Republic of Slovenia
Ministry of higher education, science and technology
Metrology Institute of the Republic of Slovenia

Report on

BILATERAL KEY COMPARISON EUROMET.M.M-K4.1

COMPARISON OF 1 kg STAINLESS STEEL WEIGHTS

Final Report

By

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Abstract:

This report summarizes the results of bilateral comparison of two 1 kg stainless steel mass standards between DFM, Denmark, and MIRS, Slovenia. The objectives were to facilitate the demonstration of metrological equivalence between the two participating national laboratories and to check the capabilities of MIRS to improve quoted calibration measurement capabilities (CMC) at 1 kg.

Both laboratories have participated in EUROMET 510 project (key comparison EUROMET.M.M-K4). Results are consistent with each other and with the key comparison reference value of the comparison CCM.M-K1 to which this comparison has been linked through the comparison EUROMET.M.M-K4.

1. Introduction

This report describes a bilateral comparison of two 1 kg stainless steel mass standards between DFM, Denmark, and MIRS, Slovenia. The objectives of the comparison were to facilitate the demonstration of metrological equivalence between the two participating national laboratories, and to check the capabilities of MIRS to improve quoted calibration measurement capabilities (CMC) at 1 kg. Both laboratories have participated in EUROMET 510 project - key comparison EUROMET.M.M-K4 to which the link was provided by DFM. The comparison has been linked to the key comparison CCM.M-K1 through EUROMET.M.M-K4.

2. Transfer standards

The transfer standards for this comparison comprise two stainless steel cylindrical weights with a knob (OIML R111 design). The weights were housed in individual wooden containers. The weights were designated LM-006 and LM-006d. They were produced by Mettler Toledo and purchased by MIRS in 1997. The volumes and centers of gravity of the standards and associated uncertainties are given as a part of the measurement report.

3. Participants

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4. Circulation scheme and time schedule

The traveling standards were transported by courier between the participating laboratories. The standards were shipped in a plastic transport case. It was the responsibility of the participating laboratories to organize the transport to the next participant. No special custom procedures were required between Slovenia and Denmark. Agreed and realized time schedule was as follows:

Measurements at MIRS, Ljubljana: August 2007
Transport to DFM, Lyngby
Measurements at DFM, Lyngby: August 2007
Transport to MIRS, Ljubljana
Measurements at MIRS, Ljubljana: October 2007

5. Technical protocol

The agreed technical protocol is given in *Annex 1*.

6. Results

Completed measurement reports containing the measurement results, data of the ambient conditions, lists of instruments used and a description of the traceability of the participant's reference standards were submitted to IRMM, which was acting as an independent collector of all results in the comparison. After all measurement were completed, the collected measurement reports were sent to DFM, which carried out the data analysis and also provided the link to CCM.M-K1 through EUROMET.M.M-K4.

MEASUREMENT RESULTS

The participating laboratories determined the mass (not conventional value) of both standards (LM-006 and LM-006d) according to their normal calibration procedures.

The values of the mass m (expressed in terms of the correction $\Delta m = m - 1$ kg) of the weights LM-006 and LM-006d measured during the comparison are shown in Table 1 and Table 2, respectively, column 4 “Results”. The tables include mass values measured in the summer 2007, when the weights were exchanged between the two participating laboratories.

The following general model for the change in the actual correction Δm_{ref} of the LM-006 and LM-006d weights during the comparison has been used [1]:

$$\Delta m_{\mathrm{ref}} = a_1 + a_2\, t + \delta m, \qquad (1)$$

where t is the time of measurement, a_1 and a_2 are constants, and δm is a time dependent random variable with expectation 0 and variance σ^2, which describes random changes in the mass of LM-006 (and LM-006d) over time. Under the assumptions $a_2 = 0$ (no deterministic drift in the value of the mass standard) and $\sigma^2 = 0$ (no random changes in the value of the mass standard), the two reference values $\Delta m_{\mathrm{ref}} = a_1$ (one for each standard) have been calculated by the method of least squares taking into account the claimed uncertainties and covariances of the measurement results [2]. The calculated reference values are given in Table 1 and Table 2 and plotted in Figure 1 and Figure 2 as a blue, solid line

LM-006

Table 1: Results of the comparison. Δm is the mass correction of the weight LM-006 measured by the specified participant at the specified date, Δm_{ref} is the corresponding reference value, and d is the normalized deviation defined as the difference $D = \Delta m - \Delta m_{ref}$ divided by the standard uncertainty of that difference. The last column indicates if a result is included in the calculation of the reference value or not (y=yes, n=no).

Meas. No. i	Lab ID	Date	**Results (LM-006)** Δm [mg]	$u(\Delta m)$ [mg]	Reference value Δm_{ref} [mg]	$u(\Delta m_{ref})$ [mg]	Normalized deviation d	Included (y/n)
1	MIRS	01.08.2007	0,626	0,035	0,636	0,013	-0,3	0,626
2	DFM	23.08.2007	0,634	0,014	0,636	0,013	-0,4	0,634
3	MIRS	01.10.2007	0,632	0,035	0,636	0,013	-0,1	0,632

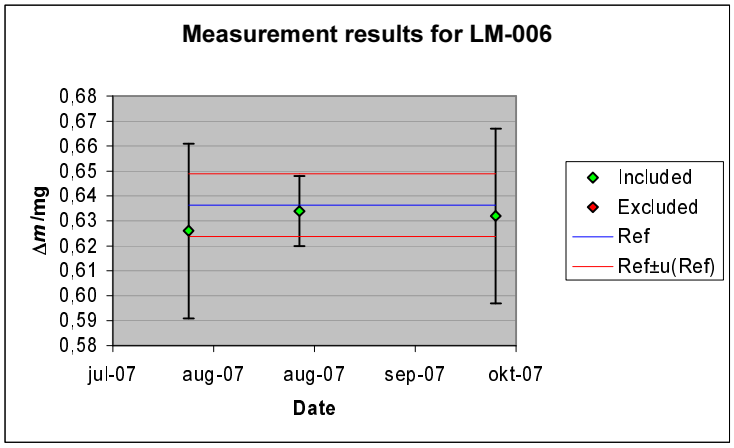

Figure 1: Measurement results for LM-006 (Δm and $u(\Delta m)$)

LM-006d

Table 2: Results of the comparison. Δm is the mass correction of the weight LM-006d measured by the specified participant at the specified date, Δm_{ref} is the corresponding reference value, and d is the normalized deviation defined as the difference $D = \Delta m - \Delta m_{ref}$ divided by the standard uncertainty of that difference. The last column indicates if a result is included in the calculation of the reference value or not (y=yes, n=no).

Meas. No. i	Lab ID	Date	**Results (LM-006d)** Δm [mg]	$u(\Delta m)$ [mg]	Reference value Δm_{ref} [mg]	$u(\Delta m_{ref})$ [mg]	Normalized deviation d	Included (y/n)
4	MIRS	01.08.2007	0,578	0,035	0,588	0,013	-0,3	y
5	DFM	23.08.2007	0,589	0,014	0,588	0,013	0,2	y
6	MIRS	01.10.2007	0,576	0,035	0,588	0,013	-0,4	y

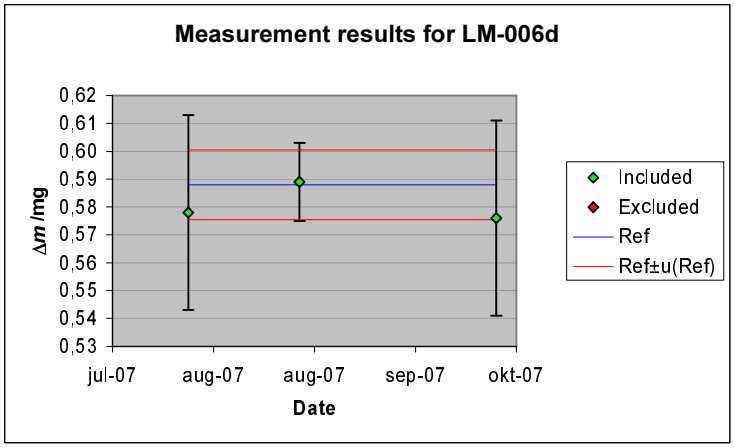

Figure 2: Measurement results for LM-006d (Δm and u(Δm))

The observed chi-square value of the least squares adjustment was χ^2_{obs} = 0,04 with ν = 4 degrees of freedom. It is therefore concluded, that the measurement results provided by the participants in this comparison are consistent taking into account the claimed uncertainties and correlation coefficients.

In order to identify potentially discrepant results [3], the normalized deviations are calculated:

$$d = \frac{\Delta m - \Delta m_{ref}}{u\left(\Delta m - \Delta m_{ref}\right)} \qquad (2)$$

A result is classified as discrepant at a 5% level of significance, if $|d| > 2$. The values of d shown in Table 1 and Table 2 and in Figure 3 indicate that no result is significantly discrepant.

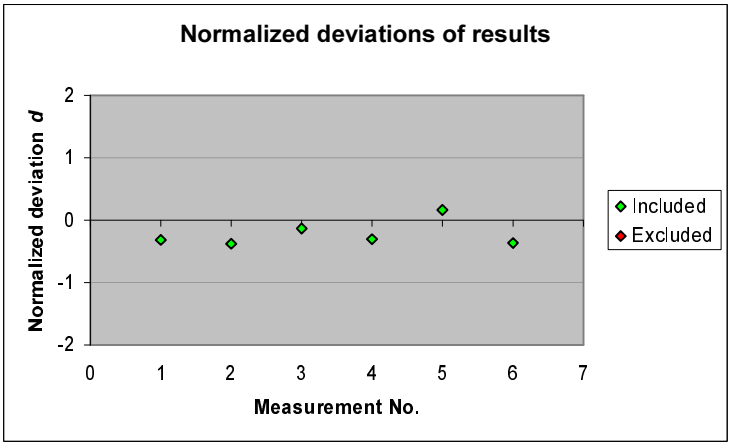

Figure 3: Normalized deviations d of results. Results with $|d| > 2$ are classified as discrepant results.

The degree of equivalence between the results and corresponding reference values are shown in Figure 4. Also this figure indicates that no result is significantly discrepant.

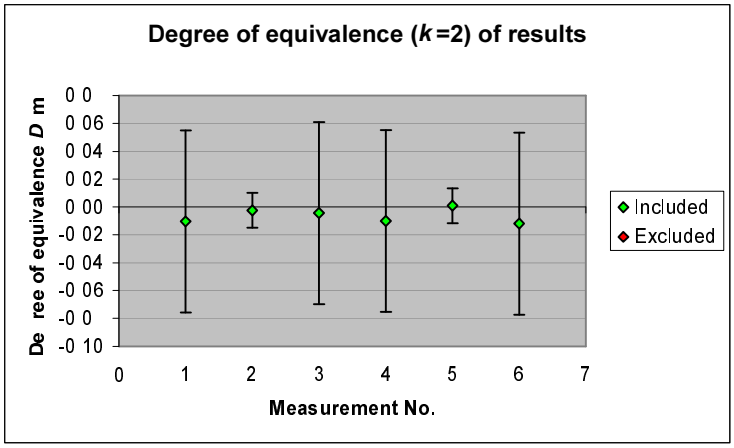

Figure 4: The degree of equivalence expressed as the deviation $D = \Delta m - \Delta m_{ref}$, and the expanded uncertainty ($k = 2$) of that deviation.

LINKED RESULTS

The results of this comparison has been linked to CCM.M-K1 through EUROMET.M.M-K4 using the method that was used to link EUROMET.M.M-K4 to CCM.M-K1, cf. ref [4], Appendix A. DFM acted as the linking laboratory; as basis for the linking, the degree of equivalence for DFM given in table 6 of ref. [4] was used:

D_{DFM} = 0.005 mg, $2u(D_{DFM})$ = 29 mg.

The correlation coefficients between D_{DFM} and the results provided by the participants in this bilateral comparison were all taken into account.

Table 3: Linked results of the comparison for mass standard LM-006. Δm is the mass correction of the weight LM-006 measured by the specified participant at the specified date; Δm_{ref} is the corresponding reference value.

Lab ID	Date	Result		Reference value	
		Δm	**$u(\Delta m)$**	Δm_{ref}	$u(\Delta m_{ref})$
	t	**[mg]**	**[mg]**	[mg]	[mg]
MIRS	1.8.2007	0,626	0,035	0,631	0,016
DFM	23.8.2007	0,634	0,014	0,631	0,016
MIRS	1.10.2007	0,632	0,035	0,631	0,016

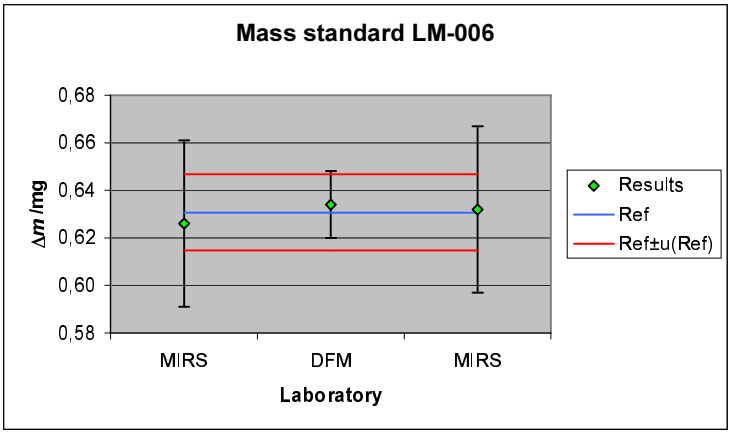

Figure 5: Linked results for mass standard LM-006

Table 4: Linked results of the comparison for mass standard LM-006d. Δm is the mass correction of the weight LM-006 measured by the specified participant at the specified date; Δm_{ref} is the corresponding reference value.

Lab ID	Date	Result		Reference value	
	t	**Δ*m*** **[mg]**	***u*(Δ*m*)** **[mg]**	Δm_{ref} [mg]	$u(\Delta m_{ref})$ [mg]
MIRS	01.08.2007	0,578	0,035	0,582	0,016
DFM	23.08.2007	0,589	0,014	0,582	0,016
MIRS	01.10.2007	0,576	0,035	0,582	0,016

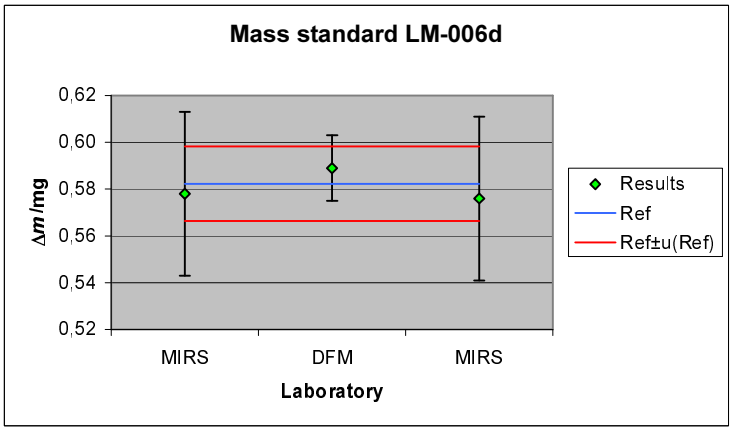

Figure 6: Linked results for mass standard LM-006d

Degrees of equivalence

	D_i [mg]	$2u(D_i)$ [mg]
DFM	0,005	0,029
MIRS	-0,004	0,073

Degrees of equivalence between pairs

	D_{ij} [mg]	$2u(D_{ij})$ [mg]
DFM-MIRS	0,009	0,058

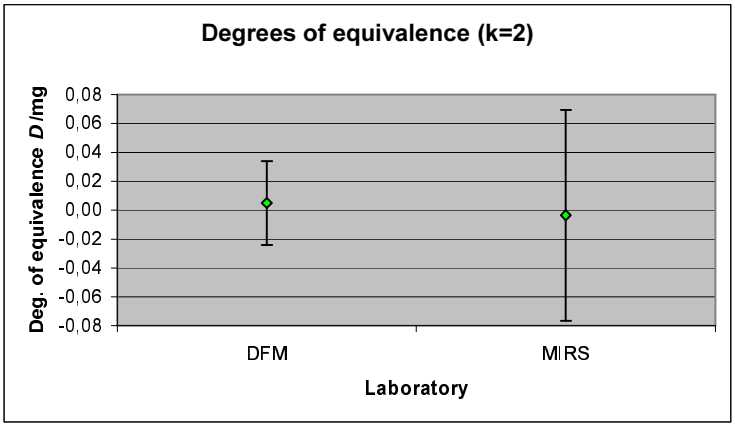

Figure 7: The degree of equivalence expressed as the deviation $D = \Delta m - \Delta m_{ref}$, and the expanded uncertainty ($k = 2$) of that deviation.

7. Conclusion

The results provided by participants in this bilateral comparison are consistent with each other and also with the key comparison reference values linked to the key comparison CCM.M-K1 through EUROMET.M.M-K4.

8. References

[1] L. Nielsen, Evaluation of the calibration history of a measurement standard, DFM-01-R5 (2001).

[2] L. Nielsen, Evaluation of measurement intercomparisons by the method of least squares, DFM-99-R39 (2001)

[3] L. Nielsen, Identification and handling of discrepant measurements in key comparisons, DFM-02-R28 (2002).

[4] M. Perkin, Report on EUROMET key comparison of 1 kg standards in stainless steel (EUROMET.M.M-K4)

Annex 1

Technical protocol for bilateral comparison of mass standards

Contents

1. Objectives

This technical protocol specifies the conditions of a bilateral comparison of a mass between DFM, Denmark, and MIRS, Slovenia. The objectives of the comparison are to facilitate the demonstration of metrological equivalence between the two participating national laboratories, and to check the capabilities of MIRS to improve quoted calibration measurement capabilities (CMC) at 1 kg. Both laboratories have participated in EUROMET 510 project (key comparison EUROMET.M.M-K4).

2. Participants

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3. Transfer standards

The transfer standards for this comparison comprise two stainless steel cylindrical weights with a knob (OIML R111 design). The weights are housed in individual wooden containers. The weights are designated LM-006 and LM-006d. They were produced by Mettler Toledo and purchased by MIRS in 1997. The volumes and centres of gravity of the standards and associated uncertainties are given as a part of the measurement report (Measurement report.doc).

4. Time schedule

Preliminary time schedule:

Measurements at MIRS, Ljubljana: July 2007
Transport to DFM, Lyngby:
Measurements at DFM, Lyngby: August 2007
Transport to MIRS, Ljubljana:
Measurements at MIRS, Ljubljana: September 2007
Draft report expected: October 2007

5. Transportation

The travelling standards are transported by courier between the participating laboratories. The standards are shipped in a plastic transport case. It is the responsibility of the participating laboratories to organize the transport to the next participant. No special custom procedures are required between Slovenia and Denmark.

6. Unpacking, handling and care of the standards

When the standards arrive at the participating laboratory, the transportation case and its contents should be checked for damage and missing items. A visual inspection of the surfaces of the standards should be made and the results noted on the measurement report. 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.

Every incident during handling of the transfer standards, where the standards may have been polluted or damaged, should be documented and communicated to the pilot laboratory as soon as possible. Also, the pilot laboratory should be informed about any delay or required change of the time schedule.

The standards should be stored at a place where they are protected from dust, aerosols and vapours all the time they are not in the balance, for example in their traveling containers or in a suitable clean environment protected from dust, drafts and vapour. If the weight is placed on a table, the table surface should be clean and covered by acid free tissue paper.

The transfer standards should be handled carefully and only ever with the appropriate tools. When being manipulated the weights should be handled with tongs or tweezers. The standards should never be touched with bare hands.

The standards should be wrapped in the acid free tissue paper provided before being put in their wooden containers.

7. Measurements

The participating laboratories shall determine the mass (not conventional value) of both standards according their normal calibration procedure. An appropriate time should be allowed for the stabilization of the weights following transportation (as specified in OIML R111 for class E1). Before the mass determination, dust particles should be removed from the surface of the standard by a clean, soft brush. No further washing should be performed. After visual inspection for dust particles on the surface, the weights should be placed in the weighing chamber of the comparator. All weighings should be performed in air. For the buoyancy correction, the air density should be determined using the laboratory's standard procedure, specified on the annexed form.

8. Reporting

A completed measurement report (the form "Measurement report.doc") for the measurement results, data of the ambient conditions, instruments used and traceability of the participant's reference standards must be submitted to the coordinator for the results within two weeks after the completion of the measurements. All collected measurement reports will be then sent to DFM, which will carry out the data analysis and also provide the link to EUROMET.M.M-K4.

The pilot laboratory will provide a first draft of the comparison report within four weeks after the receipt of the results of the data analysis.

9. Financial aspects and insurance

Each participating laboratories is responsible for its own costs for the measurements, transportation and travel insurance as well as any damage that may occur within its country.