Interim report for Deliverable 4.3.3: Storage of primary mass standards to optimise stability.

1. Introduction

The aim of this task was to develop and optimise protocols for the medium-term storage of primary mass standards between apparatus (watt balance, kilogram comparator and Avogadro experiments) running under vacuum conditions.

Having constructed transfer and storage vessels based on the criteria identified in task 4.1, optimised protocols for the storage of mass standards were outlined and validated by a short-term (6-month) evaluation of the surface condition of weights stored following the protocols developed with weights stored in air. This report describes the progress made in meeting this deliverable up until the end of November 2014.

2. Participants and artefacts

A list of the participating laboratories is given in table 1.

Table 1: List of participating laboratories for deliverable 4.3.3

National Physical Laboratory	NPL
Laboratoire national de métrologie et d'essais	LNE
Physikalisch-Technische Bundesanstalt	PTB
Conservatoire national des arts et metiers	CNAM

Two stainless steel kilogram mass standards were selected by each participant for use in this 6 month storage evaluation; one for storage in air and one for storage in inert gas. Participants with surface analysis facilities also selected separate stainless steel samples for use in surface analysis measurements if the kilogram mass standards were too large to be accommodated by the surface analysis equipment. Each participant also selected a reference standard with a well-known mass gain for monitoring the change in mass of the two stainless steel kilograms.

3. Procedure for Deliverable 4.3.3

3.1 Initial cleaning of the artefacts

The two stainless steel kilograms were cleaned at the start of the measurements using one of the cleaning methods described in deliverable 4.2.3. The samples used in the surface analysis measurements were also be cleaned using this procedure and at the same time as the two kilograms.

3.2 Handling and storing the artefacts

Two pairs of gloves were worn when handling the artefacts. The first layer was either a pair of latex or nitrile gloves and the second (outer) layer consisted of a pair of special DuraCLEAN with Lycra gloves (as used in the NMIA silicon sphere cleaning method).

Any visible dust on the kilograms was removed using a soft brush.

Two storage vessels were required; one for storage in air and another for storage in inert gas (nitrogen).

3.3 Measurement procedure

The two stainless steel kilograms were compared with the reference standard before they are cleaned so that the mass loss due to cleaning could be calculated. Participants performing surface analysis measurements also performed these measurements on the surface samples before cleaning them.

After cleaning the two stainless steel kilograms were left to stabilise for 48 hours before weighing in air against the reference standard (if the kilograms were not stable after this time then a longer start delay may be necessary). The surface samples were analysed straight after cleaning. The two kilograms were then transferred into their respective storage vessels along with the respective surface samples. After sealing the storage vessels they were evacuated to remove the air within them and then filled with either dry air or dry nitrogen to a pressure above atmospheric pressure of about 110 000 Pa (i.e. 10% above atmospheric pressure). (Although it is not strictly necessary to evacuate the air storage vessel, since it already contains air, doing this will enable a fair comparison with the nitrogen stored artefacts).

The two stored kilograms and surface samples were then left within their respective storage vessels for a period of 6 months. The pressure within the storage vessels was monitored at regular intervals and additional gas was added so that the pressure stayed at about 10 % above atmospheric pressure.

At the end of the 6 month period the two kilograms should be transferred to a mass comparator and compared again in air with the reference standard after allowing 48 hours for them to

stabilise. Surface analysis on the surface samples should be done as soon as possible after removing them from the storage vessels.

4. Interim progress for T4.3.3 up until November 2014

4.1 NPL progress

NPL has built two inert gas / vacuum compatible storage vessels with a vacuum sight glass lid and two valves on the side of each vessel allowing connection to a gas supply or vacuum pump. The inserts for the storage vessels that secure the weights in place are manufactured from natural PEEK material. NPL has selected two OIML shaped stainless steel kilogram artefacts for evaluation in this trial and four stainless steel surface samples. Information on the stainless steel kilogram artefacts and stainless steel surface samples is given in Table 2.

Table 2. Information on the stainless steel artefacts used at NPL for Task 4.3.3

Type	Identification	Density / kg m^{-3}	Dimensions / mm
OIML shape kilogram artefacts	59 and 59D	7 855	Height = 57.96
Small samples for surface analysis	Coupon 2, 3, 4 and 5	8 000	Diameter = 13 Thickness = 1

The two OIML shaped kilograms identified as 59 and 59D were initially weighed in air against stainless steel standards traceable to the International Prototype Kilogram (IPK). They were then cleaned along with the four coupons using the same two stage cleaning process used in the sorption comparison for Task 2.2 [1]. This consisted of initial washing in 99.9 % ethanol in an ultrasonic bath for 5 minutes followed by rinsing under a jet of distilled water for a further 5 minutes. After leaving the artefacts in air to stabilise for 48 hours they were compared again with the reference standards and the four surface samples were analysed using X-ray Photoelectron Spectroscopy (XPS). One of the OIML shaped kilograms (59) and coupons 2 and 3 were placed within one of the storage vessels. The storage vessel was sealed, evacuated and then filled with filtered compressed air to a nominal pressure 10 % above atmospheric pressure. The other OIML shaped kilogram (59D) and coupons 4 and 5 were placed inside the other storage vessel. This storage vessel was also sealed, evacuated and then filled with filtered dry nitrogen to a nominal pressure 10 % above atmospheric pressure.

4.2 CNAM Progress

CNAM has performed the first part of the protocol by storing stainless steel artefacts in air and nitrogen during 6 months. Gravimetric measurements have also been performed. Final results expected in April 2015.

4.3 PTB Progress

PTB have measured the mass of their stainless steel kilogram artefacts before and after cleaning and have placed one of them in a storage vessel filled with dry air and another filled with dry nitrogen for a period of 6 months.

5. References

[1] Berry J, Borys M, Firlus M, Green R, Malengo M, Mecke M, Meury P-A and Züda J 2014 Analysis of the correlation of sorption coefficients to pressure *National Physical Laboratory* Report ENG 50