

AVOGADRO CONSTANT DETERMINATION FOR A REDEFINED KILOGRAM

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ABSTRACT

Today the mass unit kilogram is still defined by the mass of an artefact, the international prototype of the kilogram. The Avogadro constant can be used to define the kilogram in terms of a fundamental constant. The International Avogadro Coordination determined the Avogadro constant in a new approach by ‘counting’ the atoms in 1 kg single-crystal spheres, which are highly enriched with the ^{28}Si isotope. The value obtained, $N_A = 6.022\,140\,82\,(18) \times 10^{23} \text{ mol}^{-1}$, is the most accurate input datum for a new definition of the kilogram. In this publication volume, mass and surface measurements are reviewed. The result of N_A is compared to determinations by watt balance experiments.

Index Terms – Avogadro constant, silicon, isotopically enriched silicon crystal, mass, density, volume, interferometer, oxide layer, new kilogram definition.

1. INTRODUCTION

Today the mass unit kilogram is still defined by the mass of the international prototype of the kilogram (IPK), which is a PtIr cylinder that can be damaged or even destroyed. Additionally, it was discovered that the mass of the official and national copies changed by about 50 μg during 100 years in comparison to the IPK [1], compare figure 1. Thus, it seems that the IPK mass changes with time. This would result also in a drift of other SI units like ampere and volt.

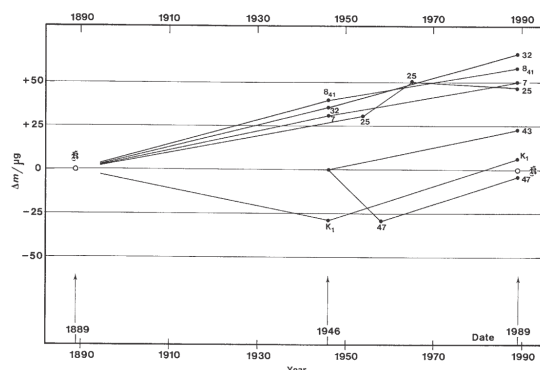


Fig. 1. Relative changes in mass of the six official copies with respect to the mass of the international prototype [1].

Two experiments are favoured to prepare and to realise a new definition of the kilogram. First, a measurement of the Planck constant h by a watt balance. This method includes a virtual comparison of a mechanical power with an electrical power, which is measured by Josephson and quantum Hall devices. If additionally to the Planck constant, the charge of the electron is defined (thus re-defining the unit of the electric current, ampere), the Josephson and quantum Hall effects can be used to realise volt and ohm. Then, the Josephson and von Klitzing constants have exact values. Additionally, the representations of the volt and the ohm based on conventional values for the Josephson and von Klitzing constants are dispensable.

The second way is to define the kilogram by the mass of an exact number of the atomic mass unit or of an exact number of specified atoms. The unit of the amount of substance, the mole, is defined as the number of atoms in 12 g of the isotope ^{12}C . This number is also the value of the Avogadro constant. Thus, a definition of the Avogadro constant N_A defines the atomic mass unit $u = m(^{12}\text{C})/12$ in terms of kilogram.

Using measurements of the molar Planck constant $N_A h$, both ways are nearly equivalent, since the molar Planck constant is known with a relative standard uncertainty of about 1×10^{-9} [2].

2. INTERNATIONAL AVOGADRO PROJECT

In the International Avogadro Project a silicon single crystal grown from isotopically enriched ^{28}Si is used to determine the Avogadro constant [3, 4] using the formula

$$N_A = 8 M / (\rho a_0^3). \quad (1)$$

In this method molar mass M , lattice parameter a_0 and density ρ of the crystal have to be measured.

The International Avogadro Project started in 2004 with the isotope enrichment by centrifugation of SiF_4 gas undertaken at the Central Design Bureau of Machine Building in St. Petersburg. Subsequently, after conversion of the enriched gas into SiH_4 , a polycrystal was grown by chemical vapour deposition at the Institute of Chemistry of High-Purity Substances of the Russian Academy of Sciences in Nizhny-Novgorod and, in 2007, the 5 kg ^{28}Si boule shown in Fig. 2 was grown by the Leibniz-Institut für

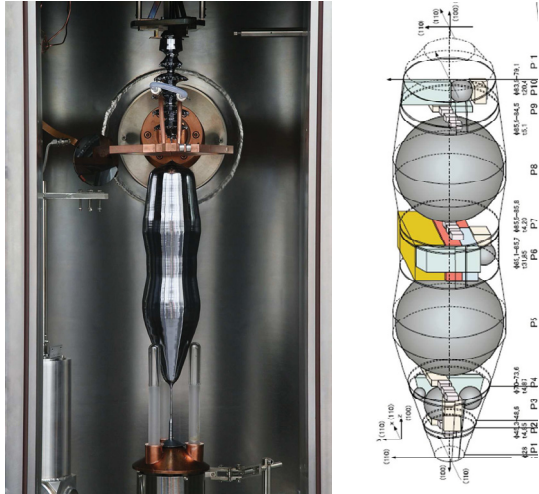


Fig. 2. The float-zone ^{28}Si crystal and its cutting plan [4]. To determine its density, two spheres were manufactured from the two bulges. To determine the lattice parameter, an x-ray interferometer was cut from the material between these spheres (see yellow part of the cutting plan).

Kristallzüchtung in Berlin [4]. The isotopically enriched of the final single crystal is higher than 0.99995.

To measure the density $\rho = m/V$ of the crystal, two kilogram prototypes shaped as nearly perfect spheres were manufactured at the Australian Centre for Precision Optics (ACPO) and named AVO28-S5 and AVO28-S8. Their mass m and volume V were accurately measured and their surfaces were chemically and physically characterized at the atomic scale. The lattice parameter and molar mass of the spheres were also measured. Additionally, the point

defects have been investigated, since the lattice spacing and density are affected by impurity atoms and vacancies which change the distances of the silicon atoms due to their individual binding conditions.

The main measurements were performed by the International Avogadro Coordination (IAC), consisting of the national metrology institutes of Australia, Germany, Italy, and Japan and the Bureau International des Poids et Mesures (BIPM). In this publication only the measurements on the spheres are reviewed. A comprehensive description of all measurements can be found in the special issue of Metrologia on the “International determination of the Avogadro constant” [5].

3. VOLUME DETERMINATION

For the volume determination of the silicon spheres a spherical Fizeau interferometer was developed at PTB [6], see figure 3. It consists of a temperature-controlled vacuum chamber and two opposing arms for illuminating and imaging optics. In these optical arms light from a multimode fibre propagates 1.6 m in free space and is then collimated. The plane wave enters the vacuum chamber through an optical window and is subsequently focused by a Fizeau lens whose focal point is carefully adjusted to the centre of the silicon sphere. As the inner surfaces of the two Fizeau lenses are formed concentrically to the focussed wavefront, a spherical etalon is formed in one case, with the sphere removed, between the pair of lenses and in the other case, with the sphere inserted, between the reference face of each lens and the corresponding surface of the sphere.

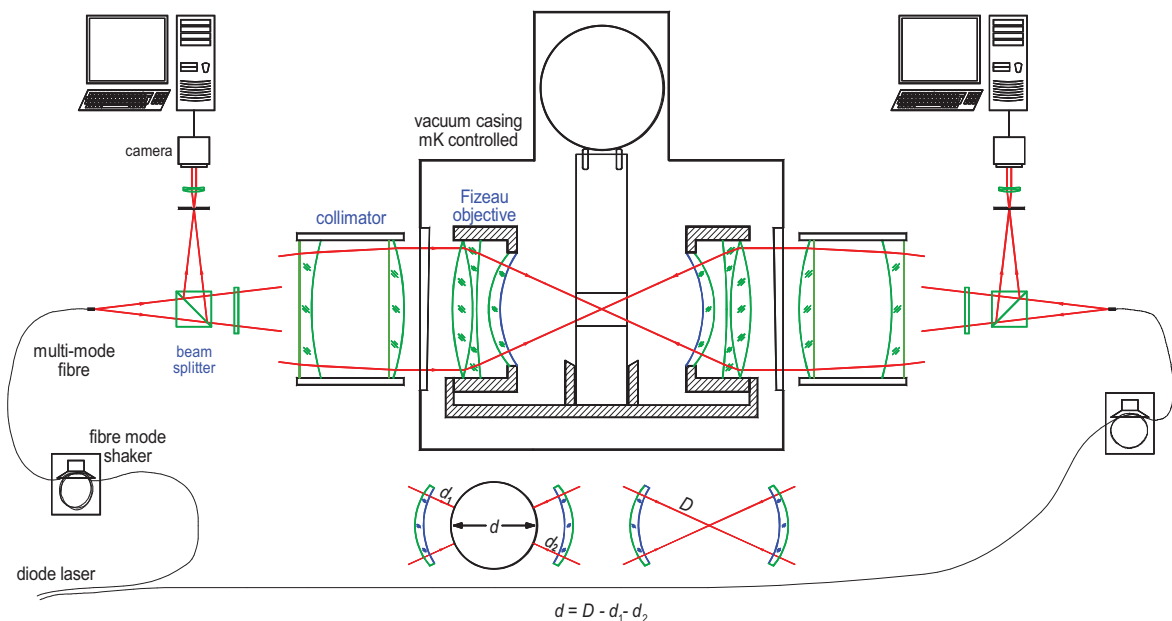


Fig. 3. Sphere interferometer of PTB [6].

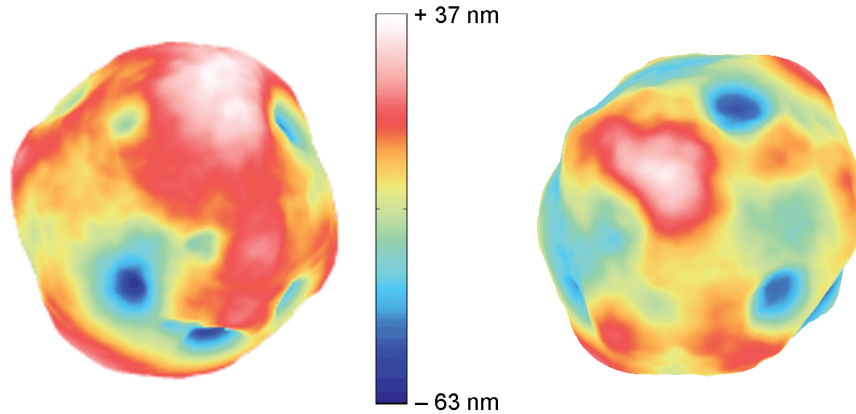


Fig. 4. Diameter topographies of the silicon spheres [4]. Peak-to-valley distances are 98 nm (AVO28-S5, left) and 90 nm (AVO28-S8, right).

Therefore, three measurements are necessary to obtain the diameter of the sphere: diameter D of the empty etalon, which is obtained with the sphere in a lifted position, and the distances d_1 and d_2 between the sphere and the reference surfaces. The diameter d of the sphere is then $d = D - d_1 - d_2$.

With this technique 10000 diameter values, only depending on the camera resolution chosen, within a 60° section of the surface can be obtained. To cover the whole sphere with diameter values and to lift it out of the beam path for measuring the length of the empty etalon, an elevation and positioning mechanism is used consisting of a liftable column which contains two pairs of plastic wheels as a support for the sphere in the upper position. Lifting and rotation around the vertical axis is done by the column itself and the rotation around the horizontal axis is done by rotation of the wheels. In the measurement position the sphere rests on a three-point support.

To achieve low uncertainties the implementation of a thermal stabilization is a basic prerequisite. In the first instance a laboratory climate control provides temperature fluctuations below 0.1 K in 24 hours. For the second level a pipe network was incorporated in the vacuum chamber housing which is flown-through by a water-glycol-mixture. The temperature of the liquid is stabilized by a thermostat to 0.01 K, so that due to the thermal inertia of the massive body of the vacuum chamber a temperature fluctuation of only a few millikelvin is reached. As the final step the interferometer itself, consisting of a rigid monolithic frame, which contains the Fizeau lenses and the three-point rest of the sphere, is coupled to the vacuum chamber with three small point contacts, so that a weak heat and energy exchange guarantees small temperature fluctuations. For the target uncertainty the temperature of the sphere needs to be measured with an uncertainty of less than 1 mK. To reduce any effects of influencing the temperature of the sphere, thermocouple pairs measure the temperature difference between the sphere and a copper block

inside the interferometer frame which acts as a reference body. The temperature of this copper block is measured by common means, i.e. an AC bridge with a 25Ω standard platinum resistance thermometer, directly calibrated at the triple point of water and the melting point of gallium.

For the determination of the volume of the two ^{28}Si spheres, 32 measurements with different orientations of respective sphere have been performed for AVO28-S5 and 44 measurements for AVO28-S8. The whole set of measurements gave 320000 and 440000 single diameter values, respectively, distributed all over the sphere and covering the topographies thoroughly. The surfaces of the spheres were subdivided into small cells of almost equal area and all measurement values inside a cell were assigned to the centre position of this cell. By doing so, maps of the topography can be determined, which are shown in figure 4 [4]. The colour scales are adapted to the respective topography and span 98 nm for AVO28-S5 and 90 nm for AVO28-S8 related to the diameter variation.

Since silicon oxidises very fast under ambient conditions, the surface of the silicon sphere is covered by an oxide layer. Due to the refractive indexes of the silicon core and of the surface layer, the incident light is affected by a phase shift on reflection so that the thickness of the surface layer is underestimated by the optical measurement. For the calculation of this optical phase shift, a layer model is used, see chapter 5. Based on layer thickness determinations, and with the optical properties of the materials, a small correction for the apparent diameter is calculated in order to determine the exact diameter of the silicon core of the sphere.

The resulting diameters and volumes were confirmed by measurements at the National Metrology Institute of Japan (NMIJ) using a different type of interferometer [5]; the weighted means of the core volumes are listed in table 1.

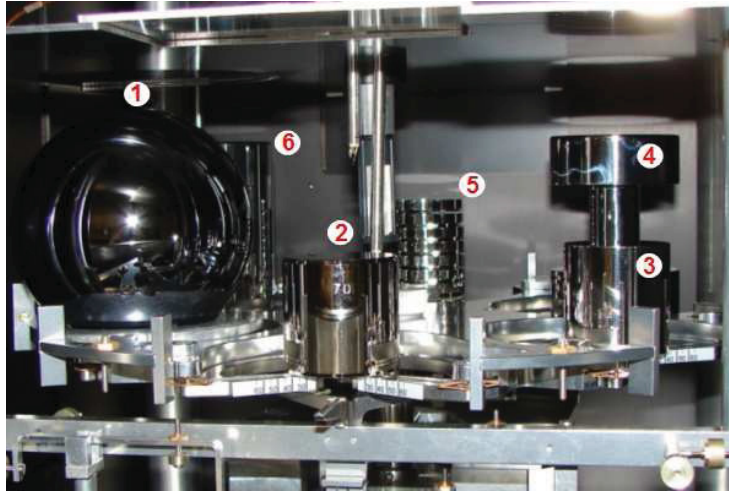


Fig. 5. Weighing chamber during the mass determination of the ^{28}Si sphere AVO28-S8 (1) with the kilogram prototype No. 70 (2), Pt/Ir sorption artefacts (3, 5) and air buoyancy artefacts (4, 6).

4. MASS DETERMINATION

The mass of the spheres was measured by comparison to Pt/Ir mass standards, which were calibrated at the BIPM traceable to the IPK. The classical mass comparison in air shows that the main uncertainty is due to the air buoyancy correction, even if the air density is determined gravimetrically by using special air buoyancy artefacts (figure 5).

Therefore, it is advantageous to determine the mass of silicon spheres under vacuum conditions [7]. Suitable vacuum mass comparators have been available since a few years ago, which make it possible to determine the mass of 1 kg silicon spheres under vacuum conditions (10^{-3} Pa to 0.1 Pa). Thereby it must, however, be taken into account that the reference mass and the silicon sphere underlie mass alterations when subjected to an air-vacuum transfer which are caused by reversible and irreversible sorption effects. The adsorption and desorption of water and hydrocarbons at the surface depend on the material, the roughness, the state of surface cleanliness and the humidity of the air. These effects can be estimated by weighing two sorption artefacts (same material and same surface properties, equal mass, surface areas as different as possible) in air and under vacuum.

The BIPM developed a special set of two 1 kg Pt/Ir sorption artefacts, calibrated in air against a 1 kg Pt/Ir prototype, that can be used to link directly the silicon sphere in vacuum to the national prototype (in air) [8]. The method takes into account the sorption correction obtained during the comparison (in vacuum) among the spheres and the sorption artefacts. In this way, air buoyancy corrections applied during all the chains of comparison are minimized ($\ll 1 \mu\text{g}$), because all comparisons in air involve Pt/Ir artefacts only. The sorption correction must be carried out on the classical Pt/Ir transfer standard, which is one of the sorption artefacts. The BIPM has fabricated a set of

1 kg Pt/Ir sorption artefacts, composed of two 1 kg objects having the same volume (to avoid air buoyancy correction), a large surface area difference and the same surface finish. One artefact consists of a stack of eight discs separated from one another by three bent rods. The second artefact is a classical prototype (a cylinder with height and diameter equal to 39 mm). The surface difference between the two artefacts is about 186 cm^2 . The PTB used similar Pt/Ir sorption artefacts for the determination of the mass of the sphere AVO28-S8 and stainless steel sorption artefacts with a surface area difference of about 57 cm^2 for the determination of the mass of the sphere AVO28-S5. The NMIJ used a stainless steel sorption artefact with a surface area difference of about 130 cm^2 .

The results obtained for the spheres AVO28-S5 and AVO28-S8, $m_{S5} = 1.000\,087\,772\,4(41) \text{ kg}$ and $m_{S8} = 1.000\,064\,730\,7(41) \text{ kg}$, demonstrate that by using sorption artefacts reference values can be achieved with a relative standard uncertainty of about 4×10^{-9} for the mass of silicon spheres under vacuum (figure 6 [4]).

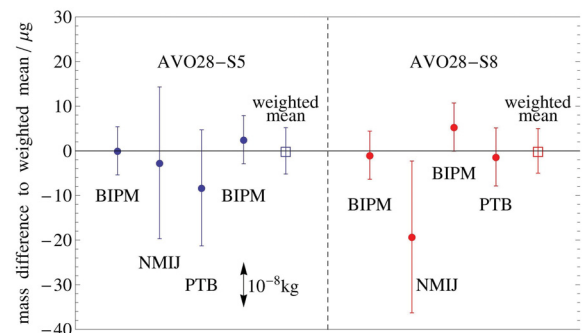


Fig. 6. Mass of the silicon spheres AVO28-S5 and AVO28-S8 weighed in vacuum by the BIPM, NMIJ, and PTB [4]. The absolute mass difference between the two spheres is $m_{S5} - m_{S8} = 23.042 \text{ mg}$. The bars give the standard uncertainties.

5. SURFACE

To determine the mass and the volume of the silicon core of the sphere, corrections for the surface layer have to be made. Mass, thickness and chemical composition of the surface layer were characterized by the following methods [9]: x-ray fluorescence analysis (XRF, in air and vacuum), near edge x-ray absorption fine structure spectroscopy (NEXAFS, vacuum), x-ray reflectometry (XRR, vacuum), x-ray photoelectron spectroscopy (XPS, vacuum), and optical spectral ellipsometry (SE, in air). For traceable determination of the oxide layer thickness, synchrotron radiation-based XRR at specific points had been chosen to calibrate the surface mapping by SE. By selecting photon energies around the oxygen K absorption edge at 543 eV, energies which are available at the PGM beamline of the PTB's laboratory at BESSY II, XRR is possible also for SiO₂ layers on quite curved surfaces. However, an unexpected metallic contamination (by copper, nickel and zinc) was detected on the sphere surfaces, which limits the total measurement uncertainty because of its significant influence on the optical constants of the sphere surface layer. NEXAFS measurements at the same beamline and XPS measurements at the Bundesamt für Metrologie (METAS, Switzerland) revealed that the metallic contaminants are present as silicides. Therefore, the direct oxide thickness determination by XRR at the oxygen K-edge on the sphere was replaced by XRF measurements, in which the oxygen K fluorescence intensity from the sphere surface was compared with that from flat samples, for which the oxide layer thickness was determined by XRR.

The surface layer of the spheres was divided for data evaluation into four sub-layers (figure 7); from top to bottom: a carbonaceous contamination layer (CL), a chemisorbed water layer (CWL), a layer of metallic silicides (ML) and a pure oxide layer (OL). The mass deposition of the metallic contamination and of carbon was also derived from XRF measurements by synchrotron radiation by comparison to flat reference samples having a layer thickness determined by XRR. XPS was used to investigate the stoichiometry of the oxide layer. In addition, XPS gave also a rough determination of the oxide-layer thickness, which was found to be in good agreement with the values measured by XRF/XRR combination. At NMIJ, the ellipsometer data were calibrated by SiO₂ layers on wafers with thicknesses certified by XRR measurements [5].

The apparent thickness of the carbonaceous layer (CL) was measured on eight spheres at BESSY by XRF in a comparison with a reference sample having a 6.5 nm carbon layer; the result is 0.212(39) nm, on the average and on the assumption of the same density as that of the reference. With an estimated density of 1.0(2) g/cm³, a mass of $m_{CL} = 15.0(3.8) \mu\text{g}$ for AVO28-S5 and of $m_{CL} = 13.1(3.3) \mu\text{g}$ for AVO28-S8

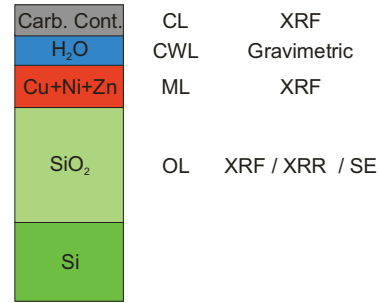


Fig. 7. Surface layer model for the characterization of the ²⁸Si spheres [9]. On the left the physical status of the sublayer is given, the abbreviations are stated in the middle. Finally, on the right, the applied methods for the quantitative determinations are listed.

was derived for the carbonaceous layer. The associated thicknesses are: $d_{CL} = 0.54(18) \text{ nm}$ (AVO28-S5) and $d_{CL} = 0.47(16) \text{ nm}$ (AVO28-S8).

For mass deposition of the chemisorbed water layer (CWL), which is the residual water on the sphere under vacuum conditions, the value 0.028(4) μg/cm² measured by Mizushima [10] was applied. To account for the difference between the characteristics of the wafer and the sphere surfaces, the uncertainty of the Mizushima's value was increased by a factor of two. A 1.0(1) g/cm³ density of the water layer was assumed. The corresponding layer mass and thickness are 7.7(2.2) μg and 0.28(8) nm, respectively.

The Ni, Cu, and Zn depositions and their binding characteristics were measured by XRF, NEXAFS, and XPS. The chemical compositions NiSi_x, CuSi_x, ZnSi_x, with $x = 1.0(1)$, were used to calculate the mass of the layer. A density of 7.2(2.0) g/cm³ of this layer was estimated. A mass of $m_{ML} = 107.1(5.2) \mu\text{g}$ for AVO28-S5 and of $m_{ML} = 107.8(5.5) \mu\text{g}$ for AVO28-S8 was derived. The associated thicknesses are: $d_{ML} = 0.54(16) \text{ nm}$ (AVO28-S5) and $d_{ML} = 0.54(17) \text{ nm}$ (AVO28-S8).

The stoichiometry of the oxide layer (OL) was measured at METAS by XPS; a pure SiO₂ layer was found. At PTB, the total oxygen surface-content was measured by XRF by comparison with a set of SiO₂ reference samples, having thickness between 2 nm and 10 nm and calibrated by XRR measurements. A 2.2(1) g/cm³ oxide density was assumed. Eventually, for observation in detail of the variation of the oxide layer thickness, SE was applied to map the whole surface layer and to generate a complete topography consisting of about 5500 data points (figure 8). Measurements at the NMIJ confirmed the results by using a spectroscopic ellipsometer that was calibrated by means of SiO₂ layers on Si wafers having thicknesses certified by a traceable XRR system. Average thicknesses of $d_{OL} = 2.88(33) \text{ nm}$ (AVO28-S5) and $d_{OL} = 2.69(32) \text{ nm}$ (AVO28-S8) were derived. The associated masses are: $m_{OL} = 92.2(12.8) \mu\text{g}$ (AVO28-S5) and $m_{OL} = 85.0(12.7) \mu\text{g}$ (AVO28-S8).



Fig. 8. Topographic maps of the surface layer thickness measured by spectral ellipsometry (not calibrated). Left AVO28-S5, right AVO28-S8. The rainbow colour code ranges from 2.0 nm (blue) to 4.5 nm (yellow).

6. RESULTS

Additionally, the isotopic composition of the ^{28}Si crystal was determined by the PTB, via isotope dilution combined with multicollector inductively coupled plasma mass spectrometry [5], and the lattice parameter was measured by a combined optical and x-ray interferometer at the Istituto Nazionale di Ricerca Metrologica (INRIM, Italy) [5].

To determine the mass of the silicon core, the mass of the surface layers, including the metallic contaminations, was subtracted from the mass of the sphere. In addition, owing to point defects, there is a difference between the mass of a sphere having Si atoms on all regular sites and the measured mass. A mass deficit of $8.1(2.4)$ μg was calculated for the AVO28-S5 and a mass deficit of $24.3(3.3)$ μg for AVO28-S8 [4]. After the surface-layer mass was subtracted and the mass deficit was added, the Si-core mass $m = m_{\text{sphere}} - m_{\text{SL}} + m_{\text{deficit}}$ is obtained (table 1).

The N_A determinations based on two spheres are summarized in table 1; they differ only by $37(35) \times 10^{-9} N_A$, thus confirming the crystal homogeneity.

Tab. 1. N_A determination [4]. Lattice parameter a , volume V , and density ρ are measured at $t_{90} = 20.0$ $^{\circ}\text{C}$ and $p = 0$ Pa.

Quantity	Unit	AVO28-S5	AVO28-S8
M	g/mol	27.97697026(22)	27.97697029(23)
a	pm	543.0996240(19)	543.0996185(20)
V	cm^3	431.059061(13)	431.049111(10)
m	g	1000.087558(15)	1000.064541(15)
ρ	kg/m^3	2320.070841(76)	2320.070998(64)
N_A	10^{23} mol^{-1}	6.022 140 95(21)	6.022 140 73(19)

By averaging these values, the final value of the Avogadro constant is

$$N_A = 6.022\,140\,82(18) \times 10^{23} \text{ mol}^{-1}, \quad (2)$$

with a relative standard uncertainty of 3.0×10^{-8} . The main uncertainty contributions are at present due to surface characterization and the volume determination, see table 2.

Tab. 2: Uncertainty budget of the Avogadro constant determination [11].

Quantity	Relative uncertainty/ 10^{-9}	Contribution/%
Molar mass	7	5
Lattice parameter	11	13
Surface	14	22
Sphere volume	23	57
Sphere mass	3	1
Point defects	4	2
Total	30	100

7. DISCUSSION

For the first time a comparison can be made between accurate values of the Planck constant derived from different experiments. Figure 9 shows our result compared with those of the most accurate measurements so far carried out: the watt-balance experiments of the National Institute of Standards and Technology (NIST - USA) [12, 13], the National Physical Laboratory (NPL - UK) [14] and of the METAS [15]. The values of the Planck constant measured by these experiments were converted into

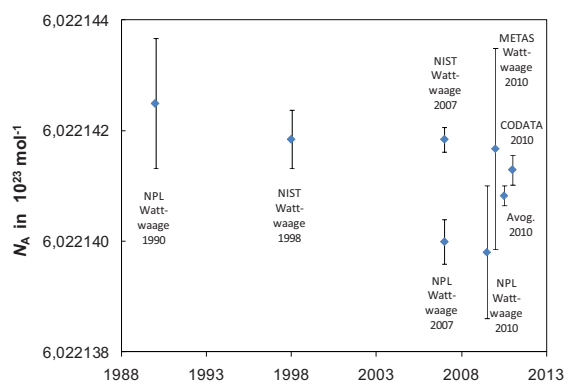


Fig. 9. Avogadro constant determinations: Comparison between the most accurate N_A values at present available. Bars represent the standard uncertainty.

the corresponding N_A values by $N_A h = 3.990\,312\,6821(57) \times 10^{-10}$ Js/mol, which has a relative standard uncertainty of 1.4×10^{-9} [2]. Figure 9 contains also the new CODATA value for N_A [16].

The new result for the Avogadro constant is a significant step also towards demonstrating a successful *mise en pratique* of a kilogram definition based on a fixed value of the Avogadro constant or of the Planck constant. The agreement between the different realizations is not yet as good as is required to withdraw the Pt-Ir kilogram prototype, but considering the capabilities already developed and the envisaged improvements, it seems realistic to achieve the targeted uncertainty in the foreseeable future.

8. ACKNOWLEDGEMENTS

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