

PAPER • OPEN ACCESS

Mass standards made of silicon and their machine-readable Digital Calibration Certificate

To cite this article: F Härtig *et al* 2018 *J. Phys.: Conf. Ser.* **1065** 082005

View the [article online](#) for updates and enhancements.

You may also like

- [Progress on accurate measurement of the Planck constant: Watt balance and counting atoms](#)
Shi-Song Li, , Zhong-Hua Zhang et al.
- [Establishment of the platinum–iridium kilogram mass standards at NMIJ after the Extraordinary Calibrations](#)
Shigeki Mizushima and Kenichi Fujii
- [Improved measurement results for the Avogadro constant using a ²⁸Si-enriched crystal](#)
Y Azuma, P Barat, G Bartl et al.



ECS
The
Electrochemical
Society
Advancing solid state &
electrochemical science & technology

DISCOVER
how sustainability
intersects with
electrochemistry & solid
state science research

Mass standards made of silicon and their machine-readable Digital Calibration Certificate

F Härtig, S Hackel, D Hutzschenreuter, K Lehrmann, R Meeß, A Scheibner and T Wiedenhöfer

Physikalisch-Technische Bundesanstalt Braunschweig und Berlin, Germany

E-mail: frank.haertig@ptb.de

Abstract. The expected re-definition of the international system of units as well as the advancing developments in the field of digitalization are fundamental for highly stable silicon mass standards and electronic transfer of its inherent metrological information. Flagships are physical standards made of highly enriched isotopic monocrystalline silicon 28 with a nominal mass of 1 kilogram. Starting from the future fundamentally fixed Planck-Constant, the number of atoms and thus the "new mass" can be directly realized by these spheres. Furthermore, years of experience in manufacturing, handling and use of silicon spheres have caused the Physikalisch-Technische Bundesanstalt to exploit the outstanding advantages of natural silicon when producing spheres for the realization and dissemination of the unit kg. The unique advantages will be of benefit for metrology institutes as well as for industrial applications. With the aim of building up a private-sector infrastructure, both, know-how and manufacturing technology have been transferred to industry. Current developments also deal with machine-readable Digital Calibration Certificates which contain all relevant information of the calibration. They will be provided with each of the silicon spheres.

1. Introduction

Since 1889, the unit mass refers to a single physical artifact called the International Kilogram Prototype (IKP) [1]. It consists of a platinum-iridium cylinder of 39 mm height and a diameter of 39 mm. Starting from this single physical body, all masses and their derived quantities, such as force, torque, pressure, density and fluids are derived. Because of this industrial and social importance, the IKP is well kept in a safe located in the Bureau International des Poids and Mesures (BIPM) in Sèvres, Paris and rarely used for measurements. Comparisons against national copies suspect that the IKP lost 50 micrograms of its mass in nearly 130 years [2]. This is motivation enough to remember old ideas which propose to define all seven SI base units by unchangeable sizes or values. For the first time, this idea was formulated in 1879 by James Clerk Maxwell. He wanted to refer all units to atomic sizes [3]:

"If, then, we wish to obtain standards of length, time, and mass which shall be absolutely permanent, we must seek them NOT in the dimensions, or the motion, or the mass of our planet, but in the wave-length, the period of vibration, and the absolute mass of these imperishable and unalterable and perfectly similar molecules."

The concept that is in use today is based on the idea of Max Planck from the year 1900 [4]:

"...with the help of fundamental constants we have the possibility of establishing units of length, time, mass, and temperature, which necessarily retain their validity for all times and cultures, even extraterrestrial and nonhuman..."



Fundamental constants are carved in stone, which finally allows the motto of the Meter Convention to be fulfilled since its initial conference on May 20, 1875 [5].

„À tous les temps, à tous les peuples“ – „for all times and for all peoples“

This idea results in two major advantages. The mass and all its derived units do not depend on a few presumable unstable physical bodies and the unit mass can be realized in the future by different physical principles and with smaller and larger masses besides one kilogram. If the recipe for realizing the mass was available to intelligent beings in the universe, they could realize the same kilogram anywhere (black holes are excluded here). Until then, the export of the IKP or a calibrated mass would have been necessary.

For the realization of the "new kilogram" on the basis of the Planck-Constant, the Physikalisch-Technische Bundesanstalt (PTB) runs two experiments based on the currently most promising approaches, the Planck-Balance and the x-ray-crystal-density (XRCD) method. The XRCD method colloquially also called Avogadro or Si-28 method is based on the realization of the kilogram via silicon spheres.

The achievements gained over the past decades in dealing with mass standards of silicon were the driver for new developments at PTB, which are the subject of this article. They include investigations on the long-term stability of the standards, their simple and robust handling such as easy to use cleaning procedures and the development of inexpensive and highly available mass standards made of natural silicon. These benefits were presented for the first time at the international workshop "round and ready" in 2016 [7] where leading metrology institutes from all over the world participate. Since end of 2016, PTB handed about a dozen silicon spheres to partners all over the globe. This enables metrology institutes to gain their own experience in handling the silicon spheres.

Moreover, these developments fall into a time when digitalization starts to play a key role in science and business. To support this, PTB developed a machine-readable digital calibration certificate which, in addition to administrative data, also covers all essential physical properties of silicon spheres.

2. Silicon mass standards

Silicon mass standards can be classified into at least three different categories. The distinction is made according to the features such as material, geometry and surface quality. Today, they are manufactured by a nominal mass of 1 kg.

The first category consists of spheres of highly enriched isotopic monocrystalline silicon ^{28}Si , used for the realization of the mass based on the Planck-Constant h , the second comprises "quasi-primary" spheres $^{\text{nat}}\text{Si}_{\text{qp}}$, made of natural monocrystalline silicon. Spheres of this category must be considered as primary realization too, as the mass will be traced back to volumetric and density measurements independent of any weighing process. The third category are secondary silicon spheres $^{\text{nat}}\text{Si}_{\text{sc}}$, equally made of natural monocrystalline silicon for industrial use. The mass will be calibrated by mass substitution methods. The spheres of these categories are distributed with various specifications, costs and accuracy.

All spheres have in common that they are extremely stable over time. Their monocrystalline structure makes it nearly impossible to be penetrated by other atoms. The silicon oxide also creates a very hard and protective surface layer. Suitable cleaning solutions and cleaning wipes ensure that all contaminants on the surface can be removed completely without damaging the silicon-oxide layer. This is an essential criterion to ensure the reproducibility of measurements.

Obtaining a silicon sphere is of course only meaningful if it is not only used for the realization but also for the dissemination of the mass. Currently, steel cylinders are used for this purpose. Calibration requires, that all significant systematic deviations of a measurement process must be determined and corrected in a traceable way to the SI. Main influences are the effective air density, which in turn depends on temperature, air pressure, humidity and gas composition, the standard's geometry, magnetic properties of the material and orientation of the mass standard during the measurement process. Most of these influences can be avoided if the measurements are carried out in vacuum. For

this purpose, suitable mass comparators are available on the market. If, on the other hand, the calibration of a mass standard is carried out against a calibrated sphere in air, it must be corrected for environmental influences. A detailed description of suitable measuring methods, basic setups of measuring instruments as well as equations for the mathematical correction of systematic influences and use of suitable buoyancy bodies are described in [8]. Within the PTB, air density is determined with a relative standard uncertainty of 10^{-4} U ($k = 1$). This requires sensors with the following specifications: pressure: 3 Pa, temperature: 0.01 K, humidity: 1 %, content of CO₂: 10^{-5} . Calibrating of a 1 kg steel cylinder by a silicon sphere leads to an uncertainty contribution of U ($k = 1$) 37 μg .

Table 1. Different categories of silicon spheres depending on material, geometry and surface quality.

	²⁸ Si	^{nat} Si _{qp}	^{nat} Si _{sc}
category	primary	“quasi-primary”	secondary
$u_{\text{rel}}(k=1)$ of mass	$2 \cdot 10^{-8}$	$3 \cdot 10^{-8}$	$3 \cdot 10^{-8}$
form error RONt	< 30 nm	< 20 nm	< 80 nm
average roughness Ra	< 0.3 nm	< 0.5 nm	< 1 nm
expected price	> 1 Mio. €	> 100 k €	> 10 k €
availability	limited, PTB	PTB/ industrial supplier	industrial supplier

In order to gain further experience in long-term stability for ²⁸Si and ^{nat}Si_{qp} spheres, PTB will regularly observe the mass change on the spherical surfaces. This is done on a specially developed XPS / XRF measuring device (XPS: x-ray photoelectron spectroscopy; XRF: x-ray fluorescence spectroscopy). Although theoretically no changes in the volume of the spheres or its densities are expected, recalibration might be appropriate before executing mass comparisons among leading institutes.

3. Digital Calibration Certificate

The Digital Calibration Certificate (DCC) serves for electronic storage and uniform interpretation of calibration results. The use of cryptographic signatures makes the DCC integer and authentic. Under the lead of the PTB, a concept is being developed that will allow these data to be handled in automated processes in the future [9]. The project will support all facilities worldwide, where the metrological traceability of measurement results shall be proved. This includes metrology institutes and designated institutes, national calibration offices, calibration laboratories and many facilities in the industry that require reliable information for customers or their quality management system.

The analogue calibration certificate has so far rarely generated a surplus value for companies since data obtained during the calibration is time-consuming and error-prone. The DCC compensates all these crucial disadvantages of its analogue counterpart. Because of its machine readability, digitally enhanced production and quality monitoring, processes are supported massively. This generates a crucial added value for all companies using the DCC.

In addition to the structure of the DCC, special framework conditions must be fixed for the transfer of data. This also includes cryptographic protection procedures. By these the electronic transmission of the contents as well as the integrity and authenticity of the contents of the DCC can be ensured.

The DCC provides an internationally recognized DCC format intended to become a worldwide standard in the entire field of metrology.

The general structure of the DCC is subdivided into four areas:

- Administrative data: This area is mandatory and fully regulated. It contains essential data of general importance such as the name of the measurement object, calibration laboratory, reference number, date and all other administrative data usually found on the first page of an analogue calibration certificate.
- Measurement results: This area is mandatory and partially restricted. It contains all measurement results provided with the calibration certificate. All measurement data which are of legal relevance have to be expressed in the SI. Alternative units such as miles, gallons, degrees of Oechsle or others are allowed as parallel information. Moreover, in this area tables, figures, equations and text can be added in any preferred language.
- Comments: This area is optional and not regulated. It allows adding any digital information which according to the calibration laboratory should also be transferred, for instance the raw measurement data of an x-ray scan which, due to the amount of data, is not part of any analogue document today.
- Analogue calibration certificate: This area is optional but recommended: It contains a digital copy of the analogue calibration certificate and shall provide users with a convenient access to a human readable document.

All areas are described in XML (Extensible Markup Language).

4. Summary

²⁸Si-spheres are predestinated for the realization of future mass realizations derived from the Planck-Constant. Due to their excellent properties, they are generally suitable as highly stable and easy-to-handle mass standards on the highest metrological level. Against this background, PTB, together with industry partners, has set up an infrastructure that can also be used to produce mass standards made of natural silicon spheres. Recent developments in digitization have been triggers to address further developments. The Digital Calibration Certificate enables a uniform and machine-readable transfer and the management of calibration data.

Acknowledgments

The authors wish to acknowledge J. Hauser GmbH & Co. KG and Häfner Gewichte GmbH for establishing a metrological infrastructure for silicon mass standards and for their participation. The Si-kg project is part of the TransMeT program which is funded by the German Federal Ministry of Economic Affairs and Environment. Additional thanks go to our colleagues from division 1 “Mechanics and Acoustics” and division 5 “Precision Engineering” from PTB for their daily work and collaboration.

5. References

- [1] BIPM; International Prototype of the kilogram; <https://www.bipm.org/en/bipm/mass/ipk/IKP>; access March 2018
- [2] PTB Mitteilungen; Experimente für das neue Internationale Einheitensystem (SI); 126. Jahrgang, Heft 2, Juni 2016
- [3] Maxwell J C; The scientific papers. Teil 2 (Hrsg. W. D. Niven), University Press, Cambridge (1890) page 225
- [4] Planck M; Ann. Physik 1 (1900) 69
- [5] BIPM; Comptes rendus des séances de la Neuvième Conférence Générale des Poids et Mesures; meter convention page 106
- [6] PTB; Round and Ready, Massenormale der PTB für das neue Kilogramm sind einsatzbereit; www.ptb.de, access 2 March 2018
- [7] OIML R 111-1; Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3 Part 1: Metrological and technical requirements; Edition 2004 []
- [8] Hackel S, Härtig F, Hornig J, Wiedenhöfer T: The digital calibration certificate. PTB-Mitteilungen 127 (4), 75–81 (2017), doi: 10.7795/310.20170499