



## FINAL REPORT SIM COMPARISON IN A MASS STANDARD

### SIM.M.M-K4

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**Abstract:** *This report summarizes the results of a SIM comparison of a 1 kg mass standard carried out between 7 NMIS. The results reported by the participants are consistent with each other and they can be linked to the comparison CCM.M-K4 with satisfactory degrees of equivalence*

### 1. General Information

The present comparison, named SIM.M.M.K4, was planned and carried out in order to evaluate the degree of equivalence in the calibration of high accuracy mass standards, and to provide evidence supporting CMCs claimed by the participants in high accuracy mass calibrations delivered by them. It is part of a more general project which includes three comparisons:

- **SIM.M.M-K4** for mass calibration of nominal value 1 kg
- **SIM.M.M-K5** for mass calibration of nominal values 2 kg, 200 g, 50 g, 1 g and 200 mg
- **SIM.M.D-K3** for volume determination of stainless steel weights of 2 kg, 1 kg, 200 g and 1 g

### 2. Data of the participant NMI and Technical Contacts

The following SIM NMIs have participated in the comparison:

Institute	Country	Technical Contact(s)
LACOMET	Costa Rica	Ramos, O; Rodríguez, S.
LATU	Uruguay	Santo, C.; Caceres, J.
INTI	Argentina	Kornblit, F; Leiblich, J.
CESMEC	Chile	García, F.; Leyton, F.
CENAM	México	Becerra, L.O.; Peña, L.M.; Luján, L.; Díaz, J.C.; Centeno, L.M.
NRC	Canada	Claude Jacques
INMETRO	Brazil	Loayza, V.M.; Cacaís, F.A.

INTI (Argentina)<sup>1</sup> has acted as the pilot laboratory

### 3. General Considerations and Procedure

A stainless steel standard, made by Masstech and provided by CENAM was used for the comparison. Its volume referred to 20 °C,  $V_{20\text{ °C}}$ , was determined by CENAM in August 2012,

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after the comparison. These result and the corresponding standard uncertainty  $u_V$  are also shown in Table 1, as well as the identification of the standard.

**Table 1.** Data associated to the standard weight

Nominal value:	1 kg
Serial number:	1866
Identification:	540161557204
$V_{20\text{ }^\circ\text{C}}$	125,414 9 cm <sup>3</sup>
$u_V$	0,002 3 cm <sup>3</sup>

The traveling standard was placed in an individual wooden case for transportation purposes, which was placed in a carrying transportation case, jointly with the standards corresponding to the comparisons SIM.M.M-K5 and SIM.M.D-K3 In all the cases, the transportation among laboratories was made by hand, by technical staff of the NMIs.

A protocol was agreed previously to the comparison. In it, instructions to travel, initial inspection in each country, store, handling and acclimatization of the standards have been specified. Particularly, the following criteria were agreed:

- The standard was not washed during the comparison.
- Each NMI applied its own method to measure the mass of the standard, in order to achieve uncertainties as low as possible, according to its capabilities.
- The CIPM-2007 formula [1] was applied by all the participants in order determine the air density. The buoyancy corrections were applied by the participants retrospectively after the circulation of the transfer standard.

#### 4. Schedule

The measurements followed the schedule shown in Table 2.

**Table 2.** Measurement order and dates

N°	Institute / Country	Date
--	CENAM / Mexico	July 2009
1	LACOMET / Costa Rica	October 2009
2	LATU / Uruguay	January 2010
3	INTI / Argentina	April 2010
4	CESMEC / Chile	July 2010
5	CENAM / Mexico	June 2011
6	NRC /Canada	February 2011
7	INMETRO/Brazil	January 2012
--	CENAM / Mexico	August 2012



## 5. Stability of the standard

The drift of the standard was evaluated from three measurements performed by CENAM, in July 2009, in June 2011, and in August 2012. The corresponding mass errors associated uncertainties are shown in Table 3. The corresponding normalized error  $E_n$  values are included too. Also, all these results are presented in Figure 1.

**Table 3.** Stability of the standard. Values obtained by CENAM

	$e / \text{mg}$	$U (k=2)$	$E_n$
July 2009	1,090	0,038	-0,1
June 2011	1,102	0,031	0,1
August 2012	1,096	0,031	0,0

It can be concluded that no significant effects associated to drifts of the standard are to be considered.

## 6. Summary of the reported results

The results sent by the participants are expressed as the mass error  $e$  from the nominal value 1 kg. They are shown in Table 4, as well as the corresponding uncertainty  $U$  (for  $k = 2$ ).

**Table 4.** Mass errors  $e$  as reported by the participants, their associated uncertainties  $U$  (for  $k = 2$ ), and non-linked reference value (*NLRV*)

	$e / \text{mg}$	$U / \text{mg}$
LACOMET	1,050	0,098
LATU	1,143	0,070
INTI	1,051	0,056
CESMEC	1,060	0,160
CENAM	1,102	0,031
NRC	1,064	0,042
INMETRO	1,050	0,100
NLRV	1,083	0,020

## 7. Data consistency and calculation of a (non-linked) reference value

In order to check the consistency among the results, a  $\chi^2$  test as proposed in [2] was applied. The conditions to apply the so-called *procedure A* were assumed. The observed sum of squares corresponding to the whole set of results was  $\chi_{obs}^2 = 7,2$  while the corresponding critical value for 6 degrees of freedom and significance level  $\alpha = 0,1$  is 10,6. So, the reported data can be considered as consistent.

The weighted average was calculated according to the same publication and has been established as the non-linked reference value of the comparison (*NLRV*). It is shown in the last row of table 4, as well as its associated uncertainty.



Then, for each participant, degrees of equivalence  $D$  and normalized errors  $E_n$  were calculated according to (1)

$$D = e - NLRV; \quad U_D = 2\sqrt{u^2 - u_{NLRV}^2}; \quad E_n = D/U_D \quad (1)$$

where  $e$ ,  $u$  are the results reported by the participant. These values are shown in Table 5. The values of  $D$  and  $U_D$  are plotted in Figure 2.

Similarly, bilateral differences between pairs of participants and bilateral normalized errors were calculated according to (2). They are shown in Tables 6A and 6B.

$$D_{ij} = x_i - x_j \quad E_{ij} = \frac{D_{ij}}{2\sqrt{u_i^2 + u_j^2}} \quad (2)$$

**Table 5.** Degrees of equivalence and normalized errors, respect to the non linked reference value

	$D / \text{mg}$	$UD / \text{mg}$	$E_n$
LACOMET	-0,03	0,10	-0,3
LATU	0,06	0,07	0,9
INTI	-0,03	0,05	-0,6
CESMEC	-0,02	0,16	-0,1
CENAM	0,02	0,02	0,7
NRC	-0,02	0,04	-0,5
INMETRO	-0,03	0,10	-0,3

**Table 6A.** Bilateral differences  $D_{ij} = x_i - x_j$ , where  $x_i$  refers to the result reported by the laboratory in the left column and  $x_j$  refers to the result reported by the laboratory in the top row

$D_{ij} / \text{mg}$	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,09					
INTI	0,00	-0,09				
CESMEC	0,01	-0,08	0,01			
CENAM	0,05	-0,04	0,05	0,04		
NRC	0,01	-0,08	0,01	0,00	-0,04	
INMETRO	0,00	-0,09	-0,00	-0,01	-0,05	-0,01

**Table 6B.** Bilateral normalized errors  $E_{ij}$  according to (2)

	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,8					
INTI	0,0	-1,0				
CESMEC	0,1	-0,5	0,1			
CENAM	0,5	-0,6	0,8	0,2		
NRC	0,1	-1,0	0,2	0,0	-0,7	
INMETRO	0,0	-0,8	0,0	-0,1	-0,5	-0,1



## 8. Link to CCM key comparisons

In order to demonstrate equivalence, the present comparison was linked to CCM.M.M.K4 [3]. CENAM and NRC have participated in it and act as linking laboratories. Their degrees of equivalence (difference  $D_0$  from K4 reference value and the corresponding uncertainty  $U_0$  for  $k = 2$ ) are shown in table 7:

**Table 7.** Degrees of equivalence of the linking laboratories in CCM.M.M.K4

	$D_0 / \mu\text{g}$	$U_0 / \mu\text{g}$
CENAM	-12	27
NRC	-14	34

The reference value linked to CCM.M.M.K4 (linked reference value) was calculated as:

$$LRV = \frac{w_C (e_C - D_{0,C}) + w_N (e_N - D_{0,N})}{w_C + w_N} \quad (3)$$

where the subscripts C and N refer to CENAM and NRC data respectively, and the weights  $w$  are calculated as:

$$w_C = \frac{1}{u^2(e_C - D_{C,0})} = \frac{1}{u_C^2 + U_{C,0}^2/4}; \quad w_N = \frac{1}{u^2(e_N - D_{N,0})} = \frac{1}{u_N^2 + U_{N,0}^2/4} \quad (4)$$

The standard uncertainty associated to the linked reference value is

$$u_{LRV} = \frac{1}{\sqrt{w_C + w_N}} \quad (5)$$

For the calculations in (4), the linking laboratories results in both comparisons were considered non-correlated.

So, (3) and (5) yield:

$$LRV = 1.099 \text{ mg}; \quad u_{LRV} = 0.017 \text{ mg}$$

Then, new degrees of equivalence were calculated, considering deviations  $D_L = e - LRV$ , and associated uncertainties  $U_L = 2\sqrt{u_x^2 + u_{LRV}^2}$ . Then, linked normalized errors  $E_{nL}$  were obtained as:

$$E_{nL} = D_L / U_L$$

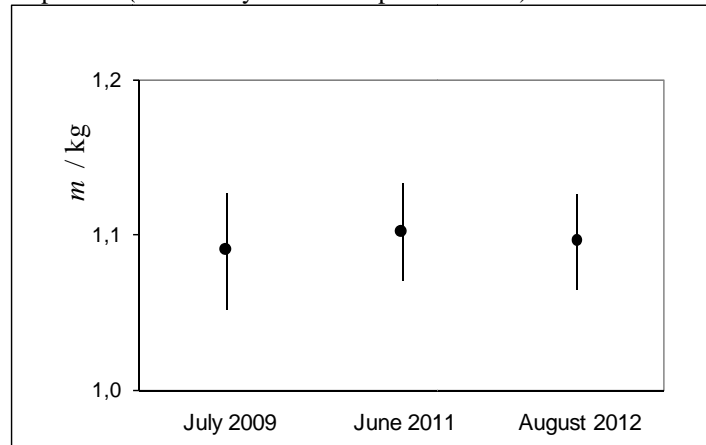


These results are shown in table 8 and in Figure 2.

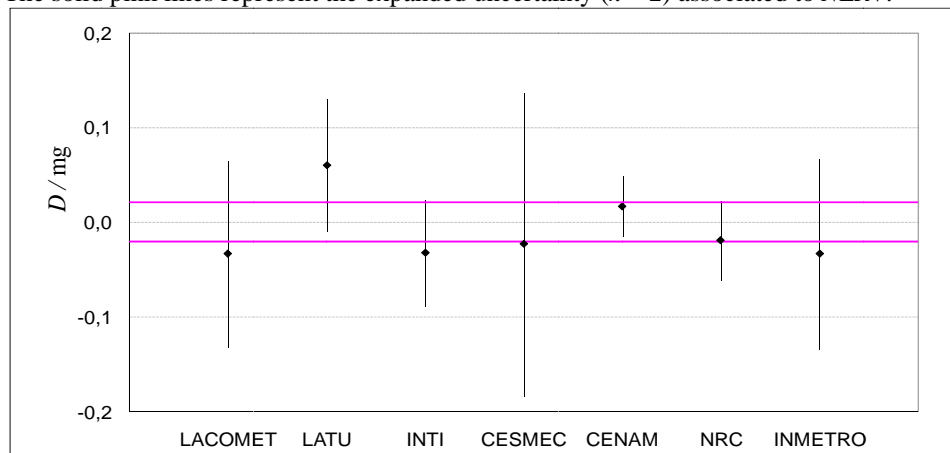
**Table 8.** Linked degrees of equivalence and normalized errors

	$D_L/\text{mg}$	$U_L/\text{mg}$	$E_{nL}$
LACOMET	-0,049	0,103	-0,5
LATU	0,044	0,077	0,6
INTI	-0,048	0,065	-0,8
CESMEC	-0,039	0,163	-0,2
CENAM	0,001	0,046	0,0
NRC	-0,035	0,053	-0,7
INMETRO	-0,049	0,105	-0,5

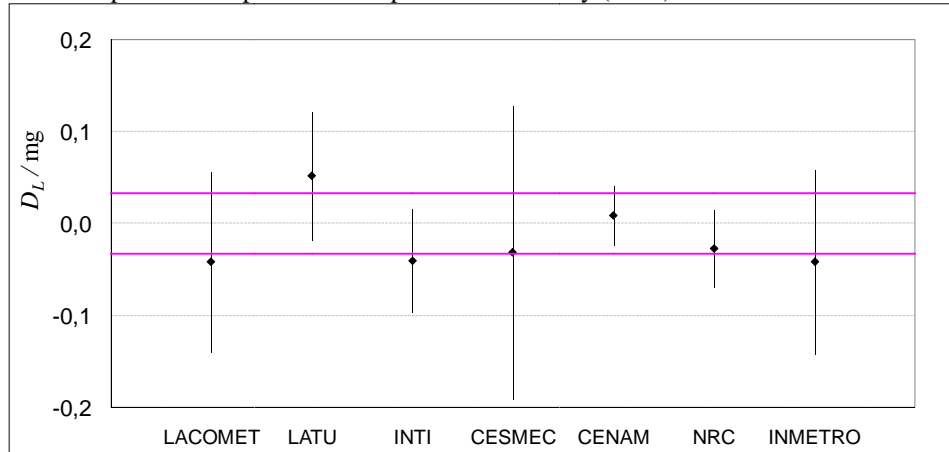
**Figure 1.** Plot showing the stability of the standard during the comparison (uncertainty bars correspond to  $k = 2$ )



**Figure 2.** Deviations  $D$  from  $NLRV$  and their associated expanded uncertainties ( $k = 2$ ). The solid pink lines represent the expanded uncertainty ( $k = 2$ ) associated to  $NLRV$ .



**Figure 3.** Deviations  $D_L$  from  $LRV$  and their associated expanded uncertainties ( $k = 2$ ). The solid pink lines represent the expanded uncertainty ( $k = 2$ ) associated to  $LRV$ .



## References

1. Picard A., Davis R.S., Gläser M., Fujii K., *Revised formula for the density of moist air (CIPM-2007)*, Metrologia **45** (2008), 149-155
2. Cox, M.G., *The evaluation of key comparison data*, Metrologia **39** (2002) 589-595
3. Luis Omar Becerra et al. *Final Report of Key comparison of 1 kg stainless steel mass standards CCM.M-K4, Organized by the Working Group on Mass Standards of the Consultative Committee for Mass and Related Quantities (CCM) 2014*, <http://kcdb.bipm.org/appendixB/AppBResults/CCM.M-K4/CCM.M-K4.pdf>