for accuracy class F_2 ;

Eight laboratories (8, 10, 11, 14, 16, 17, 21, and 22) have only one inappropriate result for accuracy class F_2 , while the laboratory 18 has two and the laboratory number 7 has all the results inappropriate.

The results obtained can be used to demonstrate the participating laboratory's measurement capability. The

participants who obtained results with E_n values outside the interval $[-1, +1]$ should analyze the reasons in order to remedy and correct them.

By analyzing the primary data and the measurement results, it is proposed that the participating laboratories with unsatisfactory results, should take corrective measures as follows [4, 5]:

- it is advisable that some participants must review the uncertainty analysis;
- in the case of a big difference between standard and test it is advisable to use additional weights so that this difference is as small as possible. Otherwise it would lead to increased uncertainty with a component associated to this difference;
- further improvement in technical skills and practice of personnel in calibrating and estimating uncertainty.

The results obtained can be used to demonstrate the participating laboratory's measurement capability and also in its own activity.

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