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EXAMINATION OF PARAMETERS THAT CAN CAUSE ERROR IN MASS DETERMINATIONS

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DEFINITION OF MASS

We present the following quotation of Condon and Odishaw [1] as a succinct definition of mass: "The property of a body by which it requires force to change its state of motion is called inertia, and mass is the numerical measure of this property."

THE MASS UNIT

According to Maxwell, "every physical quantity (mass in the present case) can be expressed as the product of a pure number and a unit, where the unit is a selected reference quantity in terms of which all quantities of the same kind can be expressed." The fundamental unit of mass is the international kilogram. At present the kilogram is realized as an artifact. Originally, the artifact was designed to have the mass of 1 cubic decimeter of pure water at the temperature of maximum density, 4 °C. Subsequent determination of the density of pure water with the air removed at 4 °C under standard atmospheric pressure (101325 pascals) yielded the present value of 1.000028 cubic decimeters for the volume of one kilogram of water.

MASS ARTIFACTS, MASS STANDARDS

The present embodiment of the kilogram is based on the French platinum Kilogram of the Archives constructed in 1792. Several platinum-iridium cylinders of height equal to diameter and nominal mass of 1 kilogram were manufactured in England. These cylinders were polished and adjusted and compared with the Kilogram of the Archives. The cylinder whose mass was closest to that of the Kilogram of the Archives was sent to the International Bureau of Weights and Measures (BIPM) in Paris and chosen as the International Prototype Kilogram in 1883. It was ratified as the International Prototype Kilogram (IPK) by the first General Conference of Weights and Measures (CGPM) in 1899. Other prototype kilograms were constructed and distributed as national prototypes. The United States received prototypes Nos. 4 and 20. All

other mass standards in the United States are referred to these. As a matter of practice, the unit of mass as maintained by the developed nations is interchangeable among them.

MASS COMPARISON

The gravitational force exerted on a balance mechanism by a standard kilogram is compared to the gravitational force exerted by an artifact of mass and density nominally equal to those of the standard kilogram to determine the mass of the artifact. That is, the balance is a mass comparator. If these forces are not equal, a second mass artifact (or a combination of artifacts) whose mass (previously determined by an iterative process) is a small fraction of that of the standard kilogram is required to calibrate the balance response in terms of the mass unit.

BUOYANCY

When the above mass comparison is made between a platinum-iridium standard and an artifact fabricated from a material of different density, the gravitational forces on the two bodies are opposed by buoyant forces whose inequality must be taken into account. Archimedes' principle [2] provides the necessary information to account for the buoyant forces. The gravitational force,

$$F_1 = Mg \tag{1}$$

is opposed by a buoyant force,

$$F_2 = \rho_a V_M g \tag{2}$$

where M is the mass of the object, and g is the acceleration due to gravity, ρ_a is the air density, and V_M is the volume of the object.

The difference between the gravitational force and the buoyant force is most conveniently expressed as

$$F = F_1 - F_2 = Mg\left(1 - \frac{\rho_a}{\rho_M}\right) \tag{3}$$

where ρ_m is the density of the object.

THE FUNDAMENTAL MASS COMPARISON RELATIONSHIP

This fundamental relationship is expressed by the following equation:

where S is the mass of the standard, ρ_S is the density of the standard, X is the mass of the object being compared to the standard, ρ_X is the density of the object, and δ' is the mass difference

$$\left[S\left(1-\frac{\rho_a}{\rho_s}\right)-X\left(1-\frac{\rho_a}{\rho_x}\right)\right]g=\delta' g \tag{4}$$

indicated by the balance. If the centers of gravity of the two weights are not in the same horizontal plane, there is a small correction due to gravitational gradient [3].

Solving the above equation for X,

$$X=\frac{\left[S\left(1-\frac{\rho_a}{\rho_s}\right)-\delta'\right]}{\left(1-\frac{\rho_a}{\rho_x}\right)} \tag{5}$$

WEIGHING DESIGNS

Conceptually, one could use the fundamental relationship to determine X from one δ' observation. However, there are a number of weighing designs [4] that allow a more precise determination of X and have the additional advantage of checking the consistency of the prototype kilograms. At NIST we have the unique advantage of having <u>two</u> prototype kilograms. That being the case, we can employ the following weighing design:

K_{20}	K_4	X_1	X_2	BAL. OBS.
1	-1	0	0	δ'_1
1	0	-1	0	δ'_2
1	0	0	-1	δ'_3
0	1	-1	0	δ'_4
0	1	0	-1	δ'_5
0	0	1	-1	δ'_6

where K_{20} and K_4 represent the prototype kilograms, X_1 and X_2 represent unknown kilograms, and the δ's are the balance observations. The plus and minus 1's are used to indicate differences between masses; for example, in the first line the 1 under K_{20} and the -1 under K_4 means that the difference in mass, $K_{20} - K_4$ is indicated by the balance observation δ'_1. This weighing design is referred to as a "4-1's series",and in general is referred to as a "combinational" weighing design. δ'_1 is the consistency check, i.e., the data is used to perform a statistical "t-test."

The solution equation for X_1 is given by the following equation:

$$X_1 = (-3\delta'_2 - \delta'_3 - 3\delta'_4 - \delta'_5 + 2\delta'_6 + 4K)/8$$

For simplicity in the above equation buoyancy has been ignored and the restraint, K, is the sum of the two Pt-Ir kilograms. The estimate of the standard deviation resulting from the least-squares process is used in calculating the random (type A) component of the uncertainty assigned to X_1 and X_2. Subsequently, to protect the prototype kilograms from wear, X_1 and X_2 can be used as working standards at NIST. As a matter of practice, X_1 and X_2 are fabricated from stainless steel and not platinum-iridium and therefore one can expect an increase in the uncertainty due to the uncertainty in the buoyancy, as shall be discussed later.

Uncertainties in the Determination of X Due to Uncertainties in Parameters in Equation 5

Equation (5) gives the relationship between X and various parameters. We undertake now to propagate the uncertainties in the various parameters using the method of Ku [5]. According to Ku:

$$(SD)^2 = \sum_i \left(\frac{\partial X}{\partial Y_i}\right)^2 (SD_i)^2 \tag{6}$$

The various partial derivatives are:

$$\frac{\partial f}{\partial S} = \frac{\left(1 - \frac{\rho_a}{\rho_s}\right)}{\left(1 - \frac{\rho_a}{\rho_x}\right)} \tag{7}$$

$$\frac{\partial f}{\partial \rho_x} = -\frac{\rho_a\left[S\left(1 - \frac{\rho_a}{\rho_s}\right) - \delta'\right]}{\rho_x\left(1 - \frac{\rho_a}{\rho_x}\right)^2} \tag{8}$$

$$\frac{\partial f}{\partial \rho_S} = \frac{\rho_a S}{\rho_S^2\left(1 - \frac{\rho_a}{\rho_x}\right)} \tag{9}$$

$$\frac{\partial f}{\partial \rho_a} = \frac{S\left(1 - \frac{\rho_a}{\rho_s}\right) - \delta'}{\rho_x\left(1 - \frac{\rho_a}{\rho_x}\right)^2} - \frac{S}{\rho_S\left(1 - \frac{\rho_a}{\rho_x}\right)} \tag{10}$$

$$\frac{\partial f}{\partial \delta'} = -\left(\frac{1}{1-\frac{\rho_a}{\rho_x}}\right) \tag{11}$$

Examination of the partial derivatives reveals the need for an uncertainty estimate for the air density term, ρ_a. The air density is calculated from the CIPM 81-91 formulation and requires knowledge of air temperature, barometric pressure, relative humidity and CO_2 content; all measured in the weighing chamber. The partial derivatives of the air density equation with respect to the above parameters are:

$$\frac{\partial \rho_a}{\partial P} = \frac{\rho_a}{P} \tag{12}$$

$$\frac{\partial \rho_a}{\partial RH} = -\frac{0.034970}{T} \tag{13}$$

$$\frac{\partial \rho_a}{\partial X_{CO_2}} = 12.011\frac{\rho_a}{M_a} \tag{14}$$

$$\frac{\partial \rho_a}{\partial T} = -\frac{\rho_a}{T} \tag{15}$$

where X_{co_2} is the mole fraction of CO_2 in the air and M_a is the apparent molecular weight of the air.

In Table 1, we give our estimates of the uncertainties that can be achieved for these parameters and have evaluated the partial derivatives. The root sum square (RSS) uncertainty is carried forward as the air density uncertainty (type A). The type B uncertainty arising from the constant parameters of the air density equation is insignificant.

In Table 2, the uncertainties in the comparison of a Pt-Ir artifact with the U.S. National Prototype kilogram are tabulated. From examination of Table 2, we see that the two major uncertainties are the uncertainty on the National Prototype kilogram provided by BIPM (the coverage factor is 1), and the imprecision of the balance.

In Table 3, the uncertainties in the comparison of a stainless steel artifact with the U.S. National Prototype kilogram are tabulated. All of the uncertainties in Table 3 are of the order of 1 microgram or higher, except for the uncertainty in the density of the National Prototype kilogram, and cause a very significant increase in the RSS uncertainty.

TABLE 1

Variable	Value	$SD(y_i)$	$SD(\rho_a)_i$, g/cm^3
Temperature (T)	295 K	5 mK	0.020×10^{-6}
Pressure (P)	100258 Pa	5.1 Pa	0.061×10^{-6}
Rel. Humidity (RH)	41%	1%	0.118×10^{-6}
CO_2 Mole Fract.	0.000440	0.000050	0.025×10^{-6}
		RSS =	0.14×10^{-6}

TABLE 2

Variable	Value	SD	$\partial x/\partial y_i$	$\left[SD(y_i)^2(\frac{\partial x}{\partial y_i})^2\right]^{1/2}$
S	1000 g	0.0000023 g	1	0.0000023 g
ρ_S	21.5 g/cm^3	0.000072 g/cm^3	-0.00260 cm^3	0.00000018 g
ρ^X	"	"	"	"
δ'	0.01 g	0.000001 g	-1	-0.000001 g
ρ_a	0.0012 g/cm^3	0.14×10^{-6} g/cm^3	0.0004651 cm^{-3}	6×10^{-11} g
			RSS=	2.5×10^{-6} g

INSTABILITY OF IPK

There is evidence that the mass of Pt-Ir prototype mass standard artifacts has changed montonically by about 50 micrograms, with respect to IPK, in the 100 years since inception of the IPK as the standard of mass [6]. Since IPK is one of the group of 40 original prototype kilograms, it is certainly conceivable that it is also changing in mass with time. We cannot know the magnitude of the change and the rate of change with time of the IPK. Insofar as mass metrology as practiced assumes the value of the IPK to be invariant and that all other mass standards are referred to the initial mass value of the IPK, mass metrology can be practiced at the level of several parts in 10^9 (see Table 2) relative to the IPK. However, if there are experiments, for example, in which the absolute mass of an object must be known to better than

50 micrograms per kilogram the present system based on the IPK, which varies in value, is inadequate.

TABLE 3

Variable	Value	SD	$SD(y_i)$	$\left[SD(y_i)^2(\frac{\partial x}{\partial y_i})^2\right]^{1/2}$
S	1000 g	0.0000023 g	1	2.3×10^{-6} g
ρ_s	21.5 g/cm^3	0.000072 g/cm^3	0.00260 cm^3	0.19×10^{-6} g
ρ_x	8.0 g/cm^3	0.000072 g/cm^3	0.01875439 cm^3	1.4×10^{-6} g
δ'	0.01 g	0.000001 g	-1	1×10^{-6} g
ρ_a	0.0012 g/cm^3	0.14×10^{-6} g/cm^3	78.5 cm^3	10.8×10^{-6} g
			RSS =	11.1×10^{-6} g

THERMAL EQUILIBRIUM

Probably the limiting systematic error remaining in the mass measurement is that due to the convective forces arising from the lack of thermal equilibrium between the mass artifact, the mass comparator, and the surrounding air [7,8]. In practice it is not possible to assure equality of temperatures of these three items; therefore, some minute convection will remain. It follows that the balance or comparator observation will always be biased by small convective forces and, therefore, the mass determination will have a systematic error on this account. Consequently, it is necessary to take precautions to approach thermal equilibrium as closely as possible. Prior to the work of Schoonover and Keller [7], these effects were usually ignored. To minimize the systematic error, safeguards, including the following, should be maintained:

1. Passive and active control of the thermal environment in and around the balance or comparator.
2. Adequate thermal "soaking" of the artifacts and the comparator mechanism.
3. Thermal sensors to assure that the safeguards are effective.

CLEANING

The mass of an artifact is dependent on the surface contamination present. Therefore, the mass and mass stability depend on the cleaning procedure prior to weighing. There are several cleaning procedures in use by BIPM and national standards laboratories.

The national prototypes of the kilogram (Platinum-Iridium) as received by the recipient country from BIPM has been cleaned and washed by BIPM using the BIPM procedures [9]. The mass assigned by BIPM is only applicable on a given date. Immediately following this date BIPM recommends adding 0.037 micrograms per day for a maximum period of three months. The National Physical Laboratory of Teddington England [10] found for its kilogram #18 a functional relationship between mass change and time, for BIPM data. The relationship is:

$$M_t = M_0 + 0.356097 \times t^{0.511678},$$

where M_t is the mass at time t after cleaning and washing, M_0 is the mass at the time of cleaning and washing, and t is the elapsed time in days. It is suggested by Plassa [10] that the above equation calculates mass values to within a few micrograms for a period of up to ten years following cleaning and washing, provided that the storage conditions can be carefully controlled.

The BIPM cleaning and washing procedures for platinum-iridium mass standard artifacts [9] involves solvent cleaning and steam washing. For cleaning, chamois leather is used which had been previously soaked for 48 hours in a mixture of equal parts ethanol and ether after which the absorbed solvent is wrung out of the leather. This soaked chamois leather is rubbed over the entire surface of the mass standard artifact. In the steam washing procedure, steam is directed to all parts of the surface of the artifact. National prototypes should be cleaned and washed using these BIPM procedures prior to use.

The BIPM practice for stainless steel mass standard artifacts omits the steam washing. However, national standards laboratories are free to clean stainless steel artifacts by whatever procedures they wish unless their stainless steel mass standards are calibrated by BIPM.

During the development of the solid object density scale [11,12,13] NBS (now NIST) studied the cleaning residue that remained on steel spheres, and vapor degreasing was found to be the superior cleaning method. The method was used in preparation for very high-precision mass measurement and diametric measurement by interferometry. Initially, inhibited 1,1,1-trichloroethane was used as a solvent for vapor degreasing. Ultimately ethanol was used due to its availabilty; if a fume hood were available, methanol could be used. NIST studies have revealed that mass measurements of stainless steel kilogram artifacts of significantly different surface areas have comparable standard deviations. This finding indicates that vapor degreasing of the disparate surface areas does not contribute uncertainty to the measurements. Numerous vapor degreasings over a period of a year did not result in different mass values. In the literature, it has been reported that alternate cleaning methods can change mass values and the variability of mass values. A newly-manufactured mass standard artifact requires more rigorous and varied initial cleaning procedures to remove effects contributed by the manufacturing processes.

MAGNETIC EFFECTS

In ultra-high-accuracy mass determination it is necessary to minimize the magnetic interaction between magnetic structures and the mass artifacts being compared. There may also be magnetic interaction between the mass artifacts and external magnetic fields; this interaction also must be

minimized. Davis [14] suggests the following strategies to reduce the force of interaction:

(a) Minimize μ_R - 1 [magnetic susceptibility] by selecting materials with low magnetic susceptibility at low fields;
(b) Minimize the volume of magnetic material in the structure; and
(c) Maximize the distance between the mass artifact and the magnetic structures or magnetic fields.

Based on handbook values for platinum and iridium, the magnetic susceptibility for 90% Pt/10% Ir is 0.00027. For AISI 316 stainless steel, the magnetic susceptibility is 0.003 and a sample of AISI 304 has been found to have a value as high as 0.038 and the alloy is slightly soluble in boiling water and in steam. Using the above strategies one should use standards manufactured from the 316 alloy. Since the magnetic susceptibility is a limiting factor, items (b) and (c) merit careful consideration. OIML suggest that for weights of class E_1, the magnetic susceptibility of the metal or alloy should not exceed 0.01 [15].

CONCLUSIONS

From the above analysis, it is clear that the calibration of Pt-Ir kilograms is the simplest of the calibrations and does not require the highest accuracy in measurement of the parameters in the air density equation. Usually the comparison of Pt-Ir kilograms is only made by BIPM. However, in the calibration of stainless steel kilograms, state-of-the-art measurements of the air density parameters are required in order to minimize the uncertainties on this account. However, if one is determining the mass of a silicon kilogram (by comparison with a stainless steel kilogram or with a Pt-Ir kilogram), such as would be done in an experiment to determine Avogadro's number, the state-of-the-art measurements are inadequate. They contribute almost all, 42 micrograms, of the uncertainty in the determination against stainless steel and 52 micrograms against Pt-Ir. For the Avogadro number experiment, the mass of the silicon kilogram should be determined in vacuum or by direct measurement of the air density [16] at the time of weighing if these relatively large uncertainties are to be avoided. Vacuum-weighing of the IPK or the national prototypes would be an unacceptable practice because mass could be lost in vacuum.

The Pt-Ir vs. stainless steel comparison requires near state-of-the-art measurements of the densities of the artifacts. The magnetic susceptibility of stainless steel should be checked to ensure that it is sufficiently low. The thermal history and thermal stability of the artifacts and their environment are crucial in the determinations of mass. The comparisons of artifacts whose densities lie between 7.8 and 8.4 g/cm^3 require less rigor because the density difference is smaller than that between Pt-Ir and stainless steel.

DISCUSSION

The 50 micrograms of instability, or possibly more, in the IPK is, at the present time, not threatening to practical mass measurements, most of which never require accuracy better than 1 part per million. As previously discussed the drift has occurred over the course of 100 years and has only been detectable in recent times. The successful development of the NIST-balance

with a precision of better than 1 part per billion was a crucial step in highlighting the problem. There is no reason to believe the drift will not continue at the present rate giving the metrology community time to find a time invariant replacement for the IPK. However, as pointed out by the recent work of the National Physical Laboratory of the UK, mercury amalgam on the surfaces of Pt-Ir artifacts may be another matter of concern [17].

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