

FINAL REPORT SIM COMPARISON IN VOLUME OF WEIGHTS SIM.M.D-K3

Kornblit, F¹; Leiblich, J.¹; Becerra, L.O.²; Peña, L.M.²; Luján, L.²; Díaz,J.C.²; Centeno, L.M.²; Ramos, O³; Rodríguez, S.³; Santo, C.⁴; Caceres, J.⁴; García, F.⁵; Leyton, F.⁵; Claude J.⁶; Loayza, V.M.⁷; Cacais,F.A.⁷

Abstract: This report summarizes the results of a comparison concerning the determination of volume of a set of four weights which nominal values are 2 kg, 1 kg, 200 g and 1 g. Seven NMIs within SIM have participated. The results reported are consistent with each other. Also, they can be used to evidence degrees of equivalence with other RMOs results once linking results are available.

1. General Information

The present comparison, named SIM.M.D-K3, 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 determination of volume of mass standards. 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	Canada	Jacques, C.
INMETRO	Brazil	Loayza, V.M.; Cacais, F.A.

INTI (Argentina)¹ has acted as the pilot laboratory

3. General Considerations and Procedure

A set of four 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.

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); ⁵CESMEC (Chile); ⁶ NRC, National Research Council (Canada), INMETRO, Instituto Nacional de Metrologia, Qualidade e Tecnologia (Brazil)



 Table 1. Data associated to the standard

Nominal	Serial	Idantification	
mass value	Number	Identification	
2 kg			
1 kg	40601140	520770772104	
200 g	40001140	320779772194	
1 g			

The traveling standards were placed in individual wooden cases for transportation purposes, which was placed in a carrying transportation case, jointly with the standards corresponding to the comparisons SIM.M.M-K1 and SIM.M.M-K5. 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 hydrostatic method was employed to measure the volume of the standards
- Measurements were done after an appropriate acclimatization time
- The CIPM-2007 formula [1] was applied by all the participants to determine the air density, in order to estimate air buoyancy effects.
- After the measurements, the travelling standards were cleaned in appropriate ways according to the transfer liquids employed in the calibrations.
- The volume thermal expansion coefficient for all the traveling standards was considered as equal to $4,80 \times 10^{-5} \text{ °C}^{-1}$.

4. Schedule

The measurements followed the schedule shown in Table 2.

N°	Institute / Country	Dates of the
		measurements
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

 Table 2. Measurement schedule



5. Summary of the reported results

The results sent by the participants, expressed as the volumes V (referred to 20 °C), and their associated standard uncertainty u_V , are shown in table 3.

	2 kg		1 kg		200g		1 g	
	V/cm^3	u_V/cm^3	V/cm^3	u_V/cm^3	V/cm^3	u_V/cm^3	V/cm^3	u_V/cm^3
LACOMET	254.674	0.040	127.243	0.021	24.857 4	0.008 2	0.126 1	0.003 3
LATU	254.663	0.005	127.242 9	0.0032	24.857 5	0.002 1	0.125 63	0.000 28
INTI	254.665	0.004	127.240 4	0.0031	24.853 0	0.001 0	0.125 99	0.000 20
CESMEC	254.668	0.004	127.237 0	0.0020	24.854 0	0.000 5	0.126 00	0.000 30
CENAM	254.664	0.004	127.237 4	0.0020	24.853 94	0.000 66	0.125 83	0.000 28
NRC	254.656	0.009	127.239 91	0.000 16	24.854 12	0.000 04	0.125 89	0.000 02
INMETRO	254.668	0.005	127.238 4	0.001 4	24.853 7	0.000 5	0.126 2	0.000 6
RV	254.6652	0.002	127.239 86	0.000 16	24.854 12	0.000 04	0.125 89	0.000 02

Fable 3.	Data	sent	by	the	partici	oants
			- /			

6. Liquids employed in the hydrostatic weighing

The laboratories employed different liquids to perform the hydrostatic weighing and different ways to determine their density. Some laboratories used pure water and calculated its density by the Tanaka's formula [2]. In the other cases the measurements of the liquids density are traceable to liquid or solid density standards. The buoyant liquids employed, their estimated density values ρ referred to 20 °C and the corresponding standard uncertainties u_{ρ} are summarized in table 4, as well as the methods and traceability sources to determine ρ .

NMI	Buoyant liquid	$ ho/{ m kg}{ m m}^{-3}$	$u_{\rho}/\mathrm{kg}\mathrm{m}^{-3}$	Method / Traceability source
LACOMET	Bidistilled and deionized water	998.191 3	0.031 4	Hydrostatic weighing / Tanaka formula [2]
LATU	Distilled water	997.581 16	0.000 43	Hydrostatic weighing / Tanaka formula [2]
INTI	Distilled water	998.278 7	0.008 4	Hydrostatic weighing / Tanaka formula [2]
CESMEC	Ciclohexane	778.602 6	0.001 5	Hydrostatic Weighing / Silicon crystal traceable to PTB
CENAM	Pentadecane	768.638	0.002	Hydrostatic weighing / Zerodur sphere, traceable to PTB
NRC (2 kg)	NRC water	998.2	0.002	Hydrostatic weighing / Tanaka formula [2] with isotopic correction
NRC (1kg, 200 g, 1 g)	FC-40	1 878.685	0.003	Comparison with a silicon cylinder / Tanaka formula [2] with isotopic correction
INMETRO (2 kg)	n-Dodecane	748.806 0	0.002 5	Hydrostatic Weighing / Silicon sphere, traceable to NMIJ
INMETRO (1 kg)	FC-40	1 882.94	0.04	Comparison with the volume of an E ₁ stainless steel weight measured at CENAM
INMETRO (200 g, 1 g)	FC-40	1 882.94	0.04	Subdivision Method / INMETRO

Table 4. Buoyant liquids employed and methods and traceability sources used	to measure their density



7. Data consistency and computation of reference values and degrees of equivalence

In order to check the consistency of the results, χ^2 tests were applied. For the different standards, the χ^2_{obs} statistics as defined in [3] were calculated. The following values were obtained:

Nominal	χ^2
value	λ_{obs}
2 kg	2.5
1 kg	5.7
200 g	4.8
1 g	1.6

The corresponding critical value for v = 6 degrees of freedom and significant level 0.05 is $\chi^2(v) = 12.6$ (the same for all the standards). Therefore, in all the cases $\chi^2_{obs} \leq \chi^2(v)$. So, all the data can be considered consistent and the assumptions to employ the so-called *Procedure A* in [3] are considered valid. Strictly speaking, not all of them are fully satisfied. INMETRO has employed a solid standard calibrated at CENAM as traceability source for 1 kg, 50 g and 1 g (see table 4). Then, the results provided by both NMIs are linked to a common standard (a CENAM's zerodur sphere). So, a positive correlation exists between them and the *Condition of Use* 2 in [3] may be considered breached. However, the uncertainty associated to that common standard is an order smaller than the uncertainties declared by both NMIs in the comparison. Then, the effect produced by that correlation is considered negligible.

Then, the weighted averages were calculated and established as the reference values of the comparison (*RV*). They are shown in the last row of table 3 with their associated uncertainties. For each participant and standard, degrees of equivalence *D* and normalized errors E_n were calculated according to (1). They are shown in tables 5A and 5B.

$$D = V - RV; \quad U_D = 2\sqrt{u_V^2 - u_{RV}^2}; \quad E_n = D/U_D$$
 (1)

where V and u_V are the results reported by the participant. Bilateral differences between pairs of participants and bilateral normalized errors can be calculated according to:

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



	21	kg	1	kg	200 g		1 g	
	D / cm^3	U_D / cm^3	D/cm^3	U_D / cm^3	D/cm^3	U_D / cm^3	D/cm^3	U_D / cm^3
LACOMET	0.009	0.080	0.003	0.042	0.003 3	0.016	0.000 2	0.006 6
LATU	-0.003	0.008	0.003	0.006	0.003 4	0.004 2	-0.000 3	0.000 6
INTI	0.000	0.006	0.001	0.006	-0.001 1	0.002 0	0.000 1	0.000 4
CESMEC	0.003	0.007	-0.003	0.004	0.000 1	0.001 0	0.000 1	0.000 6
CENAM	-0.001	0.006	-0.002	0.004	0.000 2	0.001 3	-0.000 1	0.000 6
NRC	-0.009	0.017	0.000 05	0.000 06	0.000 00	0.000 01	0.000 00	0.000 01
INMETRO	0.003	0.009	-0.001 5	0.002 8	-0.000 4	0.001 0	0.000 3	0.001 2

Table 5A. Degrees of equivalence

Table 3D. Values of En										
	2 kg	1 kg	200 g	1 g						
LACOMET	0.1	0.1	0.2	0.0						
LATU	-0.3	0.5	0.8	-0.5						
INTI	0.0	0.1	-0.6	0.3						
CESMEC	0.3	-0.7	-0.1	0.2						
CENAM	-0.1	-0.6	-0.1	-0.1						
NRC	-0.5	0.8	0.4	0.0						
INMETRO	0.3	-0.5	-0.4	0.3						

Table 5B Values of F.

8. Link to CCM key comparisons

CCM comparisons in density of solids are not available yet. However, there is a planned one, named CCM.D-K3 (see [4]). Once it is complete, the current comparison could be linked to it for nominal values common to both them.

9. Plots.

The results indicated in table 4.A are plotted in figures 1, 2, 3, and 4. In those plots, the dotted pink lines represent the expanded uncertainty U_{RV} associated to RV. In some of them, both lines are too closed to be distinguished for the scale chosen.









Figure 2. Plot of the measured results for the 1 kg standard











9. 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
- Tanaka. M; Girard. G.; Davis. R.; Peuto. A.; Bignell. N.; *Recommended table for the density of water between 0 C and 40 C based on recent experimental reports;* Metrologia 38 (2001). 301-309.
- 3. Cox. M.G.. The evaluation of key comparison data. Metrologia 39 (2002) 589-595
- 4. <u>http://kcdb.bipm.org/AppendixB/KCDB_ApB_search_result.asp?search=1&met_idy=6&</u> <u>bra_idy=23&cmt_idy=1&ett_idy_org=16&epo_idy=0&cou_cod=0</u>