Ensuring the stability of primary mass standards during the implementation of the new definition of the kilogram

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Abstract. In November 2018 the General Conference on Weights and Measures (CGPM) will meet and are likely to ratify the revision of the international system of units (SI). This represents a major change to the way the base SI units are defined and realised, a major consequence being that the last of the definitions based on a material artefact, the international prototype of the kilogram, will be “retired” and the “new” kilogram will be realised in terms of the Planck constant via the Kibble balance or X-ray crystal density (XRCD) experiment. While the revision of the SI and the redefinition of the kilogram will almost certainly be endorsed by the CGPM there are issues around the current agreement of the Kibble balance and XRCD experiments. This will mean that in order to implement the kilogram redefinition a consensus value for the “new” kilogram will need to be adopted in order that a consistent value for the SI unit of mass be maintained. In order to maintain this consistency the storage, monitoring and use of current primary mass standards will be critical and this paper outlines ways in which the stability of artefact based mass standards can be optimised by careful storage and monitoring using surface analysis techniques and quartz crystal microbalance technology.

1. Introduction

The General Conference on Weights and Measures (CGPM) will meet in November 2018 to discuss potential revisions to the international system of units (SI). In the revised SI all units will be defined in terms of a set of seven reference constants, to be known as the “defining constants of the SI”. This will result in a simpler and more fundamental definition of the entire SI, and dispenses with the last of the definitions based on a material artefact – the international prototype of the kilogram which defined the SI unit of mass [1]. The “new” kilogram will be related to the Planck constant and can be realised via the X-ray crystal density (XRCD) experiment [2] by counting the atoms in a silicon sphere or via the Kibble balance [3] which realises the unit of mass with relation to quantum electrical phenomena.

In 2016 a pilot study was conducted by the BIPM comparing the Kibble balance and XRCD realisation experiments [4] which showed that the results of the experiments could be compared using conventional mass standards transferred in air between the participants. The results of the comparison demonstrated that, at that time, there was good equivalence between participants’ values for the kilogram generated for a fixed, agreed, value of the Planck constant. However, since the completion of the pilot study new values (for the Planck constant) have been published by most of the participants and there is now a significant discrepancy between results [5], most notably between the two experiments with the lowest uncertainties (the NRC Kibble balance and the XRCD experiment as...
realised by the PTB). For this reason, rather than further delay the redefinition of the kilogram, the BIPM Consultative Committee for Mass and Related Quantities (CCM) agreed that a “consensus value” for the new kilogram should be used after the redefinition until such time that “the CCM determines that the dispersion of the results from individual realization experiments is compatible with the uncertainties of the individual realizations”. The consensus value will be determined based on contributions from all realisation experiments and from the mass scale as maintained by the BIPM. This will ensure continuity in the absolute value of the SI unit of mass and agreement between NMIs disseminating the unit.

The current realisation experiments are complicated and time consuming to execute and so do not represent a means of continuous access to the new SI kilogram. In practice conventional mass artefacts will be relied upon for the short-term maintenance and dissemination of the mass scale, both at the BIPM or in individual NMIs. Thus the short-term stability of artefact mass standards is crucial for the continuity of the mass scale and for the comparison and integration of primary realisation experiments.

2. Continuing requirements for artefact mass standards

The revision of the SI represents a major change to the way the base units are defined and realised, a significant consequence being that the last of the definitions based on a material artefact, the international prototype of the kilogram, will be “retired” and the “new” kilogram will be realised in terms of the Planck constant via the Kibble balance or XRCD experiment. While the revision of the SI and the redefinition of the kilogram will almost certainly be endorsed by the CGPM the current agreement of the primary realisation experiments as reported to CODATA in 2017 [6] will be an issue meaning a consensus value will be used until agreement can be reached [7].

In order to be able to maintain and disseminate such a consensus value reliance will still be placed on artefact mass standards as a means to compare the primary realisation experiments and to provide access to the SI mass unit on a continuous basis. It is recognised that artefact standards stored in air gain mass at a rate of about 1 µg/year if carefully stored. The aim of the research presented in this paper is to investigate whether weights stored in inert gas are more stable and to implement means of monitoring mass stability without the need to remove the weight from the storage medium to weigh it.

3. Inert gas storage of primary mass standards

Figures 1 and 2 show one of the vessels designed by NPL to store primary, platinum-iridium and tungsten, mass standards in inert gas. The vessel is constructed from standard vacuum compatible components with bespoke PEEK inserts to support the standards. The apparatus is controlled by a laptop which reads the internal vessel pressure using a pressure sensor and operates a control valve to admit pure gas to the vessel. The control software allows the vessel at to be maintained at a pressure above ambient to ensure the purity of the storage medium. Control pressure of 1.2 bar (minimum) and 1.4 bar (maximum) have been chosen. The intended storage period for weights is between 1 year and 5 years.
4. Monitoring the surface condition of mass standards during storage

Figure 3 shows an aerial view of the internal construction of the storage vessel. Housed under the PEEK weight support is a quartz crystal microbalance (QCM) sensor. This is designed to provide real-time feedback on the depth of surface overlayer accretion during the inert gas storage. Also shown in the picture is the XPS coupon which is a disc of approximately 1 cm diameter manufactured from the same material, and to the same surface finish, as the mass standard being stored. X-ray photoelectron spectroscopy (XPS) measurements will be made on this coupon before and after the inert gas storage period to provide further information on both the depth and composition of the surface layers accreted during storage.
5. XPS and QCM calibration against gravimetric measurements
An issue with XPS and QCM measurements is realising traceability to the SI. Additional the model normally used to calculate film thickness for QCMs, the Sauerbrey equation, is only really valid for solid tightly bonded films so is not suitable for calculating typical contamination layers which would be accreted on mass standards during storage [8], which mostly consist of hydrocarbons [9]. A method of heating mineral oil to temperatures of about 150 °C in a sealed container and suspending XPS coupons, QCM crystals and mass standards in the container was devised to accelerate the contamination of the surfaces. Thus surface contamination could be applied in a controlled way and the changes in response of the QCM crystals and the overlayers as measured by XPS could be correlated with gravimetric measurements made on the mass standards. QCM crystals coated with aluminium and gold were tested and both had sensitivities of the order of 0.01 µg/cm³ meaning changes equivalent to about 0.5 µg on a platinum-iridium kilogram could be detected. Overlayer thickness can be estimated from XPS measurements with uncertainties of about ± 0.2 nm equivalent to about ± 0.3 µg on a platinum-iridium kilogram. XPS measurements also give additional information about the chemical composition of the surface contamination.

6. Conclusions
Following the redefinition of the kilogram a consensus value will be used for the unit of mass as realised by the Kibble balance and X-ray crystal density experiments at NMIs undertaking this work. In order to compare the various experiments and calculate the consensus value and to disseminate this value artefact standards will be relied on to make these comparisons and to maintain a stable unit of mass on an ongoing basis. For this reason the effective storage of mass standards to maintain temporal stability is vital.

NPL has developed a simple apparatus for the storage of mass standards in an inert gas environments, designed to improve the medium term (1 to 5 years) stability of these standards. The storage apparatus incorporates a quartz crystal microbalance for real-time monitoring of surface contamination and a facility to store XPS coupons. A method for the controlled contamination of surfaces has been devised and this has been used to characterise the response of QCM crystals and XPS measurements with respect to the SI via gravimetric measurements made on mass standards exposed to contamination at the same time. Thus the likely surface contamination of primary mass standards stored in inert gas can be characterised in real-time using a QCM and XPS measurements made before and after the storage period can be used to give information on the chemical composition of any accreted contamination layers.

7. References
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