Technical protocol July 2020

EURAMET Comparison of Stainless Steel Multiples and Sub-Multiples of the Kilogram
5 kg, 100 g, 10 g, 5 g, 500 mg

Comparison: EURAMET.M.M- K7
Technical protocol

Contents

1 Introduction

1.1 Aim

The scope of this key comparison is the calibration of stainless steel mass standards of multiples and sub-multiples of the kilogram by the participating institutes. The objectives of the comparison are to validate the calibration measurement capabilities (CMCs) of the participants and to provide a basis for the submission and review of updated or new CMCs.

The comparison will be undertaken within the EURAMET RMO and, to underpin the Mutual Recognition Agreement (MRA), will be linked to the Consultative Committee for Mass comparison CCM.M-K7. Linking laboratories will be CEM, INRIM, METAS and PTB.

Purpose of this document is:

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

The purpose of a key comparison is to compare the calibration results of the participating institutes. It is not required that each participant adopts precisely the same measurement conditions or procedures. Therefore, this protocol specifies the procedures necessary for the comparison, but not for the measurements.[1]

1.2 Organisation

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1.3 Steering Committee (SC)

The support group is composed of the contact persons at the NPL, DFM, PTB, INRIM, CEM, and METAS (detailed below). Its task is to assist the Pilot Laboratory in drafting the technical protocol, in making decisions to solve problems encountered during the process of the KC and for compiling the draft A and draft B comparison reports.

[1] http://www.bipm.org/utils/common/CIPM_MRA/CIPM_MRA-D-05.pdf

2 Contacts

2.1 Pilot, Steering Committee and Linking Laboratories

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3. Timetable

3.1 Measurement Schedule

Table 1. Measurement schedule for the comparison

Activity	Deadline *
Protocol agreed among participants	Sept 2020
CCM approval of the protocol	Sept 2020
Finalisation of the participants, timetable. Complete measurements of the Pilot Laboratory	Oct 2020
Circulation and measurements by participants	Nov 2020 to May 2021
Interim measurements by Pilot Laboratory	May 2021
Circulation and measurements by participants	June 2021 to Oct 2021
Final measurements by Pilot Laboratory	Dec 2021
Preparation of Draft A	6 weeks after completion of measurements
Circulation of Draft A for comments/corrections	4 weeks for comments
Production of Draft B	4 weeks after the last comments
Circulation of Draft B for comments/corrections	
Final report	2 weeks after Draft B approval (aim April 2022)

*The deadline corresponds to the end of the month shown in table 1.

3.2 Plan of circulation

The travelling standards will be sent to each participating institute. The key comparison will be organized in two consecutive “petals” as outlined below. Each petal contains at least one linking laboratory and the Pilot Laboratory will undertake stability measurement of the travelling standards between the two petals.

Each laboratory has three weeks for the measurements and one week for the transport to the next laboratory. The circulation schedule has been arranged so that the transportation distance between any two successive laboratories is minimized.

CEM, INRIM, METAS and PTB will serve as linking laboratories.

In the case of unexpected delays, the institute involved must give details to the Pilot Laboratory as soon as possible giving details of the cause and expected duration of the delay. If the delay is considered too long, the Pilot Laboratory will attempt to accommodate the delay by rescheduling the participation of the institute in question. Each participant is expected to report its results to the Pilot Laboratory within 4 weeks after completing the measurements.

4 Travelling standards

The travelling standards comprise a set of stainless steel mass standards of nominal values 5 kg, 100 g 10 g, 5 g and 500 mg. Each participating NMI is asked to determine the mass of the travelling standards against their own **stainless steel** standards.

The properties of the standards have been determined by UME and are listed in the Appendix of this protocol.

The mass stability of the standards will be regularly monitored by UME prior to the start and at the mid-point of the comparison. No cleaning of the travelling standards should be carried out. If any participant believe that the weights have been contaminated either in transit or while at an NMI they should contact the Steering Committee immediately.

5 Evaluation

All the result will be corrected to take into account any drift of the travelling standards as determined by the pilot laboratory. A reference value will be calculated for the comparison based on a weighted mean value for all participants. A link to (the reference values) of CCM.M.K-7 will be established using the results of the linking laboratories in both comparisons.

6 General Information and procedure

6.1 Transport of the transfer standards

Each laboratory is responsible for the organisation of the transport of the weights to the next participant. Each participating institute shall cover the costs of the measurements, transport and any customs charges as well as any damage that may occur within its country. The overall costs of organizing the comparison, including the supply of the transfer devices, is borne by the Pilot institute in collaboration with the support committee.

Insurance of transfer of the standard is a decision of the participants, taking into account the risk for any damage that might occur within its country. In case of any transportation problems of a travelling standard, the institute sending the standard must report to the Pilot Laboratory as soon as possible with the necessary data: e.g. the cause of the problem, the expected delay or the eventual damage to the standard. The institute sending the standard and the institute receiving the standard must strive to resolve it in order to minimize its impact. In case of any damage to the standard, the institute sending it must return the standard to the Pilot Laboratory for expert advice.

As a general rule, the travelling standards are to be carried by hand between institutes. If the cost of transfer by hand is not acceptable to the sending institute the option of shipping the travelling standards by using a safe Carrier Company, which can deal with customs formalities, can be used in exceptional circumstances.

Essential customs formalities will be handled through a temporary importation form.

Detailed arrangements for each time the standards are transported will be made between the sending institute, the receiving institute and the Pilot Laboratory. Email information should contain: the method of transportation of the weights (carried by hand or by a carrier company), date the standards left the country and the expected date of entry into the country of the next participant.

The Pilot Laboratory will provide photographs of the open boxes so that customs and airport security will be able to visualize the contents. In addition, the institute sending the standards, the receiving institute and the Pilot Laboratory will collaborate to supply a short letter describing the standards and their metrological importance. The letter should be written in English and in the language of the customs and security officers and should be typed on official institute stationery (see example in ANNEX VI).

In the event that the containers must be opened for inspection: Place the box on a table or counter. Open the top of the box. Close it as soon as possible. They shall be manipulated only with special tools, special gloves. They should never be touched with bare hands. A brief message should be sent to the Pilot Laboratory and the receiving institute if any problems occur during transport (customs, airport security or other).

When the standards arrive at the participating laboratory the suitcase and its contents should be checked for damage and missing items.

The standards should only be manipulated with special tools or special gloves. They should *never* be touched with bare hands or placed on a contaminated surface.

A visual inspection of the surfaces should be made, and the results noted on the corresponding page. The Pilot Laboratory should be informed about the arrival and departure time and about the result of the visual inspection as soon as possible by email.
The standards should be stored protected from dust, aerosols and vapours at all times.
NEVER CLEAN THE TRANSFER STANDARDS.

The procedure of receiving and unpacking the standards are in annex II.
The procedure of packing the standards is in annex III.
The visual inspection procedure of the standards is in annex IV.

2.2 Measurements to be performed

Each participating institute is asked to determine the mass in air of two travelling standards.

Any dust should be removed by using a clean brush provided by the Pilot Laboratory. The mass must not include any visible surface contamination. Visible surface contamination must be reported immediately to the Pilot Laboratory.
Determine the mass of the travelling standards to the best of your ability. There are few requirements that must be followed, although sufficient data must be taken to complete the reports. (annex VII).

The participants must follow these guidelines:

- Comparisons should be made between the travelling standards and your stainless steel reference standards (a sub-division weighing process may be used).
- Always use the gloves provide by the Pilot Laboratory or your own gloves and forceps

- The (**true) mass** of the travelling standards should be determined by weighing in air against the participants (stainless steel) reference standards.
- In order to achieve a good mass stability, it is recommended that the travelling standards are placed in the balance and allowed to stabilise. (Minimum of one full day recommended before carrying out the first measurement).
- There is no need to check the magnetic properties of the travelling standards.
- Use the brush provided to remove any visible dust from the surface of the standard.

2.3 Reporting, Paperwork

The forms (provided in the form of an excel file) of the measurement results, data of the ambient conditions, instruments used, and traceability of the participant's reference standards should be filled out and sent to the Pilot Laboratory within one month after the measurements are completed.

- Each participant shall determine the mass of the travelling standards with respect to its own reference standards in stainless steel.
- Each participant will supply the Pilot Laboratory with the general information requested. Please use the Excel table provided.
- Institutes should report their results in air, traceable to the International Prototype of the Kilogram, calculate their uncertainties in a coherent way. Please use the Excel table provided.
- A complete table of uncertainties (including estimated degrees of freedom) is requested. Please use the Excel table provided.
- The uncertainty must include details of traceability to the International Prototype of the Kilogram[2]. All uncertainties should be reported in accordance with the 'Guide to the expression of Uncertainty in Measurement'[3] (GUM) using a coverage factor of unity ($k = 1$).
- The results should be sent to the Pilot Laboratory.

Every incident, where the transfer standards may have been damaged, should be documented and communicated to the Pilot Laboratory immediately. The Pilot Laboratory should also be informed about any delay in the comparison schedule.

[2] Note that at the time of the comparison traceability for new (BIPM) calibrations will be to the kilogram "consensus value". The calculation of the consensus value has been selected by the CCM Task Group to ensure continuity of the global mass scale and so the change in traceability will have no impact on the equivalence of results in this comparison. The increase in uncertainty when transferring from IPK traceability to the consensus value (and the potential increase in NMIs CMCs) will be dealt with as a separate exercise.

[3] JCGM 100:2008, Evaluation of measurement data - Guide to the expression of uncertainty in measurement, 2008. http://www.bipm.org/en/publications/guides/gum.html

3. Annexes

I. Time schedule,
II. Transportation protocol (arrival)
III. Transportation protocol (departure)
IV. Visual inspection, record of the surfaces of the transfer standards
V. Data determined by the Pilot Laboratory
VI. Example form for security control
VII. Forms for general data, traceability, calibration results, and environmental data during calibration, the recommended form for reporting the uncertainty budget and the details of the used instruments and description of the measuring room.

Annex I. Time schedule

Table 1. Timetable for the comparison

Leg	NMI	Date of arrival	Date of departure	Deadline for sending the results
1	BIM (Bulgaria)	2 Nov 2020	27 Nov 2020	15 Jan 2021
	METROSERT (Estonia)	1 Dec 2020	15 Jan 2021	15 Feb 2021
	BEV (Austria)	18 Jan 2021	12 Feb 2021	12 Mar 2021
	MIRS (Slovenia)	12 Mar 2021	9 Apr 2021	9 May 2021
	METAS (Switzerland)	12 Apr 2021	7 May 2021	7 June 2021
	UME (Pilot) (Turkey)	10 May 2021	4 June 2021	4 July 2021
2	MIKES (Finland)	6 July 2021	10 Sept 2021	10 Oct 2021
	CEM (Spain)	13 Sept 2021	8 Oct 2021	8 Nov 2021
	LATMB (Latvia)	11 Oct 2021	5 Nov 2021	5 Dec 2021
	ILNAS (Luxembourg)	8 Nov 2021	3 Dec 2021	3 Jan 2022
	PTB (Germany)	6 Dec 2021	21 Jan 2022	21 Feb 2022
	INRIM (Italy)	25 Jan 2022	18 Feb 2022	18 Mar 2022
	UME (Pilot) (Turkey)	22 Feb 2022	21 Mar 2022	21 Ap 2022
3	SE (Ukraine)	25 Mar 2022	21 Ap 2022	21 May 2022
	INPL (Israel)	25 Ap 2022	20 May 2022	20 June 2022
	DMDM (Serbia)	24 May 2022	20 June 2022	20 July 2022

Leg	NMI	Date of arrival	Date of departure	Deadline for sending the results
	DZM (Croatia)	24 June 2022	21 July 2022	21 Aug 2022
	SASO-NMCC (Saudi Arabia)	25 July 2022	19 Aug.2022	19 Sept 2022
	UME(Pilot) (Turkey)	23 Aug 2022	16 Sept. 2022	16 Oct 2022
4	BOM (Macedonia)	20 Sept 2022	17 Oct 2022	17 Nov 2022
	IMBIH (Bosnia and Herzegovina)	21 Oct 2022	18 Nov 2022	18 Dec 2022
	MBM Montenegro	22 Nov 2022	19 Dec 2022	19 Jan 2023
	(RMI) Belgium	23 Dec 2022	19 Jan 2023	19 Feb 2023
	(IPQ) Portuguese	23 Jan 2023	19 Feb 2023	19 Mar 2023
	UME(Pilot) (Turkey)	23 Feb 2023	-	

Notes on the schedule:

- The schedule has been compiled taking into account the availability of the participants, geographic proximity and custom formalities.

Annex II. Transportation protocol (arrival)

List of the content of the transport container:

- Five transfer standards of nominal values 5 kg, 100 g 10 g, 5 g and 500 mg
- Glove supplied by the pilot laboratory.
- Lens cleaning tissues surrounding the transfer standards.
- Lens cleaning tissue sheet to place under/above the standards.
- Lens cleaning tissues as reserve.
- (Inspection forms, report forms, technical protocol will be sent electronically).

The standards should only be manipulated with special tools or special gloves. They should *never* be touched with bare hands or placed on a contaminated surface.

Procedure of unpacking (arrival)

1. Visual inspection of the case (damaged, clean). If not clean please carefully clean it.
2. Take the suitcase into the mass laboratory.
3. Prepare a clean surface.
4. Open the case and check for damage and missing items.
5. Check the content (list below).
6. Open the envelope (for your laboratory) with the gloves and lens papers (assigned to you).
7. Remove the box from the suitcase and the protecting bag and place it on a clean surface.
8. Open the box (with clean hands or normal gloves)
9. Put on the gloves supplied by the Pilot Laboratory
10. Put a lens cleaning tissue on the surface.
11. Remove the lens paper and using the gloves or the forceps provided place the standard on the lens cleaning tissue and covered with a bell jar (or with a clean lens cleaning tissue).
12. Perform steps 7-12 for the next standard.
13. Perform the visual inspection of the surfaces of the standards using a record sheet (Annex IV).
14. (Never clean the standards).
15. If the standard is not placed into the balance after inspection, it should be placed on the lens cleaning tissues and covered by a bell jar.

Illustrations:

Form to be fill out on arrival of the standards

Name of NMI:	
Name of Contact Person:	
Full mailing address:	
Telephone:	
e-mail:	
Current date:	

Date of arrival:		From NMI:	
Transportation box: (e.g. damaged, missing items)			
Package of the weights was according to the requirements?			
Surface conditions: (use record sheets)			
Other remarks:			

Annex III. Transportation protocol (departure)

The standards should only be manipulated with special tools or special gloves. They should *never* be touched with bare hands or placed on a contaminated surface.

1. Carry out a visual inspection of the surfaces using a record sheet (Annex IV).
2. Place a lens cleaning tissue (supplied by the Pilot Laboratory) on a clean surface. Please use new tissue.
3. Using gloves (supplied by the Pilot Laboratory) or forceps , place the standards on top of the tissue
4. Wrap the standard into the lens paper.
5. Put the standard into the box and gently close the lid.
6. Put the box into the protecting bag and into the suitcase.
7. Repeat steps 1-6 for the other standards.
8. Place the gloves and envelopes for other participants inside the suitcase.
9. Close the suitcase and secure it with the cable ties.

Illustrations:

Form to be filled out on departure of the standards

Name of NMI:	
Name of Contact Person:	
Full mailing address:	
Telephone:	
e-mail:	
Current date:	

Date of sending:		To NMI:	
The brush, the forceps and envelopes of other NMI are packed			
Package of the weights was according to the requirements?			
Surface conditions: (use record sheets if there any changes)			
Other remarks:			

Annex IV. Visual inspection, record of the surfaces of the transfer standards

A visual inspection of the surfaces shall be made, and the results shall be reported immediately after receiving and before sending the standards using this protocol by sending it to the Pilot Laboratory by email (scanned in PDF format).

Procedure:

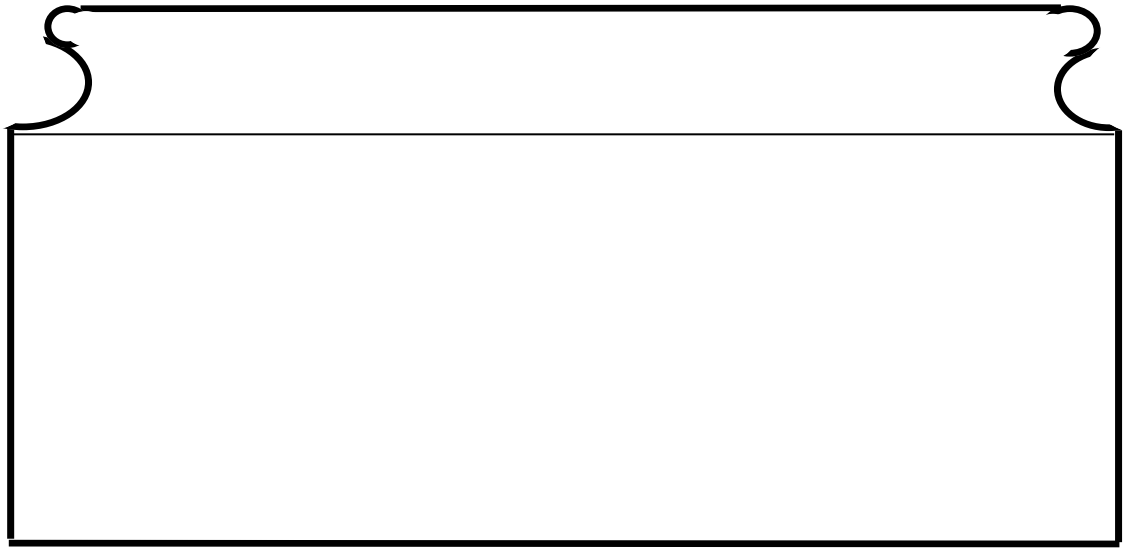
1. Put the standards on clean lens cleaning paper as described.
2. Always use gloves or forceps provided by the Pilot Laboratory.
3. In case of dust on the surface, use only the brush provided by the Pilot Laboratory to remove the visible particles. DO NOT CLEAN THE TRANSFER STANDARDS.
4. Record on the diagrams any mark(s) seen on the travelling standard (scratches, contamination, etc.)
5. Scan the page and send it to the Pilot Laboratory before starting any measurements.

Sample inspection sheet.

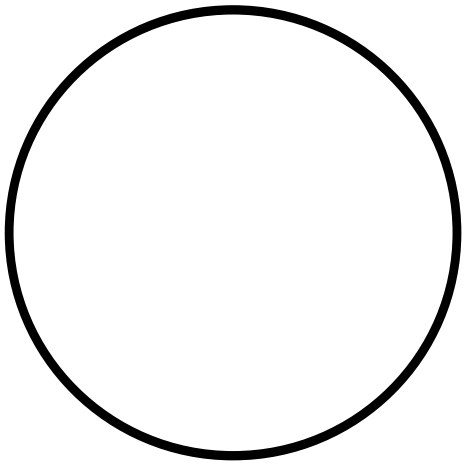
Identification of the standard	
Institute	
Carried out by	
Date	
Notes:	

5000 / 100 / 10 / 5 gram

Top surface



Base surface

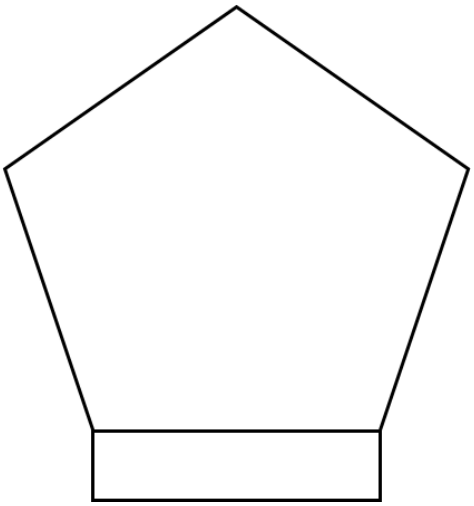


Sample inspection sheet.

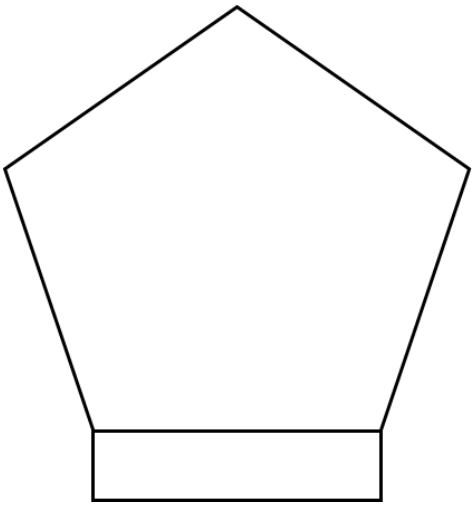
Identification of the standard	
Institute	
Carried out by	
Date	
Notes:	

500 mg

Front Surface



Reverse Surface



Other Comments:

Annex V. Data determined by the Pilot Laboratory.

The transfer standard set consists of weights of nominal values 5 kilograms, 100 grams, 10 grams, 5 grams and 500 milligrams. Details of each mass standard are given in table 2.

The participants will be supplied with the following data:

- Volume of each mass standard at 20 °C
- Coefficient of cubical expansion
- The gravity height of each mass standards
- The magnetic properties of all travelling standards as the volume magnetic susceptibility and the magnetic polarization.

All data have been determined by UME.

Table 2. Information on the five travelling standards.

Nominal Value *g*	Identi-fication	Volume at 20 °C cm^3	Uncertainty (*k=2*) cm^3	Coefficient of cubic expansion $10^{-6}\ °C^{-1}$	CofG height *mm*	Volume magnetic susceptibility	Magnetic polarization less than μT
5 000	K1	636.303	0.026	48.0	64.0	0.00269	0.01
100	K1	12.51880	0.00040	48.0	17	0.00373	0.06
10	K1	1.24360	0.00030	48.0	8.2	0.00393	0.06
5	K1	0.62460	0.00025	48.0	-	0.00491	0.18
0.5	K1	0.05814	0.00058	54.2	-	-	-

Uncertainty (*k*=2) of the gravity height is less than 2 mm.

Annex VI Example form for security control

This is an example document for security purposes when the transfer standards are carried by hand (for air travel).

Additional forms can be provided (in the language of the sender/recipient country) to the person who will have to assure the mass transfer. This model should be adapted in accordance with the experts and NMIs involved in the transfer.

To whom it may concern DD MM YYYY, City, Country

I the undersigned, *Name of expert* of the Mass Department of the *Name of NMI*, certify that *Name of person accompanying weights* is carrying a set of stainless steel mass standards of values 5 kg, 100 g 10 g, 5 g and 500 mg from *Name of (sending) NMI* to the National Metrology Institute of *Recipient country*:

Name of the Institute:
Address:
Tel:
e-mail:

These mass standards are made of stainless steel and have nominal material value but an enormous scientific value. They are very delicate (fragile) and may not be manipulated by unauthorized persons. Improper handling during transportation would render them useless. The container has been designed to protect the masses during their transport so that they remain clean.

It is essential that they travel in the cabin as hand luggage and NOT in the baggage hold. They are safe and do not pose any risk to the health and safety of passengers travelling on the same flight.

Pilot Laboratory and contact:
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TÜBİTAK UME
Gebze Yerleşkesi,
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Annex VII

All reporting forms are provided only electronically in excel format.

Forms are for general data, traceability, calibration results, and environmental data during calibration, the recommended form for reporting the uncertainty budget and the details of the used instruments and description of the measuring room.

All the reporting shall be preferably done by using the provided excel file.