THE FINAL REPORT FOR APMP.M.M-K6.1: KEY COMPARISON OF 50 kg STAINLESS STEEL MASS STANDARD

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ABSTRACT

This report describes the result of an Asia Pacific Metrology Programme (APMP) regional comparison of 50 kg stainless steel mass standard carried out among 3 laboratories as a collaboration among inter-Regional Metrology Organization (RMO) programs. One weight of nominal value 50 kg has been prepared by CENAM (Mexico) of Sistema Interamericano de Metrología (SIM) for this APMP comparison as an international collaboration. The reported results by participants are consistent with each other and with the key comparison reference value (KCRV) of comparison, CCM.M-K6 to which this comparison has been linked.

1. INTRODUCTION

This key comparison was suggested by NIM (China) in APMP and based on a decision of CCM Key comparison (KC), CCM.M-K6. KRISS (Korea) was recognized as the pilot laboratory in this key comparison and invited CENAM (Mexico) in SIM to participate in this key comparison in August of 2016. CENAM was the pilot laboratory of the CCM key comparison and provided kindly one weight of 50 kg mass standards for this KC. Because these two laboratories also participated in the CCM.M-K6, they were expected to act as the linking laboratories in this regional KC to CCM.M-K6.

The way of evaluating result was agreed among the participants in advance and indicated in the protocol. The protocol was prepared finally on November 11, 2016.

2. PARTICIPANTS

Table 1 shows the contact and contributed persons information in each participant.

Table 1. Detail information of participants.

Country	Institute	Representative Contact	Contributed persons
Korea	Korea Research Institute of Standards and Science (KRISS)	Sungjun Lee lsjun@kriss.re.kr	Kwang Pyo Kim
Mexico	Centro Nacional de Metrologia (CENAM)	Luis Omar Becerra lbecerra@cenam.mx	Luis Manuel Ramírez
China	National Institute of Metrology (NIM)	Ruilin Zhong zhongrl@nim.ac.cn	Jian Wang

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Figure 1. The package of transfer standard provided by CENAM.

3. TRANSFER STANDARD

The travelling standard for this comparison was provided by CENAM and was a cylindrical shaped 50 kg weight made in one piece of stainless steel, as shown in Figure 1. The transfer standard had been monitored regularly and confirmed at CENAM. Some values of volume, density and magnetic properties of the weights were also measured at CENAM before the circulation of the weights. The physical data of the travelling standards are listed in Table 2.

The transfer package consisted of 1) 50 kg standard weight protected by 2) aluminum container with air-tight valve and 3) Sponge material, 4) eyebolt for 5) the handling fork, 6) two spanners, 7) box with gloves and brush, and 8) outer container made of hard-plastic and locked its cover by 9) two padlocks. The size and weight of transfer package was approximately 67.3 cm × 67.3 cm × 70 cm and 113.5 kg.

When the package arrived at the participating laboratory, a visual inspection of the surfaces was made and then the results were reported to the pilot lab using the corresponding paper sheet annexed in protocol.

Table 2. The physical quantity of the transfer standard

Identification	K6-01
Nominal Value	50 kg
Density at 20 ºC / Standard uncertainty	8009.15 kg/m^3 / 0.28 kg/m^3
Volume at 20 ºC / Standard uncertainty	6242.86 cm^3 / 0.22 cm^3
Coefficient of volumetric thermal expansion	0.000045 / K
Magnetic susceptibility (χ)	< 0.01
Magnetization	< 1 μT
Surface roughness (R_z)	< 0.5 μm
Surface roughness (R_a)	< 0.1 μm
Height	289 mm
Diameter	185 mm
Height of centre of gravity above base	163 mm

4. CIRCULATION OF THE TRANSFER STANDARDS

Table 3 shows the actual schedule for the circulation of transfer standard. The transfer standard was delivered to participant laboratories by a commercial courier without arranging ATA-Carnet for customs clearance when crossing international borders.

Table 3. The schedule of measurement

Start Date	End Date	Country
Dec. 1, 2016	Feb. 25, 2017	Mexico
Mar. 3, 2017	Mar. 22, 2017	Korea*
Jun. 19, 2017	Jul. 21, 2017	China
Aug. 3, 2017	Oct. 10, 2017	Korea**
Oct 24, 2017	Nov. 15, 2017	Mexico

* The transfer package was sent to China on May 22, 2017. It delayed in exportation process for obtaining a special permission from a boarder control of China.

** Stability check for a short time at KRISS.

5. MASS COMPARATORS USED BY PARTICIPANTS

The weighing instruments used by participating laboratories are listed in Table 4. It is worth mentioning that KRISS had used a different comparator from that used in CCM.M-K6 (Sartorius, CC50001S-L with resolution of 1 mg).

Table 4. Weighing Instruments of participants

NMI	Manufacturer	Model	Capacity	Resolution
KRISS	Mettler-Toledo	AX64004	64 kg	0.1 mg
CENAM	Mettler-Toledo	AX64004	64 kg	0.1 mg
NIM	Mettler-Toledo	AX64004	64 kg	0.1 mg

Note: Based on the information provided by participants without any verification. Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by any of the participating organizations nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

6. REPORTED RESULTS FROM PARTICIPANTS

The reported result are expressed as the mass error, e_i (for i-th lab), in reference to 50 kg nominal value. Table 5 shows the mass error and its associated standard uncertainty u_i (at $k = 1$).

In the consideration of reported values of first and second measurements at KRISS and CENAM, it can be concluded that no significant effect associated with drift in the transfer standard need be considered.

Based on the data of Table 5 and Reference 1, a chi-square test was performed to evaluate the consistency of reported results. The observed chi-square (χ_{obs}) value was calculated as 0.65. By consideration of the chi-square values (5.99) at 2 degrees of freedom and significance level of 0.05, the consistency was found in this set of measurement.

Table 5. The raw data reported by the participants

NMI	Mass Error (50 kg) in mg	Uncertainty ($k=1$) in mg
CENAM	2.2	2.2
KRISS	-0.4	1.8
NIM	0.3	1.5
KRISS	-0.6	1.8
CENAM	1.3	2.1

7. ANALYSIS OF RESULTS

The non-linked reference value $NLRV$, the degrees of equivalence D_i , its associated expanded uncertainty of degrees of equivalence $U_{D,i}$, and normalized errors $E_{n,i}$ could be calculated for the i -th laboratory according to Reference 1, as already indicated in the protocol. Here, the weighted mean was chose as the best estimator in evaluating $NLRV$ using mass error and its standard uncertainty indicated in Table 5. The result of degree of equivalence with its uncertainty and normalized error were shown in Table 6 and depicted in Figure 2.

$$NLRV = 0.41 \text{ mg} \tag{1}$$

$$D_i = e_i - NLRV \tag{2}$$

$$U_{D,i} = 2 \times \sqrt{u_i^2 - u_{NLRV}^2} \tag{3}$$

$$E_{n,i} = D_i / U_{D,i} \tag{4}$$

Table 6. The deviation from $NLRV$ with uncertainty and normalized error

NMI	D_i in mg	$U_{D,i}$ in mg	$E_{n,i}$
KRISS	-0.87	2.95	-0.29
CENAM	1.38	3.77	0.37
NIM	-0.07	2.18	-0.03

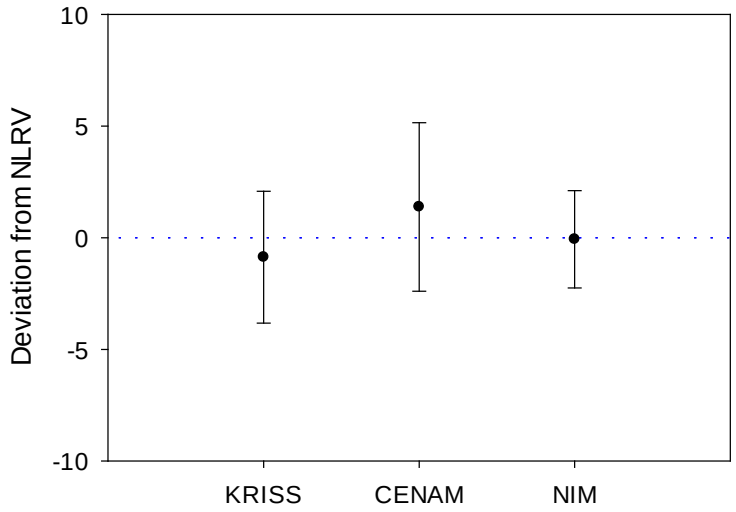

Figure 2. Deviation from $NLRV$.

8. LINK TO CCM.M-K6

In order to show the equivalence with CCM key comparison, the author referred a similar analysis published in previous regional key comparison [2]. KRISS and CENAM have participated in CCM.M-K6 and acted as linking laboratories. Their degrees of equivalence, $D_{0,i}$ and its associated expanded uncertainty $U_{0,i}$ for the i-th laboratory shown in CCM comparison are indicated in Table 7 [3].

Table 6. Degree of equivalence of linking labs in CCM.M-K6

NMI	$D_{0,i}$ in mg	$U_{0,i}$ in mg
KRISS	-3.06	8.78
CENAM	0.90	2.28

The reference values of the current comparison linked to CCM.M-K6 (Linked Reference Value, LRV) could be calculated as the weighted mean of $e_i - D_{0,i}$ for the i-th laboratory as shown in the following equations.

$$LRV = \frac{w_{KRISS}(e_{KRISS} - D_{0,KRISS}) + w_{CENAM}(e_{CENAM} - D_{0,CENAM})}{w_{KRISS} + w_{CENAM}} \tag{5}$$

$$w_i = \frac{1}{u^2(e_i - U_{0,i})} = \frac{1}{u_i^2 + U_{0,i}^2 / 4} \tag{6}$$

If there was no correlation between CCM and regional comparisons, the standard uncertainty associated to the linked reference value (LRV) could be expressed as the following equation.

$$u_{LRV} = \frac{1}{\sqrt{w_{KRISS} + w_{CENAM}}} \tag{7}$$

By inserting all data in Table 6, LRV and u_{LRV} could be calculated as 1.21 mg and 2.20 mg, respectively. In the same way with evaluating non-linked degree of equivalence, the linked degree of equivalence, $D_{L,i} = e_i - LRV$ could be obtained easily. The linked normalized error could be estimated with the following equation.

$$E_{nL,i} = \frac{D_{L,i}}{2\sqrt{u_i^2 + u_{LRV}^2}} \tag{8}$$

Table 7. The linked degree of equivalence with its expanded uncertainty and linked normalized error

NMI	$D_{L,i}$ in mg	$U_{DL,i}$ in mg	$E_{nL,i}$
KRISS	-1.71	5.63	-0.30
CENAM	0.54	6.10	0.09
NIM	-0.91	5.27	-0.17

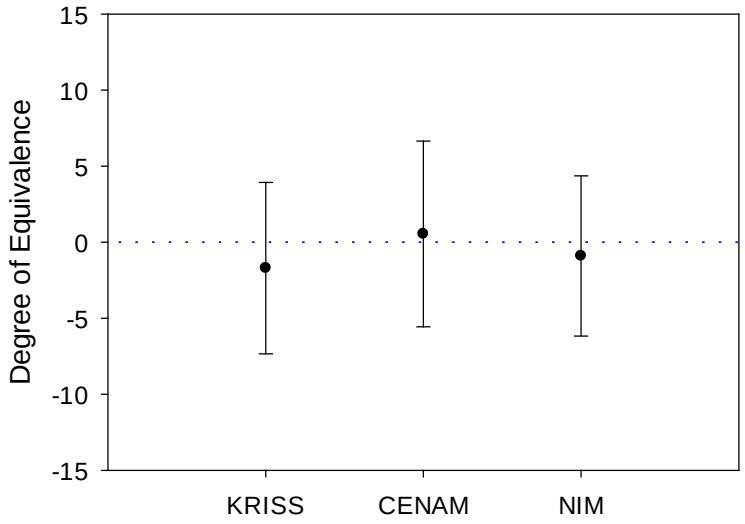

Figure 3. The linked degree of equivalence

9. SUMMARY AND CONCLUSIONS

This report summarizes the procedure and analysis of APMP.M.M-K6.1, a Key Comparison of 50 kg stainless steel mass standard. The result of each participant is consistent at the nominal value of 50 kg. The result of NIM is also consistent with KCRV of CCM.M-K6.

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