



## Final Publishable JRP Summary for SIB05 NewKILO

### Developing a practical means of disseminating the redefined kilogram

#### Overview

In 2018 the kilogram will be redefined in terms of a natural constant rather than being defined by a physical object. The redefinition will be realised in a vacuum, but will have to be transferred and disseminated in in-air conditions. This project developed new primary mass standards, new methods to clean and monitor such mass standards, and procedures and equipment to transfer the mass standards between vacuum and non-vacuum conditions, in order to minimise any uncertainties introduced into the dissemination of mass after the redefinition. The results of this project will help ensure the redefinition is successful, and that the kilogram can be disseminated to National Measurement Institutes and end users with the highest degrees of accuracy achievable.

#### Need for the project

The kilogram, the SI base unit of mass upon which all mass measurements made globally are ultimately based, is currently defined by a platinum-iridium cylinder kept in Sèvres near Paris, known as the International Prototype Kilogram (IPK). Copies of the IPK have been distributed globally to disseminate the kilogram. In 2018 the definition will change, and the kilogram will be redefined in terms of a natural constant, rather than a physical artefact. The redefinition will fix the value of the Planck constant and either the Kibble balance approach can be used to realise the unit of mass in terms of the fixed Planck constant, or the X-ray crystal density approach can be used to realise the unit of mass via the Avogadro constant (and thus with relation to the fixed Planck constant).

Although the definition will no longer be based on a physical artefact, artefacts will still be used as transfer standards, to transfer the definition from the location in which it is realised to National Measurement Institutes (NMIs) around the world. To ensure the continuity of the mass scale following the redefinition, the redefined value must also be based on the IPK, i.e. the exact proportion of the Planck and/or Avogadro constant must be chosen that results in the same mass value as the IPK.

The IPK and its copies are currently stored under in-air conditions and most mass measurements are made in in-air conditions, however the redefinition will be realised in a vacuum (both the Kibble balance and X-ray crystal density approaches are performed in a vacuum). This means that the IPK must be transferred between in-air and vacuum conditions in order to fix the redefinition to the IPK, and that mass standards must be moved from vacuum to in-air conditions to disseminate the redefinition to NMIs. These different environmental conditions will influence the value of the standards, and will introduce a degree of inaccuracy and uncertainty into the process by which the kilogram is disseminated via physical standards. In addition, Kibble balances produce strong magnetic fields, so mass standards to be used with Kibble balances must be non-magnetic.

#### Scientific and technical objectives

The aim of this project was to develop the practical infrastructure and procedures to successfully implement the kilogram redefinition, in particular (i) to ensure the continuity of the mass unit between existing and new realisations of the kilogram by providing a means of accurately fixing the Planck (and Avogadro) constant with reference to the International Prototype Kilogram (IPK) and (ii) to develop the metrological infrastructure to enable the dissemination of the new realisation of the kilogram at the NMI level with uncertainty contributions of less than two hundred millionths of a kilogram ( $u_{\text{rel}} < 2 \times 10^{-8}$  kg at  $k=1$ ). The following objectives were set:

1. Develop and evaluate artefacts suitable for the determination of the Planck and the Avogadro constants to provide traceability to the IPK and for the maintenance and dissemination of a redefined kilogram.

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2. Provide appropriate procedures and apparatus for the mass transfer between in-vacuum experiments (Kibble balance apparatus and vacuum mass comparators) and to in-air experiments (comparison with the IPK and dissemination of the unit to end-users).
3. Develop and adapt surface analysis techniques e.g. X-Ray Photoelectron Spectroscopy, (XPS), Ellipsometry, Contact Angle Spectrometry, (CAS) and overlayer models for the accretion of contamination on mass standards (including silicon spheres).
4. Evaluate the mass stability of suitably stored mass artefacts and develop the metrological infrastructure for the (medium term) maintenance of the mass unit and its dissemination based on different realisations (via a pool of artefacts which may be held at a number of NMIs and key comparisons).
5. Develop and validate methods to allow the reproducible cleaning (to less than 5 µg) of primary mass standards, including optimisation of non-contact cleaning techniques such as the use of UV activated ozone and gas plasma techniques.
6. Identify and evaluate the uncertainty components inherent in the *mise-en-pratique* and their propagation through the dissemination chain for the kilogram and its multiples and sub-multiples.

## Results

### 1. Develop and evaluate artefacts suitable for the determination of the Planck and the Avogadro constants to provide traceability to the IPK and for the maintenance and dissemination of a redefined kilogram.

Potential materials for next generation mass standards need to be suitable for use in a vacuum and in high magnetic fields (both of which occur in the Kibble balance), and should offer mass stability at least equal to the current IPK and its copies.

The mechanical and magnetic properties of a range of materials were evaluated, including iridium, mono- and poly-crystalline tungsten, U720 nickel alloy, a tertiary alloy of gold and platinum, gold and rhodium plated copper, together with the three materials currently used for mass standards (platinum-iridium, stainless steel and silicon).

Tungsten proved to have suitable mechanical and magnetic properties, and surface finishes better than can be achieved with the current standards. Pure iridium samples also exhibited a good surface finish and had low magnetic susceptibility compared with platinum iridium alloy, however, its hardness makes it very difficult and time consuming to polish, and it is difficult to obtain high-quality pure iridium. The other materials investigated offered more limited benefits, therefore next generation mass standards were manufactured from poly-crystalline tungsten and pure iridium.

### 2. Provide appropriate procedures and apparatus for the mass transfer between in-vacuum experiments (Kibble balance apparatus and vacuum mass comparators) and to in-air experiments (comparison with the IPK and dissemination of the unit to end-users).

The redefined kilogram will be realised experimentally under vacuum conditions, and the primary mass standards stored at National Measurement Institutes are also likely to be stored under vacuum conditions. However, the current mass standards are not stored in a vacuum, and most mass measurements are made in air conditions. To ensure that mass measurements can be reliably traced back to the redefined kilogram and to mass standards stored and used in vacuum, and to ensure that the new definition can be linked to the IPK, methods are needed that allow mass measurements made in vacuum and air conditions to be accurately and reliably compared.

Optimised procedures and techniques for the transfer of mass artefacts between vacuum and air were developed, with the aim minimising the measurement uncertainty introduced.

- Direct transfer of masses between air and vacuum was shown to be the most practical procedure. The inclusion of an intermediate nitrogen stage did not significantly improve the repeatability of the transfer or the stability of the transfer standard, and added to the complexity of the transfer process.
- Transfer of weights stored in nitrogen directly to vacuum (without exposure to air, using a glove box) provided the most repeatable results in terms of sorption (contamination), and the best weight stability.

- The influences of vacuum, load-lock apparatus, and glove boxes on mass standard surface sorption was evaluated for seven different vacuum balance assemblies at five National Measurement Institutes – with no apparatus-dependent effects detected.
- The effect of vacuum pressure on the surface desorption (loss of material) from mass standards (relative to values in air), was also assessed. Over the pressure range 0.1 Pa to 0.001 Pa no change in desorption was observed and so this vacuum pressure range is recommended for realising and disseminating the redefinition.
- The use of sorption artefacts (with different surface areas) is recommended to determine the sorption effects on the mass standards in real time. The artefacts are transferred along with primary mass standards of the same material, and provide an accurate measure of any sorption that occurs.
- A protocol for the transfer of weights under inert gas was evaluated for the first time by a comparison of stainless steel masses weighed in vacuum, carried out by seven NMIs. The masses were transported either under inert gas, using enclosures designed and manufactured in the project, or else conventionally in air according to current comparison protocols. The results showed no discernible advantage in transporting the weights in inert gas compared to in air, and interestingly, some of the air transported weights showed the best stability.

3. Develop and adapt surface analysis techniques e.g. X-Ray Photoelectron Spectroscopy, (XPS), Ellipsometry, Contact Angle Spectrometry, (CAS) and overlayer models for the accretion of contamination on mass standards (including silicon spheres).

Complementary surface analysis techniques (X-ray photoelectron spectroscopy, X-ray reflectance, thermal desorption, spectral ellipsometry and atomic force microscopy) were successfully used to characterise the surfaces of samples and mass standards, to measure residual contamination from manufacturing, and to monitor surface contamination during cleaning and storage. An ellipsometry vacuum cell was also constructed to enable real time interrogation of surfaces under dynamic pressure conditions to allow the detailed modelling of surface desorption process for a range of substrates. Quartz crystal microbalance technology was also used to monitor the reversible sorption effects of transfer between air and vacuum and the short term recontamination of surfaces, stored in different media, after (non-contact) cleaning with UV/Ozone and plasm techniques. An extensive report is available detailing the use of these surface analysis techniques in particular to monitor the recontamination of cleaned surfaces under various storage conditions.

4. Evaluate the mass stability of suitably stored mass artefacts and develop the metrological infrastructure for the (medium term) maintenance of the mass unit and its dissemination based on different realisations (via a pool of artefacts which may be held at a number of NMIs and key comparisons).

The IPK and its copies are currently stored in air, but storage in a vacuum or in an inert gas, such as nitrogen, was investigated to determine whether it might improve the stability of physical mass standards.

Storage in air, in vacuum and in nitrogen was assessed. Weighing and surface analysis by X-ray photoelectron spectroscopy were used to monitor the changes in the mass and the depth of the contamination overlayer. After cleaning, nitrogen showed a slight advantage over air storage, with the overlayer taking longer to form. Vacuum storage showed the most rapid and least predicable accretion of the overlayer. Therefore storage in nitrogen is recommended if mass standards are to be stored for long periods (at least 6 months), but storage in air is adequate for shorter periods as it provides improved ease of access to the standards.

5. Develop and validate methods to allow the reproducible cleaning (to less than 5 µg) of primary mass standards such as the use of UV activated ozone and gas plasma techniques.

The accretion of contaminants on the surface of mass standards over time is a key factor affecting their stability, and it is necessary to periodically remove the contaminants by cleaning.

The efficacy of current contact cleaning methods depends on and varies with user skill. The project developed and evaluated alternative non-contact cleaning techniques using both UV-activated ozone and low-pressure oxygen and hydrogen plasma. These methods were implemented and trialled by project partner institutes.

Results from the new cleaning techniques revealed significant differences between institutes, highlighting the necessity for careful implementation of the apparatus and procedures. When used at the Swiss NMI (METAS)

and UK NMI (NPL) the plasma and UV/ozone techniques respectively have been shown to give very repeatable and effective results. The results from the participants also confirmed the variability of the current manual cleaning techniques. Further research in this area is recommended.

6. Identify and evaluate the uncertainty components inherent in the *mise-en-pratique* and their propagation through the dissemination chain for the kilogram and its multiples and sub-multiples.

Rigorous uncertainty analysis has been carried out on the processes that will be involved in the dissemination of the redefined kilogram, most notably the additional vacuum to air transfer stage. The way in which surface sorption corrections are measured and applied to this stage has been evaluated and guidance has been published on issues affecting the calculation of the associated uncertainty.

### Actual and potential impact

#### Dissemination of results

Project results and recommendations were shared with stakeholders both directly and via a workshop held in Sarajevo in 2015. The workshop was attended by over 60 participants from European NMIs, academia and industry, including major weight and mass balance manufacturers, such as Mettler Toledo, Sartorius, and Häfner. In particular, advice was given to weight manufacturers on the suitability of new materials for next generation mass standards, to balance manufacturers on transfer and storage apparatus, and to the BIPM on weight storage and transfer for their ensemble of mass standards, and for a pilot study comparing primary realisation experiments. Results have also informed the BIPM's development of the written *mise en pratique* for the redefined kilogram, the instructions that will allow the redefinition to be realised in practice in NMIs.

20 peer reviewed papers have already been published, together with 4 articles in trade journals or popular media, and a further 3 articles published on external websites. 11 presentations have been given at key international conferences and a further 20 presentations at external seminars and workshops. Presentations at two EURAMET CCM meetings have shared the project's results with non-European NMIs, and have made them aware of the likely impact on the kilogram redefinition. The paper "Preparations for the Forthcoming Redefinition of the Kilogram" was awarded the Worshipful Company of Scientific Instrument Makers' Prize for the best paper published by the Institute in 2014 on the development or application of scientific instrumentation. 2 good practice guides on weighing in vacuum and the storage of primary mass standards have been published, summarising the output of the project for end users in the NMI mass community, and for weight and balance manufacturers. Additionally a Guide to the impact of the redefinition of the kilogram for end users has been made available on the project website.

#### Impact on the measurement science, scientific and industrial communities

Next generation mass standards have been developed, with high-quality surfaces, low magnetic susceptibility, and good stability, suitable for dissemination of the redefined kilogram. Poly-crystalline tungsten mass standards of 100 g, 500 g and 1 kg have been produced by the weight manufacturer Häfner, and by the UK's National Physical Laboratory and the National Research Council of Canada, and have already been provided to the BIPM for use in their Kibble balance experiments. The French NMI, LNE, have produced pure iridium mass standards which will be used in the future dissemination of mass in France. Tungsten has been recommended as a suitable material for mass standards and is likely to be used more widely in the future.

The recommendations for an optimum operating pressure range for Kibble balances and vacuum mass standards, and the optimised protocols for the transfer of standards from vacuum to air, will improve repeatability and minimise uncertainties in the dissemination of the kilogram. The protocol for transfer of weights under inert gas and the first comparison of 'weighing in vacuum' can be used as the basis for future 'mass in vacuum' comparisons such as the pilot study being organised by the BIPM for the comparison of primary realisation experiments. The universally compatible mass storage and transfer enclosures and protocols developed in the project have allowed comparisons to be undertaken with mass standards transported under inert gas, and will improve and add flexibility to the design of the next generation of Kibble balances and vacuum mass comparators. The report on vacuum mass comparators (and associated equipment) currently in use by NMIs and the design of the enclosures has already been used by a major balance manufacturer Mettler-Toledo to improve their design of equipment for the storage and transport of masses in vacuum. Recommendations on weighing in vacuum are now available in a Good Practice Guide.

The use of a range of complementary techniques for characterising the surfaces of mass standards, and for determining the evolution of contaminant overlayers, can be used in the future to predict drift in the mass of standards without the need for direct weighing. Such measurements could be used, for example, to corroborate unexpected changes in the values of primary mass standards which will be beneficial in the future maintenance and dissemination of the mass scale (the importance of this development is demonstrated by the recent issues with the maintenance of the current mass scale and the significant changes in the scale seen at last year's *Extraordinary Calibrations* using the International Prototype Kilogram undertaken at the BIPM).

Real-time surface sorption measurements under dynamic pressure conditions are now possible using a specially developed density ellipsometry cell and using quartz crystal micro-balance (QCM) technology. The QCM was also used to evaluate the recontamination of mass standards, stored in different media after cleaning. These measurements have provided further insight into the mechanisms which cause changes in the values of mass standards and enabled recommendations to be made on transfer and storage processes.

Apparatus to implement new non-contact cleaning techniques has been developed by a number of project partners. The manner in which these cleaning methods are implemented affects the quality of the results, and precise guidance on constructing and commissioning of UV/ozone and H-plasma cleaning apparatus has been provided. Optimisation of the use of these techniques will lead to more repeatable cleaning of primary mass standards, which will ensure consistency in the way in which the redefined kilogram is disseminated by NMIs.

Rigorous uncertainty analysis has been carried out on the processes that will be involved in the dissemination of the redefined kilogram, most notably the additional vacuum to air transfer stage. The way in which surface sorption corrections are measured and applied to this stage has been evaluated and guidance given on issues affecting the calculation of the associated uncertainty. The results showed that standards can be transferred between vacuum and air with a degree of uncertainty which supports the overall uncertainty target for dissemination of the new realisation of the kilogram at NMI level.

#### Potential future impact: Redefining the kilogram.

This project has made significant contributions to the redefinition of the kilogram in 2018. The project has developed a practical means of comparing the proposed realisation against the existing mass standards, ensuring continuity in mass measurements by linking the IPK to the Planck and Avogadro constants. The project has also developed methods to support the dissemination of the redefinition, and will enable a seamless transition, invisible to end users in industry. Ultimately, the redefinition of the kilogram will provide primary standards with enhanced accuracy, capable of supporting every finer mass measurements, vital to the development of new technologies in fields including quantum computing, nanotechnology and biotechnology.

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