I CIMMEC
1º CONGRESSO INTERNACIONAL DE METROLOGIA MECÂNICA
DE 8 A 10 DE OUTUBRO DE 2008
Rio de Janeiro, Brasil

FIRST REALIZATION OF THE MASS SCALE IN BRAZIL

Victor M. Loayza [1], Fábio L. Cacais [2], Vivian R. Corrêa [3]

[1] Inmetro, Rio, Brasil, vmloayza@inmetro.gov.br
[2] Inmetro, Rio, Brasil, facacais@inmetro.gov.br
[3] Inmetro, Rio, Brasil, vrcorrea@inmetro.gov.br

Abstract: The mass scale is realized from 1 kg reference mass standard by applying the subdivision method to a set of mass standards. The subdivision method is performed by means of a series of comparisons involving combinations of those mass standards. The sequence of comparisons is specified according to properly chosen weighing designs covering the entire range of nominal values of the set. The mass value for the stainless steel 1 kg reference mass standard used for this purpose was determinated from the brazilian prototype K66 [1]. This article describes the applied procedure.

Key words: Mass scale, subdivision method, weighing designs, least squares.

1. INTRODUCTION

One of the main tasks of the Mass Laboratory of INMETRO, the National Institute of Metrology of Brazil, is 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.

Formerly, national reference mass standards were constituted by a set of mass standards, calibrated by the NPL-UK, thus, traceability from the international prototype of the kilogram was obtained through the UK national prototype K18.

For the first time in Brazil traceability from the international prototype of the kilogram was derived from the Pt-Ir brazilian national prototype of the kilogram K66 to realize the mass scale (multiples and submultiples).

2. METHODOLOGY

For realizing the mass scale it was used a set of stainless steel mass standards of nominal values from 500 g to 1 mg. The standards of this set has OIML shape [2] and includes two 200 g discs and one 100 g disc. Their identification codes, shapes and marking are shown on table 1. The 1 kg stainless steel reference mass standard for this procedure was calibrated against the brazilian prototype K66 [1] and its identification code is R-PP062.

The weighings were performed on a mass comparator Mettler Toledo AT1006 which uses the principle of electromagnetic force compensation. It has a resolution of 1 μg and performs the mass comparisons by means of an automatic load exchanger.

Table 1. Characteristics of the stainless steel set

Nominal Value	Identification Code	Shape	Marking
500 g	PP062	OIML	
200 g	PP061	Disc	O
200 g	PP061	Disc	o
100 g	PP061	Disc	O
100 g	PP062	OIML	
50 g	PP062	OIML	
20 g	PP062	OIML	*
20 g	PP062	OIML	
10 g	PP062	OIML	
5 g	PP062	OIML	
2 g	PP062	OIML	*
2 g	PP062	OIML	
1 g	PP062	OIML	
500 mg	PP062	wire	
200 mg	PP062	wire	^
200 mg	PP062	wire	
100 mg	PP062	wire	
50 mg	PP062	wire	
20 mg	PP062	wire	^
20 mg	PP062	wire	
10 mg	PP062	wire	
5 mg	PP062	wire	
2 mg	PP062	wire	^
2 mg	PP062	wire	
1 mg	PP062	wire	

A climate station, Meteorlabor Klimet A30, was used to measure the environmental air parameters within the weighing chamber of the mass comparator. The metrological characteristics of the climate station sensors are shown in Table 2.

Table 2. 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

2.1. Weighing designs

Ideally it would be necessary just one kind of weighing design, for example, C.8 of Cameron *et al* [3], to perform all the comparisons corresponding to the six decades, but due to limited height of the weighing chamber (95 mm) it was not possible to mount the combination of standards for the first decade (500 g to 100 g). Then it was chosen the weighing design C.10 for the first decade and disc shape standards were introduced. For other decades the C.8 weighing design was used. The weighing designs C.10 and C.8 are shown, respectively, in tables 3 and 4.

Table 3. Weighing design C.10

Comparison	500 g	200 go	200gO	100 g	100 g	Σ 100g
1	+	-	-	-	-	+
2	+	-	-	-	+	-
3	+	-	-	+	-	-
4	+	-		-	-	-
5	+		-	-	-	-
6		+	-	+	-	
7		+	-	-		+
8		+	-		+	-
Restraint	+	+	+	+		

Table 4. Weighing design C. 8

Comparison	5×10^n g	2×10^n g	2×10^n g *	10^n g	$\Sigma\ 10^n$ g
1	+	-	-	-	
2		+	-	+	-
3		+	-	-	+
4		+	-		
5		+		-	-
6			+	-	-
7				+	-
Restraint	+	+	+	+	
n = 1, 0, -1, -2, -3 (for n = -3, the summation becomes only one standard)					

These weighing designs form a matrix system of linear equations per decade.

2.2. Mathematical model for the true mass value difference

In order to obtain true 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 air buoyancy which act upon the weights during weighing.

$$\Delta m = \Delta I \cdot S + \rho_{ar} \cdot \Delta V \cdot (1 - \alpha \cdot \Delta T) \qquad (1)$$

where:

Δm true mass difference between the arrangements of mass standards (m_i - m_j)
ΔI indication differences displayed by the comparator
S balance sensitivity
ρ_{ar} air density during the comparisons
ΔV volume difference between the arrangements of mass standards at 20 ºC
α coefficient of the volume expansion
ΔT temperature variations of mass standards in relation to reference temperature of 20 ºC

The indication differences were obtained from a series of six ABBA weighing cycles for each pair of mass standards combinations involved in the weighing designs.

2.3. Least squares method

From the weighing designs and the true mass differences, equation (1), the following matrix of weighing equations can be obtained:

$$\boldsymbol{Y} = \boldsymbol{X}.\boldsymbol{\beta} + \boldsymbol{e} \qquad (2)$$

where:
$\boldsymbol{Y}$ vector of the true mass differences
$\boldsymbol{X}$ design matrix
$\boldsymbol{\beta}$ vector of the unknown mass values
$\boldsymbol{e}$ vector of the unknown errors of the observations

The mass values for the standards were obtained from the solution of equation (2) using the classic least squares analysis with Lagrange multipliers considering the mass value of the appropriate restraint Bich [4].

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

By the least squares analysis the normal equations are defined as:

$$\boldsymbol{X'X.b} = \boldsymbol{X'.Y} \qquad (3)$$

where:

$\boldsymbol{b}$ is the vector of the estimated unknown mass values
$\boldsymbol{Y}$ vector of the true mass differences

$\boldsymbol{X}$ design matrix
$\boldsymbol{X'}$ is the transpose of $\boldsymbol{X}$

Due to $\boldsymbol{Y}$ is a vector of mass differences, equation (3) doesn't provide a unique solution for $\boldsymbol{b}$ unless a restraint mass value be considered.

The restraint mass value is a linear combination between elements of $\boldsymbol{\beta}$ vector.

$$\boldsymbol{r'}.\boldsymbol{\beta} = M_R \quad (4)$$

where:

$\boldsymbol{r'}$ is the row vector which performs the linear combinations in elements of $\boldsymbol{\beta}$ vector
M_R is the restraint mass value

After introducing the restraints by means of Lagrange multipliers , the new normal equations obtained are:

$$\begin{pmatrix} \boldsymbol{X'X} & \boldsymbol{r} \\ \boldsymbol{r'} & 0 \end{pmatrix} \cdot \begin{pmatrix} \boldsymbol{b} \\ \lambda \end{pmatrix} = \begin{pmatrix} \boldsymbol{X'Y} \\ M_R \end{pmatrix} \quad (5)$$

where:

λ is the Lagrange multipliers
$\boldsymbol{r}$ is a column vector, transpose of $\boldsymbol{r'}$

The general solution for equation (5) may be written as follows:

$$\begin{pmatrix} \boldsymbol{b} \\ \lambda \end{pmatrix} = \begin{pmatrix} \boldsymbol{C} & \boldsymbol{h} \\ \boldsymbol{h'} & 0 \end{pmatrix} \cdot \begin{pmatrix} \boldsymbol{X'Y} \\ M_R \end{pmatrix} \quad (6)$$

where:

$\boldsymbol{C}$ is a matrix which performs combinations between elements of $\boldsymbol{X'Y}$ matrix
$\boldsymbol{h}$ is a vector whose elements weight the restraint mass value

$\begin{pmatrix} \boldsymbol{C} & \boldsymbol{h} \\ \boldsymbol{h'} & 0 \end{pmatrix}$ is the inverse matrix of $\begin{pmatrix} \boldsymbol{X'X} & \boldsymbol{r} \\ \boldsymbol{r'} & 0 \end{pmatrix}$

Thus, the general form of the least squares' solution by Lagrange multipliers for the estimated unknown mass values is:

$$\boldsymbol{b} = \boldsymbol{CX'}.\boldsymbol{Y} + \boldsymbol{h}.M_R \quad (7)$$

2.4. Systematic effects

Systematic effects in comparisons of mass standards arise mainly due to air buoyancy, magnetic, thermal and the mass comparator effects.

All of these effects were corrected as follows.

2.4.1. Mass comparator effect

A limited resolution, non-linearity and excentricity of the mass comparator can cause systematic errors in the displayed differences. Such systematic erros are considered as having zero value but an uncertainty value was considered for them.

The mass comparator 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.

2.4.2. Air buoyancy effect

The air buoyancy correction is obtained from the mass standards volume and the determined air density values.

- the air density was determined from CIPM 2007 equation [5];
- the values for volume of mass standards and their uncertainty are shown in Table 5.

Table 5. Volume of mass standards

NominalValue	Volume at 20 °C cm^3	u_c
500 g	62,8	0,2
200 g	25,1	0,1
100 g	12,6	0,1
50 g	6,28	0,02
20 g	2,51	0,01
10 g	1,26	0,01
5 g	0,628	0,002
2 g	0,251	0,001
1 g	0,126	0,001
500 mg	0,0628	0,0002
200 mg	0,0251	0,0001
100 mg	0,0126	0,0001
50 mg	0,00628	0,00002
20 mg	0,00251	0,00001
10 mg	0,00126	0,00001
5 mg	0,000628	0,000002
2 mg	0,000251	0,000001
1 mg	0,000126	0,000001

2.4.3. Thermal effects

All weights were kept inside weighing chamber, with the mass comparator turned on, for a time long enough to reduce any effect on weighing results arisen from temperature differences between the mass comparator, the mass standards and the surrounding air, Gläser [6].

2.4.4. Magnetic effects

Since weighings are executed on a electromagnetic force compensated mass comparator and mass standards are made of stainless steel alloy an unsuspected vertical magnetic force could be influencing the results [7].

In order to avoid this, the magnetic susceptibility of each mass standard was measured using a susceptometer developed by the BIPM, Davis [8].

All magnetic susceptibility measured values for the stainless steel standards were lower than the permissible limit for OIML class E_1 weights.

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.

2.5.1. Type A evaluation of the uncertainty

The type A evaluation of the uncertainty is based on a statistical data analysis [9]. Such statistical analysis performed by the least squares method provides the variance-covariance matrix ψ_A of the mass values arisen from the weighing designs [10].

A general form of the variance-covariance matrix is:

$$\psi_A = (\boldsymbol{CX'XC'}).\sigma^2 \quad (8)$$

where:

$\boldsymbol{C}'$ is the transpose matrix of $\boldsymbol{C}$

σ^2 is the variance of the elements of the $\boldsymbol{Y}$

Equation (8) is obtained on the hypothesis that there isn't random covariance between elements of vector of the true mass differences $\boldsymbol{Y}$.

A variance σ^2 is estimated from of:

$$\mathrm{s}^2 = \frac{(\boldsymbol{Y} - \boldsymbol{Xb})' \cdot (\boldsymbol{Y} - \boldsymbol{Xb})}{\mathrm{r} - \mathrm{c} + 1} \quad (9)$$

where:

s^2 is the estimated variance

r is the number of rows from $\boldsymbol{X}$

c is the number of columns from $\boldsymbol{X}$

2.5.2. Type B evaluation of the uncertainty

The type B evaluation of the uncertainty isn't based on statistical analysis, but on all the available information about possible variations in the input quantities. This information is contained in $\boldsymbol{Y}$ and M_R, in the equation (7).

The variance-covariance matrix based on the type B evaluation, ψ_B, was determined considering the contributions due to indication differences, air density, mass standards volume, mass comparator effects and restraint's mass value.

The general form for the estimated mass standards values b_i obtained from equation (7) is:

$$b_i = \kappa_i \cdot M_{\mathrm{R}} + S \cdot \sum_{k=1}^{r} \gamma_{ik} \cdot \Delta I_k + \sum_{k=1}^{r} \sum_{l=1}^{c} \nu_{ik} \cdot \eta_{kl} \cdot \rho_{ar_k} \cdot V_l \cdot (1 + \alpha \cdot \Delta T_k) \quad (10)$$

where:

M_R is the restraint mass value

ΔI_k is the difference displayed by mass comparator in the k-*th* comparison in the weighing design

S balance sensitivity

ρ_{ark} is the air density in the k-*th* comparison in the weighing design

V_l is the *l-th* volume of mass standard

ΔT_k is the difference of the temperature in relation to 20 ºC in the k-*th* comparison in the weighing design

k_i is a constant coefficient which weights the restraint mass value

γ_{ik}, ν_{ik} *e* η_{kl} are constants coefficients elements from matrix product $\boldsymbol{CX'}$

The uncertainties, u_B, arisen by the type B evaluation were obtained from GUM's law of propagation of uncertainty [9] applied to equation (10), as shown below:

$$u_B^2(b_i) = \sum_{l=1}^{N} \left(\frac{\partial b_i}{\partial w_l} \right)^2 \cdot u^2(w_l) + 2 \cdot \sum_{l<p} \frac{\partial b_i}{\partial w_l} \cdot \frac{\partial b_i}{\partial w_p} \cdot cov(w_l, w_p) \quad (11)$$

where:

u^2 is the square of the standard uncertainty

w_l, w_p represents the input quantities

cov is the a covariance between the input quantities w_l and w_p

u_B is the type B uncertainty

For any two mass values b_i and b_j there is a dependence on the air density, comparator sensitivity, indications difference displayed by the comparator, the coefficient of the volume expansion, the volume of the standards, the estimated temperature of the thermal equilibrium and the restraint mass value. This dependence makes these mass values correlated.

The covariance between two mass values is defined as, equation (12):

$$cov(b_i, b_j) = E(b_i \cdot b_j) - E(b_i) \cdot E(b_j) \quad (12)$$

where E is the expected value.

The covariance term between any two mass values b_i, b_j is obtained from equation (13):

$$cov(b_i, b_j) = \sum_{l=i}^{N} \sum_{p=1}^{M} \frac{\partial b_i}{\partial w_l} \cdot \frac{\partial b_j}{\partial w_p} \cdot cov(w_l, w_p) \quad (13)$$

where:

$cov(b_i,b_j)$ is the a covariance between the mass values

$cov(w_l,w_p)$ is the a covariance between the input quantities w_l and w_p

From variances and covariances obtained, a variance-covariance matrix, based on a type B evaluation, ψ_B can be written as:

$$\psi_B = \begin{bmatrix} u_B^2(b_1) & cov(b_1,b_2) & \cdots & cov(b_1,b_s) \\ \vdots & u_B^2(b_2) & & \vdots \\ \vdots & & \ddots & \vdots \\ symmetric & \cdots & \cdots & u_B^2(b_s) \end{bmatrix} \quad (14)$$

For the uncertainty of 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:

$$\psi = \psi_B + \sigma_p^2 . \boldsymbol{I} \quad (15)$$

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

$$\psi_c = \psi + \psi_A \quad (16)$$

3. RESULTS

The result of this work, the assigned mass and uncertainty values for the set of mass standards, are shown on Table 6.

Table 6. Results

Mass standard nominal value	Assigned mass value g	u_c mg	Marking
500 g	500,000 241	0,143	
200 g	199,999 898	0,057	O
200 g	200,000 005	0,057	o
100 g	100,000 012	0,029	O
100 g	100,000 053	0,029	
50 g	50,000 027	0,015	
20 g	20,000 016	0,006	*
20 g	20,000 012	0,006	
10 g	10,000 005	0,003	
5 g	4,999 996	0,002	
2 g	2,000 005	0,001	*
2 g	2,000 004	0,001	
1 g	1,000 003	0,001	
500 mg	0,500 000 0	0,0007	
200 mg	0,199 998 8	0,0005	^
200 mg	0,200 002 4	0,0005	
100 mg	0,100 001 1	0,0005	
50 mg	0,049 999 4	0,0003	
20 mg	0,019 998 1	0,0002	^
20 mg	0,019 999 0	0,0002	
10 mg	0,010 001 3	0,0001	
5 mg	0,005 001 0	0,0006	
2 mg	0,002 001 8	0,0005	^
2 mg	0,001 999 8	0,0005	
1 mg	0,001 001 0	0,0005	

Figure 1 shows the combined variance-covariance matrix graphs for each decade. In these graphs axes, in the horizontal, plane correspond to the nominal values.

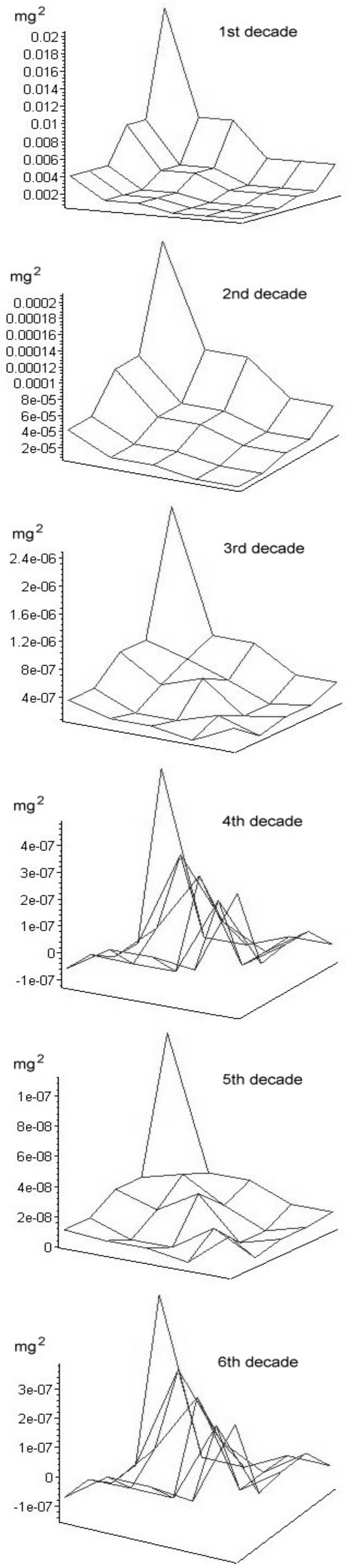

Fig. 1. Variance-covariance graphs per decade.

Each node shown in the graph corresponds to a value of variance (diagonal nodes) or covariance (off-diagonal nodes).

The results can be validated by comparison with check weights formerly calibrated by the NPL as shown on Table 7.

Table 7. Assigned mass values, their uncertainties and normalized error (E_n) obtained against check weights mass values

Assigned mass value g	***U* (*k*=2) mg**	**Check weights g**	***U* (*k*=2) *mg***	E_n
500,000 241	0,286	500,000 301	0,050	-0,21
50,000 027	0,030	50,000 032	0,008	-0,16
4,999 996	0,004	4,999 995	0,001 2	0,24
0,500 000 0	0,0014	0,500 001 2	0,000 6	-0,79
0,049 999 4	0,0006	0,049 999 1	0,000 6	0,35
0,005 001 0	0,0012	0,005 001 1	0,000 6	-0,07

4. CONCLUSION

The Inmetro's mass scale from 1 kg to 1 mg has been realized and linked to the prototype K66 mass value through the reference mass standard R-PP062.

For the higher nominal values for which the buoyancy effect is more significant, obtained uncertainty of the mass values is high due to estimated value for the volume of mass standards. Calibration in volume will improve the uncertainty values.

Comparison with mass values of the check weights from Table 7, shows the compatibility between obtained results and earlier mass values.

The obtained results show that the mathematical procedure by using least squares method was properly applied and that the laboratory facilities are adequate. Thus the process can be considered reasonably under control.

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

REFERENCES

1. Loayza V. M., Cacais F.L., Corrêa V.R., "Mass values of 1 kilogram stainless steel mass standards traceable to the brazilian national prototype of the kilogram", to be presented in I CIMMEC - I Congresso Internacional de Metrologia Mecânica, Brazil, October 2008.

2. OIML R111-1, "Weights of classes E_1, E_2, F_1, F_2, M_1, M_{1-2}, M_2, M_{2-3} and M_3, Part 1: Metrological and technical requirements" , Edition 2004 (E).

3. Cameron J. M., Croarkin M. C. and Raybold R. C., "Designs for the Calibration of Standards of Mass", Natl. Bur. Stand. (U.S.), Tech. Note 952, June 1977.

4. W. Bich, Variances, Covariances and Restraints in Mass Metrology, Metrologia, vol. 27, pp. 111-116, 1990.

5. Picard A., Davis R. S., Gläser M. and Fujii K., "Revised formula for the density of moist air (CIPM – 2007)", Metrologia, vol. 45, pp. 149-155, 2008.

6. Gläser M., "Change of the mass of weights arising from temperature differences", Metrologia, vol. 36, pp. 183-197, 1999.

7. Davis R. and Gläser M., "Magnetic properties of weights, their measurements and magnetic interactions between weights and balances", Metrologia, vol. 40, pp. 339-355, 2003.

8. Davis R. S., "New Method to Measure Magnetic Susceptibility", Meas. Sci. Technol., Vol. 4, pp. 141-147, 1993.

9. BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, "Guia para Expressão da Incerteza de Medição", Terceira Edição, 2003.

10. Bich W., Cox M.G. and Harris P.M., "Uncertainty Modeling in Mass Comparison", Metrologia vol. 30, pp. 495-502, 1993/94.