PROTOCOL FOR THE CCM KEY COMPARISON OF MASS STANDARDS CCM.M-K6 piloted by CENAM-MEXICO

1. OUTLINE

This inter-laboratory comparison is based on the decision of the "*Comité Consultatif pour la Masse et les Grandeurs Apparentées*" (CCM) during the 11th meeting held in April 2008 at the BIPM.

For this key comparison the CENAM-Mexico agreed to act as pilot laboratory, and later the NPL-United Kingdom accepted to be the support laboratory.

The aims of this key comparison are to compare the results obtained by participating laboratories in calibration of 50 kg stainless steel weight and to repeat the exercise realized in 2001 – 2002 with the key comparison CCM.M-K3.

In July 2009, two stainless weights of 50 kg nominal value were donated by Sartorius AG to be used them as travelling standards in this key comparison.

2. PURPOSE OF THIS DOCUMENT

The purposes of this document are:

- to define the organization of the comparison
- to provide instructions for the participants for the transport and handling of the transfer standards and
- to explain the way for reporting results.

3. PARTICIPANTS

Ten National Metrology Institutes will take part to this key comparison. Among the participants, four are SIM members, four are EURAMET members and two are APMP members.

The participating laboratories are listed in table 1.

Table 1. Participant laboratories of the comparison

National Institute of Metrology	Acronym	Technical Contact
Instituto Nacional de Metrologia, Normalização e Qualidade Industrial Av. Nossa Senhora das Graças, 50 – Duque de Caxias – RJ Prédio 3 – Divisão de Metrologia Mecânica- Dimec BRAZIL – 25250-020	**INMETRO**	Víctor M. Loayza vmloayza@inmetro.gov.br; lamas@inmetro.gov.br Tel: + 55 21 2679 9023 Fax: + 55 21 2679 1505

Centro Nacional de Metrología km. 4,5 Carretera a los Cués, Mpio. El Marqués, C.P. 76241 Querétaro, MEXICO	**CENAM**	Luis Omar Becerra lbecerra@cenam.mx Tel: + 52 442 211 05 00 ext. 3602, + 52 442 211 05 73 Fax: + 52 442 211 05 68
National Physical Laboratory Hampton Road, Teddington, Middlesex, TW11 0LW, UNITED KINGDOM	**NPL**	Stuart Davidson stuart.davidson@npl.co.uk Tel: + 44 208 943 6224 Fax: + 44 208 614 0535
National Metrology Institute of Japan Mechanical Metrology Division National Metrology Institute of Japan AIST Tsukuba Central -3, Umezono, Tsukuba, Ibaraki 305-8563, JAPAN	**NMIJ**	Kazunaga Ueda Jianxin Sun kazunaga-ueda@aist.go.jp; j.sun@aist.go.jp Tel: + 81 29 861 4153 Fax: + 81 29 861 4399
Korea Research Institute of Standards and Science 1 Doryong-Dong Yseong-Gu, Daejeon, KOREA	**KRISS**	Jin Wan Chung jwchung@kriss.re.kr Tel: + 82 42 868 5111 Fax: + 82 42 868 5012
Istituto Nazionale di Ricerca Metrológica Mechanical Division Strada delle Cacce 73, 10135 Torino, ITALY	**INRIM**	Andrea Malengo a.malengo@inrim.it Tel: + 39 011 3919 944 Fax: 39 011 3919 937
Physikalisch-Technische Bundesanstalt Bundesallee 100, 38116 Braunschweig, GERMANY	**PTB**	Michael Borys michael.borys@ptb.de Tel: + 49 531 592 1110 Fax: + 49 531 592 691110
National Research Council Canada, Institute for National Measurement Standards Montréal Road, M-36 Ottawa, Ontario CANADA K1A 0R6	**NRC**	Claude Jacques claude.jacques@nrc-cnrc.gc.ca Tel: +1 613 993 9330 Fax: +1 613 952 1394
Centro Español de Metrología C/Alfar, 2, 28760, Tres Cantos, Madrid SPAIN	**CEM**	Nieves Medina A. Lumbreras mmedina@cem.mityc.es alumbreras@cem.mityc.es Tel: + 349 1807 4789 Fax: + 349 1807 4807
National Institute of Standards and Technology 100 Bureau Dr, MS 8221 Gaithersburg, MD 20899 UNITED STATES OF AMERICA	**NIST**	Zeina J. Jabbour zeina.jabbour@nist.gov Tel: 301 975 6624 Fax: 301 417 0514

4. TRAVELLING STANDARDS

The travelling standards for this comparison are two 50 kg weights, each made in one piece of stainless steel, cylindrical shaped (Fig. 1).

Fig.1. Travelling standard

4.1 Characterization of the travelling standards

Values of volume, density and magnetic properties of the weights were measured at CENAM before the circulation of the weights. The data of the travelling standards are listed in table 2.

Table 2. Data of the travelling standards

Identification	K6-01	K6-02
Nominal Value	50 kg	50 kg
Density at 20 ºC *	7 949.75 kg/m^3	7 964.49 kg/m^3
Standard uncertainty	0.28 kg/m^3	0.28 kg/m^3
Volume at 20 ºC *	6 289.50 cm^3	6 277.87 cm^3
Standard uncertainty	0.22 cm^3	0.22 cm^3
Magnetic susceptibility (χ) *	< 0.01	< 0.01
Magnetization *	< 1 µT	< 1 µT
Surface roughness Rz	< 0.5 µm	< 0.5 µm
Surface roughness Ra	< 0.1 µm	< 0.1 µm
Height	288 mm	288 mm
Diameter	185 mm	185 mm
Height of centre of gravity above base	124.7 mm	124.7 mm

* Values measured at Pilot laboratory.

At the beginning and at the end of the circulation, the masses of both weights will be measured at the pilot laboratory.

5. TRANSPORTATION OF THE TRAVELLING STANDARDS

For this comparison two weights will be circulated between participants. The circulation will be done in two petals according to Tables 3 and 4.

One 50 kg weight will be circulated within each petal. At the beginning, both travelling standards will be measured at CENAM-Mexico and NPL-UK. These measurements will be used to link the results of participating laboratories of both petals.

CENAM will measure the mass of the standards at the beginning and at the end of the circulation in order to evaluate their possible drift.

Table 3. Petal 1, timetable for the circulation of the standard K6-01

NMI	Date of Arrival	Date of departure	Sending of results
CENAM/Mexico		2011-04-01	
NPL/UK	2011-04-15	2011-05-06	2011-06-03
NRC/Canada	2011-05-20	2011-06-10	2011-07-08
NIST/United States	2011-06-24	2011-07-15	2011-08-12
INMETRO/Brazil	2011-07-29	2011-08-19	2011-09-16
KRISS/Korea	2011-09-02	2011-09-23	2011-10-21
NMIJ/Japan	2011-10-07	2011-10-28	2011-11-25
CENAM/Mexico	2011-11-11		

Table 4. Petal 2, timetable for the circulation of the standard K6-02

NMI	Date of Arrival	Date of departure	Sending of results
CENAM/Mexico		2011-04-01	
NPL/UK	2011-04-15	2011-05-06	2011-06-03
PTB/Germany	2011-05-20	2011-06-10	2011-07-08
INRIM/Italy	2011-06-24	2011-07-15	2011-08-12
CEM/Spain	2011-07-29	2011-09-23	2011-10-21
CENAM/Mexico	2011-10-07		

Tables 3 and 4 give the dates of arrival and departure of the travelling standards and the date for sending results.

The circulation of the weights will be done by a Courier Company. Each participant will take care of the transportation of the travelling standard to the next participating laboratory according to the timetable of tables 3 and 4. When the package enters or leaves the country, the appropriate forms should be filled in very carefully under the auspices of the two laboratories concerned.

6. TIMETABLE OF THE COMPARISON

Table 5. Participant laboratories of the comparison

Activity	**Date**
Protocol agreed	February 2011
Measurements at the pilot laboratory	March 2011
Circulation and measurements by participants	April 2011
Return to pilot laboratory and control measurements	October – December 2011
Reception of the last measurements results reported by participants	November 2011
Analysis of results reported by participants	January – April 2012
Elaboration of Draft A	May – July 2012
Circulation of Draft A for comments/corrections	August 2012 – November 2013
Elaboration of Draft B	December 2012 – January 2013
Circulation of Draft B for comments/corrections	February – March 2013
Final report	April 2013

7. RECEIPT OF THE TRAVELLING STANDARD

7.1 Receipt the package

Upon receipt of the package, the laboratory should send by fax or e-mail to the pilot and dispatching laboratories a filled “Arrival of the travelling standard” and “List of content at arrival” forms given in appendix A.

7.2 Opening the package

At the arrival of the standard, the weights should be unpacked according to the following steps

1. Move the package into the mass laboratory
2. Open the two padlocks and the six throw latches of the plastic box,
3. Remove the inner container (aluminum mass standard case) from the outer container. In order to remove the mass standard container, lift it using lifting means.
4. Gently put the mass standard container in a carriage avoiding any shock.
5. Take the mass standard handling fork and the set of tools out of the outer container
6. Open the manual valve by unscrewing its cover.
7. Remove the eight socket head cap screws with the fitted spanner of the set of tools.
8. Lift very carefully the cover of the case using lifting means.
9. Lift very carefully the mass standard from the case using the handling fork.
10. Fill the “Travelling standard visual inspection form” given in appendix C of this protocol.
11. Close again the mass standard case with its cover and its screws.

Note: Additional information concerning the package, (photos, dimensions and total weight), as well as the combination of the padlocks will be sent soon.

7.3 Visual inspection

The travelling standard shall be examined on receipt, and any scratches or other marks shall be recorded in the “travelling standard visual inspection form” given in appendix C. This form should be sent by fax or e-mail to the pilot laboratory within 24 hours after the inspection.

8. MEASUREMENTS

The standards should be manipulated with pincers or other appropriate tools. They should never be touched with bare hands. When not in the balance, the standards should be stored at such a place where they are protected from dust, aerosols and vapor, for example on a plate covered with a clean bell jar.

Measurements should be done after an appropriate acclimatization time and according to own weighing procedure and results should be reported on the forms annexed (Annex D and F).

Before mass determination, dust particles should be removed from the surface of the standard by a soft brush.

The participating laboratories shall determine the mass of the travelling standard. For the buoyancy correction, the air density should be determined using the CIPM 2007 formula.

9. REPORTING RESULTS

The forms of Annexes D, E and F (calibration results, Environmental data during calibration, characteristics of the balance and instruments used for the calibration, traceability and uncertainty budget) should be copied, filled in and returned to the pilot laboratory within one month after the measurements.

10. DISPATCH OF THE TRAVELLING STANDARD

After completion of the measurements the participant laboratory should pack the travelling standard and send it to the next laboratory according to timetable of table 3 and 4.

Within 24 hours after dispatch, the dispatching laboratory should fill and send to the pilot laboratory the “Departure of the travelling standard” and “List of content as departure” forms given in appendix A.

11. ANNEXES

- Annex A. List of content of package
  - A.1. Arrival of the travelling standard
  - A.2. List of content as arrival
  - A.3. Departure of the travelling standard
  - A.4. List of content as departure

- Annex B. Transportation form (arrival), contact in the laboratory
  - B1. Conditions of the weights at arrival

- Annex C. Travelling standard visual inspection form

- Annex D. Calibration results, environmental data during calibration
  - D.1. Calibration results
  - D.2. Environmental data during calibration
  - D.3. Characteristics of the balance used for the measurements and instruments for measurement the environmental conditions
  - D.4. Characteristics of the equipment for the measurement of the environmental conditions (for the air density evaluation)

- Annex E. Traceability

- Annex F. Uncertainty budget

ANNEX A. LIST OF CONTENT OF PACKAGE

Laboratory:		Date:	
Filled by:			

Fill the form and send it to the pilot laboratory and to the previous lab within 24 hours from package receipt.

A.1. Arrival of the travelling standard

Delivery by (Courier Company):	
Arrival date:	

A.2. List of content as arrival

	YES	NO
Outer container (plastic box)		
Was the outer container in good conditions?		
Was the container locked with the two padlocks?		
Mass standard handling fork		
Was the handling fork in the plastic bag?		
Mass standard aluminum case		
Was the mass standard aluminum case in good conditions?		
Mass standard		
Two spanners for the socket head cap screws (M8 x 1.25)		

ANNEX A. LIST OF CONTENT OF PACKAGE

Laboratory:		Date:	
Filled by:			

A.3. Departure of the travelling standard

Delivery by (Courier Company):	
Departure date:	

A.4. List of content as departure

	YES	NO
Outer container (plastic box)		
Was the outer container in good conditions?		
Was the container locked with the two padlocks?		
Mass standard handling fork		
Was the handling fork in the plastic bag?		
Mass standard aluminum case		
Was the mass standard aluminum case in good conditions?		
Mass standard		
Two spanners for the socket head cap screws (M8 x 1.25)		

ANNEX B. TRANSPORTATION FORM (ARRIVAL), CONTACT IN THE LABORATORY

Laboratory:		Date:	
Filled by:			

Fill the format and send it the pilot laboratory and to the previous lab within 24 hours from package receipt.

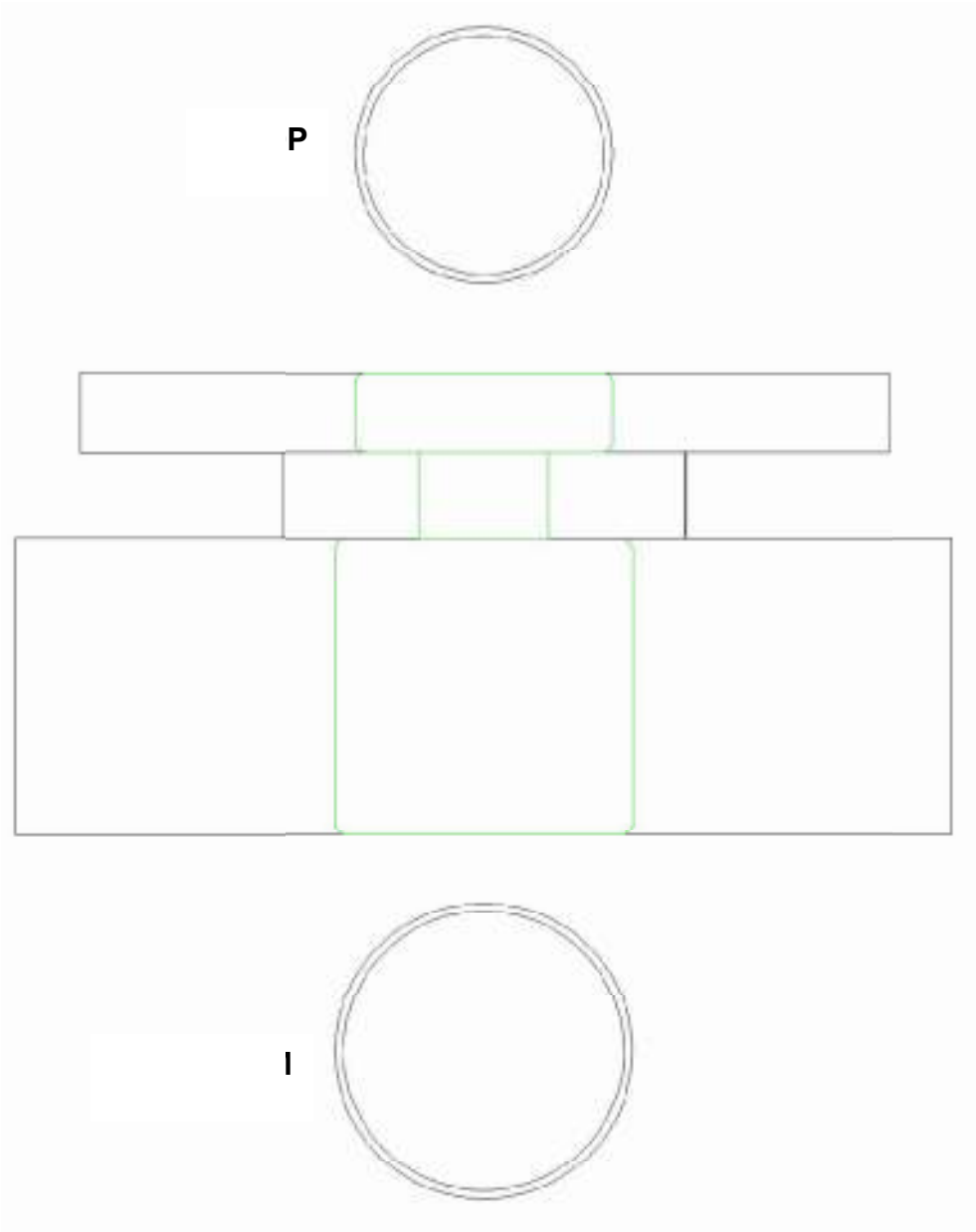
B1. Conditions of the weights at arrival

Package and mass standard case:	
Surface conditions:	
Observations:	

ANNEX C. TRAVELLING STANDARD VISUAL INSPECTION FORM

Laboratory:		Date:	
Filled by:			

Record on the diagrams any mark seen on the travelling standard (scratches, contamination, etc…), and send it the pilot laboratory.



ANNEX D. CALIBRATION RESULTS, ENVIRONMENTAL DATA DURING CALIBRATION

Laboratory:		Date:	
Filled by:			

D.1. Calibration Results

Nominal Value	Mass, m	Standard uncertainty, u_m	Number of measurements	Number of effective degrees of freedom, v_{eff}
50 kg	50 kg + mg	mg		

D.2. Environmental data during calibration

Insert the maximum, minimum and average values of the measured quantities.

Parameter	Minimum	Maximum	Average
Air density (kg/m^3)			
Temperature (ºC)			
Pressure (kPa)			
Dew point (ºC) or Relative Humidity (%)			
CO_2 (x10^{-6})			

D.3. Characteristics of the balance used for the measurements and Instruments for measurement the environmental conditions

Manufacturer	Type	Range	Resolution	Standard deviation*

* Please indicate the weighing procedure and number of measurements

D.4. Characteristics of the equipment for the measurement of the environmental conditions (for the air density evaluation)

	Manufacturer	Type	Range	Resolution	Standard uncertainty *u*
Temperature t /ºC					
Barometric pressure p /Pa					
Relative humidity h_r, %					
Mole fraction of CO_2 (if appropriate)					

ANNEX E. TRACEABILITY

Laboratory:		Date:	
Filled by:			

Insert the standards including additional weights used for the calibration and its traceability to the international prototype of the kilogram.

Identification	Mass m	Uncertainty u_m (1σ)	Volume V	Uncertainty u_V (1σ)	Date of Calibration

Add lines as necessary.

ANNEX F. UNCERTAINTY BUDGET

Laboratory:		Date:	
Filled by:			

The uncertainty should be evaluated and combined following the GUM (JCGM 100:2008, Evaluation of measurement data — Guide to the expression of uncertainty in measurement).

Parameter	Standard uncertainty of component *u* / mg
Mass (correction) of the standard	
Stability of mass standard	
Compensation weight	
Stability of compensation weight	
Weighing reproducibility	
Position effect (automatic loading comparator)	
Comparator resolution	
Air density	
Travelling standard density	
Mass standard density	
Other…	

Add lines as necessary.