FINAL REPORT

Supplementary Comparison

COMPARISON IN 50 kg MASS STANDARDS

SIM.M.M-S12
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SUPPLEMENTARY COMPARISON

FINAL REPORT OF COMPARISON IN 50 kg MASS STANDARDS
AMONG CESMEC - INACAL - CENAM
SIM.M.M-S12

Fernando Garcia G.[1], Francisco García L[1]. Luz Cori Almonte[2], Donny Taipe Araujo[2], Aldo Quiroga Rojas[2], Luis Omar Becerra[3], Luis Manuel Peña[3].

1 Centro de Estudios de Medición y Certificación de Calidad, CESMEC / Chile
2 Instituto Nacional de Calidad, INACAL / Perú
3 Centro Nacional de Metrología, CENAM / México

1. General Information

This report describes the results of the supplementary comparison SIM.M.M-S12, planned and carried out to evaluate the difference among the laboratories and the reference in the calibration of 50 kg mass standards.

This comparison was undertaken by CESMEC (Chile) as pilot laboratory; CENAM (México) as support laboratory; and INACAL (Peru).

The travelling standard was a 50 kg weight, provided by CESMEC.

The standard was circulated among the NMIs. Each NMI determined their mass and uncertainty using their procedures and methods. The measurements were carried out from September 2012 to September 2014.

2. Data of participant NMIs and Technical Contacts

The participating laboratories and their respective technical contacts are listed in Table 1:

Table 1: Information about participants

	Country	Technical contact(s)
CESMEC	Chile	Fernando García G., Francisco García L.
INACAL	Peru	Luz Cori Almonte; DonnyTaipe Araujo; Aldo Quiroga Rojas
CENAM	Mexico	Luis Omar Becerra; Luis Manuel Peña

3. Travelling standard

The traveling standard, provided by CESMEC, was a stainless-steel weight of cylindrical shape with a lifting knob, with nominal value of 50 kg.

Volume and density of the weight were determined by CENAM by means of geometrical measurements, as shown in Table 2.

Table 2: Volume and density values of the weight

	Value	**Standard uncertainty** $(k = 1)$
Volume	6289,2 cm³	9,5 cm³
Density	7954 kg/m³	12 kg/m³

4. Circulation Schedule

The circulation schedule of the traveling standard is shown in Table 3. The planning schedule according to the comparison protocol was modified due to difficulties in the transportation and retention of the standards at customs in some countries.

Table 3: Comparison program

	Arrival date	**Departure Date**
CESMEC	--	2012-11
INACAL	2012-11	2013-06
CENAM	2013-06	2014-09
CESMEC	2014-09	--

5. Surface damages of the standard:

Each NMI at its reception and departure examined the travelling standards in order to register all marks and damages during circulation. No significant damage was reported by the laboratories.

6. Measurement conditions and methods

The upper and lower limits for environmental conditions in calibration reported by the participating laboratories are listed in Table 4.

Table 4: Environmental conditions during each calibration

	CESMEC	INACAL	CENAM	CESMEC
Temperature / ºC	21,5 – 21,9	22,51 – 22,75	17,98 – 19,23	23,2 – 23,4
Pressure / hPa	946,53 – 946,34	994,2 – 995,9	809,13 – 811,75	949,13 – 950,00
Relative humidity / %	47,7 – 51,1	51,3 – 53,0	34,01 – 38,26	42,4 – 43,8
Air density / kg m^{-3}	1,112 – 1,114	1,165 – 1,167	0,9609 – 0,9680	1,108 – 1,111

Resolution of weighing and environmental conditions instruments used by the different laboratories are shown in Table 5.

Table 5: Resolution of instruments used in this comparison

	CESMEC	INACAL	CENAM
Weighing / mg	10	5 / 0,2	0,1
Temperature / ºC	0,1	0,01	0,01
Humidity / %	0,1	0,1	0,01
Pressure / Pa	1	10	0,1

INACAL reported two different resolutions for the weighing because they used two different instruments to carry out the calibration by subdivision method (see Table 7).

Table 6 shows the traceability of the mass standards used by the NMIs, and date of last calibration.

Table 6: Traceability and date of last calibration of standards

	CESMEC	INACAL	CENAM
Traceability	PTB	NRC	BIPM
Date	2010	2012	2009 - 2012

The measurement method used by each laboratory are according to Table 7.

Table 7: Measurements methods reported by each laboratory

	CESMEC[1]	INACAL	CENAM
Measurement method	Direct comparison	Subdivision	Subdivision with ABBA weighing cycles

In case of CENAM, in order to simplify the presentation of the uncertainty budget for this report, even when the traveling standard was calibrated by subdivision method, the uncertainty budget was estimated as if the traveling standards were calibrated by direct comparison method. It is important to note that there is no significant difference between the combined uncertainty estimated by both methods (subdivision and direct comparison).

CENAM and INACAL measured the mass value and CESMEC measured the conventional mass value of the traveling standard. All the participants converted their respective values to conventional mass (CENAM and INACAL) or to mass (CESMEC) using OIML D28 (E) Edition 2004.

7. Measurements results

Table 8 shows the mass correction value reported by the NMIs and its associated standard uncertainty ($k = 1$).

Table 8: Mass correction

| Participant laboratory | 50 kg | |
	Correction mg	Uncertainty ($k = 1$) mg
CESMEC – initial	25 953	40
INACAL	25 885	15
CENAM	25 899	10
CESMEC - final	25 997	40

Table 9 shows the conventional mass error found by the NMIs and its associated standard uncertainty ($k = 1$).

Table 9: Conventional mass correction

| Participant laboratory | 50 kg | |
	Correction mg	Uncertainty ($k = 1$) mg
CESMEC – initial	25 910	40
INACAL	25 842	15
CENAM	25 855	10
CESMEC - final	25 954	40

For the pilot institute, the average value is according to Table 10. The results from initial and final correction of the pilot institute agree each other, considering the measurement

uncertainty. According to this, no drift correction was applied to calculate the reference value.

The uncertainty value reported by CESMEC was according to OIML R111-1 class F_1 weight, calculated as 1/3 of the maximum permissible error for that weight class.

Table 10: Mass and Conventional Mass correction for the pilot institute

Pilot laboratory	50 kg			
	Mass		**Conventional Mass**	
	Correction **mg**	**U** **($k = 2$)** **mg**	**Correction** **mg**	**U** **($k = 2$)** **mg**
CESMEC	25 975	80	25 932	80

Since the pilot institute perform two measurements of the traveling standard, it was necessary to calculate the combined expanded uncertainty of the result, that was calculated as the mean (R_{cesmec}) of the initial (R_i) and final (R_f) measurement, according to Table 9.

$$R_{\text{cesmec}} = \frac{R_i + R_f}{2} \qquad (7.1)$$

And the expanded uncertainty of R_{cesmec}, considering the correlation between each initial and final measurement, will be according to eq. 7.2.

$$U(R_{\text{cesmec}}) = 2u(R_{\text{cesmec}}) = 2\frac{1}{2}\sqrt{u^2(R_i) + u^2(R_f) + 2u(R_i)u(R_f)} \qquad (7.2)$$

In this case, $u(R_i) = u(R_f) = u(R_{i \text{ or } f})$, and the expanded uncertainty of the mean R_{cesmec} will be equal to the expanded uncertainty of the initial or final measurement.

$$U(R_{\text{cesmec}}) = \sqrt{4u^2(R_{i \text{ or } f})} = 2u(R_{i \text{ or } f}) = 80 \text{ mg} \qquad (7.3)$$

Uncertainty analysis was according to GUM [3]. The uncertainty contributions assigned by each NMI are shown in table 11, using the form shown in Annex D of the comparison protocol. It was allowed for any institute to include any uncertainty contribution that was not included in the Annex.

Most of the laboratories considered the same influence magnitudes (uncertainty contributions) for the estimation of the uncertainty. This guarantees a greater homogeneity in the determination of the combined uncertainty.

Table 11: Uncertainty contributions informed by each institute

INFLUENCE MAGNITUDE x_i	$u(x_i)$ / mg			
	CESMEC - initial	INACAL	CENAM	CESMEC - final
Mass standard	12,500	1,100 00	3,5	15,000
Instability of the mass standard		1,270 17	0,87	
Density of the mass standard	-1,628 8		0,48	0,827 3
Density of the mass under test	4,914 2		-9,15	4,914 2
Air density	-0,038 0	0,047 24		-0,033 8
Mass difference	3,600 9			31,135 9
Sensitivity of the balance				
Resolution of the balance		4,085 75		
Repeatability		1,041 54		
Eccentricity				
Reproducibility				
Fitting				
Method		8,755 12		
Volume of mass standard		0,349 48		
Volume of mass under test		11,066 71		

8. Results Analysis

The aim of the comparison is to demonstrate the consistency of the results provided by the laboratories with the reference value.

The uncertainty values associated with the mass measurements reported by the participating laboratories appears in Table 12. In addition, it showed the maximum calibration uncertainty for F_1, E_2 and E_1 OIML accuracy classes [1].

Table 12. Expanded uncertainties reported by each NMI, and typical uncertainty according to OIML.

Institute	$U(k = 2)$ mg
CESMEC	80
INACAL	30
CENAM	20
U (F_1)	80
U (E_2)	25
U (E_1)	8

The expanded uncertainty of CESMEC is according to the measurement of each calibration (initial and final measurements has the same uncertainty). It is equal to two times the standard uncertainty.

The reference value was proposed by the pilot laboratory and calculated from results of participants, by weighted mean of largest consistent values, according to M. G. Cox [6]:

$$y = \frac{x_1/u^2(x_1) + \cdots + x_N/u^2(x_N)}{1/u^2(x_1) + \cdots + 1/u^2(x_N)} \tag{7.4}$$

Where,

y : is the weighted mean of the institutes' measurements
x_i : is the institutes' measurements
$u(x_i)$: is the standard uncertainty of the institutes' measurements
N : is the number of participating institutes (in this case $N = 3$)

And the standard uncertainty of the weighted mean $u(y)$ was calculated from:

$$\frac{1}{u^2(y)} = \frac{1}{u^2(x_1)} + \cdots + \frac{1}{u^2(x_N)} \tag{7.5}$$

In order to establish the reference value of the comparison and its uncertainty, it was assumed that the conditions to use the Procedure A detailed in the article by M. G. Cox [6] were accomplished. The reference value and its uncertainty are showed in Table 13.

Table 13: Reference value and uncertainty, according to eqs. 7.1 and 7.2

	y mg	$u(y)$ mg
Correction in mass	25 898,02	8,15
Correction in conventional mass	25 854,36	8,15

A chi-squared test was applied, in order to establish an overall consistency check of the results obtained. The observed chi-squared value χ_{obs} was calculated as [6]:

$$\chi_{\text{obs}}^2 = \frac{(x_1 - y)^2}{u^2(x_1)} + \cdots + \frac{(x_N - y)^2}{u^2(x_N)} \tag{7.6}$$

And the results after applying eq. 7.3 are according to Table 14.

Table 14: Observed chi-squared values for mass and conventional mass

	χ_{obs}^2
Mass	4,42
Conventional mass	4,45

For this case, the chi-squared value for reference is 5,99 tacking into account a number of degrees of freedom equal to 2. Since $Pr\{\chi^2(2) > \chi^2_{\text{obs}}\} > 0,05$, the consistency check does not fail, and the reference value is accepted, together with its uncertainty.

Then, the degrees of equivalence d_i and its expanded uncertainty $U(d_i)$ can be calculated as:

$$d_i = x_i - y \tag{7.7}$$

$$U(d_i) = 2\sqrt{u^2(x_i)^2 - u^2(y)^2} \tag{7.8}$$

Where $i = 1,2,3$ is the participating institute. Table 15 presents the obtained results, after aplying eqs. 7.7 and 7.8.

Table 15: Observed chi-squared values for mass and conventional mass

Institute	Mass		Conventional Mass	
	d_i mg	$U(d_i)$ mg	d_i mg	$U(d_i)$ mg
CESMEC	76,98	78,32	77,64	78,32
INACAL	-13,02	25,19	-12,36	25,19
CENAM	0,98	11,60	0,64	11,60

With these values, it is possible to calculate the normalized error E_n, as a criteria for the agreement of the reported results of each institute (x_i) and the reference value (y) using eqution 7.9.

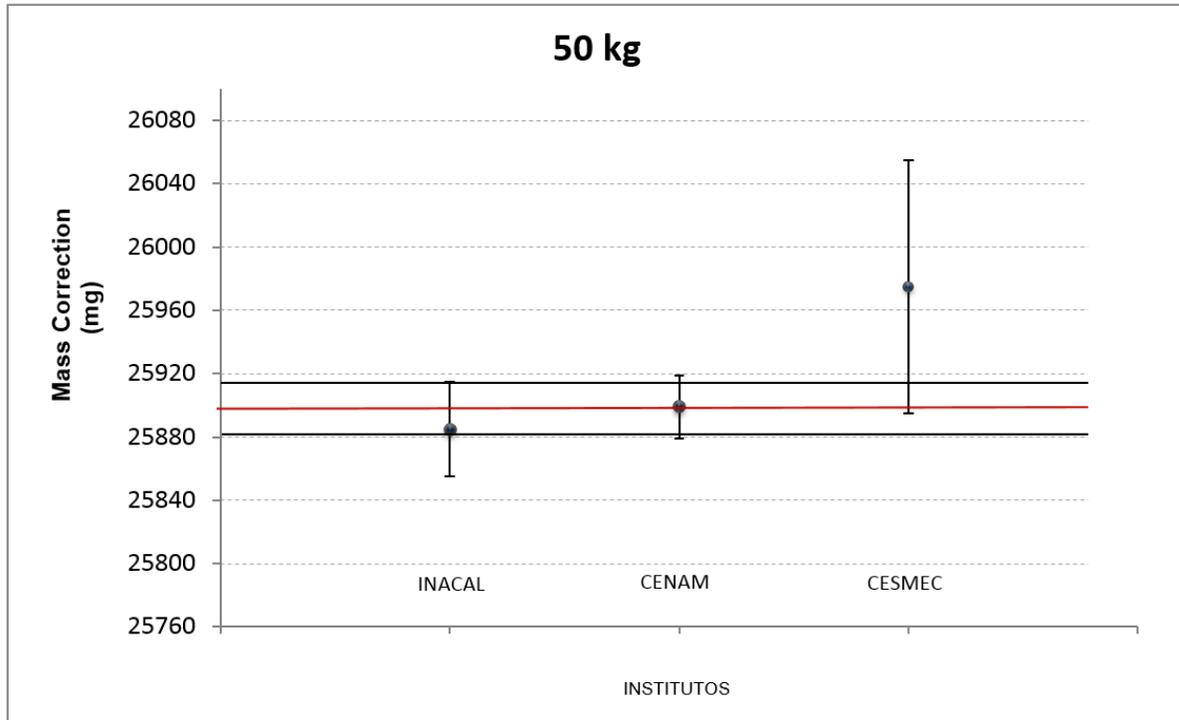$$E_{\text{n},i} = \frac{d_i}{\sqrt{U(y)^2 + U(x_i)^2}} \tag{7.9}$$

The numerical results are shown in Table 16.

Table 16: Normalized error in conventional mass

| NMIs	NOMINAL VALUE	$|E_{\text{n}}|$	$|E_{\text{n}}| \leq 1$
CESMEC		0,99	YES
INACAL	50 kg	0,49	YES
CENAM		0,06	YES

9. Graphical Analysis

The relation between the accepted reference value and the results reported by the participants is show in Graph 1 and 2 for nominal value 50 kg.

Graph 1: Mass correction and uncertainty reported by each institute. The red line indicates the reference value and dashed black lines its uncertainty at $k = 2$.

Each institute reported a uncertainty according to its capabilities and CMC. In Graph 1 can be seen that the results are similar between them, and the reference value is a good estimative for the comparison. It can be observed that the weighning reference value is according to the capabilities of the laboratories; CENAM has the lower uncertainty, and it reported value has more influence over the reference value that CESMEC, who has the largest uncertainty.
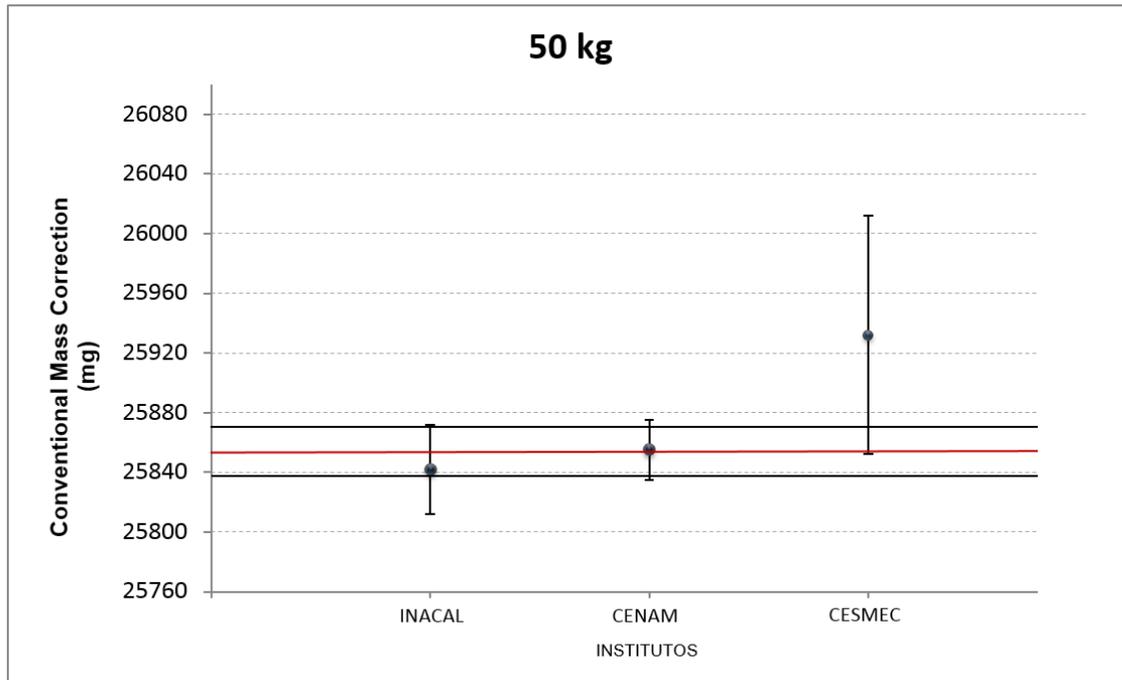
Graph 2: Conventional mass correction and uncertainty reported by each institute. The red line indicates the reference value and dashed black lines its uncertainty at $k = 2$.

For this comparison, all the participants have a normalized error (E_n) lower than 1, which means that there is agreement between them and the reference value. From Graph 1, it can be observed that the agreement of the measurements is consistent, since each value (with its uncertainty) share, at least, a common interval, with a confidence of 95%.

10. Conclusions

The comparison was successful. The reference value was calculated by weighted mean of largest consistent values, according to M. G. Cox [6], and CENAM reported the density reference value for the 50 kg weight. There was no significant deviation in the measured mass of the travelling standard. There is no normalized errors bigger that 1, which means that exist an agreement of the results between each of them and the reference value. Graphical analysis shows the relation between the institutes and the reference value, and also, the normalized error.

All the participants checked that the results were consistent with their published CMC values.

11. References

[1]   OIML R111, Weights of classes E_1, E_2, F_1, F_2, M_1, M_2, M_3, 2007
[2]   Revised formula for the density of moist air (CIPM-2007); *Metrologia* **45** (2008) 149-155.
[3]   JCGM 100:2008, Evaluation of measurement data - Guide to the expression of uncertainty in measurement, 2008.
[4]   http://www.bipm.org/utils/common/documents/CIPM-MRA/CIPM-MRA-D-05.pdf
[5]   EA-4/02, Expression of the Uncertainty of Measurement in Calibration, 1999 http://www.european-accreditation.org/n1/doc/ea-4-02.pdf
[6]   Maurice Cox, The Evaluation of key comparison data, Metrologia 2002, Volume 39, Number 6, pages 589 - 595.
[7]   ISO/IEC 17043:2010, Conformity assessment -- General requirements for proficiency testing.