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

Background
The mass of the international prototype of the kilogram is drifting with respect to the mass of its official copies (Figure 1). Therefore, in 2011, the 24th General Conference on Weights and Measures recommended that the International System of Units (SI) is upgraded in terms of fundamental constants.

Need for the project
Both the watt-balance experiment – to measure the Planck constant – and the counting of the atoms in $^{28}$Si spheres – to measure the Avogadro constant – are under consideration to monitor the prototype mass-stability and to realise the kilogram.

The watt-balance experiments determine the $h/m$ ratio, where $m$ is the mass of the international prototype of the kilogram. In other experiments, the quotient $h/m$ is determined, where $m$ is the mass of an atom or of a particle. Since only the relative atomic masses are well known, these experiments deliver the value of the $N_A h$ product. In this case, the link to macroscopic masses is made by measuring the Avogadro constant, $N_A$, by silicon spheres of known composition, volume, and lattice parameter.

Prior to the start of this project, three experiments went close to the accuracy required to make the kilogram redefinition possible: two watt-balance determinations of $h$ carried out at National Institute of Standards and Technology (NIST – USA) and the National Research Council (NRC – Canada) and a measurement of $N_A$ carried out by the International Avogadro Coordination (IAC). Although neither NIST nor NRC was completely out of scale, the measurement results were inconsistent: their differences were larger than the combined standard uncertainties. This impaired the kilogram realisation to within the uncertainty required to ensure continuity to mass metrology and indicated that an error was made in at least one experiment.

Scientific and technical objectives
The project addresses the following scientific and technical objectives:
- To fine-tune and adjust the European watt balances to their maximum performance level.
- To carry out $h$ measurements with $5 \times 10^{-8}$ relative uncertainty via independent watt-balance experiments.
- To challenge atom-counting experiments and confirm the measured $N_A$ value and its uncertainty or identifying error/s.
- To repeat the $N_A$ determination by simultaneously reducing the relative uncertainty to $1.5 \times 10^{-8}$.
- To investigate, understand, and possibly resolve, the inconsistency between the determinations of $h$ and $N_A$.
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.

To demonstrate consistent kilogram realisations based on both the \( h \) and \( N_A \) determination.

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.

**Expected results and potential impact**

This project aims to strengthen the confidence in the measured values of \( N_A \) and \( h \) and in the network of the recommended values of the fundamental constants of physics. This is a prerequisite for the foundation of the International System of Units (SI) on fixed values of fundamental constants of physics.

NRC demonstrated the watt-balance technology at \( 1.9 \times 10^{-8} \) \( h \) accuracy and is close to demonstrating a practical realisation of the kilogram. NIST reported a determination, which is in substantial agreement with the values obtained by NRC and IAC. Although no reason has been found for the shift with respect to the previous value, this agreement increases the confidence in the realisations of the kilogram from the Planck constant.

This project aims to increase the measurement accuracies and capability of Europe, a result that is essential to give the firmest ground to the kilogram redefinition. It is expected that a repetition of the \( N_A \) determination will have a relative uncertainty of \( 1.5 \times 10^{-8} \). Stress tests are expected to highlight mistakes or hidden assumptions such that they can be excluded, or identified and eliminated.

At the end of 2014, using the two re-polished spheres of the IAC, a new determination of \( N_A \) was provided with a relative standard uncertainty of \( 2.0 \times 10^{-8} \), which falls within the targeted uncertainty necessary for the redefinition of the kilogram. With respect to the 2011 determination, whose uncertainty did not yet catch this uncertainty goal, the new determination benefitted from many improvements in the measurements constituting the X-ray-crystal-density method.

Metals contaminating the spheres surface after the first polishing were removed by Freckle™ etch and PTB reshaped the spheres up to a peak-to-valley diameter variation below 70 nm (Figure 2).

Concerning the bulk contaminants, INRIM and ANSTO are carrying out nuclear activation analyses. For 42 out of the 90 possible contaminants the detection limit is at the 1 ng/g level or better. By infrared measurements, PTB determined the nitrogen contamination estimating a contribution to the mass of the spheres of 1 \( \mu \)g. The molar mass determination was improved substituting the aqueous organic solvent Tetramethylammonium hydroxide (TMAH) for the previously used aqueous NaOH, which was probably responsible of the discrepancy in the ratio \( x(28\text{Si})/x(30\text{Si}) \) between the measurements done by NRC, on one side and PTB, NMIJ and NIST on the other. The variability of the isotopic composition across the AVO28 boule is also under investigation by three different NMIs. The first results show a substantial uniformity.

INRIM have repeated measurements of the \( ^{28}\text{Si} \) lattice parameter and have excluded systematic errors and halved the uncertainty. The main novelties were: a new optical interferometer, a new bridge for temperature measurements, the on-line monitor of the laser-beam alignment and a better noise-to-signal ratio. NMIJ investigated the lattice spacing homogeneity by a self-referenced lattice comparator. Two samples – from the ‘tail’ and a region close to the seed of the originally grown \( ^{28}\text{Si} \) single crystal – were analysed, as well as the crystal of the X-ray interferometer used for the lattice parameter determination. The correlation between the lattice distortion and contaminant concentration will be investigated using micro Fourier Transform Infrared Spectroscopy (FT-IR).

*Figure 2: Topography of the AVO-28 S5 sphere having a cuboctahedron profile.*
The fixed point cells of INRIM and PTB were compared to establish their temperature difference. INRIM, PTB and NMIJ also compared their thermometers in order to link the extrapolations to 20 °C of the measurements of the lattice parameter (INRIM) and the sphere volume (PTB and NMIJ).

The surface of the spheres were more accurately characterised using spectral ellipsometry, a fast technique that, to achieve an accuracy below 1 nm in the layers thickness determination, needs calibration based on other surface analysis methods like X-ray reflectometry (XRR) and X-ray fluorescence (XRF).

Although the volume measurement is still one of the dominant contributors to the error budget, the reduced diffraction uncertainty allowed a relative uncertainty of $1.6 \times 10^{-8}$ to be reached. This improvement owes to the sphere polishing that reduced the diameter variations from 98 nm to 38 nm.

To monitor the prototype and kilogram-realisations’ stability and to link the atom-counting and watt-balance experiments to the prototype’s mass, BIPM carried out an extraordinary calibration campaign. The BIPM prototypes, 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 international prototype. The BIPM estimated the offset, about 35 μg, for the mass calibrations that supported the $h$ and $N_A$ measurements. NMIJ examined the effect of the washing procedure, pointing out how a minimum repetition of three washings is likely necessary to stabilise the total mass. It was observed that after each of the three washings a mass loss varying between 3 μg and 8 μg is detected.

Based on the knowledge gained with previous apparatus, METAS is realising a new watt balance (Figure 4). Collaborators EPFL-LSRO, CERN, and Mettler-Toledo are supporting the design and characterisation of the balance components. The commissioning, assembling and integration of the watt-balance parts (coil, magnet, translation system, load cell, mass handler) is currently going according to plan and first tests of the moving-mode operation have started.

LNE, with support from CNAM and OBSPARIS, brought into operation a watt balance from the previous iMERA-Plus project e-Mass. During 2014, LNE assembled watt-balance elements that were separately designed, developed, and tested, and carried out a determination of the Planck constant. The measured value $h = 6.6260688(20) \times 10^{-34}$ Js is affected by a relative standard uncertainty of $3.1 \times 10^{-7}$ that originates mainly from the voltage and velocity measurements and the uncertainty introduced by the suspension alignment. The improve-

---

**Figure 3:** In sight of a future mise en pratique of the kilogram, values of $h$ determined by the watt balance and $^{28}$Si spheres, through $N_A h$, are summarised.

**Figure 4:** The METAS watt balance.
ments programmed in order to reduce the uncertainty include: i) voltage measurements made directly against a 1 V programmable Josephson voltage standard (PJVS), ii) better verticality of the laser beams (going through the position detectors and the corner cubes of the coil) using a mercury pool and a 1.1 m focal length telescope, iii) the use of a 500 g mass artefact, made from Pt–Ir alloy, provided by the English company Johnson–Matthey and polished, adjusted and calibrated by BIPM.

The project has so far submitted 35 publications to peer-reviewed journals and made 56 presentations at international meetings and conferences. Eight early-stage researchers, PhD or post-doc students have been trained and several training courses/workshops have been held focussing on watt balances, measurement methods and uncertainties and the new definition of the kilogram and its "mise en pratique". Project activity has also been included in clips by the Open University (UK) and Japanese and Swiss TV.

Throughout the project, the international committee for weights and measures has been updated with the project results to advance the redefinition of the International System of Units. Updates have included the status of i) the Avogadro and watt balance experiments; ii) the mise en pratique; and iii) the extraordinary calibrations using the international prototype of the kilogram.

<table>
<thead>
<tr>
<th>JRP start date and duration:</th>
<th>01 September 2012, 3 years</th>
</tr>
</thead>
<tbody>
<tr>
<td>JRP-Coordinator:</td>
<td>Giovanni Mana, Dr, INRIM</td>
</tr>
<tr>
<td></td>
<td>Tel: +39 0113919728</td>
</tr>
<tr>
<td></td>
<td>E-mail: <a href="mailto:g.mana@inrim.it">g.mana@inrim.it</a></td>
</tr>
<tr>
<td>JRP website address:</td>
<td><a href="http://www.inrim.it/know">http://www.inrim.it/know</a></td>
</tr>
<tr>
<td>JRP-Partners:</td>
<td>JRP-Partners:</td>
</tr>
<tr>
<td>JRP-Partner 1 INRIM, Italy</td>
<td>JRP-Partner 5 OBSPARIS, France</td>
</tr>
<tr>
<td>JRP-Partner 2 CNAM, France</td>
<td>JRP-Partner 6 PTB, Germany</td>
</tr>
<tr>
<td>JRP-Partner 3 METAS, Switzerland</td>
<td>JRP-Partner 7 AIST, Japan</td>
</tr>
<tr>
<td>JRP-Partner 4 LNE, Franc</td>
<td>JRP-Partner 8 IOM, Germany</td>
</tr>
<tr>
<td>REG-Researcher 1</td>
<td>Fred Pietag, Germany</td>
</tr>
<tr>
<td>associated Home Organisation:</td>
<td>IOM, Germany</td>
</tr>
<tr>
<td>REG-Researcher 2</td>
<td>Hendrik Paetzelt, Germany</td>
</tr>
<tr>
<td>associated Home Organisation:</td>
<td>IOM, Germany</td>
</tr>
<tr>
<td>REG-Researcher 3</td>
<td>Luciano Colombo, Italy</td>
</tr>
<tr>
<td>associated Home Organisation:</td>
<td>UNICA, Italy</td>
</tr>
</tbody>
</table>

The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.