The FINAL REPORT For

AFRIMETS Key Comparison (AFRIMETS.M.M-K7) of

Five Stainless steel Mass Standards:

5 kg, 100 g, 10 g, 5 g and 500 mg

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1. INTRODUCTION

This key comparison is based on a decision of TCM of AFRIMETS on July 2015 in South Africa. TCM agreed that NIS-Egypt will pilot this comparison by providing the technical support and contributing to the link to the CCM comparison. The comparison was consisting of one set of five stainless steel mass standards with nominal values: 5 kg, 100 g, 10 g, 5 g and 500 mg. These masses are owned by the LPEE-LNM and chosen to be the transfer mass standards. The pilot laboratory (NIS) measured the volume and the magnetic properties of the standards before the circulation and had reported these values to the participants. The pilot laboratory has also verified the stability of all travelling standards for one year.

Six laboratories measured the set of travelling standards between September 2018 and December 2019.

2. PARTICIPANTS

The number of participating institutes in this AFRIMETS.M.M-K7 is six including the pilot laboratory (NIS). The participating institutes and contact persons are summarized in Table 1.

Table 1. List of the participating NMIs and contact persons.

National Institute of Metrology	Country	Acronym	Postal Address	Contact Person
National Institute of Standards	Egypt	NIS	El Sadat(tersa)St.,Giza,Egypt P.O.Box 136 Giza - code 12211	Sayed Emira
Federal Institute of Metrology	Switzerland	METAS	Switzerland , Lindenweg 50, CH-3003 Berne-Wabern	Christian Wuethrich
The National Metrology Laboratory of the Public Laboratory for Tests and Studies	Morocco	LPEE-LNM	km 7, Route d'El Jadida – Casablanca	Samira Souiyam
Zambia Metrology Agency	Zambia	ZMA	Zambia Metrology Agency, Plot 4526, Freedom Way. Lusaka, Zambia	Daniel M. Mutale
National Metrology Institute of South Africa	South Africa	NMISA	NMISA, CSIR Campus, Building 5 Meiring Naude Road, Brummeria, Pretoria 0182. South Africa	Thomas Mautjana
Kenya Bureau of Standards	Kenya	KEBS	Kenya Bureau of Standards, Popo Road, Off Mombasa Road, P.O. Box 54974-00200, Nairobi, Kenya	Tonui David

3. TRANSFER STANDARDS

3.1 Mass standards

The transfer standards consist of five masses of nominal values 5 kg, 100 g, 10 g, 5 g, 500 mg, which were marked differently on the surface except 500 mg shown as in figure1. The material of these masses is stainless steel which has the OIML shape [1].

The transfer standard was monitored regularly for more than one year at the pilot lab. It showed excellent stability without any overshoot in monitoring.

Table 2 summarizes the relevant technical data on volume and magnetic susceptibility for each mass provided by pilot laboratory. Other quantities can be assumed. For example, the coefficient of volume expansion (stainless steel) could be 0.000045 K^{-1}.

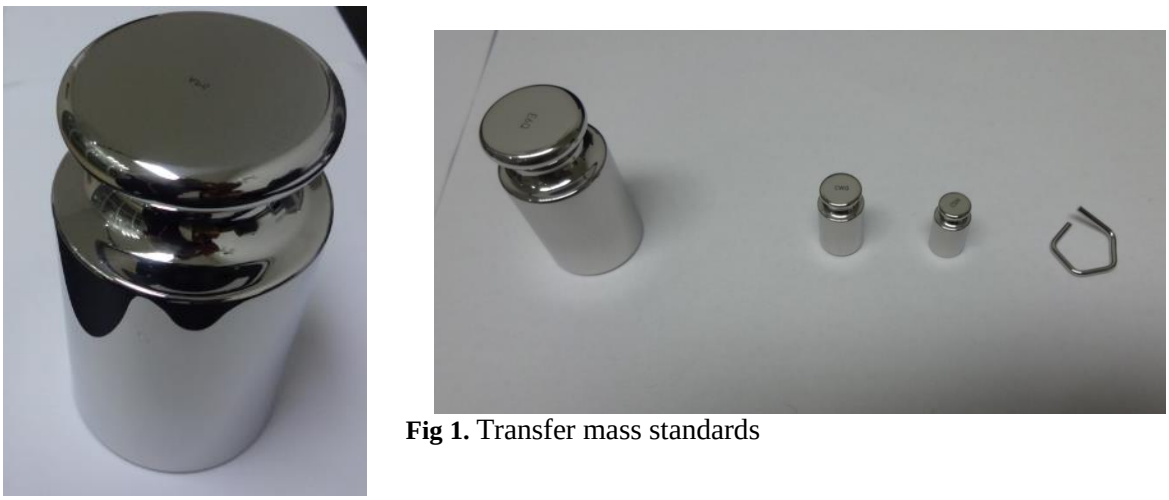

Fig 1. Transfer mass standards

Table 2. Characteristics of the transfer mass standards used for the comparison.

Nominal	Marking on Surface	Shape	Volume / cm^3 (Uncertainty, k =2 / cm^3)	Magnetic susceptibility(*)
5 kg	AP5	OIML	624.30 (0.10)	0.00443
100 g	E6Q	OIML	12.4876 (0.0015)	0.00495
10 g	CWQ	OIML	1.2489 (0.0007)	0.00461
5 g	NS3	OIML	0.6244 (0.0006)	0.00410
500 mg	--------	OIML（wire)	0.0629 (0.0005)	------

*) measured with a commercial susceptometer (Sartorius).

3.2 Transfer Standard Package

The transfer Package consists of one carrying box which includes the mass set box. The carrying box is from aluminum with dimensions 50 cm × 50 cm × 25 cm contains black plastic box with dimensions 35 cm × 37 cm × 18 cm and separate holes for each standard mass and the total weight is about 13 kg, figure 2, shows the five masses (5 kg, 100 g, 10 g, 5 g, 500 mg), In addition to one tweezer, one glove and two brushes.

Figure 2. Carrying case of mass traveling standards used for the AFRIMETS.M.M-K7 comparison.

4. CIRCULATION OF THE TRANSFER STANDARD

Table 3 shows the final schedule confirmed by all participants for the circulation of transfer standard. NIS carried out a stability check for these standards before the comparison and after the conclusion of work. Four weeks were allowed for each laboratory to carry out the measurements and one week for transport between participants.

Table 3. The sequence of circulation and measurement schedule

Institute	country	Start	End
NIS	Egypt	10 / 9 / 2018	5 / 10 /2018
METAS	Switzerland	16 / 10 /2018	1 / 12 /2018
LPE - LNM	Morocco	21/ 12/ 2018	1/ 3/ 2019
ZMA	Zambia	28/ 3/ 2019	1/ 5 / 2019
NMISA	South Africa	9/ 5 / 2019	1/ 7/ 2019
KEBS	Kenya	29 / 7/ 2019	4 / 10 / 2019
NIS	Egypt	7 / 10 / 2019	30 /12/ 2019

5. MASS COMPARATORS USED BY PARTICIPANTS

The weighing instruments used by the participating laboratories are listed in Table 4 based on the information provided by the participants.

Table 4. Weighing instruments of participants

Nominal	NMI	Manufacturer	Type	Capacity	Resolution
5 kg	NIS	Sartorius	CC10000U-L	10050 g	0.01 mg
	METAS	Mettler-Toledo	AT10005	10011 g	0.01 mg
	LPE-LNM	Sartorius	CCE10000S	10 kg	0.1 mg
	ZMA	Sartorius	CCE5004	5100 g	0.2 mg
	NMISA	Mettler Toledo	AT10005	10 011 g	0.01 mg
	KEBS	Mettler Toledo	AX10005	10 kg	0.01 mg
100 g	NIS	Mettler-Toledo	AT 1006	1011 g	0.001 mg
	METAS	Mettler-Toledo	M one	1001.5 g	0.0001 mg
	LPE-LNM	Sartorius	CCE1005	1 kg	0.01 mg
	ZMA	Sartorius	CC1000S-L	1000 g	0.001 mg
	NMISA	Mettler Toledo	AX1006	1011 g	0.001 mg
	KEBS	Mettler-Toledo	AX 1006	1011 g	0.001 mg
10 g	NIS	Mettler-Toledo	AT 21	22 g	0.001 mg
	METAS	Mettler-Toledo	AT106	111 g	0.001 mg
	LPE-LNM	Sartorius	CCE66	60 g	0.001 mg
	ZMA	Sartorius	CC111	110 g	0.001 mg
	NMISA	Mettler Toledo	AT106H	111 g	0.001 mg
	KEBS	Sartorius	CC50	50 g	0.001 mg
5 g	NIS	Sartorius	CC6	6 g	0.0001 mg
	METAS	Mettler-Toledo	A5	5.1 g	0.0001 mg
	LPE-LNM	Sartorius	CCE6	6 g	0.0001 mg
	ZMA	Sartorius	CCE6	6.1 g	0.0001 mg
	NMISA	Mettler Toledo	UMX5	5.1 g	0.0001 mg
	KEBS	Sartorius	CC50	50 g	0.001 mg
500 mg	NIS	Sartorius	CC6	6 g	0.0001 mg
	METAS	Mettler-Toledo	A5	5.1 g	0.0001 mg
	LPE-LNM	Sartorius	CCE6	6 g	0.0001 mg
	ZMA	Sartorius	CCE6	6.1 g	0.0001 mg
	NMISA	Mettler Toledo	UMX5	5.1 g	0.0001 mg
	KEBS	Sartorius	S4	4 g	0.0001 mg

6. REPORTED RESULTS FROM PARTICIPANTS

The results and uncertainties provided by each of the participants for each of the nominal mass values are shown in Table 5. Each result is shown as the difference between the mass determined by the participant (m) and the nominal mass value (m_o), in mg. The uncertainties (u_i) are given in mg at $k = 1$.

Table 5. The measured mass value and its standard uncertainties reported by the participants.

Laboratory	5 kg		100 g		10 g		5 g		500 mg	
	$m-m_0$ /mg	u_i/mg	$m-m_0$ /mg	u_i/mg	$m-m_0$ /mg	u_i/mg	$m-m_0$ /mg	u_i/mg	$m-m_0$ /mg	u_i/mg
NIS	4.38	0.12	0.008	0.003	0.009	0.002	0.0126	0.0011	0.0079	0.0007
METAS	4.19	0.11	0.009	0.0011	0.009	0.0007	0.0123	0.0008	0.0082	0.0005
LPEE	4.80	1.25	0.010	0.025	0.011	0.010	0.0094	0.0075	0.0086	0.004
ZMA	4.70	6	0.051	0.042	0.007	0.016	0.011	0.012	0.0052	0.0075
NMISA	4.49	0.41	-0.002	0.008	0.004	0.0032	0.0104	0.0026	0.0046	0.0013
KEBS	4.40	0.4	0.020	0.01	0.019	0.003	0.009	0.003	0.008	0.001

7. ANALYSIS OF RESULTS

7.1 Comparison Reference Value:

The key comparison reference value (KCRV)was determined as the weighted mean in accordance with ISO Guide ISO 13528:2015 [2] in which the uncertainties reported by the participants have been assumed to be credible. The reasons for selecting this method are the approximately equal competence for all the participating laboratories and furthermore, the distribution of the results reported by the participating laboratories has a normal shape, i.e. no distinct modes were detected in the observed results. This implies that these reported results are reliable and could be used to estimate the reference value and its uncertainty using the appropriate statistical method as described below in this report.

7.2 Determination of the weighted mean (y)

The weighted mean of the institutes' measurements was determined using the inverses of the squares of the associated standard uncertainties as the weights [3-5]:

$$y = \frac{x_1/u^2(x_1) + \cdots + x_N/u^2(x_N)}{1/u^2(x_1) + \cdots + 1/u^2(x_N)}. \quad (1)$$

7.3 Determination of the standard deviation u(y) associated with y from

$$\frac{1}{u^2(y)} = \frac{1}{u^2(x_1)} + \cdots + \frac{1}{u^2(x_N)}. \quad (2)$$

7.4 Consistency check

Applying the chi-squared test to carry out an overall consistency check of the obtained results

7.3.1) An overall consistency of the results using the chi-square test was applied to all *n* calibration results

$$\chi^2_{obs} = \sum_{i=1}^{n} \frac{(x_i - y)^2}{u^2(x_i)} \quad (3)$$

7.3.2) Assign the degree of freedom

$$\upsilon = N-1$$

Where N is the number of participants

7.3.3) The consistency check is regarded as failed if:

$$\Pr\{\chi^2(v) > \chi^2_{obs}\} < 0.05 \quad (4)$$

Where:

Pr Denotes "probability of" and $\chi^2(v)$ is the inverse of the chi-square cumulative distribution function with degree of freedom specified by v for the probability of 0.05 (corresponding to the 95% level of confidence). In this case, the participant with the highest value of χ^2_{obs} is excluded from the next round of evaluation and then a new reference value, reference standard uncertainty, and chi-squared values are calculated again without the excluded laboratory.

7.3.4) If the consistency check does not fail:

Then y was accepted as KCRV x_{ref} and u(y) was accepted as the standard uncertainty $u(x_{ref})$ associated with x_{ref} .

The five reference values and their associated uncertainties are shown in Table 6.

Table 6. Reference values and associated uncertainties for AFRIMETS.M.M-K7

Nominal value	Reference value / mg	Uncertainty / mg (k =1)
5 kg	5 000 004.29	0.08
100 g	100 000.009	0.001
10 g	10 000.009	0.000 6
5 g	5 000.012 1	0.000 6
500 mg	500.008 2	0.000 4

7.5 Determination of the degrees of equivalence:

When the consistency check passes, for each laboratory results, the degree of equivalence of institute i as the pair of value $(d_i, U(d_i))$ between each laboratory and the KCRV (x_{ref}) is calculated using the following formulas [4]:

$$d_i = x_i - x_{ref} \quad (5)$$

$$U(d_i) = 2 \times u(d_i) \quad (6)$$

Where $u(d_i)$ is calculated from

$$u^2(d_i) = u^2(x_i) - u^2(x_{ref}) \quad (7)$$

In general, discrepancy values can be identified if it gives the following

$$E_i = |d_i|/2u(d_i) > 1.0 \quad (8)$$

Differences between each participant and the reference value (degrees of equivalence d_i), together with their associated uncertainties (k = 2) are shown in Table 7.

Table 7. Degree of equivalence and associated uncertainties (k = 2).

Laboratory	5 kg		100 g		10 g		5 g		500 mg	
	d_i /mg	u_i/mg	d_i /mg	u_i/mg	d_i /mg	u_i/mg	d_i /mg	u_i/mg	d_i /mg	u_i/mg
NIS	0.09	0.18	-0.00076	0.0056	-0.00023	0.0038	0.00047	0.0018	- 0.00034	0.0012
METAS	- 0.10	0.16	0.0001	0.0008	-0.00023	0.0006	0.00017	0.0010	- 0.0004	0.0007
LPEE	0.51	2.50	0.00124	0.0500	0.00177	0.0200	-0.00273	0.0150	0.00036	0.0080
ZMA	0.41	11.99	0.0422	0.0840	-0.0022	0.0320	-0.0011	0.0240	- 0.0030	0.0150
NMISA	0.20	0.81	-0.0108	0.0159	-0.0057	0.0063	-0.00173	0.0051	0.00196	0.0025
KEBS	0.11	0.79	0.0112	0.020	0.0098	0.006	-0.0031	0.0059	- 0.00024	0.0019

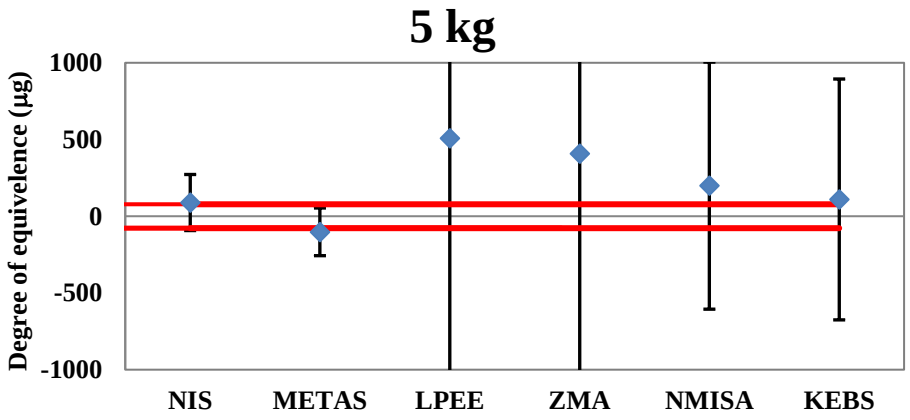

Figure 3. Degree of Equivalence for 5 kg

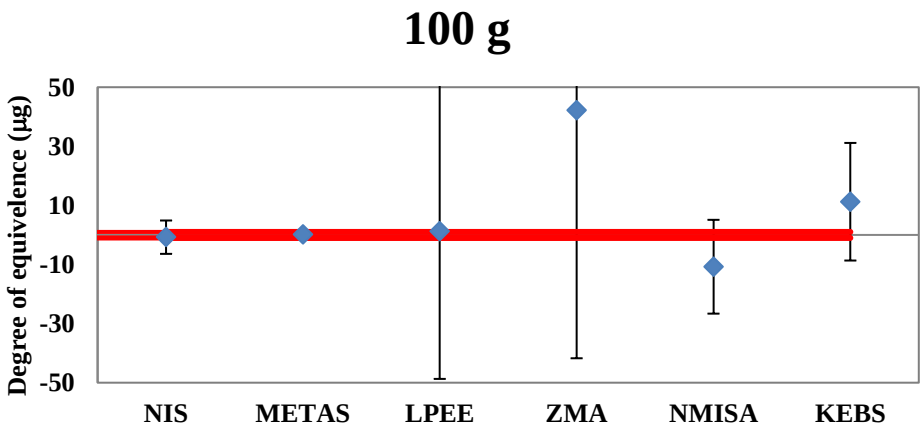

Figure 4. Degree of Equivalence for 100 g

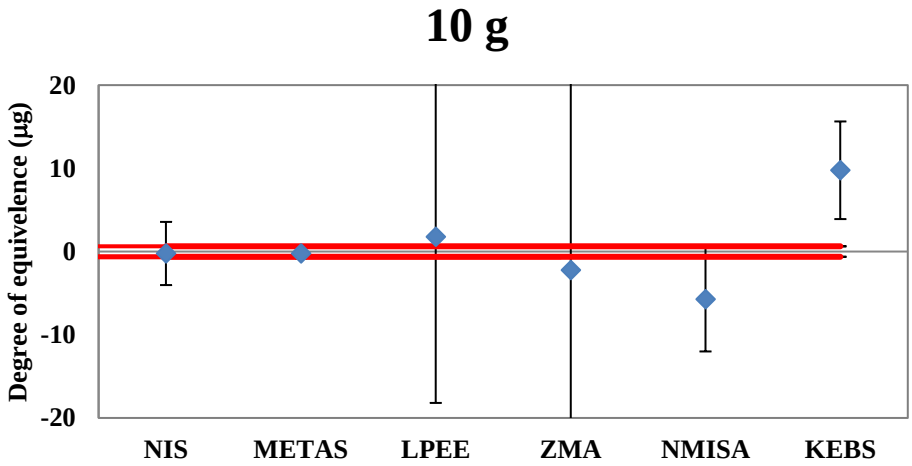

Figure 5. Degree of Equivalence for 10 g

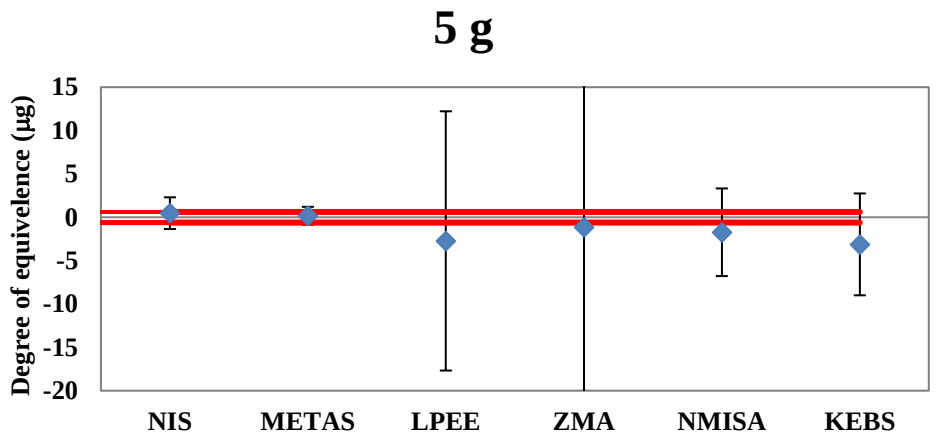

Figure 6. Degree of Equivalence for 5 g

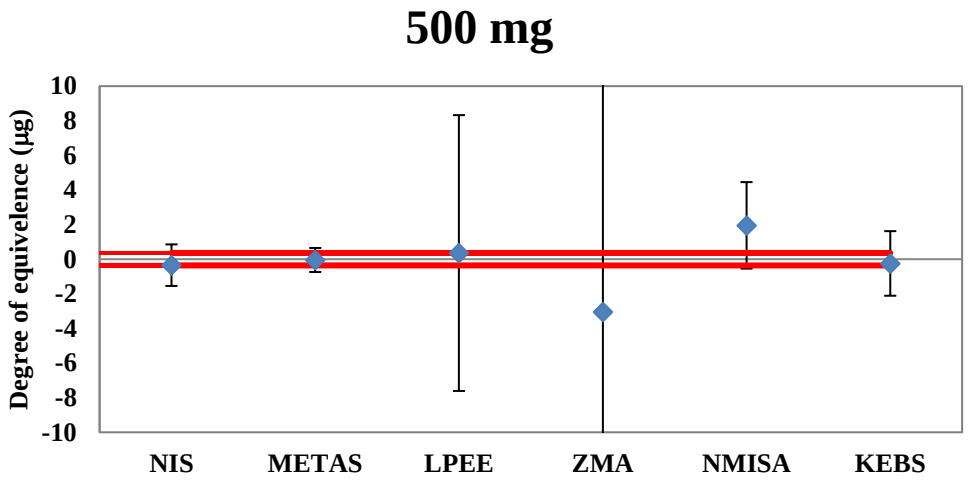

Figure 7. Degree of Equivalence for 500 mg

8. Linking with the CIPM KCRV

There are two of participants were participating in the CIPM key comparison of mass standards CCM.M-K7, NIS and METAS. The mass standards were used in CCM.M-K7 are same nominal value of mass standards in this comparison (AFRIMETS.M.M-K7).

The outcome of both comparisons, that were linked using NIS results, is graphically presented in Figures 8-9. The linking procedure and its uncertainty analysis are based on the principles which are given in the papers of Elster et al, [7], Kharitonov et al, [8] and Decker et al, [9].

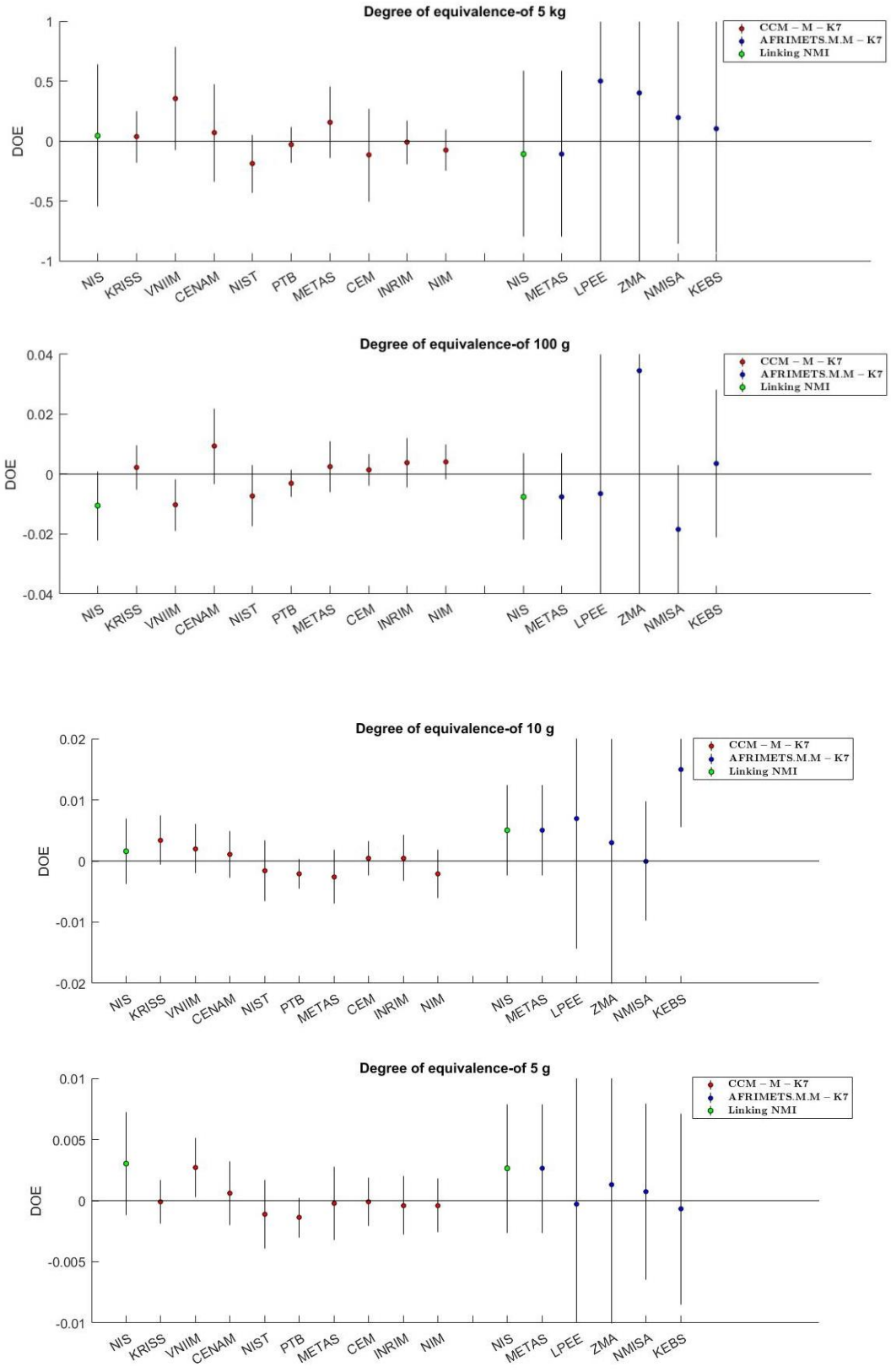

Figure 8. Degree of equivalence with respective KCRV for 5 kg, 100 g, 10 g and 5 g.

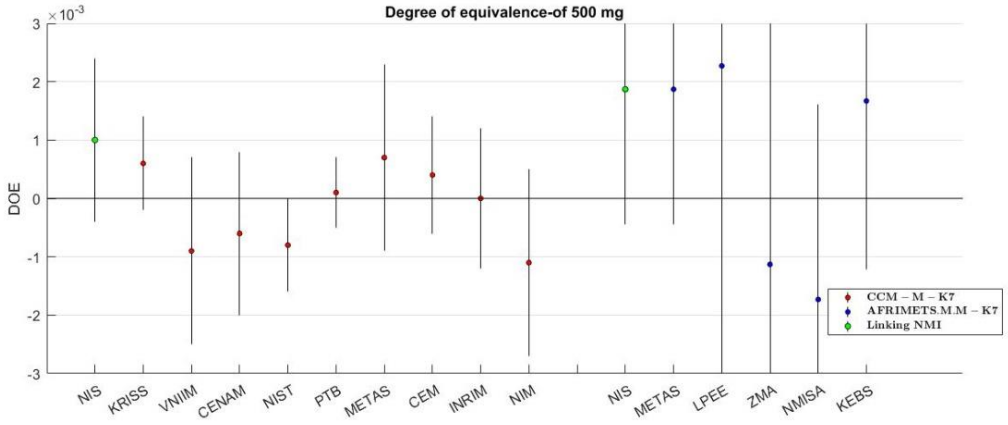

Figure 9. Degree of equivalence with respective KCRV for 500 mg.

Conclusion:

This report summarizes the procedure and analysis of AFRIMETS.M.M-K7, a Key Comparison of 5 kg, 100 g, 10 g, 5 g and 500 mg stainless steel mass standards. One set of transfer standards were prepared, monitored, and circulated to the participant laboratories. No serious problems were encountered during the course of the key comparison. The comparison reference value was determined as the weighted mean according to section [7].

Finally, one of six laboratories, KEBS, was not consistent with the key comparison reference values at 10 g, within their expanded uncertainties with a coverage factor, $k = 2$, as shown in figure 5 and figure 8 (KEBS do not have CMC entries in the KCDB).

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- Dr. Shaker Gelany, NIS
- Dr. Ahmed Hashad, NIS
- Eng. Mohammed Hamdy, NIS
- Mr. Ahmed Shawkey, NIS

References:

1. Weights of classes E1, E2, F1, F2, M1, M1-2, M2, M2-3 and M3, International recommendation OIML R111-1, OIML, 2004.
2. ISO 13528,"Statistical methods for use in proficiency testing by interlaboratory comparison", 2015.
3. Sungjun Lee, The final report for CCM.M-K7: key comparison of 5 kg, 100 g, 10 g, 5 g and 500 mg stainless steel mass standards, 2017, Metrologia, 54.
4. Cox M.G., The evaluation of key comparison data, Metrologia, 2002, 39, 589-595.
5. K Weise and W Wöger, Removing model and data non-conformity in measurement evaluation, 2000 Meas. Sci. Technol. 11 1649.
6. Nielsen L., Evaluation of measurement intercomparisons by the method of least squares, Technical Report DFM-99-R39, Danish Institute of Fundamental Metrology, 2000.
7. Elster C., Link A., Wöger W., Proposal for linking the results of CIPM and RMO key comparisons, Metrologia, 2003, 40, 189-194
8. Kharitonov I.A., Chunovkina A.G., Evaluation of regional key comparison data: two approaches for data processing, Metrologia, 2006, 43, 470-476.
9. Decker J.E., Steele A.G., Douglas R.J., Measurement science and the linking of CIPM and regional key comparisons, Metrologia, 2008, 45, 223-232.