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A COMPARISON BETWEEN SEVEN NATIONAL MEASUREMENT INSTITUTES TO EVALUATE INERT GAS TRANSFER OF MASS STANDARDS AGAINST CURRENT BEST PRACTICE

BERRY, J J (NPL), BENTOUATI, D (LNE), BORYS, M (PTB), HÖGSTRÖM, H (MIKES), KAÇMAZ, S (UME), MALENGO, A (INRIM), MARTI, K (METAS), MECKE, M (PTB), SILVESTRI, Z (CNAM), SNOPKO, L (SMU) AND ZŮDA, J (CMI)

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National
Measurement
System

A comparison between seven National Measurement Institutes to evaluate inert gas transfer of mass standards against current best practice

Berry, J J (NPL), Bentouati, D (LNE), Borys, M (PTB), Högström, R (MIKES), Kaçmaz, S (UME), Malengo, A (INRIM), Marti, K (METAS), Mecke, M (PTB), Silvestri, Z (CNAM), Snopko, L (SMU) and Zůda, J (CMI)

ABSTRACT

A study between seven National Measurement Institutes was undertaken to evaluate the benefit of transporting stainless steel standards between institutes in inert gas/vacuum compatible storage vessels compared with current comparison best practice. One transfer standard in each set circulated was also cleaned before the measurements to compare this approach with current best practice. The results showed significant correlation in the absolute mass values obtained for the standards with the institute at which they were measured, suggesting that the mass changes observed were principally due to the realisation of the mass scale at each institute. After normalising the results to reduce the influence of the reference mass values no significant differences were found between the different transport methods and in the case of one group of measurements current best practice demonstrated the best stability.

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National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

Approved on behalf of NPLML by
Stuart Davidson, (Science Area Leader - Mass).

CONTENTS

1 INTRODUCTION

It is anticipated that in the next few years the SI unit of mass, the kilogram, will be defined in relation to a fundamental physical constant of nature, namely the Planck constant (h), and will thenceforth be realised using a watt balance method [1] and an x-ray crystal density (XRCD) approach [2a, 2b]. This will replace the current realisation, which relates the kilogram to the mass of a physical artefact, the International Prototype Kilogram (IPK), manufactured from an alloy of platinum and iridium (Pt/Ir) [3]. At present the current definition of the kilogram is realised in air at ambient conditions of temperature, pressure and humidity and so in terms of improving mass stability there is little advantage in transferring mass artefacts between laboratories either under vacuum or in inert gas. In order to achieve the uncertainty required for redefinition both the watt balance and XRCD methods require the weighing of masses in a vacuum. Therefore the storage and transfer of artefacts in either vacuum or inert gas offers the potential to reduce uncertainty in the traceability chain back to the primary mass standard. It is critical to reduce the uncertainty in the traceability chain as the Consultative Committee for Mass and Related Quantities (CCM) has recommended that the standard uncertainty level required of the primary realisation experiments should be no larger than 2 parts in 10^8 [4] which is much larger than the estimated uncertainty of 3 parts in 10^9 calculated during the recent measurement of the IPK and its six official copies [5]. Gläser *et al* [6] discussed the impact of the redefinition of the kilogram and its higher realisation uncertainty of 2 parts in 10^8 as well as the increased uncertainties in the dissemination chain from the primary realisation experiments to the National Measurement Institutes (NMIs) working standards for calibrating customer masses. They found that providing the CCM requirements are met and NMIs closely control uncertainties in the traceability back to the primary mass standards, their customers that require the lowest uncertainty calibrations would still receive calibration uncertainties that do not affect the uncertainties of calibrations they perform in turn for their customers.

Research evaluating the stability of mass artefacts made from Pt/Ir and stainless steel has been studied in detail and this work has been summarised by Davidson [7]. Fen *et al* [8] have studied the stability of mass standards transported in air and although their results showed small mass losses in the transfer

standards their study suggested that with modest precautions against environmental extremes transport by air courier was not likely to result in unacceptable mass shifts. Schwartz [9] studied sorption phenomena in vacuum by means of gravimetric and ellipsometric measurements and found that the mass increased at a rate of 0.012 μg cm^{-2} day^{-1} and attributed this mass increase to oil particles originating from the bearings of the turbo-molecular vacuum pump used. Davidson [10] studied the medium-term stability of Pt/Ir artefacts stored in air and vacuum using gravimetric weighing and X-ray Photoelectron Spectroscopy (XPS), and found that the rate of contamination by hydrocarbons in air was similar to that found in vacuum. Fuchs *et al* [11] used XPS to study the accretion of carbon compounds on Pt/Ir artefacts and electroplated Au artefacts after the application of either oxygen plasma cleaning or hydrogen plasma cleaning. After cleaning the Pt/Ir and Au samples were stored in air, argon or vacuum and the amount of carbon compounds accreted on the surface was measured over a period of several years. They found similar levels of carbon compounds formed on the surface of the samples for all three storage media with the exception of the vacuum stored, oxygen plasma cleaned samples which formed higher levels of carbon contamination. Berry *et al* [12] compared the transfer and storage of Pt/Ir, stainless steel and silicon artefacts in air and argon. In the static cycling tests, where the artefacts remained on the comparator, they found little evidence of a contaminative mass gain either in air or in inert argon gas. This contrasted with the tests that transferred stainless steel and silicon artefacts between the comparator and storage vessels where significant mass gains were observed in the air transferred and stored artefacts compared with a small mass decrease in the argon transferred and stored artefacts.

The aim of this work was to develop and optimise protocols for the transfer of mass standards between laboratories with experiments (watt balance and XRCD) running under vacuum conditions or with vacuum mass comparators. The transfer standards were stainless steel kilogram mass standards which were calibrated under vacuum conditions at the participating NMIs. The transfer of the standards between the first group of participants was undertaken in both air and nitrogen to evaluate the benefits of inert gas transportation on weight stability and to identify any additional complications in using this transfer process. The transfer standards of the second group were transported in air and measured in vacuum. To allow comparison with current comparison best practice one of the in-air masses was

cleaned by each participant to investigate this comparison procedure as an effective protocol for future mass comparisons.

2 PARTICIPATING INSTITUTES AND DESCRIPTION OF THE TRANSFER STANDARDS

2.1 PARTICIPATING INSTITUTES

The United Kingdom's National Physical Laboratory (NPL) was the pilot laboratory and supplied the transfer standards. The comparison was split into two groups of participating NMIs and these participants are listed in Table 1. The NMIs in group 1 compared a transfer standard transported in nitrogen gas with current comparison best practice and the NMIs in group 2 compared a transfer standard transported in air with current comparison best practice.

Table 1. List of participating institutes

Group 1 participants	
National Physical Laboratory (NPL) (pilot laboratory)	United Kingdom
Federal Institute of Metrology (METAS)	Switzerland
Physikalisch-Technische Bundesanstalt (PTB)	Germany
Insituto Nazionale di Ricerca Metrologica (INRIM)	Italy
Group 2 participants	
National Physical Laboratory (NPL) (pilot laboratory)	United Kingdom
Turkiye Bilimsel ve Teknolojik Arastirma Kurumu (TUBITAK)	Turkey
Cesky Metrologicky Institut Brno (CMI)	Czech Republic
Laboratoire national de métrologie et d'essais (LNE)	France

2.2 DESCRIPTION OF THE TRANSFER STANDARDS

The mass standards used in this comparison were cylindrical and OIML [13] shape stainless steel kilogram standards and information on the standards is given in Table 2.

Table 2. Information on the transfer standards

Mass ID	Shape	Surface area / cm^2	Surface area uncertainty ($k = 1$) / cm^2	Volume / cm^3	Volume uncertainty ($k = 1$) / cm^3	Cubic coefficient of thermal expansion / x 10^{-6} °C^{-1}
			Group 1			
62D	Cylindrical	139.1	2.3	124.279 8	0.000 9	46
62DD	Cylindrical	139.1	2.3	124.279 8	0.000 8	46
62TD	Cylindrical	139.1	2.3	124.279 2	0.000 9	46
			Group 2			
56D	OIML	147.3	4.0	124.226 6	0.001 1	46.5
56DD	OIML	147.3	4.0	124.230 4	0.001 1	46.5
56TD	OIML	147.3	4.0	124.224 0	0.001 1	46.5

3 EXPERIMENTAL PROCEDURE

3.1 CLEANING PROCEDURE

All of the transfer standards were cleaned by the pilot laboratory prior to the start of the comparison using a two-stage cleaning process. The first cleaning stage involved washing the standards in an ultrasonic bath filled with reagent grade ethanol with a purity of at least 99% for a period of 5 minutes. The second stage involved rinsing the standards with distilled water for about 5 minutes with the standards inverted half way through to rinse the bottom surface. The standards were then left to dry in air with large droplets removed using lens tissue paper.

No further cleaning was performed on transfer standards 62D, 62DD, 56D and 56DD during the comparison. 62TD and 56TD were cleaned by each participant before the start of their measurements using the same procedure described above.

3.2 STORAGE AND TRANSFER PROCEDURE

Transfer standards 62DD, 62TD, 56DD and 56TD were stored and transported in air using wooden weight boxes as shown in Figure 1, used typically for transporting standards in conventional comparisons. Specially constructed vessels capable of storing artefacts in inert gas were used to transport the group 1 transfer standard identified as 62D and the group 2 standard identified as 56D as shown in Figure 2. The group 1 standard 62D was transferred in a dry nitrogen environment and was not exposed to air at any point during the comparison. The transfer of 62D from the storage vessel to a participant's vacuum mass comparator was achieved through the use of a glove box transfer device attached to the comparator. 56D was also transferred in an inert gas compatible vessel but instead of being filled with nitrogen its storage vessel was filled with ambient air. A summary of the storage, transfer and cleaning procedures for each of the transfer standards used by both group 1 and group 2 participants is given in Table 3.

Table 3. Storage, transfer and cleaning procedures for the mass standards.

Mass ID	Procedures
	Group 1
62D	Storage and transfer in dry nitrogen in a sealed vessel. Standard must be transferred from the storage vessel to vacuum without exposure to air (i.e. using a glove box transfer device).
62DD	Conventional air storage and transportation.
62TD	Conventional air storage and transportation. Standard cleaned using ethanol as a solvent in an ultrasonic bath by each NMI before the start of their measurements
	Group 2
56D	Storage and transfer in air in a sealed vessel.
56DD	Conventional air storage and transportation.
56TD	Conventional air storage and transportation. Standard cleaned using ethanol as a solvent in an ultrasonic bath by each NMI before the start of their measurements

Figure 1. Stainless steel kilogram transfer standard in a conventional wooden transfer box.

Figure 2. Stainless steel kilogram in an inert gas / vacuum compatible transfer vessel.

3.3 MEASUREMENT PROCEDURE

For each participant the transfer standards, along with the participant's own reference mass standard traceable to the International Prototype Kilogram (IPK), were transferred to the weighing chamber of the mass comparator and were then pumped down to vacuum (target pressure ~ 0.01 Pa). It is worth noting that in April 2015 the Bureau International des Poids et Mesures (BIPM) issued NMIs with revised certificate values for kilograms calibrated at the BIPM between 2003 and 2013 following the calibration campaign in 2014 of the official copies against the IPK [5]. The reference values used by the participants are traceable to the original certificate values supplied by the BIPM. Group 1 participants were required to transfer the nitrogen stored standard (62D) to vacuum in the weighing chamber via a nitrogen filled glove box and load-lock to ensure the standard was not exposed to air. After leaving the transfer standards to stabilise in vacuum for a period of 24 hours the mass differences between them were measured in addition to the mass differences between the transfer standards and each participant's reference standard for the purpose of determining the absolute mass in vacuum.

After completing the weighing in vacuum all the transfer standards were returned back to air with the exception of 62D which was transferred back to its nitrogen storage vessel using the nitrogen filled glove box and load lock.

4 RESULTS AND DISCUSSION

4.1 GROUP 1 RESULTS AND DISCUSSION

The measured mass differences between the transfer standards for group 1 with associated standard uncertainties are given in Table 4 and the absolute mass in vacuum of the transfer standards with associated standard uncertainties are given in Table 5. The mass difference between the mass measured by the participants in vacuum for the three transfer standards and the mass measured initially at NPL is shown graphically in Figure 3. The trend in the change in absolute mass in vacuum was similar for all three transfer standards. This suggested that the changes observed were more dependent on the mass scale realised at each NMI rather than the different transport techniques employed or the procedure used to clean one of the standards before the measurements. The difference

in mass values measured by NPL at the end of the comparison to the values measured at the start gave an indication of the drift of the transfer standards and suggested that the transfer standards had gained between 10 µg and 30 µg during the course of the comparison.

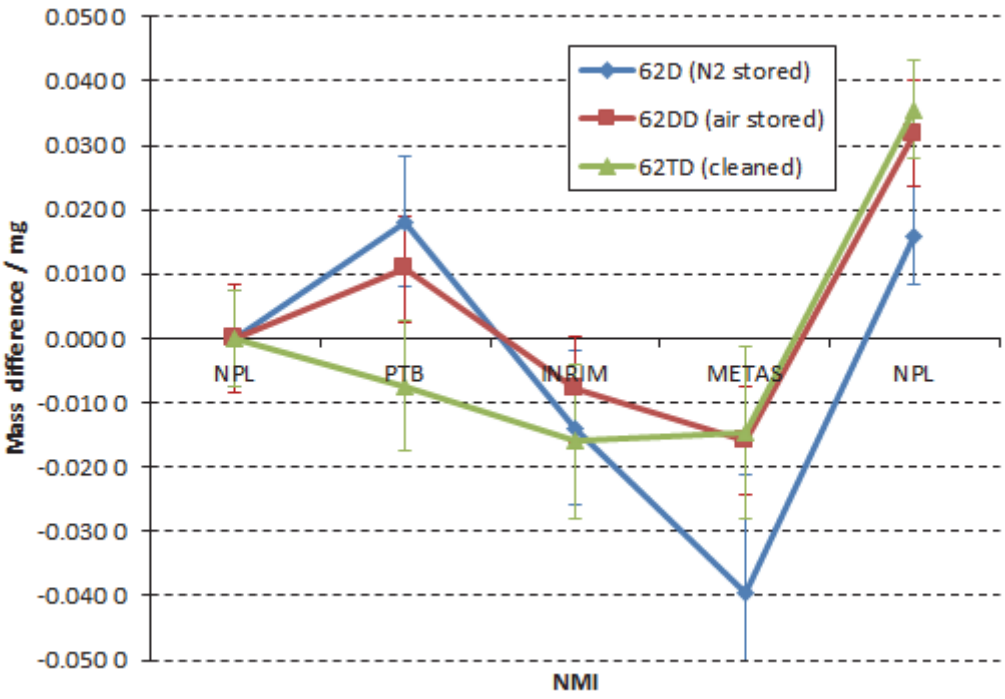

Figure 3. Mass difference in vacuum of the three group 1 transfer standards measured at four NMIs with respect to the initial mass at NPL. Error bars represent the standard uncertainty (k = 1).

To enable an examination of the differences between the storage/transfer techniques without the bias in the results caused by the influence of the reference mass value used at each NMI, the results have been normalised using the following formula:

$$M_N = M_X - (M_Y - M_Z)$$

Where: M_N = normalised mass in vacuum of the transfer standard (each NMI)

M_X = uncorrected mass in vacuum of the transfer standard (each NMI)

M_Y = average mass in vacuum of all the transfer standards (each NMI)

M_Z = average mass in vacuum of all the transfer standards (all NMIs)

The group 1 normalised mass values in vacuum for the transfer standards with standard uncertainties are given in Table 6 and the change in normalised mass in vacuum relative to the initial mass

measured at NPL is shown graphically in Figure 4. The standard uncertainties in the normalised mass values are assumed to be the same as those supplied by the participants for the original absolute mass values.

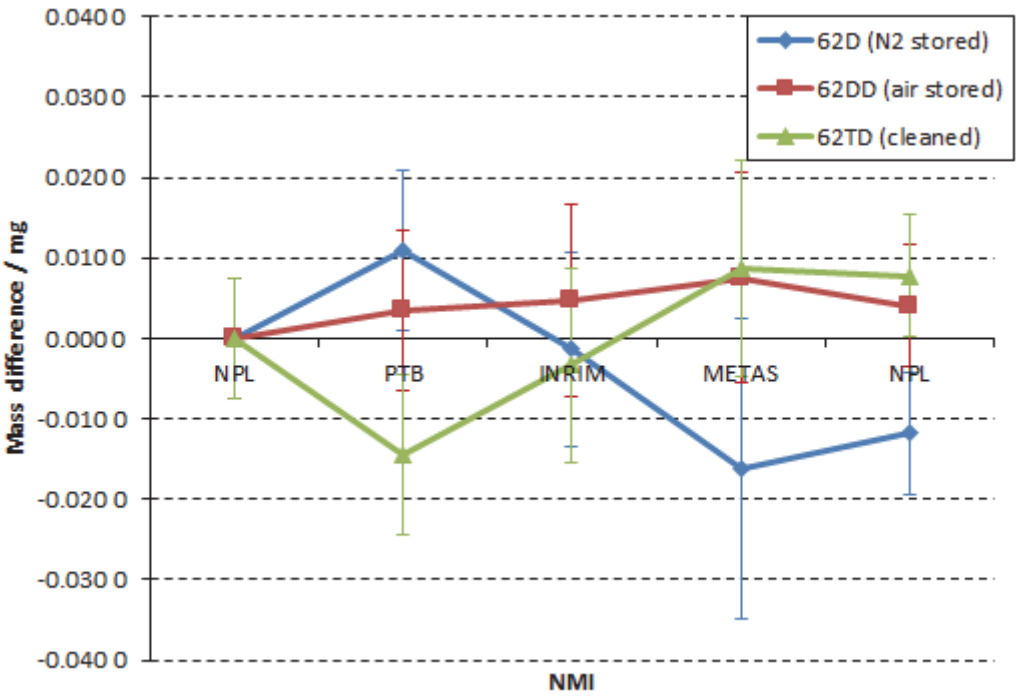

Figure 4. Mass difference in vacuum of the three group 1 transfer standards measured at four NMIs with respect to the initial mass at NPL. Mass values have been normalised to reduce the influence of variations caused by the participants' reference standard values. Error bars represent the standard uncertainty (k = 1).

The stability in the normalised mass values was typical of that occurring in other comparisons of stainless steel transfer standards such as the Consultative Committee for Mass and Related Quantities (CCM) key comparison of stainless steel standards [14]. Comparing the normalised mass values for the three transfer standards showed no advantage in either nitrogen storage or cleaning the standards before the measurements. In fact the standard transported conventionally in air (62DD) showed the best stability of all the transfer standards. A complementary study evaluating the stability of artefacts stored for 6-months in air, nitrogen and vacuum has also demonstrated that the air stored artefacts were the most stable [15]. This was attributed to either an increase in contamination from the extra manipulation involved in transferring the artefacts to storage in nitrogen or vacuum, or from the clamping materials used to secure the artefacts in the nitrogen/vacuum environment.

4.2 GROUP 2 RESULTS AND DISCUSSION

The group 2 measured mass differences between the transfer standards with associated standards uncertainties are given in Table 7 and the absolute mass in vacuum of the transfer standards with associated standard uncertainties are given in Table 8. The change in absolute mass in vacuum for the three transfer standards relative to the mass measured initially at NPL is shown graphically in Figure 5.

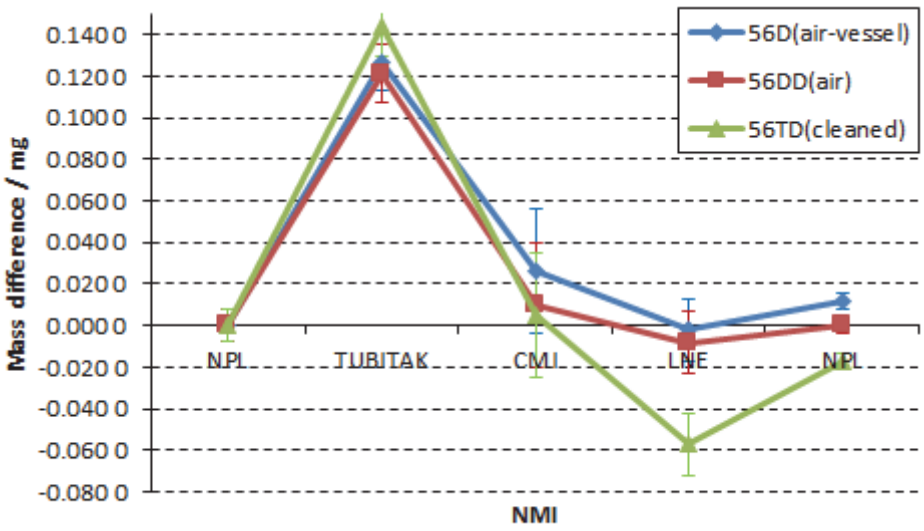

Figure 5. Mass difference in vacuum of the three group 2 transfer standards measured at four NMIs with respect to the initial mass at NPL. Error bars represent the standard uncertainty (k = 1).

The trend in the change in absolute mass in vacuum was similar for all three transfer standards. This suggests that the changes observed were due to the reference mass in vacuum value applied at each institute rather than differences between the transfer standards due to the different storage/transport techniques employed. This was similar to the trends observed in the group 1 data (§4.1) and suggests again that the changes observed were due to a correlation with the mass scale realised at each NMI rather than differences between the transfer standards due to the different storage/transport techniques employed.

To enable an examination of the differences between the storage/transfer techniques without the bias in the results caused by the influence of the mass scale of each NMI, the results have been normalised in the same way as performed for the group 1 results (§4.1). The normalised group 2 data is shown in Figure 6 and the stability in the normalised data is again similar to that found in similar comparisons

such as the CCM key comparison of stainless steel standards [14]. Comparing the normalised mass values of the three transfer standards showed no particular differences between the conventionally transported standard, the standard transported in the special sealed vessel and the standard cleaned before the measurements.

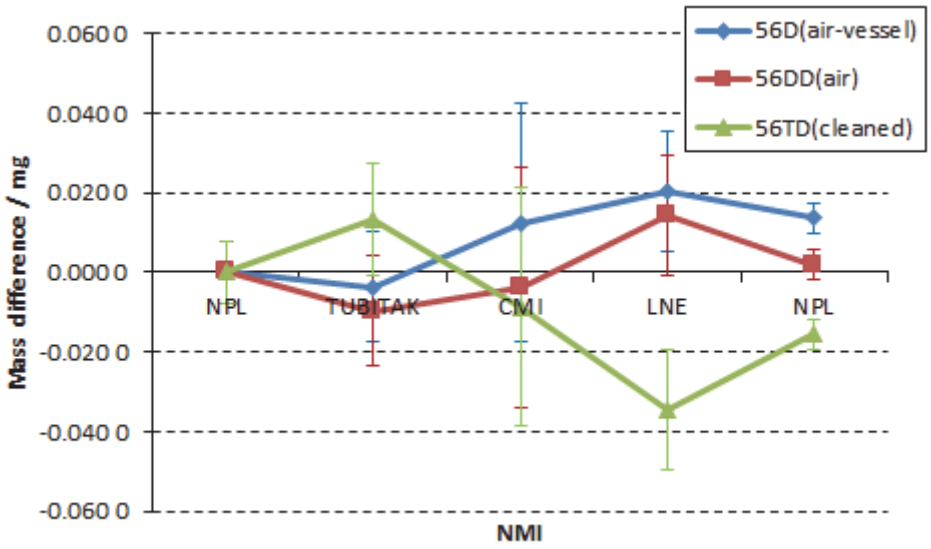

Figure 6. Mass difference in vacuum of the three group 2 transfer standards measured at four NMIs with respect to the initial mass at NPL. Mass values have been normalised to reduce the influence of variations caused by the participants' reference standard values. Error bars represent the standard uncertainty (k = 1).

5 CONCLUSIONS

Work has been undertaken to compare the transfer of stainless steel standards in inert gas/vacuum compatible storage vessels with standards transferred according to current comparison protocols. Two groups of participants performed measurements on two sets of stainless steel transfer standards. Group 1 compared a standard transported in nitrogen inside an inert gas storage vessel with one transported conventionally in air and Group 2 compared a standard stored and transported in air inside an inert gas storage vessel with one transported conventionally in air. An additional standard in each group was also transported conventionally in air but cleaned before the measurements at each participating NMI using an ultrasonic bath with ethanol as a solvent, to investigate whether this protocol offered improved consistency of results.

The trend in the change in absolute mass of all three transfer standards measured by both groups of participants was highly correlated with the participating NMI. This suggested that the differences in

the mass determinations of the transfer standards were mainly related to the mass scale realised at each NMI rather than any changes caused by the storage/transport techniques used or cleaning of one of the transfer standards before the measurements. The results from the participants were normalised in an attempt to remove the correlation in the results with the participating NMIs mass scale. After normalising the data the stability of the transfer standards during the comparison could be more easily seen. No significant improvement in mass stability was observed in the inert gas/vacuum vessel transferred steel standard and the standard that was cleaned before each measurement compared with the conventionally air transported standard. In fact in the group 1 measurements the conventionally air transported mass demonstrated the best stability. Therefore for the purpose of performing comparisons between NMIs the current best practice of transporting them in air in conventional boxes is still recommended.

6 ACKNOWLEDGEMENTS

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8 TABLES OF RESULTS

Table 4. Group 1 mass differences measured in vacuum between the transfer standards with associated standard uncertainties.

NMI	Date	Mass difference (62D-62DD) / mg	Standard uncertainty ($k = 1$) / mg	Mass difference (62D-62TD) / mg	Standard uncertainty ($k = 1$) / mg	Mass difference (62DD-62TD) / mg	Standard uncertainty ($k = 1$) / mg	Pressure / Pa
NPL	15-Jul-14	-0.309 8	0.000 5	-0.184 4	0.000 5	0.125 4	0.000 5	2.32E-3
PTB	10-Nov-14	-0.302 3	0.001 0	-0.158 8	0.001 0	0.143 5	0.001 0	5.00E-3
INRIM	18-Jan-15	-0.315 8	0.001 2	-0.182 4	0.001 2	0.133 4	0.001 2	1.00E-2
METAS	17-Mar-15	-0.328 2	0.000 2	-0.214 6	0.000 2	0.114 9	0.000 2	1.00E-6
NPL	11-Apr-15	-0.325 6	0.000 5	-0.203 9	0.000 5	0.121 8	0.000 5	1.33E-2

Table 5. Group 1 absolute mass measured in vacuum for the transfer standards with associated standard uncertainties.

NMI	Date	Absolute mass 62D (Nitrogen) / g	Standard uncertainty ($k = 1$) / mg	Absolute mass 62DD (air) / g	Standard uncertainty ($k = 1$) / mg	Absolute mass 62TD (cleaned) / g	Standard uncertainty ($k = 1$) / mg	Pressure / Pa
NPL	15-Jul-14	999.998 821	0.008	999.999 130	0.008	999.999 005	0.008	2.32E-3
PTB	10-Nov-14	999.998 839	0.010	999.999 141	0.010	999.998 998	0.010	5.00E-3
INRIM	18-Jan-15	999.998 807	0.012	999.999 122	0.012	999.998 989	0.012	1.00E-2
METAS	17-Mar-15	999.998 781	0.019	999.999 114	0.013	999.998 990	0.013	1.00E-6
NPL	11-Apr-15	999.998 838	0.008	999.999 162	0.008	999.999 040	0.008	1.33E-2
Standard deviation		0.000 024		0.000 018		0.000 021		

Table 6. Group 1 normalised mass values measured in vacuum for the transfer standards with associated standard uncertainties.

NMI	Date	Normalised mass 62D (Nitrogen) / g	Standard uncertainty ($k = 1$) / mg	Normalised mass 62DD (air) / g	Standard uncertainty ($k = 1$) / mg	Normalised mass 62TD (cleaned) / g	Standard uncertainty ($k = 1$) / mg	Pressure / Pa
NPL	15-Jul-14	999.998 820	0.008	999.999 130	0.008	999.999 005	0.008	2.32E-3
PTB	10-Nov-14	999.998 831	0.010	999.999 134	0.010	999.998 990	0.010	5.00E-3
INRIM	18-Jan-15	999.998 819	0.012	999.999 135	0.012	999.999 001	0.012	1.00E-2
METAS	17-Mar-15	999.998 804	0.019	999.999 138	0.013	999.999 013	0.013	1.00E-6
NPL	11-Apr-15	999.998 809	0.008	999.999 134	0.008	999.999 012	0.008	1.33E-2
Standard deviation		0.000 011		0.000 003		0.000 010		

Table 7. Group 2 mass differences measured in vacuum between the transfer standards with associated standard uncertainties.

NMI	Date	Mass difference (56D-56DD) / mg	Standard uncertainty ($k = 1$) / mg	Mass difference (56D-56TD) / mg	Standard uncertainty ($k = 1$) / mg	Mass difference (56DD-56TD) / mg	Standard uncertainty ($k = 1$) / mg	Pressure / Pa
NPL	16-Jun-14	0.015 2	0.000 5	-0.302 1	0.000 5	-0.317 3	0.000 5	1.89E-2
TUBITAK	23-Jan-15	0.021 9	0.000 3	-0.321 3	0.000 2	-0.345 7	0.000 3	1.00E-2
CMI	27-Mar-15	0.033 0	0.000 5	-0.281 0	0.000 5	-0.312 5	0.000 5	1.00E-3
LNE	03-Jun-15	0.021 7	0.000 7	-0.246 9	0.000 7	-0.268 4	0.000 7	5.27E-2
NPL	24-Jun-15	0.027 1	0.000 5	-0.273 0	0.000 5	-0.299 9	0.000 5	1.18E-2

Table 8. Group 2 absolute mass measured in vacuum for the transfer standards with associated standard uncertainties.

NMI	Date	Absolute mass 56D (air) / g	Standard uncertainty ($k = 1$) / mg	Absolute mass 56DD (air) / g	Standard uncertainty ($k = 1$) / mg	Absolute mass 56TD (cleaned) / g	Standard uncertainty ($k = 1$) / mg	Pressure / Pa
NPL	16-Jun-15	999.998 979	0.008	999.998 964	0.008	999.999 281	0.008	1.89E-2
TUBITAK	29-Jan-15	999.999 106	0.014	999.999 085	0.014	999.999 425	0.014	1.00E-2
CMI	27-Mar-15	999.999 006	0.030	999.998 974	0.030	999.999 287	0.030	1.00E-3
LNE	03-Jun-15	999.998 978	0.015	999.998 956	0.015	999.999 224	0.015	5.27E-2
NPL	24-Jun-15	999.998 991	0.008	999.998 964	0.008	999.999 264	0.008	1.18E-2
Standard deviation		0.000 058		0.000 052		0.000 076		

Table 9. Group 2 normalised mass values measured in vacuum for the transfer standards with associated standard uncertainties.

NMI	Date	Normalised mass 56D (air) / g	Standard uncertainty ($k = 1$) / mg	Normalised mass 56DD (air) / g	Standard uncertainty ($k = 1$) / mg	Normalised mass 56TD (cleaned) / g	Standard uncertainty ($k = 1$) / mg	Pressure / Pa
NPL	16-Jun-15	999.999 003	0.008	999.998 988	0.008	999.999 306	0.008	1.89E-2
TUBITAK	29-Jan-15	999.999 000	0.014	999.998 979	0.014	999.999 319	0.014	1.00E-2
CMI	27-Mar-15	999.999 016	0.030	999.998 984	0.030	999.999 297	0.030	1.00E-3
LNE	03-Jun-15	999.999 024	0.015	999.999 002	0.015	999.999 271	0.015	5.27E-2
NPL	24-Jun-15	999.999 017	0.008	999.998 990	0.008	999.999 290	0.008	1.18E-2
Standard deviation		0.000 008		0.000 018		0.000 015		