FINAL REPORT
SIM COMPARISON IN MASS STANDARDS
SIM.M.M-K5

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Abstract

This report summarizes the results of a SIM comparison of mass standards carried out between 7 NMIS. Five standards with nominal values 2 kg, 200 g, 50 g, 1 g and 200 mg have been circulated by the NMIs. The results reported by the participants are consistent with each other and with the key comparison reference value of comparison CCM.M-K5 to which this comparison has been linked.

1. General Information

The present comparison, named SIM.M.M-K5, 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 comprises three comparisons:

- **SIM.M.M-K1** 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,1 g

2. Data of the participant NMIs and Technical Contacts

The following SIM institutes have participated in the comparison:

Institute	Country	Technical Contact(s)
LACOMET	Costa Rica	Ramos, O; Rodríguez, S.
LATU	Uruguay	Santo, C.; Cáceres, 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-INMS	Canada	Jacques, C.
INMETRO	Brazil	Loayza, V.M.; Cacais, F.A.

INTI (Argentina)[1] has acted as the pilot laboratory

[1]INTI, Instituto Nacional de Tecnología Industrial (Argentina), contact e-mail: ferk@inti.gob.ar, [2] CENAM, Centro Nacional de Metrología (México); [3]LACOMET, Laboratorio Costarricense de Metrología; [4] LATU, Laboratorio Tecnológico del Uruguay (Uruguay); [5]CESMEC (Chile); [6]NRC, National Research Council (Canada), [7] INMETRO, Instituto Nacional de Metrologia, Qualidade e Tecnologia (Brazil)

3. General Considerations and Procedure

A set of five stainless steel standards, made by Masstech and provided by CENAM was used for the comparison. The nominal values and identifications are shown in Table 1. In June 2012, once measurements were finished, CENAM determined the volume of the standards referred to 20 ºC, V. These results and the corresponding standard uncertainties u_V are shown in Table 1.

Table 1. Data associated to the standard weights

Nominal value	Serial Number	Identification	V / cm^3	u_V / cm^3
2 *kg*	1867	540161558204	251,051	0,024
200 *g*	1865	540161556204	25,1487	0,0024
50 *g*	1864	540161555204	6,2624	0,0008
1 *g*	1863	540161554204	0,1258	0,0005
200 *mg*	S/N	540140212102	0,025 0	0,0002

The traveling standards were placed in individual wooden cases for transportation purposes, which were placed in a carrying transportation case, jointly with the standards corresponding to the comparisonsSIM.M.M-K1 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 standards were not washed during the comparison.

- The institutes applied their own method to measure the mass of the standards, in order to achieve uncertainties as low as possible, according to its capabilities. That means that each laboratory applied subdivision methods with different weighing designs.

- The CIPM-2007 formula [1] was applied by all the participants to determine the air density, in order to estimate buoyancy effects.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 schedule

N°	Institute / Country	Dates of the measurements
--	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
--	CENAM / Mexico	June 2011
5	NRC /Canada	February 2011
6	INMETRO/Brazil	January 2012
7	CENAM / Mexico	July 2012

5. Stability of the standards

The drift of the standards of 2 kg, 200 g, 50 g, and 1 g was evaluated from three series of measurements performed by CENAM, in July 2009, in June 2011, and in July 2012. The standard of 200 mg was only measured in June 2011 and July 2012.The mass errors *e* obtained their associated uncertainties and normalized errors values E_n are listed in Table 3 are plotted in Figures 1A to 1E (the uncertainty bars in them correspond to k = 2).

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

	Date	*e* / mg	*U* (*k*=2)	E_n
2 kg	July 2009	2,79	0,17	-0,1
	June 2011	2,83	0,13	0,1
	July 2012	2,82	0,13	0,0
200 g	July 2009	0,305	0,014	-0,4
	June 2011	0,324	0,016	0,3
	July 2012	0,317	0,014	0,1
50 g	July 2009	0,076	0,004	0,0
	June 2011	0,076	0,006	0,0
	July 2012	0,077	0,005	0,1
1 g	July 2009	0,004	0,001	0,0
	June 2011	0,004	0,001	0,0
	July 2012	0,004	0,001	0,0
200 mg	June 2011	0,000 1	0,000 7	0,2
	July 2012	-0,000 2	0,000 7	-0,2

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

6. Summary of the reported results

The results sent by the participants are expressed as the mass error *e* of each standard, taken from their nominal values. They are shown in Table 4, as well as the associated uncertainties *U* (for $k = 2$).

Table 4. Mass errors *e* as reported by the participants, and the corresponding uncertainties *U*

	2 kg		200g		50 g		1 g		200 mg	
	e/mg	*U*/mg	*e*/mg	*U*/mg	*e*/mg	*U*/mg	*e*/ µg	*U*/ µg	*e*/ µg	*U*/ µg
LACOMET	2,54	0,84	0,312	0,020	0,080	0,022	3,2	3,0	-0,2	1,3
LATU	2,52	0,68	0,315	0,024	0,071	0,006	4,4	2,6	0,4	1,6
INTI	2,87	0,20	0,315	0,018	0,077	0,010	4,3	2,0	-0,4	1,2
CESMEC	2,50	0,30	0,315	0,030	0,074	0,010	4,3	3,0	0,0	2,0
CENAM	2,82	0,13	0,317	0,014	0,077	0,005	4,2	1,0	0,0	0,7
NRC	2,66	0,11	0,317	0,028	0,073	0,014	3,4	1,4	-0,3	1,4
INMETRO	2,77	0,22	0,312	0,022	0,070	0,006	3,8	1,4	-0,2	0,6
NLRV	2,73	0,07	0,315	0,008	0,073	0,0029	3,9	0,6	-0,1	0,4

7. Data consistency and computation of (non-linked) reference values

In order to check the consistency of the results, χ^2 tests as proposed in [2] were applied. The assumptions to employ the so-called *Procedure A* were considered valid. For the different standards, the sums of squares corresponding to the whole set of results yielded the following values of the χ^2 statistic:

Nominal value	χ^2_{obs}
2 kg	8,6
200 g	0,3
50 g	4,6
1 g	1,4
200 mg	1,0

while the corresponding critical value for 6 degrees of freedom and significance level $\alpha = 0,1$ is 10,6. So, the data were considered consistent in all the cases.

Following [2] the weighted averages were calculated and established as the (non-linked) reference values of the comparison, called *NLRV*. They are shown in the last row of table 3, as well as their associated uncertainties.

Then, for each participant and standard, degrees of equivalence *D* and normalized errors E_n were calculated according to 1:

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

being e,u the results reported by the participant. D, U_D and E_n are shown in Tables 5A and 5B. The values of D and their associated uncertainties are plotted in figures 2A to 2E. In each plot, Here, NLRV is represented as the zero line and their associated uncertainty as solid pink lines.

Table 5A. Degrees of equivalence respect to the non-linked reference value

	2 kg		200 g		50 g		1 g		200 mg	
	D / mg	U_D / mg	D / mg	U_D / mg	D / mg	U_D / mg	D / µg	U_D / µg	D / µg	U_D / µg
LACOMET	-0,19	0,84	-0,003	0,018	0,007	0,022	-0,7	2,9	-0,1	1,2
LATU	-0,21	0,68	0,000	0,023	-0,002	0,006	0,5	2,5	0,5	1,5
INTI	0,14	0,19	0,000	0,016	0,004	0,010	0,4	1,9	-0,3	1,1
CESMEC	-0,23	0,29	0,000	0,029	0,001	0,010	0,4	2,9	0,1	2,0
CENAM	0,09	0,11	0,002	0,012	0,003	0,005	0,2	0,8	0,1	0,6
NRC	-0,07	0,08	0,002	0,027	0,000	0,014	-0,5	1,3	-0,2	1,4
INMETRO	0,04	0,21	-0,003	0,021	-0,004	0,005	-0,1	1,3	-0,1	0,5

Table 5B. Values of En respect to the non-linked reference value

	2 kg	200 g	50 g	1 g	200 mg
LACOMET	-0,2	-0,2	0,3	-0,2	-0,1
LATU	-0,3	0,0	-0,4	0,2	0,4
INTI	0,8	0,0	0,4	0,2	-0,2
CESMEC	-0,8	0,0	0,1	0,1	0,1
CENAM	0,8	0,2	0,7	0,3	0,2
NRC	-0,9	0,1	0,0	-0,4	-0,1
INMETRO	0,2	-0,1	-0,8	-0,1	-0,2

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

$$D_{ij} = x_i - x_j \qquad E_{ij} = \frac{D_{ij}}{2\sqrt{u_i^2 + u_j^2}} \tag{2}$$

Table 6A.1.2 kg standard. Bilateral differences $D_{i\,j} = x_i - x_j$, 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} / mg	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	-0,020					
INTI	0,336	0,356				
CESMEC	-0,040	-0,020	-0,376			
CENAM	0,281	0,301	-0,055	0,321		
NRC	0,124	0,144	-0,212	0,164	-0,157	
INMETRO	0,230	0,250	-0,106	0,270	-0,051	0,106

Table 6A.2.200 g standard. Bilateral differences $D_{i\,j} = x_i - x_j$, 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} / mg	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,003 0					
INTI	0,003 0	0,000 0				
CESMEC	0,003 0	0,000 0	0,000 0			
CENAM	0,004 9	0,001 9	0,001 9	0,001 9		
NRC	0,005 0	0,002 0	0,002 0	0,002 0	0,000 1	
INMETRO	0,0000	-0,0030	-0,0030	-0,0030	-0,0049	-0,0050

Table 6A.3.50 g standard. Bilateral differences $D_{i\,j} = x_i - x_j$, 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} / mg	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	-0,009 0					
INTI	-0,003 0	0,006 0				
CESMEC	-0,006 0	0,003 0	-0,003 0			
CENAM	-0,003 5	0,005 5	-0,000 5	0,002 5		
NRC	-0,006 7	0,002 3	-0,003 7	-0,000 7	-0,003 2	
INMETRO	-0,010 5	-0,001 5	-0,007 5	-0,004 5	-0,007 0	-0,003 8

Table 6A.4.1 g standard. Bilateral differences $D_{i\,j} = x_i - x_j$, 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} / µg	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	1,2					
INTI	1,1	-0,1				
CESMEC	1,1	-0,1	0,0			
CENAM	1,0	-0,2	-0,1	-0,1		
NRC	0,2	-1,0	-0,9	-0,9	-0,8	
INMETRO	0,6	-0,6	-0,5	-0,5	-0,4	0,4

Table 6A.5.200mg standard. Bilateral differences $D_{i\,j} = x_i - x_j$, 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} / µg	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,6					
INTI	-0,2	-0,8				
CESMEC	0,2	-0,4	0,4			
CENAM	0,2	-0,4	0,4	0,0		
NRC	-0,1	-0,7	0,1	-0,3	-0,3	
INMETRO	0,0	-0,6	0,2	-0,2	-0,2	0,1

Table 6B.1.2 kg standard. Bilateral normalized errors E_{ij} according to (2)

	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,0					
INTI	0,4	0,5				
CESMEC	0,0	0,0	-1,0			
CENAM	0,3	0,4	-0,2	1,0		
NRC	0,1	0,2	-0,9	0,5	-0,9	
INMETRO	0,3	0,3	-0,4	0,7	-0,2	0,4

Table 6B.2.200 g standard. Bilateral normalized errors E_{ij} according to (2)

	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,1					
INTI	0,1	0,0				
CESMEC	0,1	0,0	0,0			
CENAM	0,2	0,1	0,1	0,1		
NRC	0,1	0,1	0,1	0,0	0,0	
INMETRO	0,0	-0,1	-0,1	-0,1	-0,2	-0,1

Table 6B.3. 50 g standard. Bilateral normalized errors E_{ij} according to (2)

	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	-0,4					
INTI	-0,1	0,5				
CESMEC	-0,2	0,3	-0,2			
CENAM	-0,2	0,7	0,0	0,2		
NRC	-0,3	0,1	-0,2	0,0	-0,2	
INMETRO	-0,5	-0,2	-0,6	-0,4	-0,9	-0,3

Table 6B.4. 1 g standard. Bilateral normalized errors E_{ij} according to (2)

	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,3					
INTI	0,3	0,0				
CESMEC	0,3	0,0	0,0			
CENAM	0,3	-0,1	-0,1	0,0		
NRC	0,1	-0,3	-0,4	-0,3	-0,4	
INMETRO	0,2	-0,2	-0,2	-0,2	-0,2	0,2

Table 6B.5. 200 mg standard. Bilateral normalized errors E_{ij} according to (2)

	LACOMET	LATU	INTI	CESMEC	CENAM	NRC
LATU	0,3					
INTI	-0,1	-0,4				
CESMEC	0,1	-0,2	0,2			
CENAM	0,2	-0,2	0,3	0,0		
NRC	0,0	-0,3	0,1	-0,1	-0,2	
INMETRO	0,0	-0,4	0,1	-0,1	-0,2	0,1

8. Link to CCM key comparisons

In order to demonstrate equivalence, the present comparison was linked to CCM.M.M.K5 [3]. CENAM, INMETRO and NRC have participated in K5 and acted as linking laboratories. Their degrees of equivalence obtained in this comparison (that is, the differences$D_0$and the corresponding expanded uncertaintiesU_0) are shown in table 7. In CCM.M.M.K5 sets of two standards (called J_X and J_Y) were measured for each nominal value. Then, the entries in table 7 correspond to average values of the respective results for J_X and J_Y.

Table 7. Degrees of equivalence of the linking laboratories in CCM.M.M-K5

	2 kg		200 g		50 g		1 g		200 mg	
	D_0 /mg	U_0 / mg	D_0 / mg	U_0 / mg	D_0 / mg	U_0 / mg	D_0 / µg	U_0 / µg	D_0 / µg	U_0 / µg
CENAM	0,019	0,134	0,000	0,010	0,001	0,006	-0,1	1,7	-0,4	0,8
INMETRO	0,028	1,201	0,003	0,120	0,002	0,030	0,6	1,5	0,2	1,4
NRC	-0,084	0,079	0,000	0,009	0,007	0,011	0,7	1,7	0,3	2,0

The reference values of the current comparison, linked to CCM.M.M.K5 (linked reference values *LRV*) were calculated as:

$$LRV = \frac{w_C\left(e_C - D_{0,C}\right) + w_I\left(e_I - D_{0,I}\right) + w_N\left(e_N - D_{0,N}\right)}{w_C + w_I + w_N}$$

where he symbol *e* refers to mass error, and the subscripts *C*, *I* and *N* refer to CENAM, INMETRO and NRC respectively. The weights w_j (for $j = C$, *I* or *N*) were calculated as:

$$w_j = \frac{1}{u^2\left(e_j - D_{j,0}\right)} = \frac{1}{u_j^2 + U_{j,0}^2 / 4} \qquad (3)$$

For the calculations in (3), the results of the linking laboratories in both comparisons were considered as non-correlated. The standard uncertainty associated to the linked reference value is

$$u_{LRV} = \frac{1}{\sqrt{w_C + w_I + w_N}}$$

The values of LRV and their associated uncertainties are shown in table 8.

Table 8. Key comparison reference values, linked to CCM.M.MK5

	2 kg	200 g	50 g	1 g	200 mg
LRV /mg	2,765	0,317	0,0738	0,0034	0,000 1
u_{LRV}/mg	0,054	0,007	0,0036	0,000 6	0,000 4

Then, the degrees of equivalence stated in table 4 were corrected by subtracting the term *NLRV-LRV* to the differences *D*, obtaining linked differences D_L. Linked normalized errors E_{nL} are now calculated as:

$$E_{nL} = \frac{D_L}{2\sqrt{u_x^2 + u_{LRV}^2}}$$

These results are shown in table 9A and 9B. The values of D_L and their associated uncertainties are plotted in figures 3A to 3E, where *LRV* is represented as the zero line and its associated uncertainty as the solid pink lines.

Table 9A. Linked degrees of equivalence

	2 kg		200 g		50 g		1 g		200 mg	
	D_L / mg	U_{DL}/mg	D_L / mg	U_{DL}/mg	D_L / mg	U_{DL}/mg	D_L / µg	U_{DL} / µg	D_L / µg	U_{DL} / µg
LACOMET	-0,225	0,85	-0,005	0,025	0,006	0,023	-0,2	3,2	-0,3	1,5
LATU	-0,245	0,69	-0,002	0,028	-0,003	0,010	1,0	2,9	0,3	1,8
INTI	0,111	0,23	-0,002	0,023	0,003	0,012	0,9	2,3	-0,5	1,4
CESMEC	-0,265	0,32	-0,002	0,033	0,000	0,012	0,9	3,2	-0,1	2,2
CENAM	0,056	0,17	0,000	0,021	0,003	0,009	0,7	1,6	-0,1	1,1
NRC	-0,101	0,15	0,000	0,032	0,000	0,016	0,0	1,8	-0,4	1,6
INMETRO	0,005	0,24	-0,005	0,027	-0,004	0,009	0,4	1,8	-0,3	1,0

Table 9B. Values of *En* respect to the linked reference value

	2 kg	200 g	50 g	1 g	200 mg
LACOMET	-0,3	-0,2	0,3	-0,1	-0,2
LATU	-0,4	-0,1	-0,3	0,3	0,2
INTI	0,5	-0,1	0,3	0,4	-0,4
CESMEC	-0,8	-0,1	0,0	0,3	0,0
CENAM	0,3	0,0	0,3	0,5	-0,1
NRC	-0,7	0,0	0,0	0,0	-0,3
INMETRO	0,0	-0,2	-0,5	0,2	-0,3

Figure 1A. Stability of the 2 kg standard

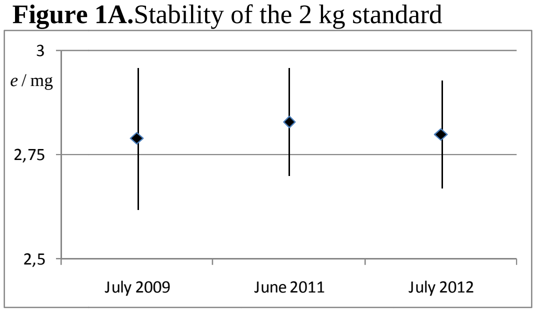


Figure 1B. Stability of the 200 g standard

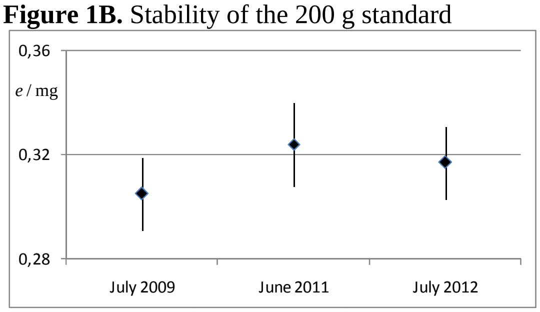


Figure 1C. Stability of the 50 g standard

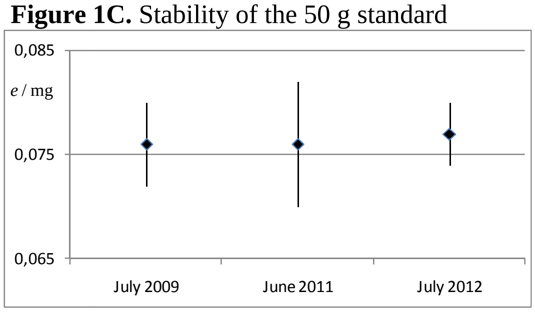


Figure 1D. Stability of the 1 g standard

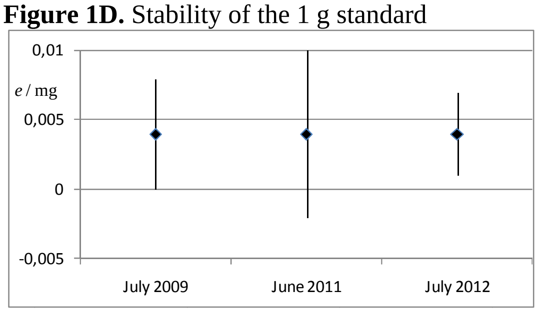


Figure 1E. Stability of the 200 mg standard

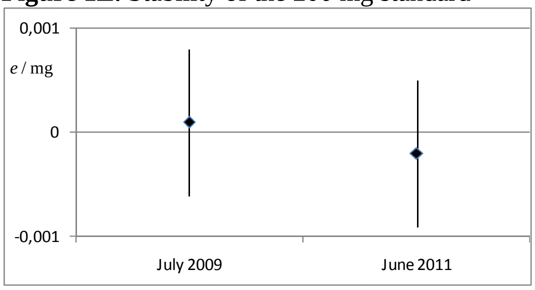


Figure 2A. Deviations from NLRV for the 2 kg standard. The solid pink lines represent the expanded uncertainty associated to N*LRV*.

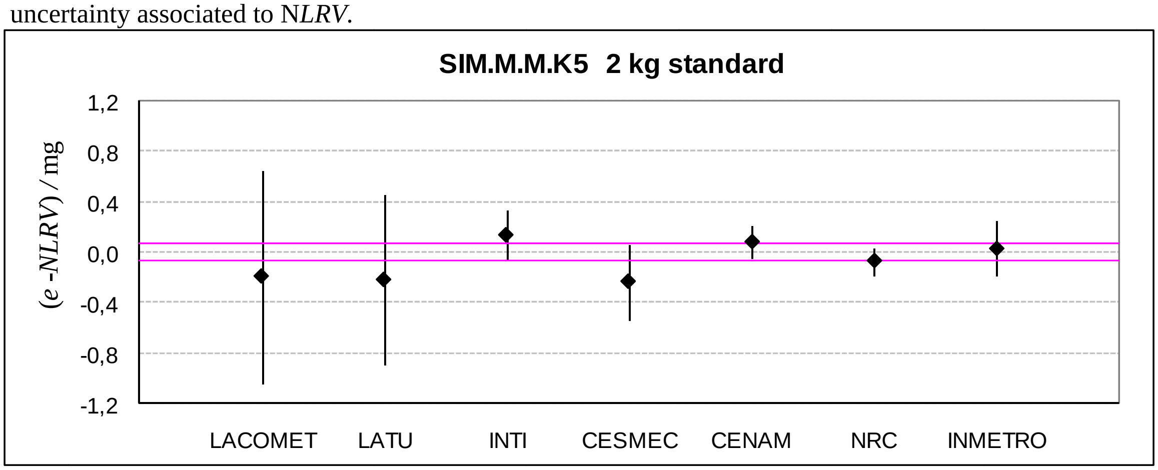


Figure 2B. Deviations from NLRV for the 200 g standard. The solid pink lines represent the expanded uncertainty associated to N*LRV*.

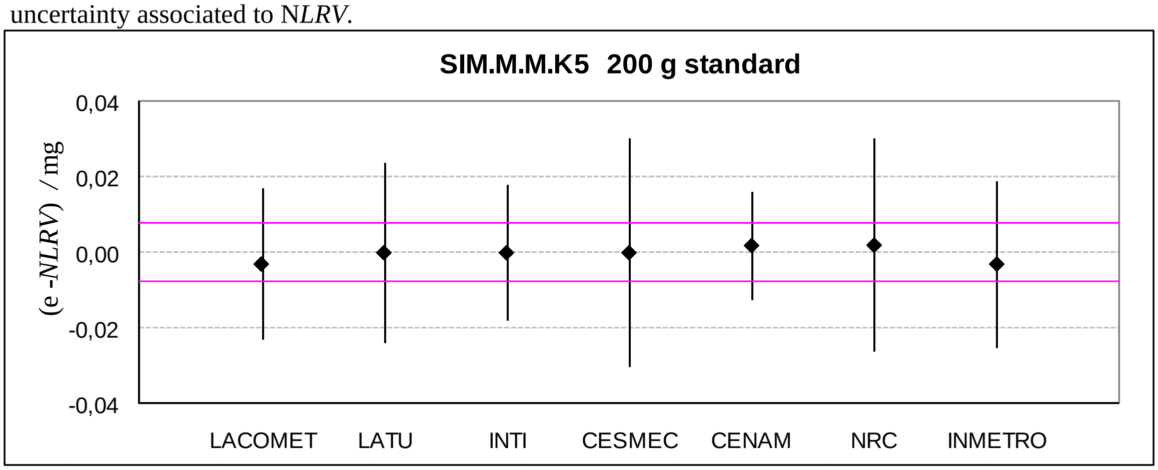


Figure 2C. Deviations from NLRV for the 50 g standard. The solid pink lines represent the expanded uncertainty associated to N*LRV*.

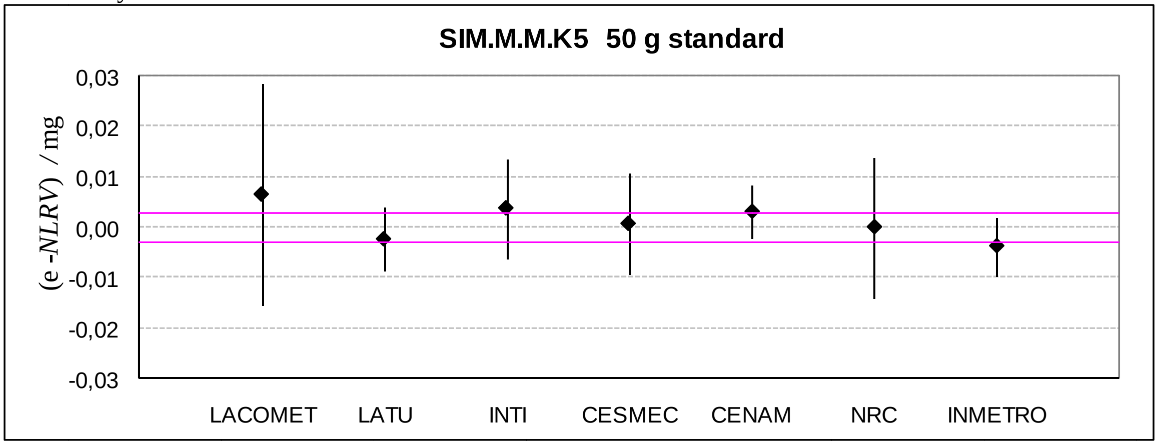


Figure 2D. Deviations from NLRV for the 1 g standard. The solid pink lines represent the expanded uncertainty associated to N*LRV*.

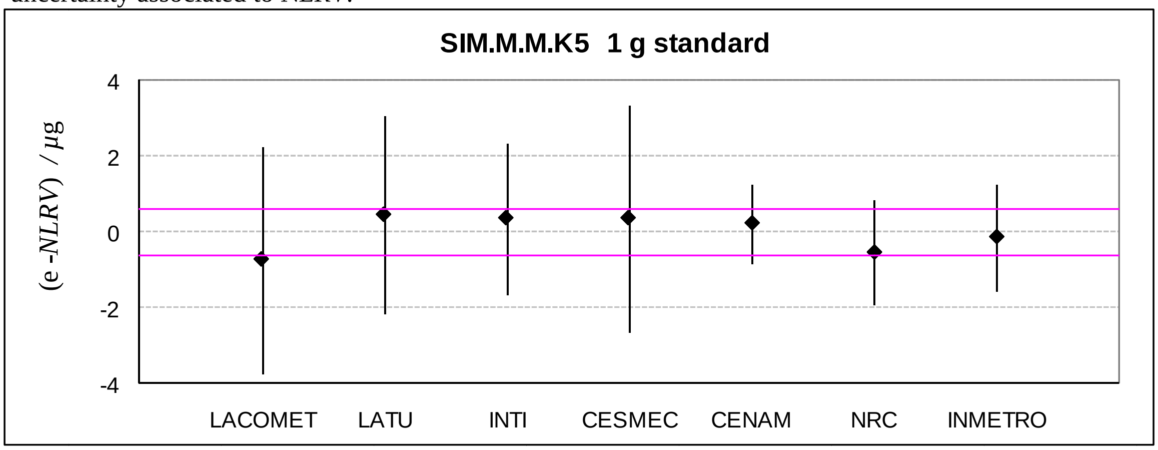


Figure 2E. Deviations from NLRV for the 200mg standard. The solid pink lines represent the expanded uncertainty associated to N*LRV*.

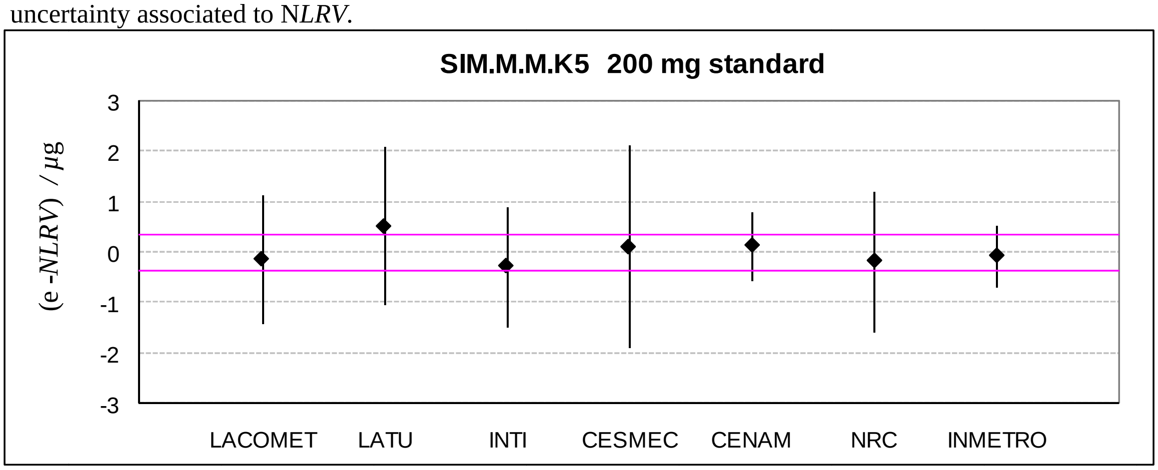


Figure3A. Deviations from LRV for the 2 kg standard. The solid pink lines represent the expanded uncertainty associated to *LRV*.

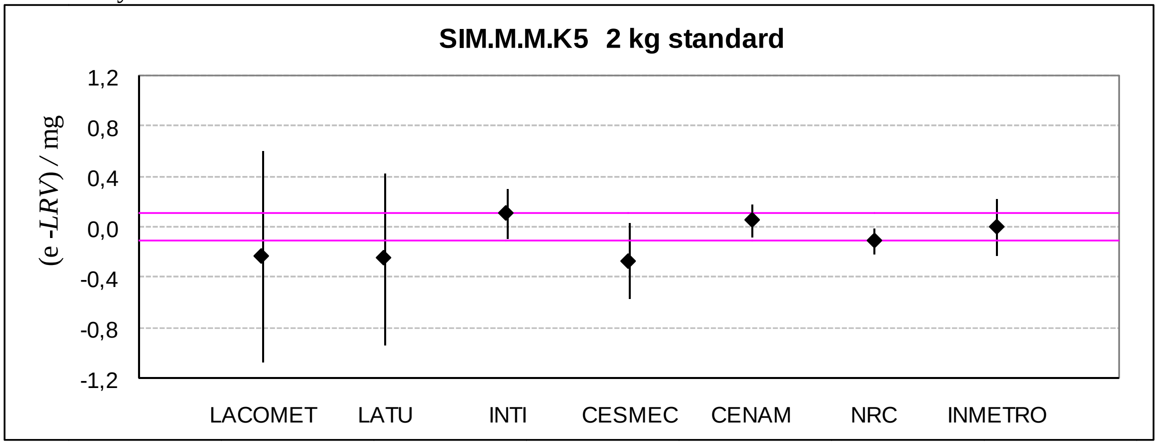


Figure 3B. Deviations from LRV for the 200 g standard. The solid pink lines represent the expanded uncertainty associated to *LRV*.

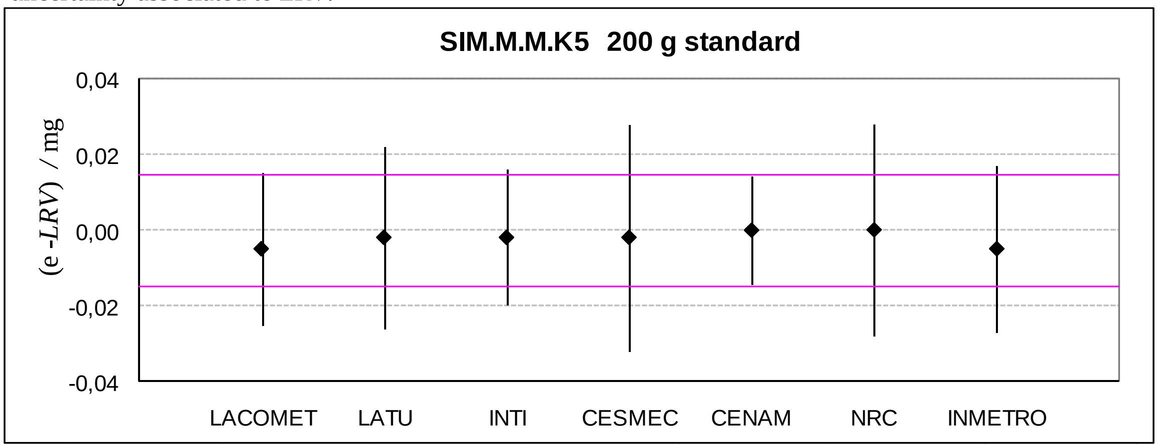


Figure 3C. Deviations from LRV for the 50 g standard. The solid pink lines represent the expanded uncertainty associated to *LRV*.

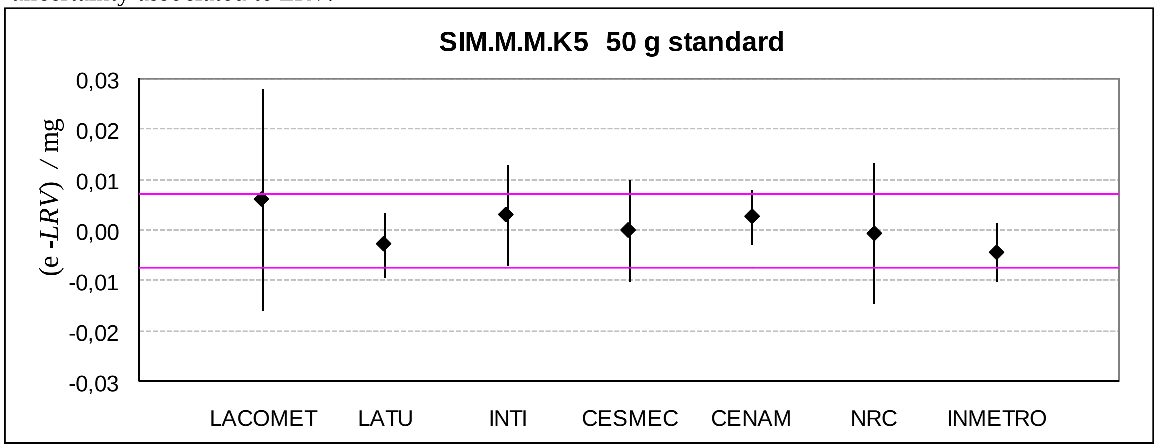


Figure 3D. Deviations from LRV for the 1 g standard. The solid pink lines represent the expanded uncertainty associated to *LRV*.

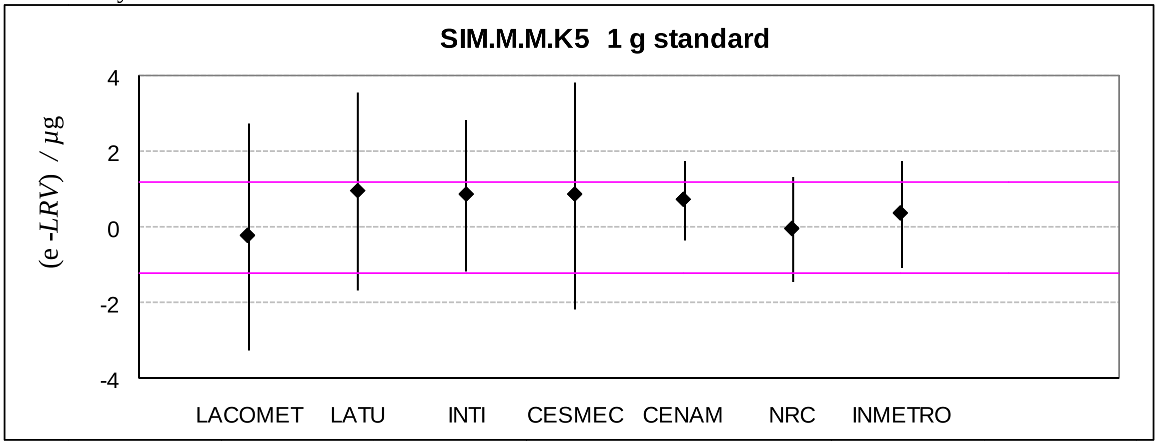
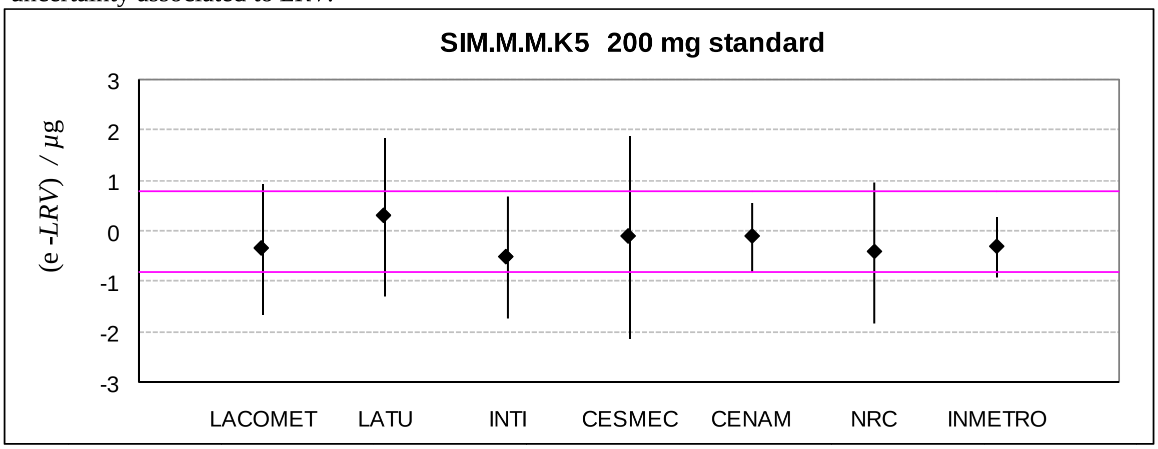


Figure 3E. Deviations from LRV for the 200 mg standard. The solid pink lines represent the expanded uncertainty associated to *LRV*.



9. References

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