INTERLABORATORY MASS COMPARISON BETWEEN LABORATORIES BELONGING TO SIM –SUB-REGIONS COORDINATED BY CENAM (SIM.7.31a & SIM.7.31b)

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Abstract: A round robin comparison in mass measurements between SIM member countries was carried out during the period April to November 2005. CENAM acted as pilot laboratory.

Six travelling standards with the following nominal values: 2 kg, 1 kg, 200 g, 50 g, 1 g and 200 mg were circulated. These travelling standards complied with the accuracy recommended for class E_2 [1,2]. The results obtained are represented in this report.

1. INTRODUCTION

A meeting of the technical contacts of SIM MWG 7 was held in Rio de Janeiro, Brazil in December 2004. At this meeting, subsequent to a proposal by the BSJ, planning was commenced for a mass comparison between SIM member countries in which at least one country from each sub region should participate. CENAM accepted the role as pilot laboratory for the mass compassion, as it had taken part in the key comparisons of the CCM of the CIPM.

The results of a comparison of six travelling standards among laboratories in the SIM region are presented in this report.

This program was coordinated by CENAM (Centro Nacional de Metrología), México. The travelling standards used are: 2 kg, 1 kg, 200 g, 50 g, 1 g and 200 mg, all of them are made of non-magnetic stainless steel.

The measurements in this comparison were carried out from April 2005 to December 2005. The CENAM contributed the travelling standards and supplied their reference values.

The density, the magnetic susceptibility, permanent magnetization, and conventional mass of all travelling standards except the density of the 200 mg were determined. A visual comparison of the surface roughness against roughness

standards proved that the travelling standards complied with the accuracy class of OIML E_2 [1,2].

The comparison protocol as well as the volume data were included in the travel container in which the standards were transported.

The SIM identification for this comparison is: **SIM.7.31a** (1 kg) and **SIM.7.31b** (2 kg, 200 g, 50 g, 1 g y 200 mg).

2. AIM OF THE PROGRAM

The aim of this comparison is to give confidence of the technical capacity of the SIM members and work in the mutual recognition agreements within the SIM and at the international level. On the other hand this comparison gives objective evidence about the technical competence of the laboratories, and it assists in identifying opportunities to improve the metrological assurance systems.

One of the problems in organizing comparisons, in which different countries are involved, is that each of them has different necessities and different capabilities; this can be seen in the wide range of uncertainty reported by the participants.

3. PARTICIPANTS

Table 1 shows the seven participating laboratories of the SIM sub-regions.

| Laboratory | Acronym | Country | SIM Sub region |
|--|-------------------------------|------------|-------------------|
| Centro Nacional de Metrología | CENAM/ Pilot laboratory | México | NORAMET |
| Bureau of Standards, Jamaica | BSJ | Jamaica | CARIMET |
| Laboratorio Costarricense de Metrología | LACOMET | Costa Rica | CAMET |
| Instituto Boliviano de Metrología | IBMETRO | Bolivia | ANDIMET |
| Centro de Estudios, Medición y Certificación de Calidad | CESMEC | Chile | SURAMET |
| Instituto Nacional de Defensa de la Competencia y de la Protección de la Propiedad Intelectual. | INDECOPI | Peru | ANDIMET |
| Instituto Nacional de Tecnología, Normalización y Metrología | INTN | Paraguay | SURAMET |

Table 1: Participating Laboratories

4. DESIGN OF THE PROGRAM AND TIME SCHEDULE

The program was designed according to the guidelines for CIPM (Comité International des Poids et Measures) key comparisons [3] and were used six travelling standards of 2 kg, 1 kg, 200 g, 50 g, 1 g and 200 mg) were used. These standards comply with the requirements of class E₂ of the International Recommendation OIML R111 [1,2]. The travelling standards were circulated in only one petal of SIM sub-regions. As pilot laboratory, CENAM determined the conventional mass of the travelling standards at the beginning and the end of the comparison.

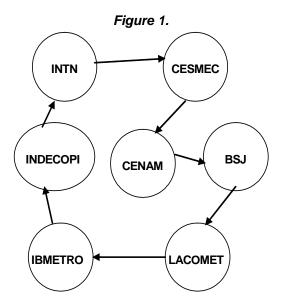
The transportation of the travelling standards to the next participant was done by hand in order to avoid any contamination or damage.

Table 2 shows the measurements scheduled and the starting dates of each laboratory.

| Table 2:- Sequence of | f the measurements |
|-----------------------|--------------------|
|-----------------------|--------------------|

| INSTITUTE | PERIOD OF MEASUREMENTS | | |
|-----------|----------------------------|--|--|
| CENAM | April-May 2005 | | |
| BSJ | May 2005 | | |
| LACOMET | June 2005 | | |
| IBMETRO | August 2005 | | |
| INDECOPI | September 2005 | | |
| INTN | October 2005 | | |
| CESMEC | November 2005 | | |
| CENAM | December 2005-January 2006 | | |

Figure 1 shows the transportation sequence and measurements of the travelling standards.



5. REPORTING BY PARTICIPANT

The measurement results were sent to the pilot laboratory in a final report where a list of the equipment used as balances and environmental conditions were included, besides the reference standard used in order to see the traceability on each laboratory.

6. STABILITY OF THE TRAVELLING STANDARDS

The pilot laboratory (CENAM) monitored the stability of the travelling standards during a period of 2 months before beginning of the laboratory measurements. No significant instability has been found, so that, the conventional mass values of the traveling standards were stable during this period.

The travelling standards were circulated among six participating laboratories without any incident that

required any return to the pilot laboratory to re measure the travelling standards.

7. REFERENCE VALUES

- **7.1** The reference values for this comparison were determined for the CENAM with an expanded uncertainty calculated as is described in 7.6.1, 7.6.2 and 7.3.
- **7.2** Before circulating the travelling standards of 2 kg, 1 kg, 200 g, 50 g and 1 g their volumes were determined at the CENAM density laboratory. The assumed density of the 200 mg weight was 7 950 kg m⁻³ as given by the manufacturer.
- **7.3** The expanded uncertainties of the reference values were obtained, according to GUM-1995 [4], as the combined standard uncertainties multiplied by the coverage factor k = 2. The expanded uncertainty corresponds to a coverage probability of approximately 95%. The uncertainty was formed from the uncertainty of measurement of the reference standard used, the weighing process and the air buoyancy correction. The uncertainty component due to long-term changes was negligible.
- **7.4** Table 3 gives the mass changes $|\Delta m|$ between the two re-calibrations by the pilot laboratory and the resulting drift uncertainties calculated by means of equation c):

| standards | | | |
|-----------|----------------|--------------|----------------|
| Nominal | Reference | $ \Delta m $ | Drift |
| Value | uncertainty | | uncertainty |
| | (<i>k</i> =2) | | (<i>k</i> =2) |
| 2 kg | 0,08 mg | 0,05 mg | 0,03 mg |
| 1 kg | 0,03 mg | 0,00 mg | 0,00 mg |
| 200 g | 0,012 mg | 0,020 mg | 0,012 mg |
| 50 g | 0,006 mg | 0,002 mg | 0,001 mg |
| 1 g | 0,002 mg | 0,000 mg | 0,000 mg |
| 200 mg | 0,001 2 mg | 0,000 4 mg | 0,000 2 mg |

| Table 3. Changes in mass of the travelling | |
|--|--|
| standards | |

7.5 The instability of the travelling standards was taken into account in the calculation of the reference values and was included in the E_n [5] value as an additional uncertainty component.

- **7.6** Therefore, the conventional mass values of the participants have to be linked to a CENAM's reference standards using the rules:
- **7.6.1** If two consecutive determinations of reference values are within the limits of the reference uncertainty, their mean value is used by all participants because we do not know when the value changed.
- **7.6.2** If two reference values m_1 and m_2 , were at times t_1 and t_2 , differ significantly, a linear drift is assumed and for a participant *i*, measuring at time t_i the mass $m_{PL,i}$ was interpolated using the following equation.

$$m_{_{PL,i}} = m_{_{PL,1}} + \left(m_{_{PL,2}} - m_{_{PL,1}}\right) \frac{t_i - t_1}{t_2 - t_1} \quad \text{ a)}$$

8. RESULTS OF PARTICIPATING LABORATORIES

- **8.1** The results were sent directly to the pilot laboratory (CENAM).
- **8.2** The results of the measurements are shown in the tabular form, see table 4 to table 9 and as a graphical representation, see 1 to 6. The E_n value [4] is obtained from the following expression.

$$E_n = \frac{m_A - m_{PL}}{\sqrt{U_A^2 + U_{PL}^2 + U_d^2}}$$
 b)

 $m_{\rm PL}$ and $U_{\rm PL}$ are the conventional mass value and uncertainty associated with the pilot laboratory are while $m_{\rm A}$ and $U_{\rm A}$ are the conventional mass value and the uncertainty associated with the participating laboratories.

A drift uncertainty U_d for the mass instability of the travelling standards is taken into account with the following equation:

$$U_d = k_v \sqrt{\left(\frac{m_{PL,2} - m_{PL,1}}{2\sqrt{3}}\right)^2}$$
 c)

8.3 In the tables from 10 to 15 are included the E_n values for all the travelling standards and for all the participants, including the pilot laboratory. The following rules were considered.

8.4 Participant A and pilot laboratory PL.

The value E_n is calculated according to equation b):

the measurements are considered as uncorrelated, in this case the denominator of (b) the equation is:

$$\sqrt{\left(U_{A}^{2}+U_{PL}^{2}+\frac{\left(m_{PL,2}-m_{PL,1}\right)^{2}}{3}\right)}$$
 d)

8.5 Participant A and B from the same petal. The value E_n is calculated according to equation b):

the measurements are considered as uncorrelated in this case the denominator from the equation b) is:

$$\sqrt{\left(U_{A}^{2}+U_{B}^{2}+U_{PL}^{2}+\frac{\left(m_{PL,2}-m_{PL,1}\right)^{2}}{3}\right)} \quad e)$$

Table 4:- Results for the 2 kg standard

| Laboratory | Reference | Laboratory | Laboratory |
|------------|------------------|--|----------------------------|
| | value | value | uncertainty |
| | m_{PL} - m_n | <i>m_l-m_n (m</i> g) | <i>U</i> ¹ (mg) |
| | <i>(m</i> g) | | |
| CENAM | | - 0,20 | 0,08 |
| BSJ | - 0,225 | - 0,18 | 2,38 |
| LACOMET | - 0,225 | - 0,24 | 1,46 |
| IBMETRO | - 0,225 | - 0,90 | 3,00 |
| INDECOPI | - 0,225 | + 0,50 | 1,00 |
| INTN | - 0,225 | + 0,20 | 3,00 |
| CESMEC | - 0,225 | - 0,20 | 1,00 |
| CENAM | | - 0,25 | 0,08 |

| Laboratory | Reference | Laboratory | Laboratory |
|------------|---------------------------------|--|----------------------------|
| | value | value | uncertainty |
| | m _{PL} -m _n | <i>m_l-m_n (m</i> g) | <i>U</i> ¹ (mg) |
| | <i>(m</i> g) | | |
| CENAM | | - 0,16 | 0,03 |
| BSJ | - 0,16 | - 0,09 | 1,28 |
| LACOMET | - 0,16 | - 0,214 | 0,046 |
| IBMETRO | - 0,16 | - 0,128 | 0,154 |
| INDECOPI | - 0,16 | - 0,20 | 0,26 |
| INTN | - 0,16 | - 0,1 | 1,6 |
| CESMEC | - 0,16 | - 0,14 | 0,50 |
| CENAM | | - 0,16 | 0,03 |

Table 6:- Results for the 200 g standard

| Laboratory | Reference value | Laboratory value | Laboratory uncertainty |
|------------|---------------------------------|------------------|---------------------------|
| | m _{PL} -m _n | $m_l - m_n (mg)$ | U_l (mg) |
| | <i>(m</i> g) | | |
| CENAM | | - 0,365 | 0,012 |
| BSJ | - 0,363 | - 0,37 | 0,24 |
| LACOMET | - 0,361 | - 0,367 9 | 0,009 6 |
| IBMETRO | - 0,356 | - 0,381 | 0,034 |
| INDECOPI | - 0,354 | - 0,36 | 0,05 |
| INTN | - 0,352 | - 0,30 | 0,30 |
| CESMEC | - 0,349 | - 0,32 | 0.10 |
| CENAM | | - 0,345 | 0.012 |

Tablet 7:- Results for the 50 g standard

| Laboratory | Reference | Laboratory | Laboratory |
|------------|---------------------------------|--|----------------------------|
| | value | value | uncertainty |
| | m _{PL} -m _n | <i>m_l-m_n (m</i> g) | <i>U</i> ¹ (mg) |
| | <i>(m</i> g) | | |
| CENAM | | - 0,061 | 0,006 |
| BSJ | - 0,060 | - 0,08 | 0,10 |
| LACOMET | - 0,060 | - 0,063 8 | 0,014 2 |
| IBMETRO | - 0,060 | - 0,052 | 0,016 |
| INDECOPI | - 0,060 | - 0,062 | 0,016 |
| INTN | - 0,060 | - 0,08 | 0,10 |
| CESMEC | - 0,060 | - 0,065 | 0,030 |
| CENAM | | - 0,059 | 0,006 |

Table 8:- Results for the 1 g standard

| Laboratory | Reference | Laboratory | Laboratory |
|------------|---------------------------------|--|----------------------------|
| _ | value | value | uncertainty |
| | m _{PL} -m _n | <i>m_l-m_n (m</i> g) | <i>U</i> ¹ (mg) |
| | <i>(m</i> g) | | |
| CENAM | | + 0,028 | 0,002 |
| BSJ | + 0,028 | + 0,030 | 0,040 |
| LACOME | + 0,028 | + 0,025 9 | 0,001 5 |
| Т | | | |
| IBMETRO | + 0,028 | + 0,026 4 | 0,003 2 |
| INDECOP | + 0,028 | + 0,027 | 0,005 |
| I | | | |
| INTN | + 0,028 | + 0,027 | 0,030 |
| CESMEC | + 0,028 | + 0,027 | 0,010 |
| CENAM | | + 0,028 | 0,002 |

| Laboratory | Reference | Laboratory | Laboratory |
|------------|---------------------------------|--|----------------------------|
| | value | value | uncertainty |
| | m _{PL} -m _n | <i>m_l-m_n (m</i> g) | <i>U</i> ¹ (mg) |
| | <i>(m</i> g) | | |
| CENAM | | + 0,000 7 | 0,001 2 |
| BSJ | + 0,000 55 | - 0,002 | 0,020 |
| LACOMET | + 0,000 55 | + 0,000 08 | 0,000 54 |
| IBMETRO | + 0,000 55 | - 0,004 4 | 0,002 2 |
| INDECOPI | + 0,000 55 | + 0,000 9 | 0,003 2 |
| INTN | + 0,000 55 | + 0,003 | 0,020 |
| CESMEC | + 0,000 55 | + 0,001 | 0,006 |
| CENAM | | + 0,000 4 | 0,001 2 |

Table 9:- Results of the standard of 200 mg

Where:

 $m_{\rm n}$ is the nominal value of the travelling standards $m_{\rm l}$ is the laboratory value of the travelling standards

 m_{PL} is the reference value of the travelling standards

9. CONCLUSIONS

Of the 36 measurements results for the mass of the travelling standards, were used for calculating the respective E_n value between participants, see tables 10 to 15 of which one are greater than one respect to pilot laboratory and two are greater than one between them, these values were calculated using the d) and e) formulas. The degree of agreement among the participants seen to be excellent, in other words, the mass measurements carried out among SIM region members do not differ significantly.

The uncertainties of each participant are plotted in graphical representation, see graphics 1 to 6 using the d) formula.

The names of the participating laboratories were included in the report as an agreement between them.

ACKNOWLEDGMENT

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| Leticia Luján Solís | CENAM |
|---------------------|--------|
| Raúl Hernández | CESMEC |

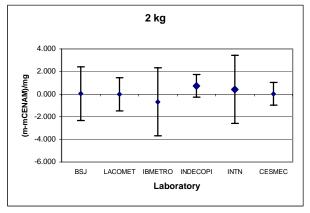
| Sheldon Walker | BSJ |
|-----------------------------|---------|
| Oscar Andrey Herrera Sancho | LACOMET |

REFERENCES

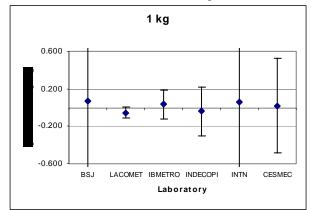
- [3] Guidelines for CIPM key comparisons http://www.bipm.fr/utils/en/pdf/guidelines.pdf
- [4] Guide to the Expression of Uncertainty in Measurement, International Organization for Standardization, Geneva, Switzerland, 1995.
- [5] W. Wöger, "Remarks on the *E*_n-criterion Used in Measurement Comparison, Internationale Zusammenarbeit.PTB-Metteilungen 1999. Pages 24-27.

APPENDIX A – GRAPHICS

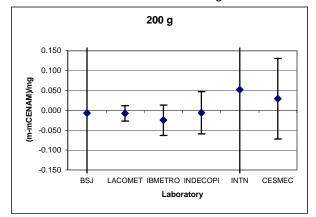
Graphic 1: The difference between the laboratory value and the reference value: 2 kg



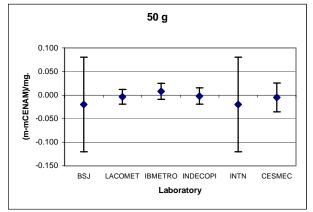
Graphic 2: The difference between the laboratory value and the reference value: 1 kg



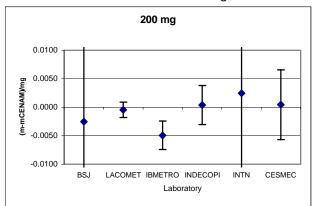
Graphic 3: The difference between the laboratory value and the reference value: 200 g



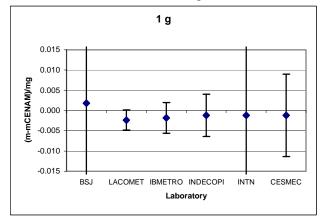
Graphic 4: The difference between the laboratory value and the reference value: 50 g



Graphic 6: The difference between the laboratory value and the reference value: 200 mg



Graphic 5: The difference between the laboratory value and the reference value: 1g



APPENDIX B – En VALUES

| 2 kg | CENAM | BSJ | LACOMET | IBMETRO | INDECOPI | INTN | CESMEC |
|----------|-------|-------|---------|---------|----------|-------|--------|
| CENAM | | 0.02 | -0.01 | -0.23 | 0.72 | 0.14 | 0.02 |
| BSJ | -0.02 | | -0.02 | -0.19 | 0.26 | 0.10 | -0.01 |
| LACOMET | 0.01 | 0.02 | | -0.20 | 0.42 | 0.13 | 0.02 |
| IBMETRO | 0.23 | 0.19 | 0.20 | | 0.44 | 0.26 | 0.22 |
| INDECOPI | -0.72 | -0.26 | -0.42 | -0.44 | | -0.09 | -0.49 |
| INTN | -0.14 | -0.10 | -0.13 | -0.26 | 0.09 | | -0.13 |
| CESMEC | -0.02 | 0.01 | -0.02 | -0.22 | 0.49 | 0.13 | |

Table 11: The En value for 1 kg

| 1 kg | CENAM | BSJ | LACOMET | IBMETRO | INDECOPI | INTN | CESMEC |
|----------|-------|------|---------|---------|----------|------|--------|
| CENAM | | 0.05 | -0.98 | 0.20 | -0.15 | 0.04 | 0.04 |
| BSJ | -0.05 | | -0.10 | -0.03 | -0.08 | 0.00 | -0.04 |
| LACOMET | 0.98 | 0.10 | | 0.53 | 0.05 | 0.07 | 0.15 |
| IBMETRO | -0.20 | 0.03 | -0.53 | | -0.24 | 0.02 | -0.02 |
| INDECOPI | 0.15 | 0.08 | -0.05 | 0.24 | | 0.06 | 0.11 |
| INTN | -0.04 | 0.00 | -0.07 | -0.02 | -0.06 | | -0.02 |
| CESMEC | -0.04 | 0.04 | -0.15 | 0.02 | -0.11 | 0.02 | |

Table 12: The E_n value for 200 g

| 200 g | CENAM | BSJ | LACOMET | IBMETRO | INDECOPI | INTN | CESMEC |
|----------|-------|-------|---------|---------|----------|------|--------|
| CENAM | | -0.03 | -0.38 | -0.66 | -0.12 | 0.17 | 0.29 |
| BSJ | 0.03 | | 0.01 | -0.05 | 0.04 | 0.18 | 0.19 |
| LACOMET | 0.38 | -0.01 | | -0.33 | 0.15 | 0.23 | 0.47 |
| IBMETRO | 0.66 | 0.05 | 0.33 | | 0.33 | 0.27 | 0.57 |
| INDECOPI | 0.12 | -0.04 | -0.15 | -0.34 | | 0.20 | 0.35 |
| INTN | -0.17 | -0.18 | -0.23 | -0.27 | -0.20 | | -0.06 |
| CESMEC | -0.29 | -0.19 | -0.47 | -0.57 | -0.35 | 0.06 | |

Table 13: The E_n value for 50 g

| 50 g | CENAM | BSJ | LACOMET | IBMETRO | INDECOPI | INTN | CESMEC |
|----------|-------|-------|---------|---------|----------|-------|--------|
| CENAM | | -0.20 | -0.25 | 0.47 | -0.12 | -0.20 | -0.16 |
| BSJ | 0.20 | | 0.16 | 0.28 | 0.18 | 0.00 | 0.14 |
| LACOMET | 0.25 | -0.16 | | 0.53 | 0.08 | -0.16 | -0.04 |
| IBMETRO | -0.47 | -0.28 | -0.53 | | -0.43 | -0.28 | -0.38 |
| INDECOPI | 0.12 | -0.18 | -0.08 | 0.43 | | -0.18 | -0.09 |
| INTN | 0.20 | 0.00 | 0.16 | 0.28 | 0.18 | | 0.14 |
| CESMEC | 0.16 | -0.14 | 0.04 | 0.38 | 0.09 | -0.14 | |

| 1 g | CENAM | BSJ | LACOMET | IBMETRO | INDECOPI | INTN | CESMEC |
|----------|-------|------|---------|---------|----------|-------|--------|
| CENAM | | 0.04 | -0.94 | -0.48 | -0.23 | -0.01 | -0.12 |
| BSJ | -0.04 | | -0.10 | -0.09 | -0.07 | -0.03 | -0.07 |
| LACOMET | 0.94 | 0.10 | | 0.13 | 0.21 | 0.01 | 0.11 |
| IBMETRO | 0.48 | 0.09 | -0.13 | | 0.10 | 0.01 | 0.06 |
| INDECOPI | 0.23 | 0.07 | -0.21 | -0.10 | | 0.00 | 0.00 |
| INTN | 0.01 | 0.03 | -0.01 | -0.01 | 0.00 | | 0.00 |
| CESMEC | 0.12 | 0.07 | -0.11 | -0.06 | 0.00 | 0.00 | |

Table 14: The *E*_n value for 1 g

Table 15: The *E*_n value for 200 mg

| 200 mg | CENAM | BSJ | LACOMET | IBMETRO | INDECOPI | INTN | CESMEC |
|----------|-------|-------|---------|---------|----------|------|--------|
| CENAM | | -0.13 | -0.34 | -1.96 | 0.11 | 0.12 | 0.08 |
| BSJ | 0.13 | | 0.10 | -0.12 | 0.14 | 0.18 | 0.14 |
| LACOMET | 0.34 | -0.10 | | -1.75 | 0.24 | 0.15 | 0.15 |
| IBMETRO | 1.96 | 0.12 | 1.75 | | 1.30 | 0.37 | 0.83 |
| INDECOPI | -0.11 | -0.14 | -0.24 | -1.30 | | 0.10 | 0.01 |
| INTN | -0.12 | -0.18 | -0.15 | -0.37 | -0.10 | | -0.10 |
| CESMEC | -0.08 | -0.14 | -0.15 | -0.83 | -0.01 | 0.10 | |