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STATUS OF MASS METROLOGY AT NIST IN 2000

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Abstract: This paper summarizes the activities in mass metrology at NIST. It includes a description of the facilities and the procedures used in the dissemination of the mass unit. This paper also discusses the research efforts to understand and characterize the stability of mass standards, especially the US national prototype kilograms K4 and K79. The on-going research for the development of improved transfer standards of mass is also reported.
Keywords: mass metrology, stability of mass standards

1 INTRODUCTION

The unit of mass, the kilogram, is the last remaining basic unit of the *Système International d'Unitès* (SI) defined by an artifact. The definition dates back to the 1901 *Conference Generale des Poids et Mesures* and it simply states [11: "The kilogram is the unit of mass, it is equal to the mass of the International Prototype of the Kilogram". The definition does not make provisions for any surface or environmental conditions. The International Prototype of the Kilogram (IPK) is a cylindrical artifact made from a platinum-iridium alloy (90 % platinum, 10 % iridium). It is kept in ambient air, under a triple bell jar, at *the Bureau International des Poids et Mesures* (BIPM) in Sèvres, France. It is subject to environmental contamination, wear due to usage, and other possible surface effects such as absorption, adsorption, andlor desorption. In addition, the IPK is always at risk of being damaged or destroyed. To make provision for the contamination problem, the kilogram definition was re-interpreted by the *Comitè International des Poids et Mesures* in 1989 [2]. The interpretation states that: "The Kilogram is equal to the mass of the International Prototype of the Kilogram after cleaning and washing using the BIPM method". This method [3], while relying on the human touch, is believed to restore the mass of the IPK to its "ideal" mass of one kilogram. At the time of the manufacture of the standard of mass, replicas were made, their masses were measured against the IPK before being distributed to the U.S. and other members of the Meter Convention. These replicas known as national prototype kilograms play the role of national mass standards. Periodically, the BIPM re-calibrates the national prototypes against IPK. The last re-calibration, known as the third periodic verification, took place between 1988 and 1992 [4]. The results, shown in Figure 1, clearly indicate a spread of values over time. This spread can be interpreted as instability in the mass unit or a possible drift of 30 µg to 50 µg over the last 100 years. For reference purposes, the value of K20, the US national standard, is indicated. This study does not give any indication whether IPK is loosing mass or the other prototypes are gaining mass. The only conclusion is that there is a relative drift in the unit of mass.

The cause of the drift is unknown. However, it is believed that it is due to material and/or surface effects that are not fully understood at this point.

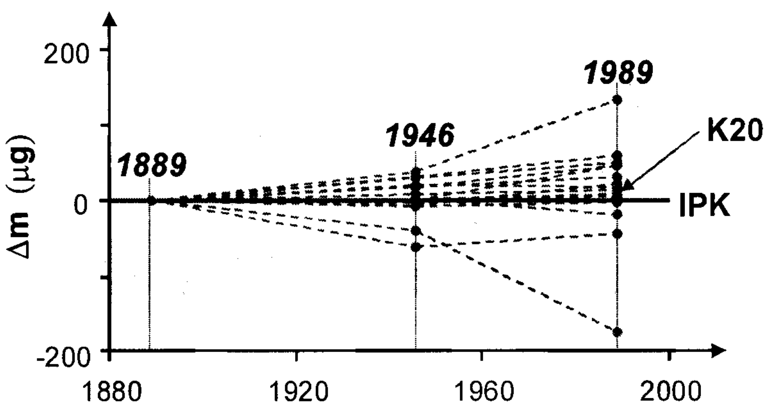

Figure 1. Results of the third periodic verification of national prototype kilograms by the BIPM

2 SURFACE PROFILES OF PRIMARY MASS STANDARDS

In an effort to quantity the stability of mass standards, we performed a characterization of the surface roughness and profiles of our national prototype kilograms K4 and K79 using non-contact surface profiling and optical microscopy techniques. K4 and K79 are representatives of the two existing types of surface finish for primary platinum-iridium kilograms. K4 is one of the first 40 replicas made; it was hand polished. K79 is representative of the newer family of Pt-Ir kilograms manufactured at the BIPM using diamond turning techniques. A summary of the results is provided here. For a more detailed account of this work, the reader is referred to reference [5].

2.1 K4

K4 is one of two mass standards allocated to the United States. The second mass standard, K20, is the national standard of mass in the United States. K4 plays the role of check standard that is used to monitor the constancy of the mass unit in the United States. Both K4 and K20 belong to the original group of 40 prototype kilograms manufactured in 1882. All 40 kilograms were manufactured from the same alloy and by the same process. We believe that the surface of K4 is therefore a good representation of the surfaces of the original national mass standards.

The machining lines on K4 are visible to the naked eye. In addition, a few scratches are notably present on the flat and cylindrical surfaces, they have been historically reported on K4 [6]. Optical microscope profiles reveal, in addition to the machining lines and numerous random scratches, wear lines due to usage on balance pans for a period spanning over more than a century. These lines can be seen in Figure 2 as short line-segments perpendicular to the machining lines. Using a white-light scanning interferometer, we measured average roughness values, R_a, ranging from 63 nm to 84 nm at different locations on the flat surfaces of K4 excluding the center. The repeatability in a single measurement location of the average roughness was 1 nm. A detailed mapping of the surface of K4 can be found in reference [5].

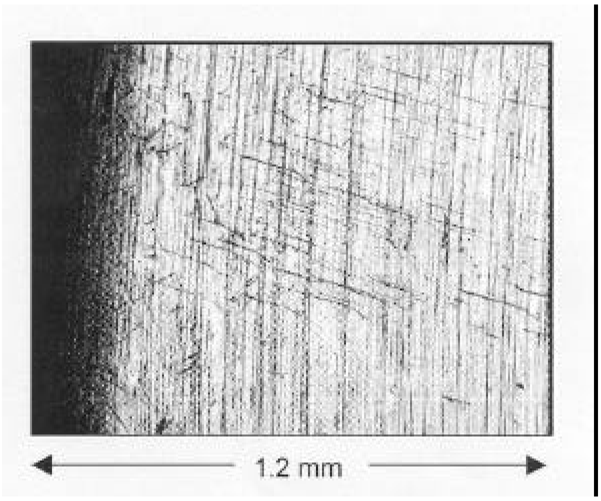

Figure 2. Optical microscopy profile of the bottom surface of K4 near the edge revealing machining lines and wear marks.

It is worth noting that in spite of the peculiar surface texture that K4 exhibits, its mass, relative to IPK, has changed only by 31 µg between two calibrations at the BIPM in 1889 and 1984 [6]. At the time of preparation of this manuscript, K4 was at the BIPM for cleaning and re-calibration. Re-examination of the surface profiles after cleaning combined with the change in mass will hopefully shed some light on the effects of cleaning on surface characteristics and possibly lead to a correlation between surface characteristics changes and mass changes for platinum-iridium mass standards.

2.2 K79

K79 was acquired by NIST in 1996. It was manufactured at the BIPM in 1986 by turning with a diamond tool. To the naked eye, the surface of K79 looks very specular in comparison with K4. When K79 was placed under the microscope, the improved surface quality was obvious, yet, some peculiarities were found.

The surface roughness was measured with a phase-measuring micro-interferometer. The average roughness, R_a, ranged form 10 nm to 15 nm at different locations on the flat surfaces of K79 with repeatability of 1 nm for a single measurement location.

In addition, the optical microscopy profiles show evidence of increasing grain size with increasing distance from the center, as shown in Figure 3. The origin of this non-uniformity in grain size is still under investigation and is most likely attributed to the interaction between the platinum-iridium artifact and the diamond tool or to Pt-Ir material properties. We observed only a few wear marks compared to the surface of K4.

While it is commonly believed that prototype kilograms with improved surface properties obtained from diamond turning are more stable than the ones hand polished, long-term history is not yet available to support this hypothesis.

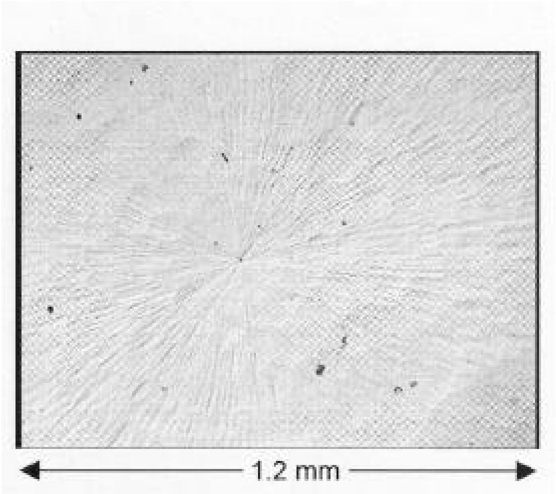

Figure 3. Optical microscopy profile of K79 at the center showing the non-uniform grain size distribution

3 DISSFMINATION OF THE MASS UNIT

The traceability of the mass unit in the US to the IPK is illustrated in Figure 4. K20 is the national standard of mass and it is directly tied to the IPK through the working standards of the BIPM. Secondary 1 kg mass standards made of stainless steel are used to disseminate from the platinum-iridium primary standards. Two sets of 1 kg stainless steel artifacts are in turn calibrated against the secondary standards and are used as working standards in the dissemination of the mass unit to multiples and sub-multiples of the kilogram using weighing designs described in detail in reference [7]. Weighing designs are aimed at transferring the mass unit to multiples and submultiples of the kilogram and achieving the smallest variance for the individual masses involved while keeping the number of observations a minimum.

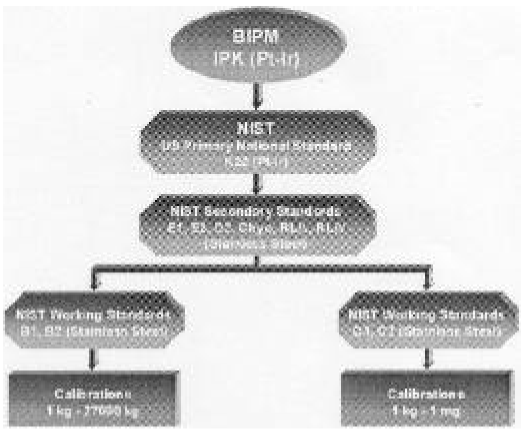

Figure 4. Traceability of NIST mass standards to the International Prototype Kilogram

A weighing design consists of multiple double-substitution weighings involving different combinations of mass standards. It incorporates artifacts with unknown mass with NIST reference and check standards. The values of the reference standards in each design are used as a restraint in the least square solution of the design. Check standards are mass standards with a known or "accepted" mass values. Check standards are treated as unknowns and their mass values are measured and compared to "accepted" values using t-test statistics. Monitoring the measured mass of an artifact with known mass value provides a monitoring of the accuracy of the measurement process. The validity of the t-test is based on the assumption that the mass of the check standard does not change from its accepted value that must be regularly updated. The standard deviation of the least-squares fit to the data obtained from the weighing design measurements is calculated and compared to the accepted standard deviation of the balance using f-test statistics. The accepted standard deviation of a balance is based on a very large number of measurements collected over a long period of time. By monitoring the scattering of the data obtained in the weighing design measurements, the f-test monitors the precision of the measurement process. The validity of the f-test relies on the assumption that the scatter of the data is typical of the scatter obtained from previous measurements using the same balance. The relative and absolute standard uncertainties in mass calibrations typically obtained at NIST from this dissemination process are shown in Figure 5 for masses ranging from 1 mg to 5 000 kg.

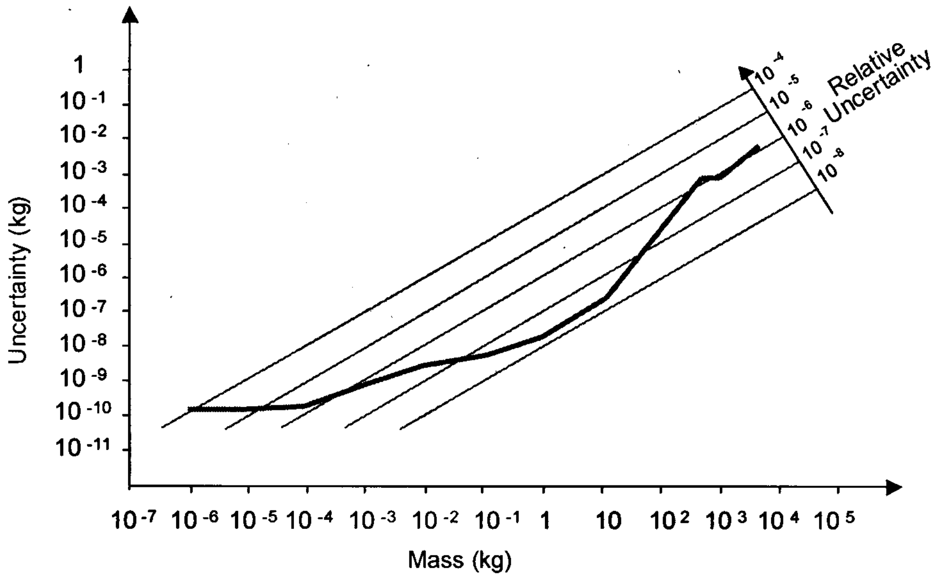

Figure 5. Absolute and relative standard uncertainties in NIST mass calibrations

Since mass measurements are generally performed in ambient air, the uncertainty at the kilogram level, is dominated by the contribution from the air buoyancy correction, which results from the large difference in volumes between Pt-Ir prototypes and stainless steel transfer standards. The uncertainty due to the air buoyancy correction at N IST is typically 10 µg for a 1 -kg stainless steel mass standard. Since this component is inherent in the uncertainty of the stainless steel working standards, it propagates to every mass measurement made in the U.S. Therefore, any significant reduction in the total uncertainty requires minimizing the uncertainty due to the buoyancy correction. Since this component is proportional to the difference in volumes between the primary Pt-Ir and Stainless steel kilograms, the key is to fabricate mass standards using a material whose density is as close as possible to that of Pt-Ir. Tungsten was chosen as a possible candidate. The nominal, manufacturer-specified, density of tungsten is 19300 kg/m^3 at 20 °C compared to 21500 kg/m^3 for Pt-Ir and 8000 kg/m^3 for stainless steel. The use of tungsten 1-kg artifacts in the dissemination from Pt-Ir would reduce the uncertainty due to the buoyancy correction to less than 1 µg. Our goal is to develop economical mass standards that meet the legal metrology requirements as specified by OIML R111 [8] and to minimize the uncertainty in the buoyancy correction. At the time of preparation of this manuscript, we were in the process of manufacturing a prototype tungsten kilogram artifact; a status update will be given at the conference.

4 FACILITIES

At NIST, electronic mass comparators, fully and partially automated, are used for calibrations in the range from 1 mg to 10 kg, while mechanical balances are used to cover the range between 10 kg and 30 kg. Partial automation refers to the automation of the data collection from the comparators and monitoring the environmental transducers, as well as the automatic analysis of the collected data while full automation includes also the remote operation of the comparators. The environmental conditions in the calibration laboratories are such that the relative humidity is set between 40% and 50% with variations of no more than 5% per 24 hours and the temperature is set between 20 °C and 22 °C with maximum variations of 0.5 °C over a period of 12 hours. Electrostatic filters are used to insure proper cleanliness with 97% filtration efficiency.

A special area is dedicated to the calibration of large weights between 30 kg and 27000 kg using mechanical balances. The temperature is maintained between 21 °C and 23 °C with maximum variations of 1.5 °C per 12 hours. Unfortunately, there is no humidity control or air filtration in this area.

A clean room facility with tight environmental control houses our state-of-the-art, fully automated and remotely operated 1 kg, 100 g, and 10 kg comparators. The environmental conditions are such that temperature is controlled to within 0.1 °C between 20 °C and 22 °C, temperature gradients are less than 0.1 °C over an elevation of 1 m, relative humidity is within 2% between 45% and 50%, and cleanliness of class 1000 accomplished with a HEPA filtration system with a 99.99% efficiency for particles of size 0.5 µm or larger.

NIST also maintains facilities for hydrostatic [9] and immersed [10] solid density measurements and for the characterization of the magnetic properties of mass standards [11].

We are currently in the process of building a new facility that will be dedicated for mass measurements under vacuum and controlled atmospheres.

4 ACKNOWLEDGEMENTS

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