kilogram NOW
Realization of the awaited definition of the kilogram

# Final Publishable JRP Summary Report for SIB03 kNOW
# Realisation of the awaited definition of the kilogram

## Overview

In 2018 the method used to define the kilogram will change. One kilogram will be defined as a ratio to Planck's constant, rather than by using a physical object, to ensure the definition can support future scientific and industrial innovation. This requires an accurate determination of Planck's constant. In 2011 two methods were used to calculate Planck's constant, but results were inconsistent and not sufficiently accurate for the redefinition. This project refined and improved the two approaches, to increase the accuracy to which they can state Planck's constant, to ensure their results are consistent, and ultimately, to ensure they can be used for the redefinition.

## Need for the project

The seven SI base units are the fundamental units of measurement from which all other measurement units are derived. They must be defined accurately, and that definition must be accessible, to ensure that measurements made throughout the world are consistent and reliable. The base units underpin all scientific and industrial measurements, and increases in the accuracy of their definition support the development of innovative products and services. For instance, a range of European industries are investing in nanotechnology to develop new products – the increasingly accurate definition of the base units ensures that reliable nano-scale measurements can be made, supporting nanotech innovation.

However, the current definition of the kilogram, the SI base unit of mass, is not capable of supporting future increases in definition accuracy, and is not sufficiently accessible. The mass of one kilogram is currently defined as the mass of the International Prototype Kilogram (IPK), a platinum-iridium cylinder kept in Paris. Although copies of the IPK have been distributed globally, access to the primary definition is limited to only those laboratories that have a copy. In addition, the mass of the IPK and its copies fluctuates over time as atoms are lost and/or adhere to their surfaces. The current definition of the kilogram is not accessible and not accurate enough to support future scientific and industrial innovation.

*Figure 1: The UK's copy of the International Prototype Kilogram (shown in protective casing)*

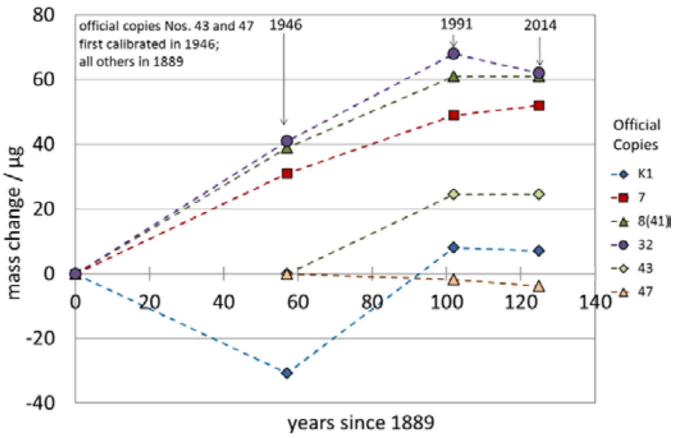


*Figure 2: Fluctuations in mass differences between the International Prototype Kilogram and its copies*

In 2011 it was decided the kilogram would be redefined in terms of Planck's constant (*h*), a naturally occurring value that can be defined to a high-degree of accuracy (Planck's constant is a measurement of energy, and energy can ultimately be related to mass by Einstein's equation $E = mc^2$). Such a definition



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would be accessible through experimentation and would support future refinements in definition accuracy. A standard laboratory method is therefore needed to determine *h*, and two approaches are under consideration, a watt-balance method and an atom-counting method. In 2011 three experiments were conducted to define *h,* two using the watt-balance approach, the other using the atom-counting method, but the results were inconsistent and not sufficiently accurate for the redefinition.

**Scientific and technical objectives**

The overall goal of this project was to support the redefinition of the kilogram in 2018 by improving the accuracy and certainty of the watt-balance and atom-counting measurements of Planck's constant. Objectives 1 and 2 refined and reduced the uncertainty of the watt-balance method, whilst objectives 3 and 4 did the same for the atom-counting approach. Objective 5 examined sources of uncertainty unique to each approach that could cause inconsistent results. Objective 6 ensured both approaches were calibrated to the IPK. Objective 7 used the results of the previous objectives to improve the consistency of the results of each approach. Objective 8 ensured the project's achievements were shared with both specialist and non-specialist audiences.

1. To fine-tune and adjust the European watt-balances to their maximum performance level
2. To carry out *h* measurements with $5\times10^{-8}$ relative uncertainty via independent watt-balance experiments
3. To challenge atom-counting experiments and confirm the measured $N_A$ value and its uncertainty or identifying error/s
4. To repeat the $N_A$ determination by simultaneously reducing the relative uncertainty to $1.5\times10^{-8}$
5. To investigate, understand, and possibly eliminate, the inconsistency between the determinations of *h* and $N_A$
6. To carry out *h* and $N_A$ determinations with comparisons traceable to the international prototype of the kilogram. This will also allow the possibility to design and begin a long-term experiment aimed at monitoring IPK stability
7. To demonstrate consistent kilogram realisations based on both the *h* and $N_A$ determination
8. To deliver the project's results to relevant metrology bodies, to disseminate them in the metrological and scientific communities, and in the wider, non-specialist, audience.

**Results**

**The watt-balance approach**

A watt-balance operates like a set of balance scales, with a one kilogram mass at the end of one arm (calibrated against the IPK), and a current-carrying coil of wire within a magnetic field at the other. The strength of the electromagnetic force generated by the interaction of the current and the magnetic field can be manipulated until it balances exactly with the kilogram mass. This value can then be related mathematically to Planck's constant.

<u>1. To fine-tune and adjust the European watt-balances to their maximum performance level</u>

To be used for the redefinition of the kilogram, a watt-balance must be able to measure *h* to an uncertainty of at least $5\times10^{-8}$ (to a five hundred millionth). Previously, in 2011, a European watt balance at the Swiss National Measurement Institute (METAS) managed an uncertainty of $3\times10^{-7}$, revealing the ultimate limit of its technology. At the project start, a second watt-balance experiment was under way at the French National Measurement Institute (LNE). Operating in test mode, it achieved an accuracy of $5\times10^{-5}$. The design and use of watt-balances therefore needs to be further refined to enable more accurate measurements.

All of the critical components of the METAS watt-balance were redesigned with the support of the Laboratoire de Systèmes Robotiques of the École Polytechnique Fédérale de Lausanne, CERN, and Mettler-Toledo. The production, assembly, and testing of the components was completed. In parallel, the LNE work focused of fine tuning of the watt balance performance. In both the experiments, problems were pinpointed, and solved through redesigning and replacing inadequate components.

2. To carry out $h$ measurements with $5\times10^{-8}$ relative uncertainty via independent watt-balance experiments

This objective has not been fully reached. The redesign and replacement of the inadequate components, and the testing of the new ones, delayed the balances' operation and prevented the measurement of the Planck's constant to the required $5\times10^{-8}$ relative uncertainty. The best measurement was completed at LNE in December 2014, with a relative uncertainty of $31 \times 10^{-8}$, still six times outside the required uncertainty. The LNE carried out an additional measurement campaign in March 2016 (outside of this project); the data analysis is currently under way.

**The atom-counting approach**

Avogadro's constant ($N_A$), the number of atoms/molecules in one mole of a substance, can also be related mathematically to Planck's constant, so an accurate definition of $N_A$ would support an accurate definition of $h$. In 2011, the International Avogadro Coordination (IAC) constructed two, pure, one kilogram silicon-28 spheres. High-precision techniques were used to determine volume and atomic structure of the spheres, so that the number of atoms within the spheres can be counted extremely accurately, and an exact measure of $N_A$ can be derived. However, when converted into the value of $h$, this result disagreed with the most accurate watt-balance value, obtained by the National Institute of Standard and Technology (USA). Therefore, these spheres were made available to the project for further investigation.

3. To challenge atom-counting experiments and confirm the measured $N_A$ value and its uncertainty or identifying error/s

As with the watt-balance approach, the design and performance of the atom-counting method needed to be refined to achieve the necessary reduction in uncertainty for measurements of $N_A$ and to investigate the inconsistency with the $h$ measurement.

The measurement of $N_A$ was improved by pushing all the techniques involved to their limits. The two silicon spheres were re-polished and measured anew. To estimate the surface effect on the measurements of the spheres, Cagliari University carried out density functional calculations of the strain and stress of silicon surfaces. The Leibniz Institute of Surface Modification and the German National Measurement Institute (PTB) developed ion-beam technologies and interferometers to re-measure the surfaces of the spheres. Molar mass measurements were repeated by substituting tetramethylammonium hydroxide for sodium hydroxide as the silicon solvent and diluent. In addition, the molar fraction of the $^{30}Si$ isotope (a potential impurity introduced by contamination by a heavier form of silicon) was confirmed by nuclear activation. The material purity was then verified experimentally with respect to a large number of contaminating elements by nuclear activation analysis with the Australian National Nuclear Research and Development Organisation. The optical interferometer used to measure the silicon lattice parameter was totally rebuilt, using different optical components and a different wavelength laser. The objective was achieved, as the project demonstrated that the counting of silicon atoms to the required level of accuracy is possible, and that no error was made in this respect in the 2011 experiment.

4. To repeat the $N_A$ determination by simultaneously reducing the relative uncertainty to $1.5\times10^{-8}$

The measurements were repeated using the refined techniques to try and achieve a measurement of $N_A$ with the required uncertainty of 15 atoms per billions ($1.5\times10^{-8}$).

The silicon-28 crystals were grown and shaped as quasi-perfect spheres, as they can be grown to a high-purity, and best suit the measurement requirements of modern electronics. However, crystals may contain impurities (such as silicon-30), interstitial atoms and vacancies, thus the counted lattice nodes will not correspond exactly to the number of silicon atoms. They were therefore characterised both structurally and chemically, and appropriate corrections were applied. The mass, thickness and chemical composition of the oxide layer covering the sphere were also taken into account; measured by optical and x-ray spectroscopy and reflectometry. The mean atomic mass of each silicon atom was measured by absolute mass-spectrometry, to account for any impurities or variations in crystal structure. The objective was almost achieved, as the refined approach allowed $N_A$ to be measured to within an uncertainty of less than 20 atoms per billion ($2.0 \times10^{-8}$).

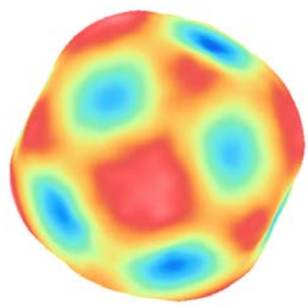

*Figure 3: Topography of the AVO-28 S5 sphere showing a cuboctahedron profile*

5. To investigate, understand, and possibly eliminate, the inconsistency between the determinations of *h* and $N_A$

The 2011 inconsistencies in the measured values of *h* and $N_A$ prompted an investigation into a number of effects that could potentially cause systematic errors in each approach.
For the watt-balance method, research focussed on the effects of: i) the finite speed of light on the operation of the free-fall gravimeters used to determine the local acceleration of gravity; and ii) the interaction between the coil and magnet on the watt-balance operation. For the atom-counting method, research focussed on i) the surface stress and strain on the lattice-parameter and sphere-volume measurements; ii) the contamination of the enriched crystal on the count of the Si atoms; iii) the NaOH dissolving and dilution matrix on the measurement of the abundance of the minority Si isotopes; and iv) the correlation of the $N_A$ measurements. Although a few of the effects studied approached the experiment sensitivity, none were found to be critical at the desired level of accuracy.

6. To carry out *h* and $N_A$ determinations with comparisons traceable to the international prototype of the kilogram. This will also allow the possibility to design and begin a long-term experiment aimed at monitoring IPK stability

The International Bureau of Weights and Measures (BIPM) carried out an extraordinary calibration campaign to ensure the results of the atom-counting and watt-balance experiments could be linked to the IPK.

BIPM prototypes, the BIPM ensemble of mass standards, and the mass standards used in the *h* and $N_A$ determinations were compared as directly as possible with the IPK, to reveal any inconsistencies in mass introduced at each key step of each method. The campaign was successful, and an offset of around 35 µg (35 billionths of a kilogram) was identified and corrected for, between the IPK and the masses that supported the *h* and $N_A$ measurements. The creation of methods to define the kilogram external to the IPK means that the watt-balance and atom-counting approaches can also be used to monitor how the mass of the IPK and its copies fluctuates over time.

7. To demonstrate consistent kilogram realisations based on both the *h* and $N_A$ determination

In addition to the project's European partner, the project team worked with the wider international metrology community outside Europe. The National Institute of Standards and Technology (NIST) in USA, and the Standards Laboratory of the Canada's National Research Council (NRC) in Canada – carried out similar stress-tests on their watt-balance experiments and repeated their *h* measurements. The 2014 watt-balance determination of *h* carried out by NRC was consistent with this project's value of $N_A$; and the measurement repetition of *h* by NIST's watt-balance resulted in an improved agreement with this project's value of *h*. Results from the two methods, in the three different locations, are converging. Therefore the impediments to a kilogram redefinition by fixing the value of Planck's constant are being removed.

8. To deliver the project's results to relevant metrology bodies, to disseminate them in the metrological and scientific communities, and in the wider, non-specialist, audience

The successful dissemination of the project's achievements are detailed in the following section.

## Actual and potential impact

Dissemination of results

Results and achievements were shared widely throughout the duration of the project. 57 papers were published in international journals, including Analytical Chemistry, the European Physical Journal, Metrologia, Journal of Physical and Chemical Reference Data, and Precision Engineering. 60 presentations were made at scientific and industrial conferences, including the Conference on Precision Electromagnetic Measurements (CPEM); the watt balance technical meeting in Ottawa, Canada; the 2015 fundamental constants meeting; and an invited presentation at the WE-Heraeus Seminar on Astrophysics, Clocks and Fundamental Constants. 23 training events were given, including internships, classes and workshops, on subjects such as the determination of the Avogadro and Planck constants, measurement methods, uncertainty evaluation, and the new definition of the kilogram. The project also contributed towards a master's thesis and three PhD theses.

Public dissemination activities included events, web articles, press releases, articles in trade/professional and the popular press, interviews, and television. These included appearances on Japanese, Swiss and UK

TV, articles on Avogadro's number and redefining the kilogram in trade and popular press, and a press release on 'More Precise Estimate of Avogadro's Number to Help Redefine Kilogram' in the United States.

Throughout the project, there were 22 participations and/or communications to metrology committees such as the Committee on Data for Science and Technology (CODATA) task group on fundamental constants, CIPM committees and working groups, and the EURAMET technical committee for mass and related quantities. The project also input into a draft documentary standard to BIPM's Consultative Committee for Mass and Related Quantities. Eight research collaborations at the European and International level were made throughout the project duration with universities, public research organisations, and a large industrial enterprise. Examples of end user uptake are the submission of the accurate value of the Avogadro constant, to which the fundamental constants from which the value of the Planck constant will be adopted will be adjusted, to the CODATA task group on fundamental constants; and the 28Si spheres (AVO28-S5 and AVO28-S8) as potential realisations of the new kilogram definition.

Impact on the redefinition of the kilogram

**Calculating *h* for the redefinition of the kilogram**

Through the work of this project, and research at NIST and NRC, the discrepancy in the calculation of Planck's constant between the two methods has been eliminated. This project's reassessment of $N_A$ using the atom-counting method, and NIST and NRC's restatement of *h* using the watt-balance approach, were accurate and in sufficient agreement to allow *h* to be used for the new definition of the kilogram. (Although this project delivered a consistent measurement of *h*, the uncertainty level was still too large to be used in conjunction with the NIST and NRC results). Based on the achievements of this project, and NIST and NRC, in August 2015 the Committee on Data for Science and Technology (CODATA) task-group on fundamental constants was able to state Planck's constant with an uncertainty of $12 \times 10^{-9}$, less than a quarter of the previous uncertainty, and within the requirements for the kilogram redefinition.

**Atom-counting as a *mise en pratique***

The project demonstrated a successful *mise en pratique* for the redefinition of the kilogram via the atom-counting method – the project's $N_A$ result was sufficiently accurate and was calculated with an uncertainty low-enough to meet the requirements set out by the BIPM International Committee for Weights and Measures (CIPM) and the Consultative Committee for Mass and Related Quantities (CCM). Although the project has not yet delivered sufficiently accurate watt-balance measurements of *h*, it has substantially progressed the European independence in this technology (to date, only the watt-balance experiment run by NRC has achieved the required accuracy).

The project has also highlighted areas in which the atom-counting method can be further refined including: i) the physical and chemical characterisation of the surface of the silicon spheres and X-ray interferometers; ii) the mass fractions of the minor isotopes and impurities, and iii) the crystallographic perfection.

Over the longer-term, the atom-counting method will make the kilogram definition accessible to any laboratory capable of carrying out surface characterisations and volume measurements. Previously, the cost of enriched silicon and the complexity of the measurement process have prevented laboratories from participating in the kilogram redefinition and the calculation of $N_A$, but relatively cheap spheres (having the same crystallographic, chemical and geometric perfections of a silicon-28 sphere) can be manufactured using natural silicon. These spheres will not be primary standards; however, they never require mass comparisons to be recalibrated. In fact, they will no longer be constrained by fears of irreversible mass changes, as with the IPK and its copies, because the mass changes can be identified, quantified and explained by the parallel observation of volume and surface changes. Eventually, contrary to the current definition, there are no (or significantly smaller) cost barriers to the usage of natural silicon mass-standards in secondary laboratories and industries. In addition, if a breakthrough will make an accurate determination of the isotopic composition of natural silicon possible, they open the door to a future spread of primary realisations.

Potential future impact

The achievements of this project have made a significant contribution to the redefinition of the kilogram in 2018. The redefinition will be more accessible than the current definition, and will support future increases in accuracy, vital to the development of scientific and industrial innovation throughout Europe. Emerging technologies, including nanotech and biotech, require accurate and reliable measurements to be made on ever finer-scales. The redefinition of the kilogram will ensure that measurements of mass, and measurements derived from mass, will be made with sufficient accuracy and certainty for these technologies and others.

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