Supplementary Comparison of the calibration of a 5 kg stainless steel standard weight between INDECOPI-PERÚ and CEM-ESPAÑA

(SIM.M.M-S10)

By

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And

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ESPAÑA

APRIL, 2012

Abstract:

This report summarizes the results of the supplementary comparison (SIM.M.M-S10) of a 5 kg stainless steel mass standard between INDECOPI-PERÚ and CEM-ESPAÑA.

The objective of the comparison is to demonstrate the metrological equivalence between both laboratories. The results of the comparison will be used to support the Calibration and Measurement Capability (CMC) at 5 kg.
1) Introduction

This report describes the results of the supplementary comparison of a 5 kg stainless steel mass standard between INDECOPI-PERÚ and CEM-ESPAÑA.

This comparison was undertaken by INDECOPI and CEM and was based on a decision taken on October 3, 2011.

The supplementary comparison was piloted by INDECOPI. This comparison was registered as an official supplementary comparison under the designation SIM.M.M-10.

The travelling standard belonged to INDECOPI. It was an OIML E2 weight.

The travelling standard was prepared by INDECOPI. INDECOPI measured the volume of the travelling standard.

The standard was circulated between both NMIs. Each NMI determined its mass and uncertainty using their procedures and methods. The measurements were carried out from January to March 2012.

2) Participants

The data of the participant laboratories are listed in table 1.

<table>
<thead>
<tr>
<th>National Metrology Institute Delivery address</th>
<th>Acronym</th>
<th>Technical Contact</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centro Español de Metrología, Área de masa Calle del Alfar n° 2 28760 Tres Cantos-Madrid SPAIN</td>
<td>CEM</td>
<td>Nieves Medina/Angel Lumbreras&lt;br&gt;<a href="mailto:mnmmedina@cem.mityc.es">mnmmedina@cem.mityc.es</a>&lt;br&gt;Tel: +34 91 807 47 89&lt;br&gt;Fax: +34 91 807 48 07</td>
</tr>
<tr>
<td>Instituto Nacional de Defensa de la Competencia y de la Protección de la Propiedad Intelectual Servicio Nacional de Metrología Laboratorio de Masas Calle de la Prosa 104, San Borja – Lima PERÚ</td>
<td>INDECOPI</td>
<td>Aldo Quiroga / Luz Cori&lt;br&gt;<a href="mailto:aquiroga@indecopi.gob.pe">aquiroga@indecopi.gob.pe</a>&lt;br&gt;Tel: +51 1 224 7800. Ext. 1662&lt;br&gt;Fax: +51 1 224 7800. Ext. 1264</td>
</tr>
</tbody>
</table>
Acknowledge: The technical contacts of participants laboratories wish to thank to Francisco J. Garcia of CESMEC-Chile (table 2) for acting as a third laboratory which received results from both participants and checked consistency before to sharing the results with INDECOPI and CEM.

<table>
<thead>
<tr>
<th>National Metrology Institute</th>
<th>Acronym</th>
<th>Technical Contact</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centro de Estudios de Medición y Certificación de Calidad División de Metrología Av. Marathon 2595, Macul Santiago,CHILE</td>
<td>CESMEC</td>
<td>Francisco J. Garcia Leoro <a href="mailto:fgarcia@cesmec.cl">fgarcia@cesmec.cl</a> Tel: + 56 2 350 21 85 Fax: + 56 2 350 21 83</td>
</tr>
</tbody>
</table>

3) Travelling standard

The travelling standard used was a stainless steel weight Class OIML E2 with nominal value 5 kg. Annex A shows the travelling standard photograph.

The travelling standard details are listed in table 3.

INDECOPI provided the following data:

- Weight volume at 20 °C.
- Thermal expansion coefficient.
- Magnetic properties of the travelling standard such as volume magnetic susceptibility and magnetic polarization.

<table>
<thead>
<tr>
<th>Manufacturer / Identification</th>
<th>Volume at 20 °C (cm³)</th>
<th>Standard uncertainty (cm³)</th>
<th>Thermal expansion coefficient (x10⁻⁶ °C⁻¹)</th>
<th>Volume magnetic susceptibility</th>
<th>Magnetic polarization /µT</th>
</tr>
</thead>
<tbody>
<tr>
<td>KERN&amp; SOHN/LM-01-004</td>
<td>638,064</td>
<td>0,033</td>
<td>50</td>
<td>0,0049</td>
<td>0,127</td>
</tr>
</tbody>
</table>

4) Circulation Schedule

The circulation schedule of the travelling standard is shown in table 4. INDECOPI started their measurements in January 2012 and the last measurements were also made by INDECOPI in March 2012.

<table>
<thead>
<tr>
<th>NMI</th>
<th>Date of arrival</th>
<th>Date of departure</th>
</tr>
</thead>
<tbody>
<tr>
<td>INDECOPI</td>
<td></td>
<td>27/01/2012</td>
</tr>
<tr>
<td>CEM</td>
<td>30/01/2012</td>
<td>17/02/2012</td>
</tr>
</tbody>
</table>
Surface damages of the standard: The travelling standard was examined by each NMI at its reception and departure in order to register all marks and damages during circulation. The data sent by the participating laboratories showed that there was no significant damage on the travelling standard.

5) Procedures and measurement methods

The measurement method used by both laboratories was the substitution method. The laboratories used their own facilities, instruments and methods. Each participant laboratory determined the corrections and associated uncertainties (in mass and conventional mass value) of the 5 kg stainless steel standard weight.

The density of the air was calculated by equation CIPM-2007[1].

The resolution of the weighing instruments used by the laboratories is shown in Table 5.

<table>
<thead>
<tr>
<th>Nominal Value</th>
<th>Laboratories</th>
<th>INDECOPI</th>
<th>CEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 kg</td>
<td></td>
<td>0.2 mg</td>
<td>0.01 mg</td>
</tr>
</tbody>
</table>

The expanded uncertainty (k=2) of the instruments used by the laboratories to measure the environmental conditions are shown in Table 6

<table>
<thead>
<tr>
<th>Climatic</th>
<th>Laboratories</th>
<th>INDECOPI</th>
<th>CEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td></td>
<td>0.35 °C</td>
<td>0.03 °C</td>
</tr>
<tr>
<td>Pressure</td>
<td></td>
<td>0.36 mbar</td>
<td>0.3 mbar</td>
</tr>
<tr>
<td>Humidity</td>
<td></td>
<td>2.9 % hr</td>
<td>0.1 °C</td>
</tr>
</tbody>
</table>

Table 7 shows the traceability and the calibration dates of the mass standards used by the laboratories, to indicate the possible correlation of the traceability source of the standards.

<table>
<thead>
<tr>
<th>Nominal Value</th>
<th>Laboratories</th>
<th>INDECOPI</th>
<th>CEM</th>
</tr>
</thead>
</table>

6) Results of the measurements

Table 8 and Figure 1 shows the mass correction found by the laboratories and its associated uncertainty for a confidence level of 95.45%. The conventional mass results obtained by the laboratories are showed by Annex B.
Uncertainty analysis has been made for both laboratories according to GUM [2].

### Table 8
Results of the Comparison

<table>
<thead>
<tr>
<th>Laboratorie</th>
<th>Nominal Value</th>
<th>Mass Correction</th>
<th>Uncertainty $k = 2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>INDECOPI 1</td>
<td>5 kg</td>
<td>17,5 mg</td>
<td>1,2 mg</td>
</tr>
<tr>
<td>CEM</td>
<td>5 kg</td>
<td>17,28 mg</td>
<td>0,54 mg</td>
</tr>
<tr>
<td>INDECOPI 2</td>
<td>5 kg</td>
<td>17,4 mg</td>
<td>1,2 mg</td>
</tr>
</tbody>
</table>

The stability of the travelling standard was assessed by INDECOPI by measuring the mass values before and after the comparison. The uncertainty due of the stability of the artefact during January to March 2012 was calculated using the next equation:

$$u(\Delta m) = \sqrt{\frac{(Y_{INDECOPI} - Y_{INDECOPI})^2}{12}}$$

where:

- $Y_{INDECOPI}$: Mass measurement by INDECOPI in January 2012
- $Y_{INDECOPI}$: Mass measurement by INDECOPI in March 2012
7) Mass Reference value of the intercomparison and $\chi^2$ test:

Table 9 includes INDECOPI mass value: $\gamma_{\text{INDECOPI}}$, CEM mass value: $\gamma_{\text{CEM}}$, and the reference mass value of the comparison: $\gamma_{\text{Ref}}$ with their uncertainties: $u(\gamma_{\text{INDECOPI}})$, $u(\gamma_{\text{CEM}})$, and $u(\gamma_{\text{Ref}})$. It also includes the observed chi squared value $\chi^2_{\text{obs}}$, as well as the probability: $P \left( \chi^2_{\text{obs}} > \chi^2_{\text{Ref}} \right)$. If this probability is more than 0.05 the measurement value is inconsistent[3].

**Table 9**
INCECOPI, CEM and Reference mass Values with their uncertainties. Evaluation for the consistency of 5 kg comparison.

<table>
<thead>
<tr>
<th>$\gamma_{\text{INDECOPI}}$</th>
<th>$\gamma_{\text{CEM}}$</th>
<th>$\gamma_{\text{Ref}}$</th>
<th>$\Delta$</th>
<th>$\chi^2_{\text{obs}}$</th>
<th>$P \left( \chi^2_{\text{obs}} &gt; \chi^2_{\text{Ref}} \right)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 kg +17.5 mg</td>
<td>0.6 mg</td>
<td>5 kg +17.28 mg</td>
<td>0.27 mg</td>
<td>5 kg +17.30 mg</td>
<td>0.26 mg</td>
</tr>
</tbody>
</table>

**Degrees of equivalence between the INDECOPI and the CEM:**

Degree of equivalence with the reference value of the intercomparison:

INDECOPI:

$$D_{\text{INDECOPI}} = \gamma_{\text{INDECOPI}} - \bar{\gamma}$$  \hspace{1cm}  $$U(D_{\text{INDECOPI}}) = 2 \sqrt{u^2(\gamma_{\text{INDECOPI}}) + u^2(\bar{\gamma})}$$

$$d_{\text{INDECOPI}} = \frac{\gamma_{\text{INDECOPI}} - \bar{\gamma}}{\sqrt{u^2(\gamma_{\text{INDECOPI}}) + u^2(\bar{\gamma})}}$$

CEM:

$$D_{\text{CEM}} = \gamma_{\text{CEM}} - \bar{\gamma}$$  \hspace{1cm}  $$U(D_{\text{CEM}}) = 2 \sqrt{u^2(\gamma_{\text{CEM}}) + u^2(\bar{\gamma})}$$

$$d_{\text{CEM}} = \frac{\gamma_{\text{CEM}} - \bar{\gamma}}{\sqrt{u^2(\gamma_{\text{CEM}}) + u^2(\bar{\gamma})}}$$

Degree of equivalence between INDECOPI – CEM:

$$D_{\text{INDECOPI-CEM}} = \gamma_{\text{INDECOPI}} - \gamma_{\text{CEM}}$$

$$d_{\text{INDECOPI,CEM}} = \frac{d_{\text{INDECOPI}} - d_{\text{CEM}}}{\sqrt{2 - 2r(d_{\text{INDECOPI}}, d_{\text{CEM}})}}$$
Degrees of equivalence between the INDECOPI and the CEM are shown in table 10, table 11, table 12, Figure 2 and Figure 3.

### Table 10
Degree of equivalence for INDECOPI with its uncertainty

<table>
<thead>
<tr>
<th>Nominal Value</th>
<th>$D_{INDECOPI}$</th>
<th>$U(D_{INDECOPI})$</th>
<th>$d_{INDECOPI}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 kg</td>
<td>0.20 mg</td>
<td>1.08 mg</td>
<td>0.37</td>
</tr>
</tbody>
</table>

### Table 11
Degree of equivalence for CEM with its uncertainty

<table>
<thead>
<tr>
<th>Nominal Value</th>
<th>$D_{CEM}$</th>
<th>$U(D_{CEM})$</th>
<th>$d_{CEM}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 kg</td>
<td>-0.02 mg</td>
<td>0.14 mg</td>
<td>-0.29</td>
</tr>
</tbody>
</table>

### Table 12
Degree of equivalence between INDECOPI and CEM with its uncertainty

<table>
<thead>
<tr>
<th>Nominal Value</th>
<th>$D_{INDECOPI-CEM}$</th>
<th>$d_{INDECOPI-CEM}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 kg</td>
<td>0.22 mg</td>
<td>0.33</td>
</tr>
</tbody>
</table>

Annex C shows the estimation of Mass Reference value of the comparison and $\chi^2$ test.
Figure 2
Degree of equivalence ($k=2$) of results

Figure 3
Normalized deviations $\delta$ of results

Note: Results with $|\delta| > 2\sigma$ are classified as discrepant results.
References


Annexes

A. Photograph
B. Results of Conventional Mass Comparison
C. Estimation of Mass Reference value of the comparison and $\chi^2$ test.
Annex A

Photograph of the travelling standard weight

5 kg stainless steel travelling standard
Annex B

Table B.1 and Figure B.1 show the laboratories results for the conventional mass comparison and their associated uncertainties.

CEM reported an uncertainty for a weight of class OIML E1 of 5 kg and INDECOPI for a weight of class OIML E2 of 5 kg.

### Table B.1
Results of Conventional Mass Comparison

<table>
<thead>
<tr>
<th>Laboratorie</th>
<th>Nominal Value</th>
<th>Convetional Mass Correction</th>
<th>Uncertainty $k = 2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>INDECOPI 1</td>
<td>5 kg</td>
<td>1,8 mg</td>
<td>1,2 mg</td>
</tr>
<tr>
<td>CEM</td>
<td>5 kg</td>
<td>1,60 mg</td>
<td>0,54 mg</td>
</tr>
<tr>
<td>INDECOPI 2</td>
<td>5 kg</td>
<td>1,8 mg</td>
<td>1,2 mg</td>
</tr>
</tbody>
</table>

### Figure B.1
Conventional Mass Correction

![Conventional Mass Correction Graph](image-url)
Annex C

The estimation of mass reference value of the comparison and $\chi^2$ test was made according the reference [3].

\[
x = \begin{pmatrix} 5 \text{ kg} & 17.5 \text{ mg} \end{pmatrix} \quad \text{(C.1)}
\]

\[
y = \begin{pmatrix} 5 \text{ kg} & 17.26 \text{ mg} \end{pmatrix} \quad \text{(C.2)}
\]

\[
\Sigma_{\text{meas}} = \begin{pmatrix} u^2(y_{\text{INDECOPI}}) & u^2(y_{\text{INDECOPI}, y_{\text{CEM}}}) \\ u^2(y_{\text{INDECOPI}, y_{\text{CEM}}}) & u^2(y_{\text{CEM}}) \end{pmatrix} \quad \text{(C.3)}
\]

The correlation between the standards weights of INDECOPI and CEM is high because the standard weight of INDECOPI was calibrated at CEM. That is the reason why the correlation shall be calculated according the equation D.6, item D4 of the reference [4].

The weighing equations (C.4) and (C.7) are correlated which is detailed below:

The CEM weighing equation of the calibration for the travelling standard is:

\[
m_t = m_{R,\text{CEM}} + \rho_a,\text{CEM}(V_e - V_{R,\text{CEM}}) + \Delta W_{1}\text{t} \quad \text{(C.4)}
\]

The INDECOPI weighing equation of the calibration for the travelling standard is:

\[
m_t = m_{R,\text{INDECOPI}} + \rho_a,\text{INDECOPI}(V_e - V_{R,\text{INDECOPI}}) + \Delta W_{1}\text{t} \quad \text{(C.5)}
\]

The CEM weighing equation of the calibration for the INDECOPI mass standard is:

\[
m_{R,\text{INDECOPI}} = m_{R,\text{CEM}} + \rho_a,\text{CEM}(V_e - V_{R,\text{CEM}}) + \Delta W_{\text{INDECOPI}} \quad \text{(C.6)}
\]

The INDECOPI weighing correlated equation of the calibration for the travelling standard is:

\[
m_t = (m_{R,\text{CEM}} + \rho_a,\text{CEM}(V_e - V_{R,\text{CEM}}) + \Delta W_{\text{INDECOPI}}) + \rho_a,\text{INDECOPI}(V_e - V_{R,\text{INDECOPI}}) + \Delta W_{1}\text{t} \quad \text{(C.7)}
\]

Then the correlated sensitivity coefficients, $c_i$, are calculated

- The sensitivity coefficients of (C.4) equations are:

\[
c_{\text{CEM}, m_{R,\text{CEM}}} = \frac{\partial m_t}{\partial m_{R,\text{CEM}}} = 1
\]

\[
c_{\text{CEM}, \rho_a,\text{CEM}} = \frac{\partial m_t}{\partial \rho_a,\text{CEM}} = V_e - V_{R,\text{CEM}}
\]

\[
c_{\text{CEM}, \Delta W_{1}\text{t}} = \frac{\partial m_t}{\partial \Delta W_{1}\text{t}} = -\rho_a,\text{CEM}
\]
The sensitivity coefficients of (C.7) equations are:

\[ c_{\text{INDECOPI}} \frac{\partial m}{\partial m_{R,\text{CEM}}} = 1 \]

\[ c_{\text{INDECOPI}} \frac{\partial V}{\partial V_{R,\text{CEM}}} = \rho_{a,\text{INDECOPI}} \]

\[ c_{\text{INDECOPI}} \frac{\partial R_{\text{CEM}}}{\partial R_{R,\text{CEM}}} = -\rho_{a,\text{CEM}} \]

The correlation INDECOPI-CEM is provided by the next equation:

\[ u(V_{\text{INDECOPI}}, V_{\text{CEM}}) = u^2(m_{R,\text{CEM}}) + \rho_{a,\text{CEM}} \cdot \rho_{a,\text{INDECOPI}} \cdot u^2(V_{\text{CEM}}) + \rho_{a,\text{CEM}} \cdot \rho_{a,\text{CEM}} \cdot u^2(V_{R,\text{CEM}}) \]

(C.8)

Information about correlated quantities and measurements:

\[ u(m_{R,\text{CEM}}) = 0.175 \text{ mg} \]

\[ u(V_{\text{CEM}}) = 0.033 \text{ cm}^3 \]

\[ u(V_{R,\text{CEM}}) = 0.010 \text{ cm}^3 \]

\[ \rho_{a,\text{CEM}} = 1.16153 \frac{kg}{m} \]

\[ \rho_{a,\text{CEM}} = 1.1685 \frac{kg}{m^3} \]

The INDECOPI-CEM correlation value is:

\[ u(V_{\text{INDECOPI}}, V_{\text{CEM}}) = 0.03216 \text{ mg}^2 \]

Then:

\[ \Sigma_{\text{new}} = \begin{bmatrix} (0.6 \text{ mg})^2 & 0.03216 \text{ mg}^2 \\ 0.03216 \text{ mg}^2 & (0.27 \text{ mg})^2 \end{bmatrix} \]

(C.9)

The uncertainty contribution due to the travelling standard stability is given by:

\[ \Sigma_{\text{object}} = \begin{bmatrix} u^2(\Delta m) & 0 \\ 0 & u^2(\Delta m) \end{bmatrix} \]

(C.10)
Therefore:

\[ \hat{y} = 5 \, \text{kg} + 17.30 \, \text{mg} \]
\[ u^2(\hat{y}) = V(\hat{y}) = 0.069 \, 07 \, \text{mg}^2 \]
\[ u(\hat{y}) = 0.26 \, \text{mg} \]
\[ V(y - \hat{y}) = \begin{bmatrix}
0.291 \, 77 \, \text{mg}^2 & -0.036 \, 90 \, \text{mg}^2 \\
-0.036 \, 90 \, \text{mg}^2 & 0.004 \, 67 \, \text{mg}^2
\end{bmatrix} \]

(C.13)

The INDECOPI normalized deviation is:

\[ d_{\text{INDECOPI}} = \frac{(15 \, \text{kg} + 17.50 \, \text{mg}) - (15 \, \text{kg} + 17.30 \, \text{mg})}{\sqrt{0.291 \, 77 \, \text{mg}^2}} = 0.37 \]

The CEM normalized deviation is:

\[ d_{\text{CEM}} = \frac{(15 \, \text{kg} + 17.28 \, \text{mg}) - (15 \, \text{kg} + 17.30 \, \text{mg})}{\sqrt{0.004 \, 67 \, \text{mg}^2}} = -0.29 \]

The correlation coefficient between INDECOPI and CEM is:

\[ r(d_{\text{INDECOPI}}, d_{\text{CEM}}) = \frac{-0.036 \, 90 \, \text{mg}^2}{\sqrt{0.291 \, 77 \, \text{mg}^2}, \sqrt{0.004 \, 67 \, \text{mg}^2}} = -1 \]

The Normalized deviation between INDECOPI and CEM is:

\[ d_{\text{INDECOPI}, \text{CEM}} = \frac{(0.37) - (-0.29)}{\sqrt{2 - 2(-1)}} = 0.33 \]

The chi square value
\[ \chi_{\text{obs}}^2 = 0.18 \]

and

\[ \Pr\{X^2(v - 1) > \chi_{\text{obs}}^2\} = 0.72 \]

**Symbols**

- \( m_t \): Mass of travelling standard
- \( m_{R,\text{CEM}} \): Mass of CEM standard
- \( m_{E,\text{INDECOPI}} \): Mass of INDECOPI standard
- \( V_t \): Volume of travelling standard
- \( V_{R,\text{CEM}} \): Volume of CEM standard
- \( V_{E,\text{INDECOPI}} \): Volume of INDECOPI standard
- \( \rho_{\text{air}} \): Air density of the \( ith \) measurement
- \( \Delta W_i \): Difference between the balance indications of the \( ith \) measurement