

ANDEAN COMMUNITY - ANDIMET
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FINAL REPORT

**COMPARISON OF MASS STANDARDS
(100 mg, 5 g, 20 g, 100 g, and 1 kg)
SIM.7.29**

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This document is a translation of the official Spanish version of the Final Report of the comparison in mass, carried out inside the Cooperation program for Technical assistance in Quality matters between the Andean Community (CAN) and the European Union (EU)

FINAL REPORT
COMPARISON OF STANDARDS OF MASS
(100 mg, 5 g, 20 g, 100 g, and 1 kg)
SIM.7.29

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1. Introduction

This report describes the results of the comparison of mass standards, carried out inside the Program of Cooperation and Technical Assistance UE-CAN in Quality, N°AECR/B7-31/IB/96/0188.

The Program of Cooperation and Technical Assistance in quality matters UE-CAN, N° AEGR/B7-31/IB/96/0188 started in 2003. An important topic of this program was the setting up of measurement comparisons of several quantities between Centro Español de Metrología (CEM) and the Andean Community (CAN). All Member countries of the Andean Community would participate in the project.

For this purpose, in May 2004, technical contacts from CAN and from CEM had set a meeting in Madrid in order to decide the criteria and detail of the comparisons, and to determine the technical protocol of these comparisons.

At that meeting, it was decided that for mass, the comparison would consist of measuring the mass and the associated uncertainty of a series of traveling standards. The traveling standards were the property of SIM and were class OIML E2 with the following nominal values: 1 kg, 100 g, 20 g, 5 g, and 100 mg.

This comparison was piloted by the Centro Español de Metrología (CEM, Spain), Co-piloted by the Centro Nacional de Metrología (CENAM, Mexico) and coordinated within the CAN by the “Instituto Boliviano de Metrología” (IBMETRO, Bolivia). Seven NMIs were participating.

The mass standards were prepared by CEM. CEM measured the volume and density of the traveling standards with the exception of the 100 mg weight, the value of which was provided by SIM.

All the standards were circulated among the NMIs. Each NMI measured the mass and its uncertainty for each weight using their procedures and methods. The measurements were carried out from September 2004 to June 2005.

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⁷ Servicio Autónomo Nacional de Metrología (SENCAMER, Venezuela)

2. List of participating NMIs

The participating laboratories and their respective technical contacts are listed below:

- *Centro Español de Metrología - CEM, España*
M. Mar San Andrés Redondo
Teresa Amar Santa Cruz
- *Centro Nacional de Metrología - CENAM, México*
Luis Omar Becerra
Jorge Nava Martínez
- *Servicio Autónomo Nacional de Metrología - SENCAMER, Venezuela*
Elías Salazar
Luis Rojas
- *Instituto Ecuatoriano de Normalización (INEN, Ecuador)*
René Chanchay
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- *Superintendencia de Industria y Comercio de Colombia (SIC, Colombia)*
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- *Instituto Nacional de Defensa de la Competencia y de la Protección de la Propiedad Intelectual (INDECOP, Perú)*
Aldo Quiroga
Juan Rodriguez
- *Instituto Boliviano de Metrología (IBMETRO, Bolivia)*
Gerson Vallejos Silva
Marco Bautista Rodas

SENCAMER, INEN, SIC, INDECOP and IBMETRO, are NMIs belonging to CAN. CEM is a NMI belonging to EUROMET and was the NMI designated by the European Union UE as the pilot laboratory for this project. CENAM took part of this comparison, as requested by CAN, in order to have recognition of the comparison within SIM.

3. Traveling standards

The traveling standards used was a set of weights Class OIML E2 belonging to SIM, with the following nominal values of 1 kg, 100 g, 20 g, 5 g, and 100 mg. The shape and material of the traveling standards are shown in table 1.

Nominal value	Accuracy Class	Material	Shape
1 kg, 100 g, 20 g, 5 g	E2	Stainless Steel	cylindrical
100mg	E2	Stainless Steel	sheet

Table 1

The traveling standards were transported from Lima to Madrid on 5TH July 2004 for their preparation and initial calibration.

In August 2004, the density and volume of the 1 kg, 100 g, 20 g, and 5 g standards were measured at the Density Laboratory of CEM. The results are shown in table 2. The density and volume for 100 mg weight were provided by SIM.

Nominal value	Volume (cm ³)	Uncertainty (k =2) (cm ³)	Density (kg/cm ³)	Uncertainty (k =2) (cm ³)
1 kg	125,887 9	0,001 6	7 943,59	0,10
100 g	12,583 6	0,000 6	7 946,85	0,40
20 g	2,517 2	0,000 5	7 945,5	1,6
5 g	0,629 7	0,000 4	7 940,2	4,8
100 mg	0,012 6	0,000 2	7 950	100

Table 2

The magnetic susceptibility of the traveling standards was not measured.

4. Circulation Schedule

The circulation schedule of the traveling standards are shown in table 3. CEM started the measurements in September 2004 and the last measurements were also made by CEM in June 2005. The original schedule was modified due to difficulties in the transportation and retention of the standards at customs in some countries.

Laboratory	Arrival date	Departure Date
Centro Español de Metrología - CEM	2004-07-05	2004-09-19
Centro Nacional de Metrología – CENAM	2004-10-25	2004-11-12
Servicio Autónomo Nacional de Metrología – SENCAMER	2004-11-13	2005-02-02
Instituto Ecuatoriano de Normalización – INEN	2005-02-03	2005-03-01
Superintendencia de Industria y Comercio - SIC	2005-03-02	2005-04-07
Instituto Nacional de Defensa de la Competencia y de la Propiedad Intelectual – INDECOPI	2005-04-08	2005-05-02
Instituto Boliviano de Metrología - IBMETRO	2005-05-03	2005-05-23
Centro Español de Metrología - CEM	2005-06-07	2005-07-01

Table 3

5. Surface damages of the standards.

The traveling standards were examined by each NMI at the reception and departure of the weights in order to register all marks and damages during circulation.

The participating laboratories sent to the pilot laboratory, the forms used to register the superficial conditions of the traveling standards, at both reception and departure. Template of the forms used to register the superficial conditions (marks and scratches) of the traveling standards, were shown in Annex IV of the Technical Protocol. The data sent by the participating laboratories showed that there was no significant damage on the traveling standards.

6. Measurement Conditions

The maximum variation of environmental conditions for calibration reported by the participating laboratories is listed in table 4. The limits recommended by the International Recommendation OIML R111 [2] are shown in tables 5 and 6. The values of temperature, relative humidity, dew point temperature and air density given in table 4 correspond to mean values during the comparison reported by participating NMIs. The relative humidity reported by SENCAMER, INEN and IBMETRO were outside the limits recommended by OMIL R111 [2].

	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
T (° C)	21,00	20,5	20	20,23	21	21,5	20	22
ΔT/h (°C)	0,02	0,2	0,3	0,6	0,5	0,5	0,5	0,03
H / tr	12,6 °C	54 %	49 %	60 %	65 %	55 %	42 %	12,4 °C
ΔH / Δtr	(11–14)°C	(50–58) %	(45–56) %	(55–64) %	(50 -70) %	(50–60) %	(35–50) %	(11–13)°C
ρ _a (kg/m ³)	1,088 4	0,955 9	0,892 0	1,135 9	0,871 4	1,166 7	0,788 3	1,097 5

Table 4

Accuracy class	Temperature variation in the laboratory during the calibration
E ₁	± 0,3 °C per hour with a maximum of ± 0,5 °C per 12 hours
E ₂	± 0,7 °C per hour with a maximum of ± 1,0°C per 12 hours
F ₁	± 1,5 °C per hour with a maximum of ± 2,0 °C per 12 hours
F ₂	± 2,0 °C per hour with a maximum of ± 3,5 °C per 12 hours
M ₁	± 3,0 °C per hour with a maximum of ± 5,0 °C per 12 hours

Table 5

Accuracy class	Range of relative humidity in the laboratory during the calibration
E ₁	40 % to 60 % with a maximum of ± 5 % per 4 hours
E ₂	40 % to 60 % with a maximum of ± 10 % per 4 hours
F	40 % to 60 % with a maximum of ± 1,5 %per 4 hours

Table 6

The weighing instruments used by the different laboratories comply with the required resolution for calibration of E2 standards. Their resolutions are shown in Table 7.

Units in mg	CEM	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO
1 kg	0,001	0,001	0,001	0,1	0,1	0,1	0,01
100 g	0,001	0,01	0,001	0,01	0,05	0,001	0,01
20 g	0,001	0,001	0,001	0,01	0,005	0,001	0,01
5 g	0,000 1	0,000 1	0,01	0,005	0,005	0,000 1	0,000 1
100 mg	0,000 1	0,000 1	0,001	0,005	0,005	0,000 1	0,000 1

Table 7

The resolution of the instruments used by the different laboratories to measure the environmental conditions are shown in table 8.

	CEM	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO
TEMPERATURE	0,001 °C	0,1 °C	0,1 °C	0,01 °C	0,1 °C	0,01 °C	0,1 °C
PRESURE	0,01 mbar	0,01 mbar	0,01 mbar	0,5 mbar	0,13 mbar	1 mbar	0,1 mbar
HUMIDITY	0,001 %	0,1 %	0,5 %	0,1 %	5,0 %	0,1 %	0,5 %

Table 8

Table 9 shows the traceability of the mass standards used by the NMIs, to indicate the possible correlation of the traceability source of the standards.

	CEM	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO
1 kg	CEM	CENAM	DKD	PTB	NIST	DKD	DKD
100 g	CEM	CENAM	DKD	PTB	NIST	DKD	DKD
20 g	CEM	CENAM	DKD	PTB	NIST	DKD	DKD
5 g	CEM	CENAM	DKD	PTB	NIST	DKD	DKD
100 mg	CEM	CENAM	DKD	PTB	NIST	DKD	DKD

Table 9

The dates of calibration of the standards used for the measurements are listed in table 10. An overdue calibration date could introduce a drift that may affect the results of the calibration.

	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM1
1 kg	2002	2004	1997	2002	2003	2003	2004	2005
100 g	2002	2004	1997	2002	1992	2003	2004	2005
20 g	2002	2004	1997	2002	1972	2003	2004	2005
5 g	2002	2004	1997	2002	1972	2005	2004	2005
100 mg	2002	2004	1997	2002	1992	2003	2004	2005

Table 10

7. Procedures and measurement methods

The measurement method used by all the laboratories was the substitution method. CEM used a minimum squares procedure to obtain the results by means of the Gaussian-Marcov estimation that uses weights of the same nominal value. This method consists of solving an over dimensioned system of equations, where each equation represents a comparison of mass standards of equal nominal value.

CENAM used the subdivision procedure, starting from one kilogram and generating a set of independent measurements that is modeled by a system of linear equations and the solution to this set of equations was found by the technique of minimum squares applying the Lagrange multipliers approximation.

The density of the air was calculated by equation CIPM 81/91 [1], or by approximate equations, according to table 11

Laboratory	Equation
CEM	CIPM 81/91
CENAM	CIPM 81/91
SIC	CIPM 81/91
SENCAMER	Approximated formula ¹
INEN	Approximated formula
INDECOP	Approximated formula ¹
IBMETRO	Approximated formula ¹

Table 11

¹ Approximated formula given by CIPM in OIML R111 [2]

8. Results of the measurements

Table 12 shows the mass error found by the NMIs and its associated uncertainty for a confidence level of 95,45 %.

The results were expressed as recommended in EA 4/02 [4], and GUM [5].

NOMINAL VALUE	100 mg		5 g		20 g		100 g		1 kg	
	Error μg	U (k=2) μg	Error μg	U (k=2) μg	Error mg	U (k=2) mg	Error mg	U (k=2) mg	Error mg	U (k=2) mg
CEM1	-20,6	1,1	22,3	3,0	0,053 6	0,005 6	0,207	0,013	1,581	0,083
CENAM	-19,50	0,60	22,3	2,0	0,055 4	0,002 8	0,198 5	0,008 3	1,561	0,020
SIC	-23,0	2,4	1	12	0,066 0	0,009 6	0,226	0,018	1,70	0,18
SENCAMER	-24,5	5,5	18,4	7,3	0,057	0,015	0,141	0,025	0,37	0,25
INEN	-33,1	5,9	21,3	9,3	0,092	0,017	0,219	0,073	1,57	0,19
INDECOPPI	-23,0	2,4	23,0	8,0	0,058	0,013	0,207	0,024	1,6	0,30
IBMETRO	-25,4	1,6	18,4	5,1	0,062	0,010	0,203	0,017	1,64	0,15
CEM2	-21,73	0,68	22,4	1,8	0,056 3	0,005 6	0,207 4	0,007 2	1,599	0,070

Table 12

Uncertainty analysis has been made for all NMIs according to GUM [5].

The uncertainty contributions assigned by the NMIs for each nominal value are shown in tables 13, 14, 15, 16 and 17 respectively, according to the format established in Annex III of the Technical Protocol.

Uncertainty contributions reported by the NMIs for 1 kg weight.

NOMINAL VALUE	1 kg	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOPPI	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i	UNIT	u $k = 1$ $u(x_i)$							
Mass of standard, P	mg	0,042	0,010	0,075	0,075	0,023	0,075	0,075	0,035
Drift of standard, δ_p	mg	0,001 6		0,043	0,043	0,061	0,087		
Volume of standard, V_p	cm ³	0,000 8	0,003 0	0,020	0,020	0,002 5	0,010	0,020	0,000 8
Volume of sample V_m	cm ³	0,000 8	0,000 8	0,000 8	0,000 80	0,000 8	0,000 8	0,000 8	0,000 8
Air density, ρ_a	mg /cm ³	0,000 16	0,000 4	0,003 0	0,069	0,036	0,000 6	0,001 5	0,000 11
Resolution of balance	mg	0,000 41	0,000 3	0,000 41	0,041	0,029	0,041	0,002 9	0,000 41
Repeatability	mg	0,000 82	0,000 4	0,000 63	0,029	0,001 3	0,053	0,001 5	0,000 63
Mass sensitivity	mg					0,000 26			
Volume of mass sensitivity	cm ³					0,000 03			
Combined uncertainty	mg	0,042	0,010	0,089	0,12	0,096	0,13	0,077	0,035

Table 13

Uncertainty contributions reported by the NMIs for 100 g weight.

NOMINAL VALUE	100 g	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i	UNIT	u $k = 1$ $u(x_i)$							
Mass of standard, P	mg	0,004 2	0,010	0,007 5	0,075	0,020	0,007 5	0,007 5	0,003 5
Drift of standard, δ_p	mg	0,004 8		0,004 3	0,004 3	0,001 0	0,008 7		
Volume of standard, V_p	cm ³	0,000 30	0,003 0	0,002 0	0,001 8	0,002 5	0,001 8	0,002 0	0,000 30
Volume of sample V_m	cm ³	0,000 30	0,000 3	0,000 30	0,000 30	0,000 30	0,000 30	0,000 30	0,000 30
Air density, ρ_a	mg /cm ³	0,000 11	0,000 4	0,003 0	0,069	0,049	0,000 6	0,001 5	0,000 11
Resolution of balance	mg	0,000 41	0,003	0,000 41	0,004 1	0,014	0,004 1	0,002 9	0,000 41
Repeatability	mg	0,000 63	0,004	0,000 63	0,003 0	0,000 59	0,001 4	0,001 5	0,000 63
Mass sensitivity	mg					0,000 26			
Volume of mass sensitivity	cm ³					0,000 030			
Combined uncertainty	mg	0,006 4	0,004 1	0,008 9	0,012	0,036	0,012	0,008 0	0,003 6

Table 14

Uncertainty contributions reported by the NMIs for 20 g weight

NOMINAL VALUE	20 g	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i	UNIT	u $k = 1$ $u(x_i)$							
Mass of standard, P	mg	0,002 1	0,004 1	0,004 0	0,004 0	0,008 0	0,004 0	0,004 0	0,002 1
Drift of standard, δ_p	mg	0,001 7		0,002 3	0,002 3	0,001 3	0,004 6		0,001 7
Volume of standard, V_p	cm ³	0,000 25	0,000 3	0,001 0	0,000 80	0,000 25	0,000 80	0,001	0,000 25
Volume of sample V_m	cm ³	0,000 25	0,000 3	0,000 25	0,000 25	0,000 25	0,000 25	0,000 25	0,000 25
Air density, ρ_a	mg /cm ³	0,000 16	0,000 4	0,003 0	0,069	0,040	0,000 6	0,001 5	0,000 16
Resolution of balance	mg	0,000 41	0,000 3	0,000 41	0,004 1	0,001 4	0,000 41	0,002 9	0,000 41
Repeatability	mg	0,000 41	0,001 2	0,000 63	0,003 8	0,003 1	0,001 4	0,001 1	0,000 41
Mass sensitivity	mg					0,000 30			
Volume of mass sensitivity	cm ³					0,000 003			
Combined uncertainty	mg	0,002 8	0,001 4	0,004 8	0,007 4	0,008 7	0,006 3	0,005 1	0,002 8

Table 15

Uncertainty contributions reported by the NMIs for 5 g weight

NOMINAL VALUE	5 g	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i	UNIT	u $k = 1$ $u(x_i)$							
Mass of standard, P	mg	0,001 3	0,001 4	0,002 5	0,002 5	0,003 6	0,002 5	0,002 5	0,000 75
Drift of standard, δ_p	mg	0,000 65		0,001 4	0,001 4	0,000 12	0,002 9		
Volume of standard, V_p	cm ³	0,000 20	0,001 3	0,000 60	0,000 50	0,000 25	0,000 50	0,000 5	0,000 10
Volume of sample V_m	cm ³	0,000 20	0,000 20	0,000 20	0,000 20	0,000 20	0,000 20	0,000 2	0,000 20
Air density, ρ_a	mg /cm ³	0,000 16	0,000 4	0,003 0	0,069	0,045	0,000 6	0,001 5	0,000 16
Resolution of balance	mg	0,000 04	0,000 03	0,004 1	0,002 0	0,001 4	0,000 41	0,000 029	0,000 04
Repeatability	mg	0,000 41	0,000 1	0,003 5	0,000 83	0,002 9	0,000 45	0,000 028	0,000 41
Mass sensitivity	mg					0,000 30			
Volume of mass sensitivity	cm ³					0,000 003 7			
Combined uncertainty	mg	0,001 5	0,001 0	0,006 2	0,003 7	0,0047	0,003 9	0,002 5	0,000 9

Table 16

Uncertainty contributions reported by the NMIs for 100 mg weight

NOMINAL VALUE	100 mg	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i	UNIT	u $k = 1$ $u(x_i)$							
Mass of standard, P	mg	0,000 35	0,001 7	0,000 75	0,000 75	0,000 33	0,000 75	0,000 75	0,000 20
Drift of standard, δ_p	mg	0,000 36		0,000 43	0,000 43	0,000 12	0,000 90		0,000 23
Volume of standard, V_p	cm ³	0,000 05	0,000 5	0,000 10	0,000 10	0,000 10	0,000 10	0,000 30	0,000 05
Volume of sample V_m	cm ³	0,000 10	0,000 1	0,000 10	0,000 10	0,000 10	0,000 10	0,000 10	0,000 10
Air density, ρ_a	mg / cm ³	0,000 16	0,000 4	0,003 0	0,069	0,041	0,000 6	0,001 5	0,000 16
Resolution of balance	mg	0,000 08	0,000 03	0,000 41	0,002 0	0,001 4	0,000 040	0,000 029	0,000 08
Repeatability	mg	0,000 041	0,000 05	0,000 71	0,001 6	0,002 9	0,000 11	0,000 028	0,000 041
Mass sensitivity	mg					0,000 30			
Volume of mass sensitivity	cm ³					0,000 003 7			
Combined uncertainty	mg	0,000 50	0,000 3	0,001 2	0,002 7	0,0030	0,001 2	0,000 8	0,000 034

Table 17

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.

INEN considered additional uncertainty contributions due to the mass sensitivity and its volume. These uncertainty contributions are negligible in comparison with the rest. Therefore, the combined uncertainty declared by INEN is not greatly affected

The estimation of the uncertainty in the case of CENAM was made by applying the propagation law of uncertainties to the matrix model, this may produce different values from the ones calculated one by one.

9. Results Analysis

The reference value has been calculated as a weighed mean of the values measured by CEM in the beginning and at the end of the comparison and the value measured by CENAM, considering the inverse of the squares of the declared uncertainty as the ponder factor.

For the nominal values of 5 g, 20 g, 100 g, and 1 kg, the drift of the mass estimated with the difference between the initial and final measurements by CEM is negligible compared with the associated uncertainty.

The 100 mg weight shows a significant difference between the initial and final measurements by CEM compared with the uncertainty. The reported values from the participating laboratories for the 100 mg weight were dispersed. That dispersion could be attributed to several reasons, among them:

- Instability of the standard due to its own material.
- A bias in the density value associated to the standard. As indicated in section 3, the density of the weight of 100 mg was not measured by CEM but was provided by SIM instead. An error in the value of density can produce errors in the buoyancy effect correction, because there are significant differences in the air densities of the different laboratories.
- The error of the mass of 100 mg is greater than the *MPE* corresponding to the E₂ class, which consequently contributes along with other factors like static or magnetic effects non attributable to masses of class E₂.
- The high magnetic susceptibility, especially shown by the milligrams masses from that set, as confirmed by SIM, a fact that we cannot confirm because it was not determined

during the comparison.

Due to the consistency between the results of CEM and CENAM, as seen in appendix 2, the reference value has been calculated from a weighed mean between both values measured in CEM and the value measured in CENAM.

The mathematic model used for the calculation of the reference value is:

$$e_{ref} = \left[\frac{\frac{e_{CEM1}}{u^2 e_{CEM1}} + \frac{e_{CEM2}}{u^2 e_{CEM2}} + \frac{e_{CENAM}}{u^2 e_{CENAM}}}{\frac{1}{u^2 e_{CEM1}} + \frac{1}{u^2 e_{CEM2}} + \frac{1}{u^2 e_{CENAM}}} \right] + \delta \Delta e_{CEM} \quad (1)$$

where:

- e_{ref} is the reference value (error of the weight)
- e_{CEM1} is the value measured in the CEM at the beginning of the comparison
- e_{CEM2} is the value measured in the CEM at the end of the comparison
- e_{CENAM} is the value measured in the CENAM
- Δe_{CEM} is the difference between the values measured in CEM at the beginning and at the end of the comparison. This value is considered as a possible drift, its estimated value, $\delta \Delta e_{CEM}$ will be null, but not its uncertainty contribution.

The combined uncertainty of the reference value, applying the propagation law of uncertainties and considering the covariance between e_{CEM1} and e_{CEM2} is:

$$u^2(e_{ref}) = \left[\frac{z}{u(e_{CEM1})} + \frac{z}{u(e_{CEM2})} \right]^2 + \left[\frac{z}{u(e_{CENAM})} \right]^2 + \left[\frac{\Delta e_{CEM}}{\sqrt{3}} \right]^2 \quad (2)$$

$$\text{where } z = 1 / \left[\frac{1}{u^2 e_{CEM1}} + \frac{1}{u^2 e_{CEM2}} + \frac{1}{u^2 e_{CENAM}} \right]$$

Equation (2) considers the maximum uncertainty of $\delta \Delta e_{CEM}$.

The expanded uncertainty, for a confidence interval of 95,45 % can be obtained from equation 3.

$$u(e_{ref}) = 2 \cdot u(e_{ref}) \quad (3)$$

It has to be considered in the results analysis, that all the NMIs have the capability to carry out calibrations of weights of accuracy class E₂ except for INEN who expressed at the beginning of the comparison that its capability of measurement is class F₁. From this situation their uncertainties were expected to be greater and not directly comparable to the others.

The uncertainty values associated with the mass measurements reported by the participating laboratories are listed in Table 18. The maximum uncertainty for conventional mass calibration established in the International Recommendation 111 of the OIML [3] expressed as the MPE/3 for the accuracy classes of E₂ and F₁ are also listed in the table 18.

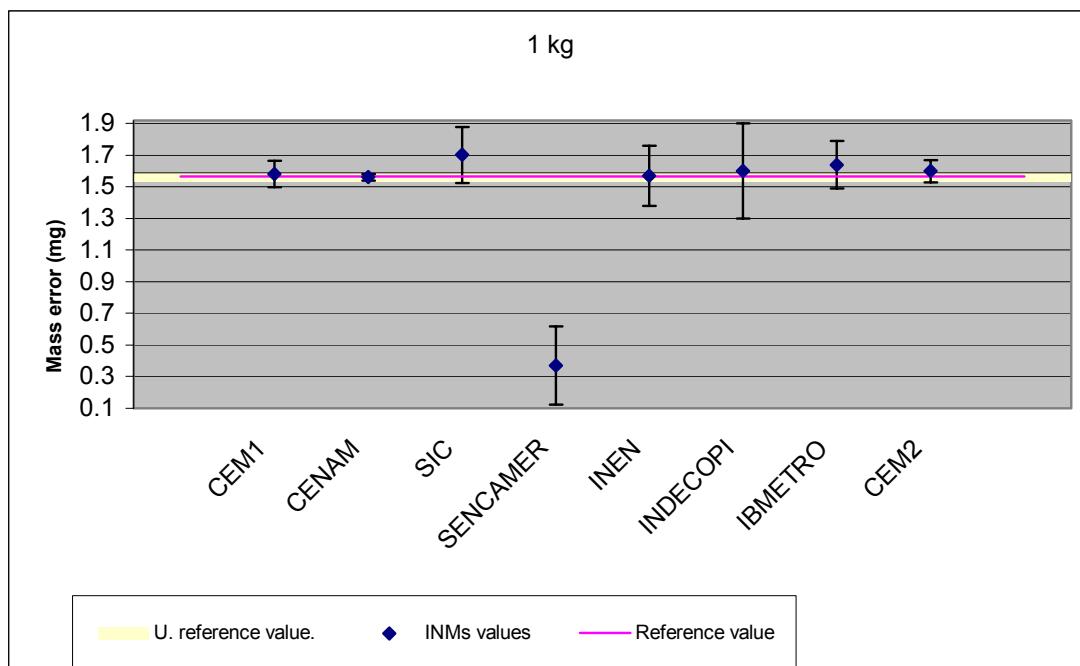
From the table, it can be seen that SENCAMER reported an expanded uncertainty for the 100 mg weight, larger than the limit recommended by OIML R111 for weights of class E₂. It has already been established that INEN calibrates in accuracy class F₁.

NOMINAL VALUE	100 mg	5 g	20 g	100 g	1 kg
	U (k=2) µg	U (k=2) µg	U (k=2) mg	U (k=2) mg	U (k=2) Mg
CEM ₁	1,1	3,0	0,005 6	0,013	0,083
CENAM	0,60	2,0	0,002 8	0,008 3	0,020
SIC	2,4	12	0,009 6	0,018	0,18
SENCAMER	5,5	7,3	0,015	0,025	0,25
INEN	5,9	9,3	0,017	0,073	0,19
INDECOPI	2,4	8,0	0,013	0,024	0,30
IBMETRO	1,6	5,1	0,010	0,017	0,15
CEM ₂	0,68	1,8	0,005 6	0,007 2	0,070
MPE/3 E ₂	5,0	16,7	0,027	0,050	0,50
MPE/3 F ₁	16,7	50	0,083	0,17	1,7

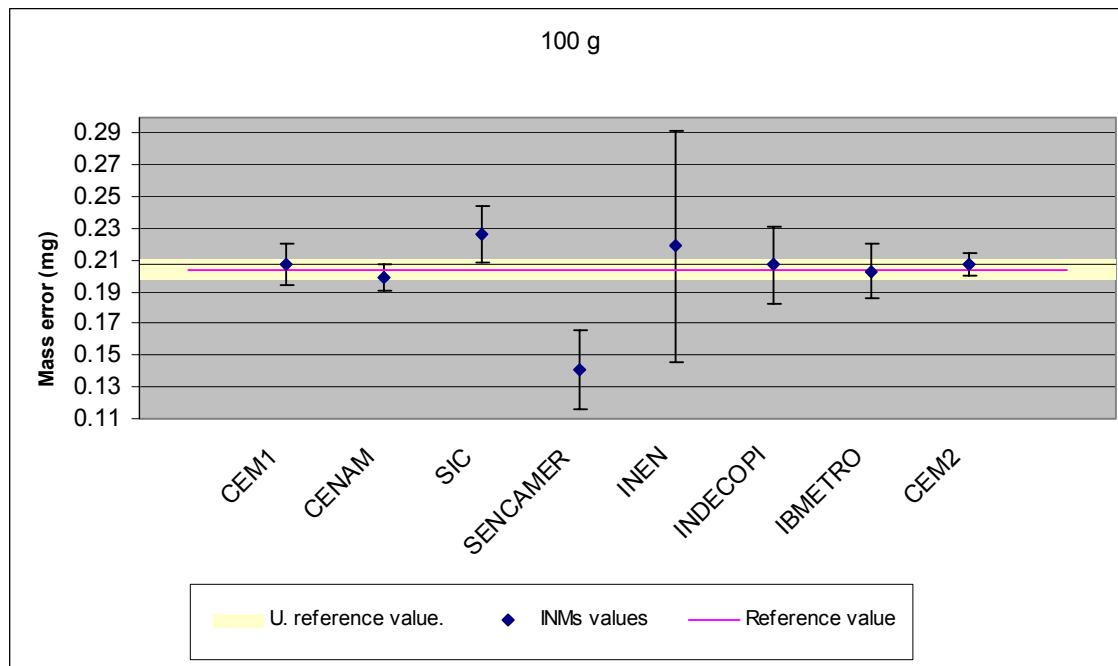
Table 18

The graphs 1, 2, 3, 4, and 5 show the representation of the results of each NMI with its uncertainties and their position relative to the reference value.

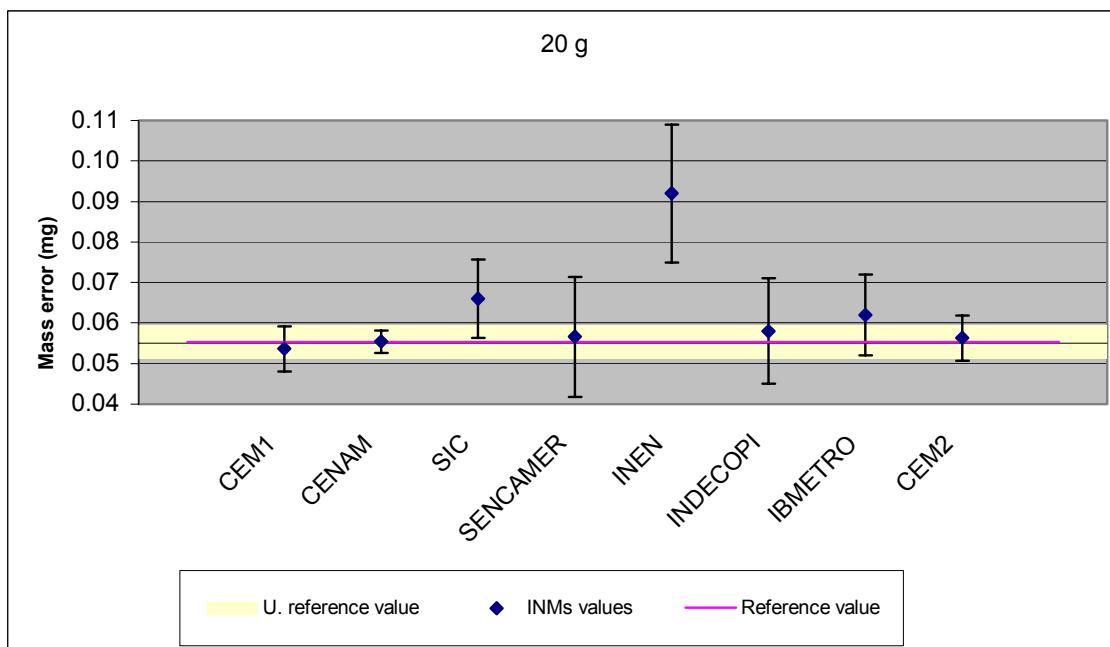
Although in some cases there are anomalous values of some laboratories with respect to the others, they have not been eliminated since the reference value has not been calculated by a general weighing.



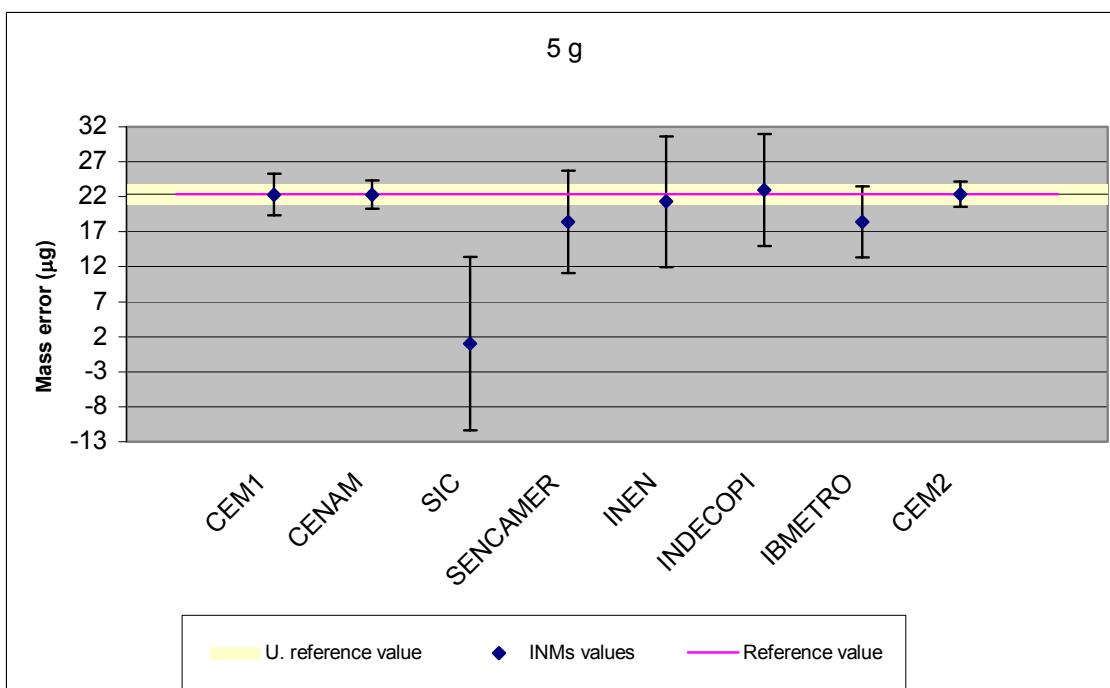
Graph 1



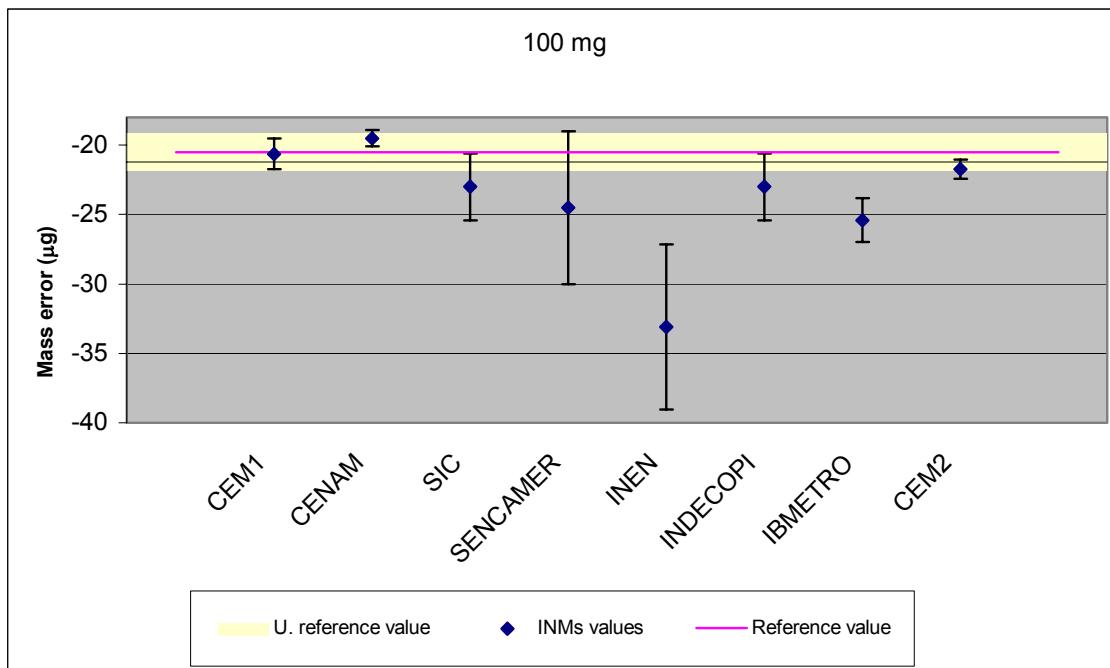
Graph 2



Graph 3



Graph 4



Graph 5

In order to verify the estimation consistency of the reference value from the weighted mean between the values of CEM and CENAM, the reference value was also estimated using other procedures. Procedures such as the arithmetic mean of CEM and CENAM values, and also the arithmetic mean of both CEM values. It can be seen in table 19 that the difference between the means was not significant.

None of the cases considered the correlation between the different countries with common traceability but the correlation between both values determined by CEM was considered.

A Montecarlo simulation was carried out with 10 000 trials to verify the consistency between the followed model and one simulated, which would include all possible covariances.

The results of this simulation can be seen in table 19 and the graphical representation of the simulation can be seen in appendix 2.

	100 mg		5 g		20 g		100 g		1 kg	
	Value	Uncert.	Value	Uncert.	Value	Uncert.	Value	Uncert.	Value	Uncert.
	µg	µg	µg	µg	mg	mg	mg	mg	mg	mg
CEM-CENAM Pondered mean	-20,5	1,4	22,3	1,5	0,055 3	0,004 1	0,204 1	0,062	1,565	0,029
CEM-CENAM Mean	-20,6	1,3	22,3	1,9	0,055 1	0,003 3	0,204 3	0,002 8	1,580	0,023
CEM Mean	-21,2	1,6	22,4	2,4	0,055 0	0,006 4	0,207	0,010	1,590	0,079
CEM-CENAM Montecarlo	-20,5	1,4	22,4	1,5	0,055 3	0,004 1	0,204 0	0,006 2	1,565	0,029

Table 19

In order to quantify the quality of the results given by the participating laboratories, it was necessary to calculate the normalized errors of measurement (or compatibility Index) with relation to the reference value and its associated uncertainty, according to the equation (4).

$$E = \frac{|e_i - e_{ref}|}{\sqrt{U^2(e_i) + U^2(e_{ref})}} \quad (4)$$

Being:

- | | |
|--------------|---|
| E | normalized error |
| e_{ref} | the reference value |
| e_i | the INM value |
| $U(e_{ref})$ | the expanded uncertainty of the reference value for $k = 2$ |
| $U(e_i)$ | the expanded uncertainty of the NMI value for $k = 2$ |

This dimensionless value should be smaller than 1. It allows comparison of the results of the participants with different uncertainties and allows the appreciation of the consistency of the obtained measurement results.

Table 20 shows the normalized errors according to the results of the NMIs for each nominal value.

NOMINAL VALUE	100 mg	5 g	20 g	100 g	1 kg
CEM ₁	0,08	0,01	0,24	0,21	0,18
CENAM	0,67	0,02	0,03	0,54	0,11
SIC	0,91	1,71	1,03	1,16	0,77
SENCAMER	0,71	0,53	0,09	2,46	4,80
INEN	2,07	0,11	2,10	0,20	0,03
INDECOP	0,91	0,08	0,20	0,12	0,12
IBMETRO	2,33	0,74	0,62	0,06	0,49
CEM ₂	0,81	0,02	0,15	0,34	0,46

Table 20

Appendix 2 contains the equivalence degree between the different participating NMIs, where the general consistency of the results can be verified.

Most of the participating countries have the intention to pursue an accreditation or to maintain the accreditation under ISO/IEC 17025, therefore it is important to consider the normalized errors given in equation (4). Since the accreditation scope declared for many laboratories is or

will be corresponding to an uncertainty declared as $\frac{mpeE_2}{3}$, (except for the INEN that will be $\frac{mpeF_1}{3}$), it is advisable also to include the calculations of the normalized error calculated with the referred uncertainty value using equation (5) to have a clear idea of their status, and what measures to take in this regard.

$U(E_2) = \frac{mpeE_2}{3}$ is the uncertainty recommended by OIML R 111 for weights of accuracy class E₂. In the case of the INEN, instead of U(E₂) it has been $U(F_1) = \frac{mpeF_1}{3}$ considered.

Table 21 expresses the normalized errors calculated according to equation (5) from the results of the NMIs for each nominal value.

NOMINAL VALUE	100 mg	5 g	20 g	100 g	1 kg
SIC	0,48	1,28	0,40	0,43	0,27
SENCAMER	0,77	0,24	0,05	1,25	2,38
INEN	0,75	0,02	0,44	0,09	0,00
INDECOPPI	0,48	0,04	0,10	0,06	0,07
IBMETRO	0,95	0,24	0,25	0,02	0,15

Table 21

Appendix 3 contains the representation of the results of each NMI with its corresponding error bars corresponding to $mep/3$ and its position in relation to the reference value.

Tables 22, 23, 24, 25, and 26 show the uncertainty percentage contributions, $\frac{u_i^2(x_i)}{u_c^2} \%$, assigned by the NMIs for each nominal value calculated from the uncertainty contributions of each influence magnitude, $u_i(x_i) = c_i \cdot u(x_i)$, and the combined uncertainty, u_c , for k = 1.

The percentage uncertainty contributions give an idea of how the partial uncertainties affect the final uncertainty reported by each NMI.

Percentage uncertainty contributions for the nominal value of 1kg:

NOMINAL VALUE	1 kg	CEM1	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i		Percent Contrib. $u_i^2(x_i) / u_c^2$						
Mass of standard, P	%	100,0	71,8	36,9	5,5	33,3	94,9	100,0
Drift of standard, δ_p	%	0,1	23,9	12,3	39,9	44,8	0,0	0,0
Volume of standard, V_p	%	0,0	4,0	0,0	0,1	0,9	4,2	0,1
Volume of sample V_m	%	0,0	0,0	0,0	0,0	0,0	0,0	0,1
Air density, ρ_a	%	0,0	0,0	34,5	8,4	0,0	0,0	0,0
Resolution of balance	%	0,0	0,0	10,9	0,0	9,9	0,1	0,0
Repeatability	%	0,0	0,0	5,4	46,9	16,6	0,0	0,0
Mass sensitivity	%				0			
Volume of mass sensitivity	%				0			
Combined uncertainty	%	100	100	100	100	100	100	100
$\sum(u_i^2(x_i) / u_c^2)$		100	100	100	101	105	99	100

Table 22

Percentage uncertainty contributions for the nominal value of 100 g

NOMINAL VALUE	100 g	CEM1	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i		Percent Contrib. $u_i^2(x_i) / u_c^2$						
Mass of standard, P	%	43,1	71,4	36,6	32,4	40,4	87,9	94,5
Drift of standard, δ_p	%	56,3	23,8	12,2	0,1	54,4	0,0	0,0
Volume of standard, V_p	%	0,2	4,0	0,0	0,4	2,9	4,0	0,7
Volume of sample V_m	%	0,2	0,1	0,0	0,0	0,1	0,1	0,7
Air density, ρ_a	%	0,0	0,0	34,6	1,3	0,0	4,0	0,0
Resolution of balance	%	0,4	0,2	10,8	0,0	12,1	13,1	1,3
Repeatability	%	1,0	0,5	6,0	67,3	1,4	3,5	3,1
Mass sensitivity	%				0,0			
Volume of mass sensitivity	%				0,0			
Combined uncertainty	%	100,0	100,0	100,0	100,0	100,0	100,0	100,0
$\sum(u_i^2(x_i) / u_c^2)$		101	100	100	101	111	113	100

Table 23

Percentage uncertainty contributions for the nominal value of 20 g

NOMINAL VALUE	20 g	CEM1	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i		Percent Contrib. $u_i^2(x_i) / u_c^2$						
Mass of standard, P	%	56,3	70,3	29,2	84,6	40,3	61,5	56,3
Drift of standard, δ_p	%	36,9	23,4	9,7	2,2	53,3	0,0	36,9
Volume of standard, V_p	%	1,0	3,6	0,0	0,1	2,0	2,5	1,0
Volume of sample V_m	%	1,0	0,2	0,0	0,1	0,2	0,2	1,0
Air density, ρ_a	%	0,0	0,0	4,3	0,6	0,0	0,0	0,0
Resolution of balance	%	2,1	0,7	30,4	0,0	0,4	32,3	2,1
Repeatability	%	2,1	1,8	26,5	12,9	4,9	4,7	2,1
Mass sensitivity	%				0,0			
Volume of mass sensitivity	%				0,0			
Combined uncertainty	%	100,0	100,0	100,0	100,0	100,0	100,0	100,0
$\sum(u_i^2(x_i) / u_c^2)$		99	100	100	100	101	101	99

Table 24

Percentage uncertainty contributions for the nominal value of 5 g

NOMINAL VALUE	5 g	CEM1	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE X_i		Percent Contrib. $u_i^2(x_i) / u_c^2$						
Mass of standard, P	%	75,1	16,3	45,7	59,9	41,1	100,0	69,4
Drift of standard, δ_p	%	18,8	5,4	15,2	0,1	55,3	0,0	0,0
Volume of standard, V_p	%	1,8	0,7	0,0	0,2	2,4	4,0	1,2
Volume of sample V_m	%	1,8	0,1	0,0	0,1	0,3	0,6	4,9
Air density, ρ_a	%	0,0	0,0	2,1	0,2	0,0	0,0	0,0
Resolution of balance	%	0,1	43,4	30,4	0,0	1,1	0,0	0,2
Repeatability	%	7,5	32,5	5,1	40,2	1,3	0,0	20,8
Mass sensitivity	%				0,0			
Volume of mass sensitivity	%				0,0			
Combined uncertainty	%	100,0	100,0	100,0	100,0	100,0	100,0	100,0
$\sum(u_i^2(x_i) / u_c^2)$		105	98	98	101	101	105	97

Table 25

Percentage uncertainty contributions for the nominal value of 100 mg

NOMINAL VALUE	100 mg	CEM1	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
INFLUENCE MAGNITUDE x_i		Percent Contrib. $u_i^2(x_i) / u_c^2$						
Mass of standard, P	%	49,0	39,1	7,7	1,3	39,1	87,9	34,6
Drift of standard, δ_p	%	51,8	13,0	2,6	0,2	56,3	0,0	45,8
Volume of standard, V_p	%	1,4	0,6	0,0	0,1	1,0	6,3	3,1
Volume of sample V_m	%	4,8	0,6	0,0	0,1	1,0	1,6	10,5
Air density, ρ_a	%	0,0	0,0	0,1	0,8	0,0	0,0	0,0
Resolution of balance	%	2,7	11,6	57,2	0,0	0,1	0,1	5,8
Repeatability	%	0,7	34,7	35,7	98,6	0,8	0,1	1,5
Mass sensitivity	%				0,0			
Volume of mass sensitivity	%				0,0			
Combined uncertainty	%	100,0	100,0	100,0	100,0	100,0	100,0	100,0
$\sum(u_i^2(x_i) / u_c^2)$		110	99	103	101	98	96	101

Table 26

The data corresponding to CENAM has not been presented since its estimation of the uncertainty was made by applying the propagation law of uncertainties to the matrix model, so it presents differences with respect to a one on one calculation and it is not consistent to do percentage estimation.

The sum of all the percentage uncertainty contributions, $\sum \frac{u_i^2(x_i)}{u_c^2} \%$, could not equal 100%

exactly, because significant figures could be lost in the rounding of each uncertainty contribution.

The estimation of the uncertainty in the case of the CENAM was made by applying the propagation law of uncertainties to the matrix model, so it presents differences with respect to a one on one calculation and also contributes to this inequality.

10. Conclusions

As a conclusion it could be stated that there is a general consistency of the measurements. The participating laboratories identified measures for improvement; some of them are already in process. For example:

Some NMIs detected a need for improved equipment and the estimation of appropriate calibration intervals of its standards, as well as the need to participate more frequently in intercomparisons.

INEN stated its need to update the traceability of its standards and to estimate their drift, as well as its recent acquisition of two new comparators with range 200 g and 1 kg and

resolutions of 1 μg and 10 μg respectively, which will represent a great improvement for its future measures.

SIC has acquired, after the comparison, a new comparator to up to 5 g and a resolution of 0,1 μg . This acquisition will support a significant improvement in its future measurements in that range.

SENCAMER declared that they suspect a possible error in the value of the 1 kg standard used in the calibration, this will be confirmed after its next calibration.

IBMETRO is requesting maintenance service for the 5 g comparator, since it suspects the comparator is malfunctioning (on the basis of reproducibility tests). They are taking the necessary actions for better control of the laboratory's environmental conditions, specifically the air's relative humidity.

11. References

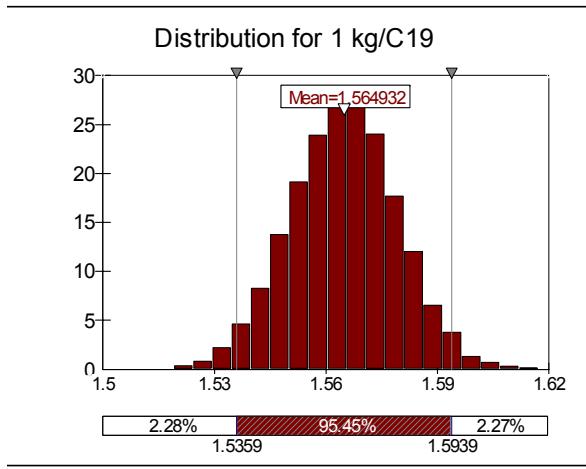
- [1] Davis R S 1992 Metrologia 29 67-70
- [2] O.I.M.L. International Recomendation n° 111. Weights of class E1, E2, F1, F2, M1, M1-2 , M2 , M2-3 and M3.; 2004.
- [3] O.I.M.L. International Recomendation n° 111. Weights of class E1, E2, F1, F2, M1, M2 , and M3., 1994.
- [4] Guide EA-4/02. Expression of the Uncertainty of Measurement in Calibration E.A. December 1999. (Clause 6.3).
- [5] 1995 Guide to the Expression of Uncertainty in Measurement (Genève: International Organization for Standardization)

Appendix 1: Brief reports of statistical data arising from the Numerical Simulation by Montecarlo Method.

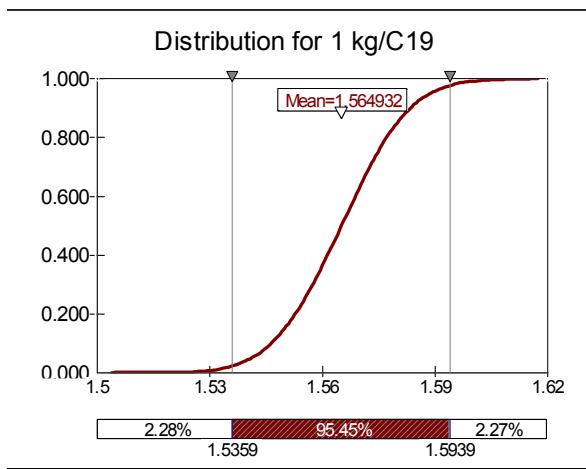
For the numerical simulation (Monte Carlo Method), formula (1) was used as the mathematical model.

Simulation Results for

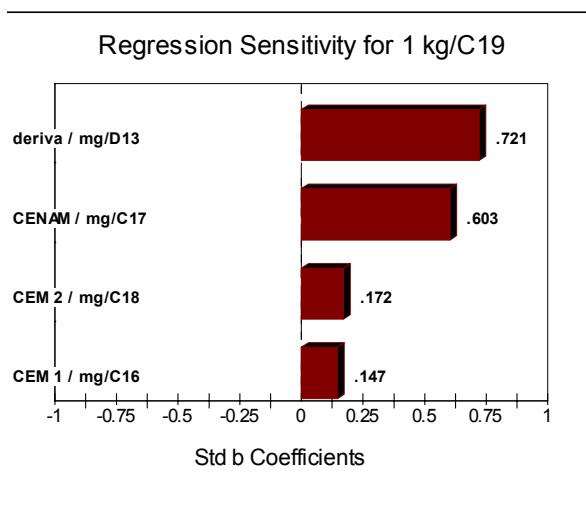
1 kg / C19



Summary Information	
Workbook Name	1 kg.xls
Number of Simulations	1
Number of Iterations	10000
Number of Inputs	4
Number of Outputs	1
Sampling Type	Monte Carlo
Simulation Start Time	15/11/2005 14:59
Simulation Stop Time	15/11/2005 14:59
Simulation Duration	00:00:09
Random Seed	1683677706

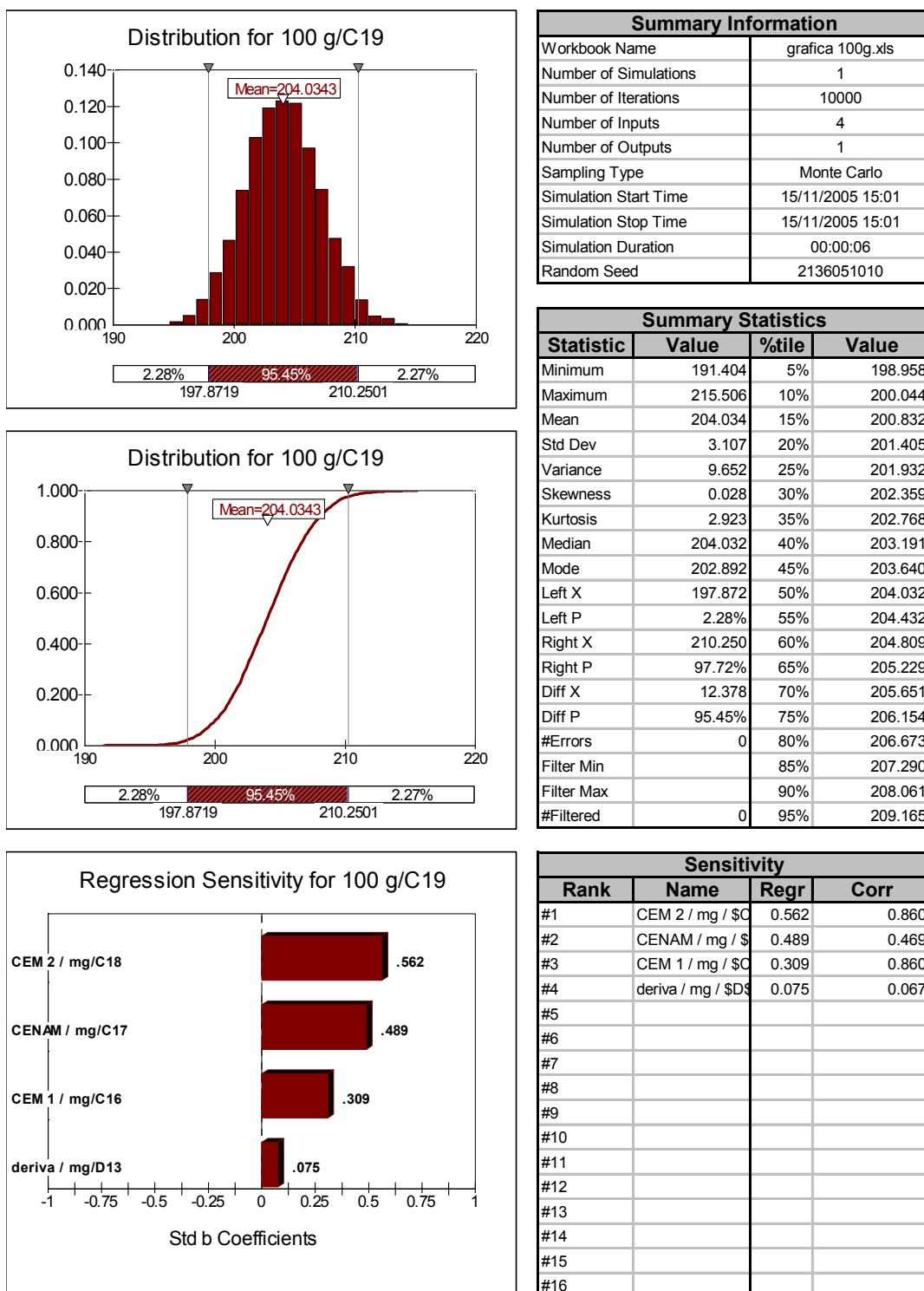


Summary Statistics			
Statistic	Value	%tile	Value
Minimum	1.5038	5%	1.5410
Maximum	1.6173	10%	1.5462
Mean	1.5649	15%	1.5499
Std Dev	0.0145	20%	1.5527
Variance	0.0002	25%	1.5552
Skewness	-0.0031	30%	1.5574
Kurtosis	3.0438	35%	1.5594
Median	1.5650	40%	1.5613
Mode	1.5432	45%	1.5632
Left X	1.5359	50%	1.5650
Left P	2.28%	55%	1.5669
Right X	1.5939	60%	1.5688
Right P	97.72%	65%	1.5706
Diff X	0.0579	70%	1.5725
Diff P	95.45%	75%	1.5747
#Errors	0	80%	1.5769
Filter Min		85%	1.5798
Filter Max		90%	1.5833
#Filtered	0	95%	1.5886

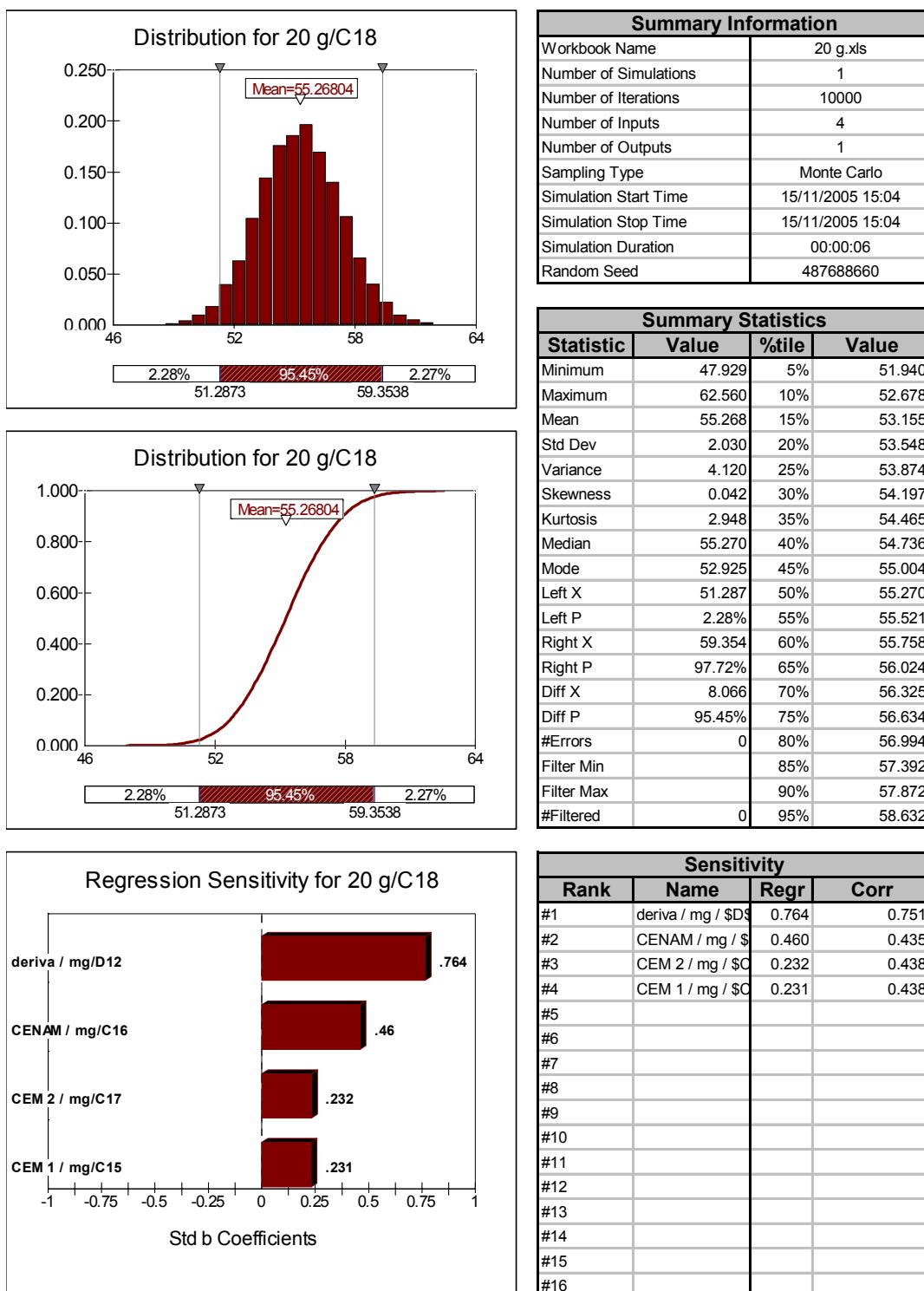


Sensitivity			
Rank	Name	Regr	Corr
#1	deriva / mg / \$D\$9	0.721	0.710
#2	CENAM / mg / \$C\$17	0.603	0.596
#3	CEM 2 / mg / \$C\$18	0.172	0.309
#4	CEM 1 / mg / \$C\$16	0.147	0.309
#5			
#6			
#7			
#8			
#9			
#10			
#11			
#12			
#13			
#14			
#15			
#16			

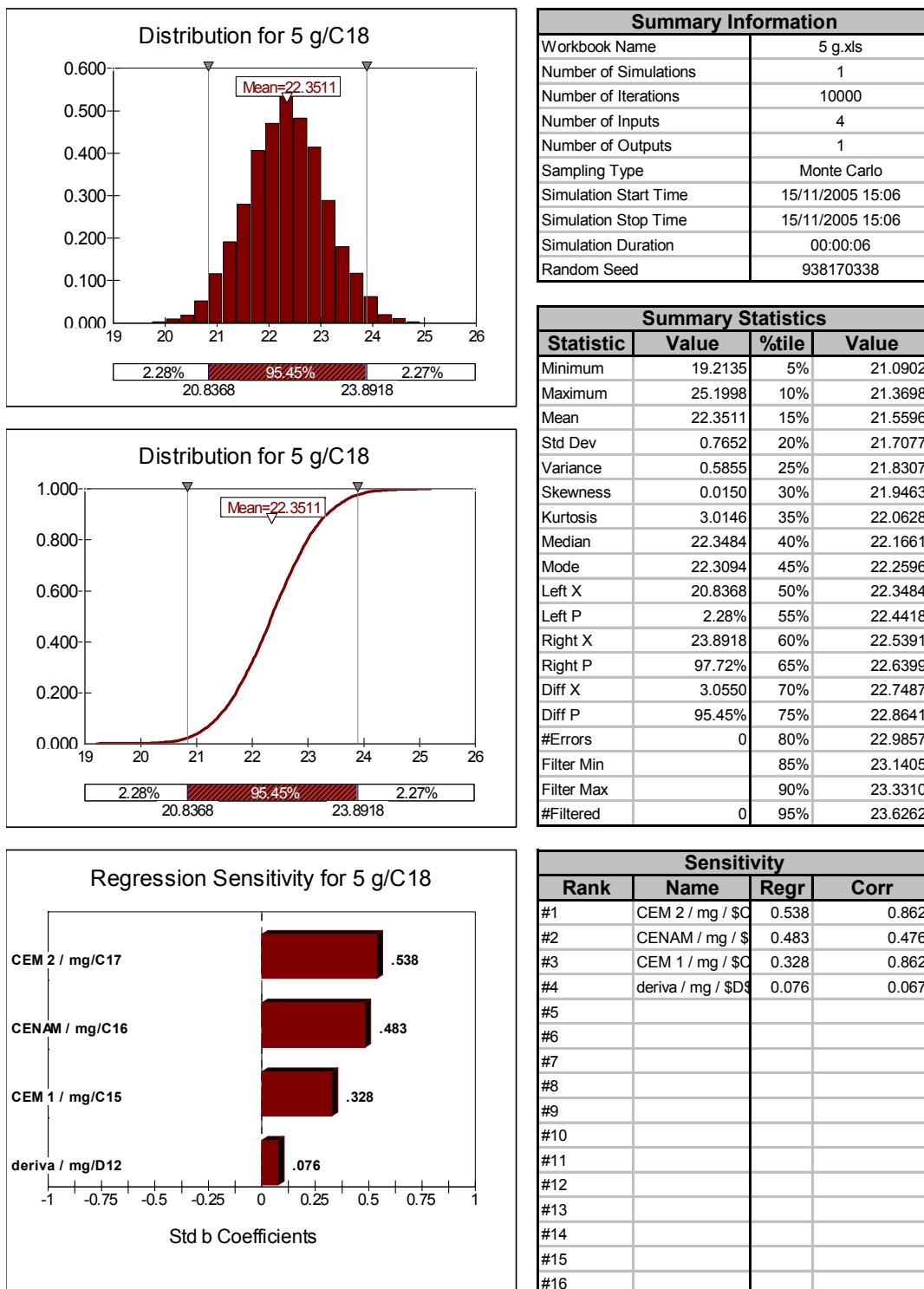
Simulation Results for 100 g / C19



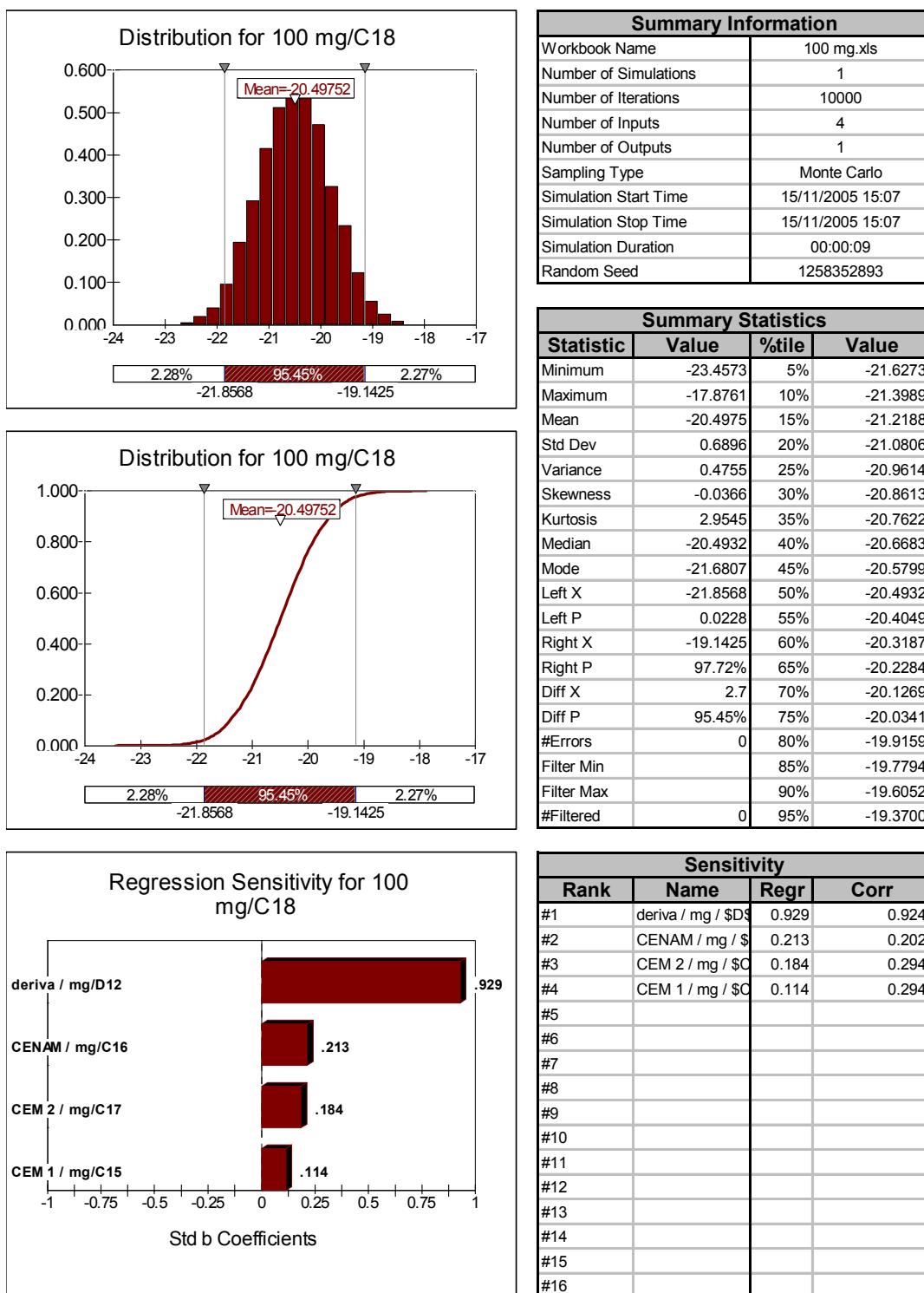
Simulation Results for 20 g / C18



Simulation Results for 5 g / C18



Simulation Results for 100 mg / C18



Appendix 2: Degrees of equivalence between the NMIs participant

Tables 27, 28, 29, 30, and 31 show the degrees of equivalence between the different NMI participants:

Degrees of equivalence for the nominal value of 1 kg

1 kg	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
CEM1	0,00	0,23	0,62	4,64	0,05	0,06	0,34	0,17
CENAM	0,23	0,00	0,79	4,80	0,05	0,13	0,52	0,53
SIC	0,62	0,79	0,00	4,38	0,51	0,29	0,27	0,54
SENCAMER	4,64	4,80	4,38	0,00	3,85	3,16	4,39	4,78
INEN	0,05	0,05	0,51	3,85	0,00	0,08	0,29	0,14
INDECOP	0,06	0,13	0,29	3,16	0,08	0,00	0,12	0,00
IBMETRO	0,34	0,52	0,27	4,39	0,29	0,12	0,00	0,25
CEM2	0,17	0,53	0,54	4,78	0,14	0,00	0,25	0,00

Table27

Degrees of equivalence for the nominal value of 100 g

100 g	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
CEM1	0,00	0,56	0,86	2,36	0,16	0,00	0,19	0,02
CENAM	0,56	0,00	1,40	2,19	0,28	0,33	0,24	0,81
SIC	0,86	1,40	0,00	2,77	0,09	0,64	0,93	0,97
SENCAMER	2,36	2,19	2,77	0,00	1,01	1,90	2,05	2,56
INEN	0,16	0,28	0,09	1,01	0,00	0,16	0,21	0,16
INDECOP	0,00	0,33	0,64	1,90	0,16	0,00	0,14	0,02
IBMETRO	0,19	0,24	0,93	2,05	0,21	0,14	0,00	0,24
CEM2	0,02	0,81	0,97	2,56	0,16	0,02	0,24	0,00

Table 28

Degrees of equivalence for the nominal value of 20 g

20 g	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
CEM1	0,00	0,29	1,12	0,19	2,15	0,31	0,73	0,34
CENAM	0,29	0,00	1,06	0,08	2,12	0,20	0,64	0,14
SIC	1,12	1,06	0,00	0,53	1,33	0,50	0,29	0,87
SENCAMER	0,19	0,08	0,53	0,00	1,57	0,07	0,30	0,02
INEN	2,15	2,12	1,33	1,57	0,00	1,59	1,52	1,99
INDECOP	0,31	0,20	0,50	0,07	1,59	0,00	0,24	0,12
IBMETRO	0,73	0,64	0,29	0,30	1,52	0,24	0,00	0,50
CEM2	0,34	0,14	0,87	0,02	1,99	0,12	0,50	0,00

Table 29

Degrees of equivalence for the nominal value of 5 g

5 g	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
CEM1	0,00	0,00	1,67	0,50	0,10	0,08	0,66	0,02
CENAM	0,00	0,00	1,70	0,52	0,10	0,08	0,71	0,03
SIC	1,67	1,70	0,00	1,21	1,31	1,49	1,30	1,71
SENCAMER	0,50	0,52	1,21	0,00	0,24	0,42	0,00	0,53
INEN	0,10	0,10	1,31	0,24	0,00	0,14	0,27	0,11
INDECOP	0,08	0,08	1,49	0,42	0,14	0,00	0,48	0,07
IBMETRO	0,66	0,71	1,30	0,00	0,27	0,48	0,00	0,74
CEM2	0,02	0,03	1,71	0,53	0,11	0,07	0,74	0,00

Table 30

Degrees of equivalence for the nominal value of 100 mg

100 mg	CEM1	CENAM	SIC	SENCAMER	INEN	INDECOP	IBMETRO	CEM2
CEM1	0,00	0,90	0,90	0,69	2,06	0,90	2,46	0,85
CENAM	0,90	0,00	1,41	0,90	2,28	1,41	3,45	2,46
SIC	0,90	1,41	0,00	0,25	1,58	0,00	0,83	0,51
SENCAMER	0,69	0,90	0,25	0,00	1,06	0,25	0,16	0,50
INEN	2,06	2,28	1,58	1,06	0,00	1,58	1,25	1,90
INDECOP	0,90	1,41	0,00	0,25	1,58	0,00	0,83	0,51
IBMETRO	2,46	3,45	0,83	0,16	1,25	0,83	0,00	2,11
CEM2	0,85	2,46	0,51	0,50	1,90	0,51	2,11	0,00

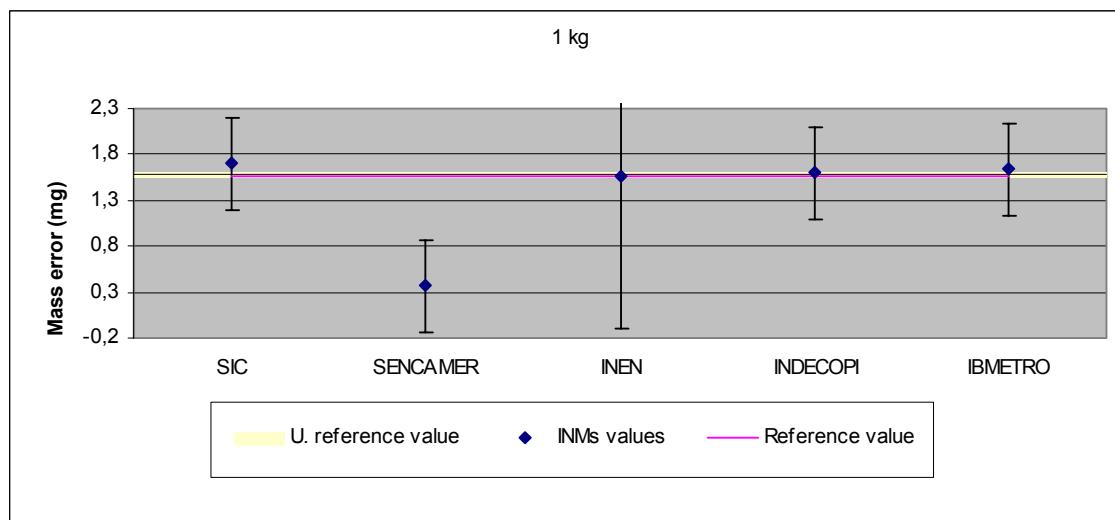
Table 31

Appendix 3: Graphical representation with the uncertainties from mpe

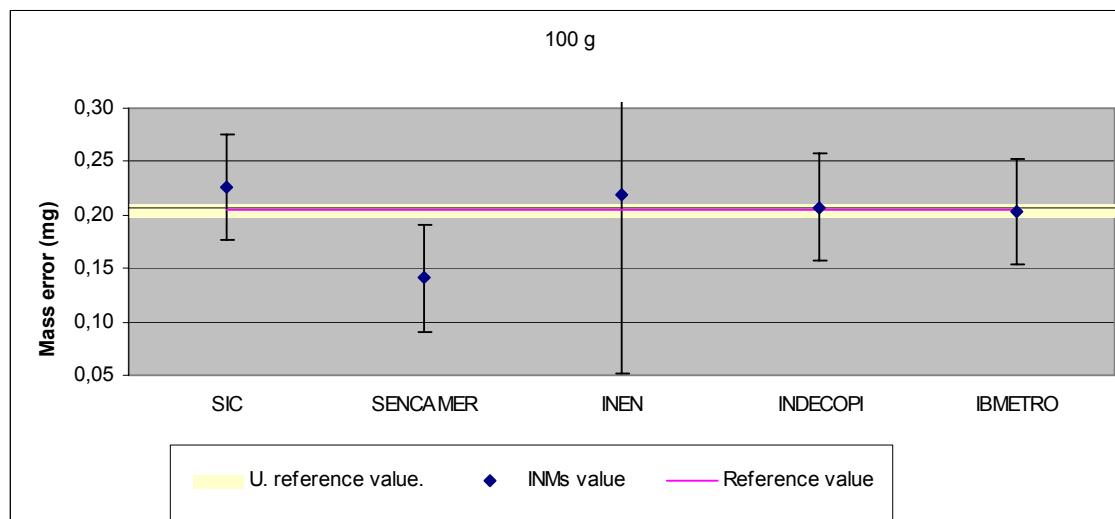
Graphs 6, 7, 8, 9 and 10 show the representation of the results of each NMI with their error bars corresponding to mpe/3 for weights of accuracy class E2 or F1 in the case of INEN, and its position in comparison to the value of reference.

In the case of SIC, IBMETRO, INDECOPPI and SENCAMER it corresponds to weights of OIML accuracy class E2 and in the case of INEN it corresponds to weights of OIML accuracy class F1.

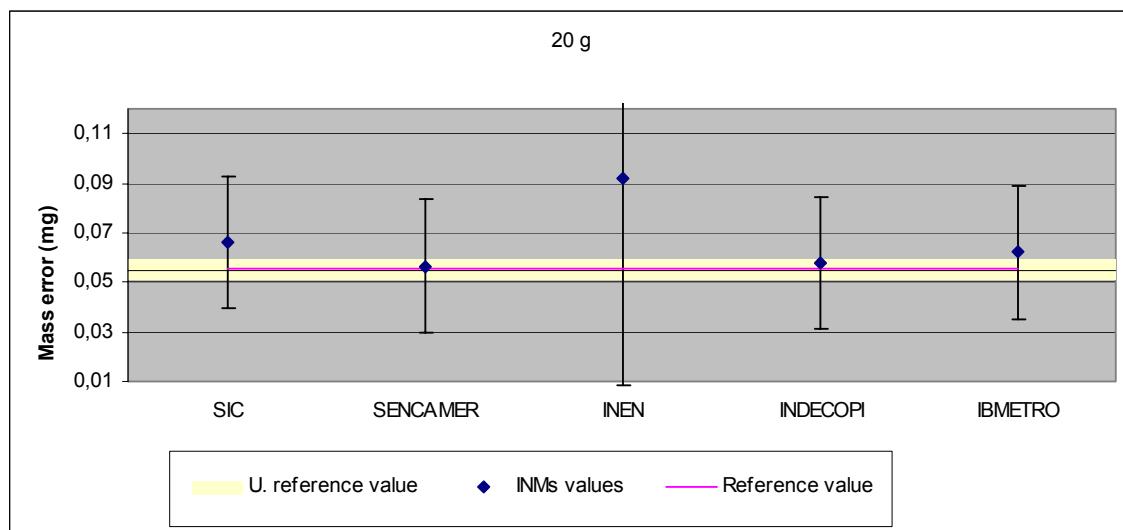
If the uncertainty given by the laboratory is greater than mpe/3, the value given by the laboratory has been kept, and the corresponding result is marked in red. It can be observed on the graphs that the results given by some NMIs continue to be outside the band of the reference value that would indicate that corrective actions have to be taken by the laboratory.



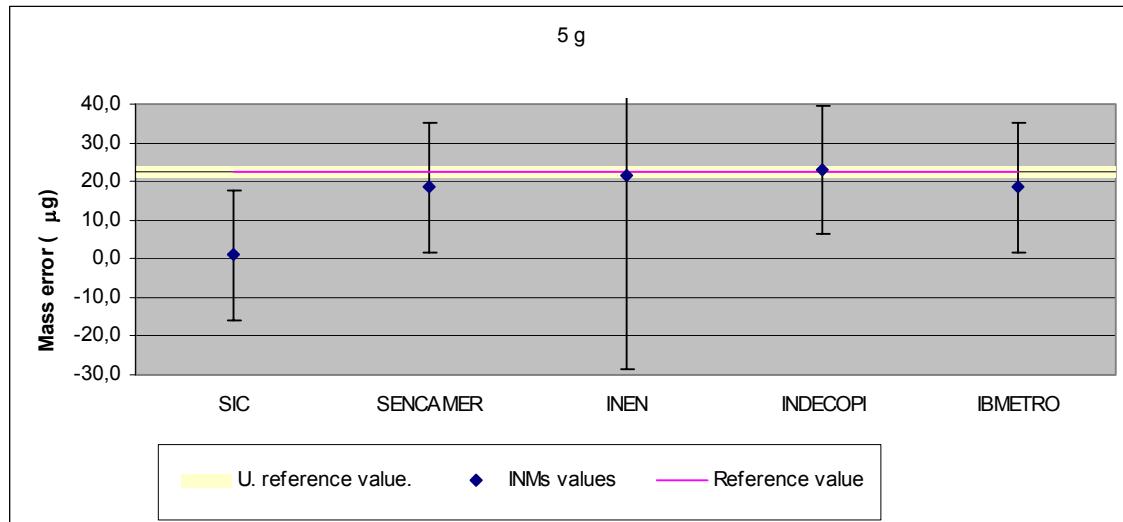
Graph 6



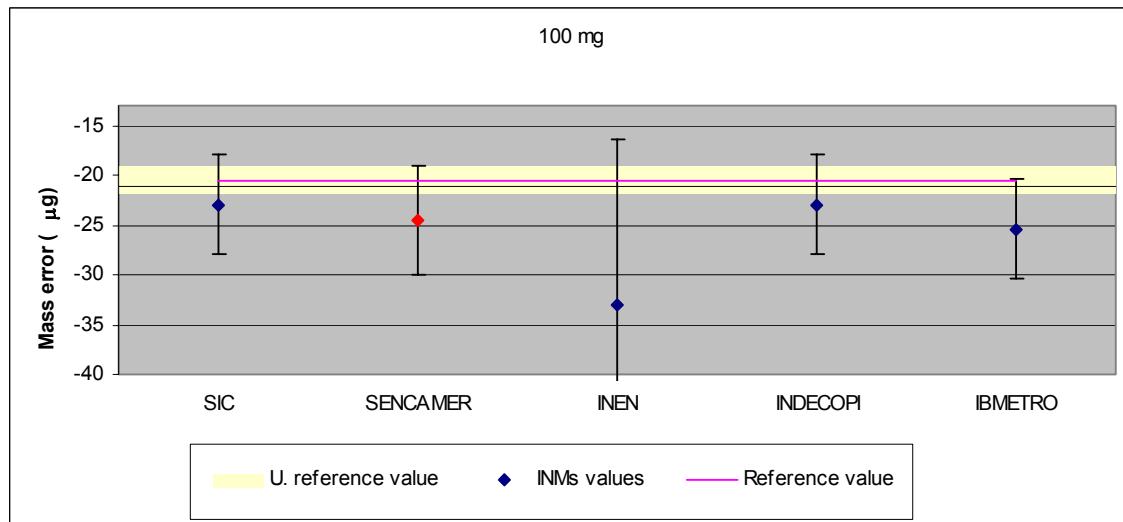
Graph 7



Graph 8



Graph 9



Graph 10

The results presented must guarantee the capacity of the participating NMIs for the calibration of weights of OIML accuracy class E2 in the case of SIC, IBMETRO, INDECOP and SENCAMER, and weights of OIML accuracy class F1 in the case of INEN.