Comparison in calibration of mass and conventional mass SIM.M.M-S11

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Abstract. The results of the supplementary comparison in calibration of mass and conventional mass SIM.M.M-S11 are reported. This comparison was organized by seven mass laboratories members of Inter-American Metrology System SIM and stainless steel standard weights with nominal values 2kg, 1kg, 200g, 50g, 1g and 200mg were chosen. These values link this comparison with the key comparisons CCM.M-K4 and CCM.M-K5. For the evaluation of the reference value and the associated uncertainty the method of least squares has been chosen, and the determination of the degrees of equivalence was calculated using the normalized deviation criteria. The results show a general consistency of the measurements in mass and conventional mass between the participant laboratories.

1. Introduction

The metrological equivalence in measurements performance by different national metrology institutes (NMIs) is evaluated through key and supplementary comparison, which are discussed and organized by the Consultative Committees of the International Committee for Weights and Measures (CIPM) and by the Regional Metrology Organizations (RMOs). In March 2012, a meeting of the working group of mass of Inter-American Metrology System (SIM) SIM MWG7 carried out in La Paz – Bolivia decided to organize two supplementary comparisons including the calculation of mass and conventional mass, density and volume for E2 standard weights made of stainless steel, both in order to guarantee the traceability for E2 standard weights. The results of the measurements in mass and conventional mass are reported in the supplementary comparison SIM.M.M-S11; the results of the measurements in volume are reported in the supplementary comparison SIM.M.D-S5. The supplementary comparison was piloted by the Servicio Ecuatoriano de Normalización (INEN, Ecuador) and the Centro Nacional de Metrología (CENAM, México) as support laboratory. Due to internal situations in INEN, after the second semester of 2013 the Instituto Nacional de Metrología de Colombia (INM-CO) continued with the activities of the pilot laboratory.

2. Participant NMIs and Traveling Standards

Seven NMIs took part in the comparison: CENAM (México), IBMETRO (Bolivia), INACAL (Perú), INM (Colombia), INEN (Ecuador), INTN (Paraguay) and LACOMET (Costa Rica); all the participant laboratories are belonging to SIM. The traveling standards used were a set of weights OIML class E2 belonging to the project FOMENTO COORDINADO DE LA INFRAESTRUCTURA DE LA CALIDAD EN LA REGIÓN ANDINA coordinated by the Physikalisch-Technische Bundesanstalt PTB and the Andean Community CAN. The nominal values selected for the comparison were 2 kg, 1 kg, 200 g, 50 g, 1 g and 200 mg. The standards weights were prepared by CENAM who
measured the volume and magnetic properties in order to verify that all of them are in accordance with the requirements for E2 accuracy class of the OIML R 111-1:2004 [1], with the exception of the 200 mg weight.

The participant laboratories determined the mass and the conventional mass and their associated uncertainties for each standard using their own facilities, instruments and procedures according to the best capability of the laboratory. The calibration was done in mass value and the conventional mass value was calculated from the mass value. Air density was determined with CIPM-2007 formula, in order to correct air buoyancy effect [2].

3. Measurement Methods and Procedure
The subdivision method was used by CENAM, INEN and INACAL. The laboratories of INTN, INM, LACOMET and IBMETRO used the direct comparison method. IBMETRO decided to do not report mass values, so only conventional mass was considered from IBMETRO for this comparison. The results reported by all the participant laboratories, as well as the uncertainty analysis, were made according to “Guide to the expression of Uncertainty in Measurements” GUM [3].

Before to determinate the reference values, a consistency check between the pilot laboratory (INEN) and the support laboratory (CENAM) was performed, in order to verify if the measurements of the pilot laboratory are equivalent to those made by the support laboratory and, in this way, to ensure the reference value established by the pilot laboratory. The normalized error criteria [4] was used to check the consistency between these measurements, according to the equation (1),

\[ E_n = \frac{\Delta x_{\text{INEN}} - \Delta x_{\text{CENAM}}}{\sqrt{U^2(\text{INEN}) + U^2(\text{CENAM})}} \]  (1)

Table 1 shows the normalized error calculated for mass and conventional mass values reported by CENAM and INEN.

| Nominal value | 2 kg | 1 kg | 200 g | 50 g | 1 g | 200 mg |
|---------------|------|------|-------|------|-----|--------|
| Normalized error in mass | 0.64 | 0.29 | -0.35 | -0.15 | -0.22 | 0.12 |
| Normalized error in conventional mass | 0.63 | 0.25 | -0.33 | -0.14 | -0.14 | 0.09 |

A progressive damage of the traveling standards and an apparent deformation of the transportation box were noticed after the end of the complete cycle of circulation (see Figure 1); this fact potentially affects the stability of the standards, especially the 200 g and 50 g weights. Measurements of traveling standards made by INEN and CENAM before and after the comparison confirmed a strong drift at the beginning of the comparison, which would happen during the transportation from Quito (Ecuador) to Lima (Perú). Because of this first values reported by INEN and CENAM were rejected, so the drift and the uncertainty due to stability of each standard were calculated considering values reported by INACAL and final measurement of CENAM.

The reference values for mass and conventional mass are calculated using the method of least squares according to the mathematical model proposed in [5], using the results reported by each participant laboratory. According to the stability of standards described in section 3.3, first results reported by CENAM and INEN are not considered in the calculations. The reference value for mass (and conventional mass) is calculated using the following equation,
\( \Delta \hat{m} = (X^T \Sigma^{-1} X)^{-1} \cdot (X^T \Sigma^{-1} Y) \) \hspace{1cm} (2)

Figure 1. Upper: Damages on the surface of standards. Left: 200 g; right: 50 g; Lower: Apparent deformation of the transportation box.

In which \( X \) is the design matrix, \( Y \) is the matrix of the results reported by each participant laboratory and \( \Sigma \) is the covariance matrix which include the uncertainty reported by the laboratories, their possible covariance and the drift of the standards. The uncertainty of the reference value for mass (and conventional mass) is calculated by the equation,

\[ u(\Delta \hat{m}) = \left[ (X^T \Sigma^{-1} X)^{-1} \right]^{1/2} \] \hspace{1cm} (3)

Finally, a chi-squared test is done for analysing the consistency of the estimated value, using the equation

\[ \chi^2_{obs} = (Y - X \cdot \Delta \hat{m})^T \cdot \Sigma^{-1} \cdot (Y - X \cdot \Delta \hat{m}) \] \hspace{1cm} (4)

The criterion used for the consistency of the estimated value is given for a confidence level of 95% with a probability \( p \) calculated using the inequality given in equation (5), with a number of degrees of freedom \( v = 5 \) for consistency of mass values and \( v = 6 \) for consistency of conventional mass values.

\[ p = P(\chi^2(v) > \chi^2_{obs}) \] \hspace{1cm} (5)

The degrees of equivalence between the participant laboratories and the reference value for mass correction (and conventional mass correction) are calculated using the normalized deviation criteria, according to the equation

\[ d_i = \frac{D_i}{u(D_i)} \] \hspace{1cm} (6)

where \( D_i \) is the difference between the value reported by each participant laboratory and the reference value, and \( u(D_i) \) is calculated using the standard uncertainty calculated by each participant laboratory, the reference value and the stability of weights.

4. Conclusions

There is a general consistency of the measurements in mass correction and conventional mass correction values between the participant laboratories, although one of these has an outlying for the conventional mass value of 1 kg weight. Other participant laboratories reported and
uncertainty larger than one third of the maximum permissible error for weights of 2 kg class E2 OIML. This comparison will help to these laboratories to identify potential problems and to take corrective actions in their procedures or methods. The strong drift in traveling standards measured in the weights during the comparison, especially in 200 g and 50 g weights, is related to the visible damage on their surface. This damage can be associated with an apparent deformation of the transportation box, but even so is difficult to explain the scratches reported in this paper.

References
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