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MASS VALUES OF 1 KILOGRAM STAINLESS STEEL MASS STANDARDS TRACEABLE TO THE BRAZILIAN NATIONAL PROTOTYPE OF THE KILOGRAM

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Abstract: Dissemination of the mass unit and realization of the mass scale begin now in Brazil with the transfer of the mass value from the national prototype kilogram K66 to a set of 1 kg stainless steel mass standards. This is performed by applying the proper weighing design. This was done for the first time in Brazil obtaining mass values for a set of three 1 kg stainless steel mass standards with reference to national Pt-Ir prototype kilogram K66.
Key words: kilogram, prototype, dissemination, weighing designs

1. INTRODUCTION

Among the main tasks of the Mass Laboratory of INMETRO, the National Institute of Metrology of Brazil, are maintaining and disseminating the SI mass unit, the kilogram, in order to provide traceability from the international prototype of the kilogram – kept by the Bureau International des Poids et Mesures – for mass measurements in Brazil.

As the first stage of dissemination process it was carried out the transfer of the mass value from K66 to a set of three 1 kg stainless steel mass standards.

For the first time traceability from the international prototype of the kilogram was derived from the Pt-Ir brazilian national prototype of the kilogram K66.

2. METHODOLOGY

The three 1 kg stainless steel mass standards used for the transfer of the mass value of the Pt-Ir prototype are identified as MP060 (Sartorius), R-PP062 (Mettler Toledo) and PR019 (Adolf Häfner). The first one has cylindrical shape and the other two have OIML format [1].

For the weighings, it was used a computer controlled electromagnetic force compensation mass comparator, Mettler Toledo AT1006. This comparator has a resolution of 1 µg and by means of an automatic load alternator with four positions it is capable of comparing up to four mass standards in a programmed sequence and for a specified number of weighing cycles and series. Standard deviations of 1 µg are regularly obtained with this comparator when comparing 1 kg stainless steel mass standards after a period of thermal stabilization within the laboratory weighing room.

Environmental parameters values were taken by an automatic data acquisition system, MeteorLabor Klimet A30, interfaced with the comparator control system so that data was taken at the time that each weighing result is obtained.

Table 1 shows the metrological characteristics of the instruments used for measurement of the ambient air parameters within the comparator's weighing chamber.

Table 1. Metrological characteristics of instruments used for air density determination

MeteorLabor Klimet A30	ID	d	u_c
Temperature	T1	0,001 °C	0,008 °C
	T2	0,001 °C	0,008 °C
Relative humidity	Dew point	0,001 °C	0,13 °C
Atmospheric pressure	P	0,001 hPa	0,025 hPa

Temperature sensors T1 and T2 are kept within the weighing chamber of the mass comparator . The relative humidity dew point sensor samples the air of the weighing chamber. The pressure sensor is placed at the level of the weighing plate. With this setup it was possible to perform all six mutual comparisons of four mass standards in a sequence or weighing design as follows, Table 2, Cameron *et al*[2].

Table 2. Weighing design

Comparison	K66	R-PP062	MP060	PP019
1	–	+		
2	–		+	
3	–			+
4		–	+	
5		–		+
6			–	+

The execution of the weighing design above provides six mass differences, one for each comparison. These absolute mass differences Δm correspond to the difference indicated by the comparator ΔI (weighing in air) corrected by the air buoyancy and the gradient gravitational effects (equation 1).

This weighing design forms a matrix of linear equations. Each mass difference was obtained from a series of six ABBA weighing cycles for each pair of mass standards involved in the weighing design.

2.1. Ambient conditions

Regarding that air buoyancy correction is the main component of uncertainty for the described process, here it is shown the performance of the ambient air control system.

The air conditioning system of the weighing room maintains temperature stable within 0,2 ºC and relative humidity stable within 3 %, both in a 24 hours period.

This ambient stability keeps temperature and relative humidity variations inside the weighing chamber lower than 0,05 ºC and 0,6 %, respectively, during each weighing series which takes about 1 hour long.

The mass comparator is not placed within an air tight chamber so, weighings were carried out under local atmospheric pressure. Pressure variations were never above 1 hPa along a weighing series of six ABBA cycles for every comparison which took about 1 hour long. The mole fraction of CO_2 was estimated to be 0,00043 within the weighing chamber. Table 3 shows the typical values for ambient air parameters, in the weighing chamber, and their largest variation during a weighing cycle.

Table 3. Typical values of ambient air parameters and their largest variation in a weighing cycle

Temperature	20 °C	0,030 °C
Relative humidity	49 %	0,15 %
Atmospheric pressure	1013 hPa	0,1 hPa

Air density was determined from these parameters and from the mole fraction of CO_2 for each weighing using the CIPM 2007 equation, [3].

Figure 1 shows a typical curve for air density variation in a 24 hours period within the comparator's weighing chamber.

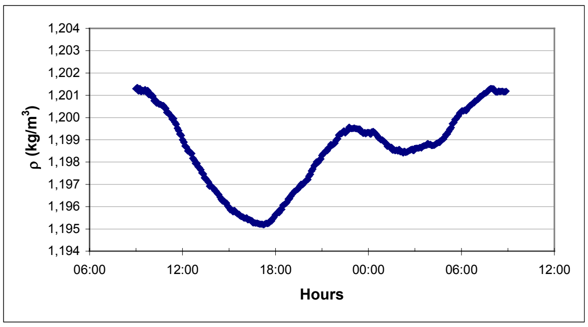

Figure 1. Typical air density curve in a 24 hours period.

2.2. Mathematical model for the absolute mass difference Δm

In order to obtain absolute mass differences Δm from the difference ΔI indicated by the comparator the mathematical model in equation (1) has been applied. This mathematical model is based on the balance of forces due to gravity and due to air buoyancy which act upon the weights during weighing.

$$\Delta m = \Delta I.S + \rho_{ar}.\left[V_i.(1-\alpha_i.\Delta T) - V_j.(1-\alpha_j.\Delta T)\right] - VN.\left(\frac{\partial g}{g.\partial h}\right).\Delta h \tag{1}$$

where:

Δm absolute mass difference between the standards (m_i - m_j)
ΔI indications difference displayed by the comparator
S balance sensitivity
ρ_{ar} air density during the comparisons
V_i , V_j volumes of mass standards at 20 ºC
α_i , α_j coefficients of volume expansion
ΔT temperature difference of mass standards in relation to reference temperature of 20 ºC
VN nominal value of mass standards
g gravitational acceleration
$\left(\frac{\partial g}{g \cdot \partial h}\right)$ relative gradient of gravitational acceleration
Δh height difference between the centers of mass

2.3. Applied corrections

2.3.1. Sensitivity

The balance sensitivity was determined before performing the complete set of comparisons. The measured value was: S = 0,99984 mg/mg with a standard uncertainty u_S = 0,00011 mg/mg.

2.3.2. Air buoyancy

The air buoyancy term in equation (1) was calculated using the following values:

- the air density, determined from CIPM 2007 equation;
- the measured volume of mass standards, shown in Table 4;

Table 4. Volume of mass standards

Mass standards	Volume at 20 °C	u_c
K66	46,4367 cm^3	0,0005cm^3 (estimated)
MP060	124,7734 cm^3	0,0010 cm^3
R-PP062	125,5161 cm^3	0,0008 cm^3
PP019	126,9556 cm^3	0,0006 cm^3

- the coefficient of volume expansion at the reference temperature of 20 ºC for all stainless steel mass standards was estimated as 48 x 10^{-6} cm^3 $ºC^{-1}$. For the prototype, the coefficient of volume expansion at 20 ºC was estimated from prototype calibration certificate [4], equation (2).

$$\alpha = (25{,}869 + 0{,}00565\ t_{90}).10^{-6}\ °C^{-1} \quad (2)$$

where:

α coefficient of volume expansion at 20 ºC

t_{90} temperature of the thermal equilibrium with air during the comparisons

2.3.3. Gravitational gradient

According to ON Measurement Report [5], the measured value of local vertical gradient of gravitational acceleration is – 1,9 x 10^{-6} s^{-2} with a standard uncertainty of 2 x 10^{-7} s^{-2}. Also, the measured value of local gravitational acceleration is 9,7874867 m/s^2 and its standard uncertainty is 4 x 10^{-7} m/s^2.

The height difference between centers of mass corresponding to mass standards R-PP062 and PP019 (OIML shape) and prototype's, was estimated to be 15 mm with a standard uncertainty of 2 mm. For the MP060 mass standard (cylinder shape) the respective estimated height difference was 8 mm with a standard uncertainty of 1 mm.

From the values shown above the gravitational gradient effect reduces the comparator indications, in mass unit, by about 2,9 µg for R-PP062 and PP019 and by about 1,6 µg for MP060.

2.3.4. Thermal effects

In order to reduce any effect arising from temperature difference on weighing results, Gläser [6], all weights were kept inside the weighing chamber, with the mass comparator turned on, about 48 hours before the weighings have been started.

2.3.5. Magnetic effects

Regarding that for the weighings it was used an electromagnetic force compensation comparator which can magnetically interact with the mass standards which are made of stainless steel alloy it was necessary to measure the magnetic susceptibility of the mass standards.

The magnetic susceptibility of each mass standard was measured using a susceptometer developed by the BIPM, Davis [7].

All magnetic susceptibility measured values for the stainless steel standards were lower than the permissible limit for OIML class E1 weights.

2.4. Solution of the linear system by restrained least squares approach

From the weighing design of Fig 1 and the absolute mass differences, equation (1), the following matrix of weighing equations can be obtained, equation (3):

$$\mathbf{Y} = \mathbf{X}.\beta + \mathbf{e} \quad (3)$$

where:

Y vector of the observed absolute mass differences

X design matrix

β vector of the unknown mass values

e vector of the unknown errors of the observations

The mass values for the standards were obtained from the solution of equation (3) using the classic least squares analysis with Lagrange multipliers considering the mass value of the reference, the prototype, as a restraint, Bich [8].

According to certificate 37/93 issued by the BIPM [4], the mass of the K66 prototype is:

1kg + 0,135 mg

$u(k=1)$ = 0,0023 mg

2.5. Uncertainty

The uncertainty estimation is obtained from the variance-covariance matrix where the diagonal elements are the variance values and the off-diagonal elements are the covariance values.

The least squares analysis provides the variance-covariance matrix ψ_A of the mass values originating from the weighing design, which is considered of type A, Bich *et al* [9].

The type B uncertainty was estimated considering the following contributions: the air buoyancy correction, the sensitivity of the mass comparator, the uncertainty of the mass of the prototype and the gravitational gradient. From the equation 1, the mass value β_i for mass standard i takes the form of equation (4):

$$\beta_i = \Delta I \cdot S + \rho_{ar}\left[Vi\cdot(1-\alpha_i\cdot\Delta T) - Vp\cdot(1-\alpha_p\cdot\Delta T)\right] - VN\cdot\left(\frac{\partial g}{g\cdot\partial h}\right)\cdot\Delta h + m_p \quad (4)$$

where m_p is the mass value assigned to the reference, the mass of the prototype. The other terms in equation (4) are the same as in equation (1).

The type B uncertainty was obtained from the law of propagation of uncertainty as reported in ISO GUM [10] applied to equation (4), on the assumption of independent input quantities values:

$$u_B^2(\beta_i) = \sum_{l=1}^{N}\left(\frac{\partial\beta_i}{\partial w_l}\right)^2 \cdot u^2(w_l) \quad (5)$$

where:

w_l represents the input quantities

u is the standard uncertainty for the respective input quantity.

u_B is the type B uncertainty

For any two mass values β_i and β_j there is a dependence on the air density, comparator sensitivity, indications difference displayed by the comparator, gravitacional effect, the mass of the prototype, the prototype's coefficient of the volume expansion, the prototype's volume and on the estimated temperature of the thermal equilibrium for the prototype and stainless steel weights. This dependence makes these mass values correlated.

The covariance between two mass values is defined as:

$$cov(\beta_i, \beta_j) = E(\beta_i \cdot \beta_j) - E(\beta_i) \cdot E(\beta_j) \qquad (6)$$

where E is the expected value.

Provided that, in this case, the input quantities are independent, thus, the covariance term between any two mass values β_i, β_j has the form of the equation (7):

$$cov(\beta_i, \beta_j) = \sum_{l=1}^{N} \frac{\partial \beta_i}{\partial w_l} \cdot \frac{\partial \beta_j}{\partial w_l} \cdot u^2(w_l) \qquad (7)$$

where w_l represents, in a general form, the input quantities and u is the standard uncertainty for the respective input quantity.

From variances and covariances obtained, a type B variance-covariance matrix of the mass values ψ_B was obtained as shown in equation (8).

$$\psi_B = \begin{bmatrix} u_B^2(\beta_1) & cov(\beta_1, \beta_2) & cov(\beta_1, \beta_3) \\ \vdots & u_B^2(\beta_2) & cov(\beta_2, \beta_3) \\ \text{symmetric} & \cdots & u_B^2(\beta_3) \end{bmatrix} \qquad (8)$$

For the uncertainties results to include the long term variability of the measurement process its estimated variance σ_p^2 was added to diagonal elements of ψ_B, thus obtaining the variance-covariance matrix ψ:

$$\psi = \psi_B + \sigma_p^2 . \boldsymbol{I} \qquad (9)$$

where $\boldsymbol{I}$ is a identity matrix. Then, the combined variance-covariance matrix ψ_c of the mass values was obtained by the sum of ψ and ψ_A.

$$\psi_c = \psi_A + \psi \qquad (10)$$

In this case, the obtained variance-covariance matrix ψ_c is:

$$\psi_c = \begin{bmatrix} 0{,}00022 & 0{,}00021 & 0{,}00021 \\ 0{,}00021 & 0{,}00022 & 0{,}00021 \\ 0{,}00021 & 0{,}00021 & 0{,}00022 \end{bmatrix} \qquad (11)$$

3. RESULTS

As a result of this work, the assigned mass and combined standard uncertainty values of the three mass standards, compared with the K66 prototype are shown on Table 5.

Table 5. Results

SS mass standards	Assigned mass value	u_c
MP060	1 kg – 0,375 mg	0,019 mg
R-PP062	1 kg + 0,474 mg	0,019 mg
PP019	1 kg + 1,896 mg	0,019 mg

The results can be validated by comparison with previous mass values for these standards as, for example, from earlier calibration certificates, Table 5.

Table 6. Results from prior calibration certificates

SS mass standards	Calibration certificate issued by / year	Assigned mass value from calibration certificate	u_c
MP060	BIPM/1997	1 kg – 0,304 mg	0,013 mg
R-PP062	NPL/1999	1 kg + 0,540 mg	0,025 mg
PP019	NPL/1999	1 kg + 1,937 mg	0,050 mg

4. CONCLUSION

The INMETRO's kilograms references mass standards have been linked to the prototype K66 mass value.

Comparison with earlier mass values of mass standards from Table 6, shows a drift in mass values which could be due to, mainly, a combination of drift of the reference mass standard, the prototype K66, and the calibrated mass standards. The prototype is now at BIPM for recalibration which will provide a new mass value for it. From this, it will be possible to determine the drift contributions.

The uncertainty values obtained are compatible with the measurement intruments, facilities of the laboratory and their earlier uncertainty values.

Next step will be to participate in interlaboratory comparisons between INMETRO and other NMIs to consolidate the whole procedure.

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