WEIGHTS

Test procedures for Class E_1 weights at the Romanian National Institute of Metrology: Calibration of mass standards by sub-division of the kilogram

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Abstract

The provision of the mass scale below one kilogram is achieved by subdivision. This paper describes one of the methods used by INM including details of the weighing techniques, weighing schemes, equipment used and the uncertainty of measurement of all the standards involved.

1 Introduction

INM is the custodian of the Prototype Kilogram No. 2. As such, it is INM's task to propagate the Romanian mass scale by subdivision and multiplication of the kilogram.

Class E_1 weights ensure traceability to the national mass standard (the value of which is derived from the International Prototype of the kilogram, maintained by the BIPM) and weights of Class E_2 and lower [1]. They are used as standards at the thirteen Romanian calibration laboratories.

2 Test procedures

The set (500...1) g of Class E_1 weights usually has the following composition:

500 g, 200 g, 200* g, 100 g
50 g, 20 g, 20* g, 10 g
5 g, 2 g, 2* g, 1 g

The 1 kg reference standard, of known mass, is used for calibration. Mass determinations are carried out by subdivision (to link standards having different nominal values up with a reference standard). Depending on the weighing scheme, this procedure requires a specific minimum number of standards. By the method of least squares adjustment, the mass departures and their standard deviations are calculated.

Weighing is always carried out as substitution weighing, i.e. single weights or combinations are always compared with another combination of the same nominal value. The difference between the balance indications has the symbol Δm and it is necessary to apply air buoyancy corrections to the observed weighing differences.

If "y" is the new corrected difference, this gives:

$$y = \Delta m + (\rho_a - \rho_o)(V_1 - V_2) \qquad (1)$$

where:

- y is the corrected indication;
- Δm is the difference in balance readings calculated from one weighing cycle (RTTR, where R is the reference standard and T is the test weight);
- ρ_o = 1.2 kg · m^{-3}, the reference air density;
- ρ_a = air density at the time of the weighing; and
- V_1, V_2 are the volumes of the standards (or the total volume of each group of weights) involved in the measurement.

In designing the scheme, all the masses from 1 kg to 1 g are broken down into decades. A weighing scheme with 12 equations per decade is used in the calibration [1]. The first decade includes the 1 kg standard.

For subsequent decades the role of the standard is taken by the "1" from the previous decade; thus the 100 g, 10 g masses become intermediate standards, whose uncertainty is propagated directly to masses in the decade they head and hence to those in subsequent decades.

With the reference standard, the mass having nominal values: 500 g, 200 g, 200* g, 100 g, Σ100 g (the sum of 50 g, 20 g, 20* g and 10 g from the next decade) shall be calibrated using a 1 kg mass comparator. The observations are of the same accuracy (for all mass comparisons the same balance was used in the first decade).

Once all the weighings have been completed, the first step consists in the formation of the design matrix.

Matrix "X" contains the information about the equations used (the weighing scheme) and matrix "Y" contains the measured differences from these equations.

Denote:

$X = (x_{ij})$;
$i = 1...n$;
$j = 1...k$;
$x_{ij} = 1, -1$ or 0;
β is (β_j) vector of unknown departures; and
Y is (y_i) vector of measured values (including buoyancy corrections).

X =

1000 g	500 g	200 g	200* g	100 g	Σ100* g
–1	1	1	1	1	0
–1	1	1	1	0	1
0	1	–1	–1	–1	0
0	1	–1	–1	0	–1
0	0	1	–1	1	–1
0	0	1	–1	1	–1
0	0	1	–1	–1	1
0	0	1	–1	–1	1
0	0	1	0	–1	–1
0	0	1	0	–1	–1
0	0	0	1	–1	–1
0	0	0	1	–1	–1

$$Y = \begin{vmatrix} y_1 \\ y_2 \\ y_3 \\ y_4 \\ y_5 \\ y_6 \\ y_7 \\ y_8 \\ y_9 \\ y_{10} \\ y_{11} \\ y_{12} \end{vmatrix}$$

$$\beta = \begin{vmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \\ \beta_5 \\ \beta_6 \end{vmatrix}$$

The first row of the matrix represents difference in mass between the +1 and the –1 weight, for example: $(500 + 200 + 200^* + 100) - 1000 = y_1$

If $(X^T \cdot X)$ is the matrix of the normal equations, this gives:

$$(X^T \cdot X) \cdot \beta = X^T \cdot Y \qquad (2)$$

where X^T is a transpose of X:

$$X^T = \begin{vmatrix} -1 & -1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 1 & 1 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 1 & -1 & -1 & 1 & 1 & 1 & 1 & 1 & 1 & 0 & 0 \\ 1 & 1 & -1 & -1 & -1 & -1 & -1 & -1 & 0 & 0 & 1 & 1 \\ 1 & 0 & -1 & 0 & 1 & 1 & -1 & -1 & -1 & -1 & -1 & -1 \\ 0 & 1 & 0 & -1 & -1 & -1 & 1 & 1 & -1 & -1 & -1 & -1 \end{vmatrix}$$

$$X^T \cdot X = \begin{vmatrix} 2 & -2 & -2 & -2 & -1 & -1 \\ -2 & 4 & 0 & 0 & 0 & 0 \\ -2 & 0 & 10 & 0 & 0 & 0 \\ -2 & 0 & 0 & 10 & 0 & 0 \\ -1 & 0 & 0 & 0 & 10 & 0 \\ -1 & 0 & 0 & 0 & 0 & 10 \end{vmatrix}$$

The next step introduces two matrices: $(X^T \cdot X)^{-1}$ is termed the inverse of $(X^T \cdot X)$ and the product $(X^T \cdot X)^{-1}X^T$.

The matrix design contains only the weighing equations. For this reason, the system can not be solved because the determinant of $(X^T \cdot X)$ is zero and the inverse $(X^T \cdot X)^{-1}$ does not exist.

To overcome this problem the Lagrangian multipliers method is applied [3, 4] which consists of adding the reference standard (restraint m_R) to the vector "Y", the Lagrangian multipliers λ to the vector "β", a line k + 1 and a column k + 1 (both containing the elements 1,0,1) to the normal equation and to the matrix X^T as follows:

$$X^T \cdot X = \begin{vmatrix} 2 & -2 & -2 & -2 & -1 & -1 & 1 \\ -2 & 4 & 0 & 0 & 0 & 0 & 0 \\ -2 & 0 & 10 & 0 & 0 & 0 & 0 \\ -2 & 0 & 0 & 10 & 0 & 0 & 0 \\ -1 & 0 & 0 & 0 & 10 & 0 & 0 \\ -1 & 0 & 0 & 0 & 0 & 10 & 0 \\ 1 & 0 & 0 & 0 & 0 & 0 & 0 \end{vmatrix}$$

$$Y = \begin{vmatrix} y_1 \\ y_2 \\ y_3 \\ y_4 \\ y_5 \\ y_6 \\ y_7 \\ y_8 \\ y_9 \\ y_{10} \\ y_{11} \\ y_{12} \\ m_R \end{vmatrix}$$

$$X^T = \begin{vmatrix} -1 & -1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 1 & 1 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 1 & -1 & -1 & 1 & 1 & 1 & 1 & 1 & 1 & 0 & 0 & 0 \\ 1 & 1 & -1 & -1 & -1 & -1 & -1 & -1 & 0 & 0 & 1 & 1 & 0 \\ 1 & 0 & -1 & 0 & 1 & 1 & -1 & -1 & -1 & -1 & -1 & -1 & 0 \\ 0 & 1 & 0 & -1 & -1 & -1 & 1 & 1 & -1 & -1 & -1 & -1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \end{vmatrix}$$

$$\beta = \begin{vmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \\ \beta_5 \\ \beta_6 \\ \lambda \end{vmatrix}$$

The inverse of $X^T \cdot X$ will be:

$$(X^T \cdot X)^{-1} = \begin{vmatrix} 0 & 0 & 0 & 0 & 0 & 0 & 1 \\ 0 & 1/4 & 0 & 0 & 0 & 0 & 1/2 \\ 0 & 0 & 1/10 & 0 & 0 & 0 & 1/5 \\ 0 & 0 & 0 & 1/10 & 0 & 0 & 1/5 \\ 0 & 0 & 0 & 0 & 1/10 & 0 & 1/10 \\ 0 & 0 & 0 & 0 & 0 & 1/10 & 1/10 \\ 1 & 1/2 & 1/5 & 1/5 & 1/10 & 1/10 & 0 \end{vmatrix}$$

The last column and row contains the factor $h_j = m_j/m_r$, the ratios between the nominal values of the unknown weights (m_j) and one of the reference (m_r).

The best estimate of β, $\langle\beta\rangle$ for an over-determined system of equations "X" is given by:

$$\langle\beta\rangle = (X^T \cdot X)^{-1} X^T \cdot Y \qquad (3)$$

3 Example of a least-squares analysis: Equipment, standards and results

3.1 Equipment

The balances used in the measurements in the range from 1 g to 500 g are listed below:

Type	Max	Standard deviation, mg	Indication
AT 1005 (Mettler)	1 kg	0.01 – 0.02	Digital
H20 (Mettler)	160 g	0.01	Optical
2405 (Sartorius)	30 g	0.002	Optical

Additionally, the mass laboratory is equipped with instruments to measure:

- the pressure, measured using a standard barometer (U = 2 mbar, k = 2);
- the relative humidity, measured using a standard psychrometer (U = 3 %, k = 2); and
- the temperature, measured using a standard thermometer (U = 0.4 K, k = 2).

From the air parameters, the air density is calculated using the equation recommended by the CIPM [2].

3.2 Standards

The 1 kg reference standard is used as the known mass for the calibration, where:

- V = 127.7398 cm^3,
  expanded uncertainty U_v = 0.0024 cm^3, k = 2.
- conventional mass m_{cr} = 0.999 996 891 kg,
  expanded uncertainty $U(m_{cr})$ = 0.044 mg, k = 2.

The observed mass differences read:

$$Y = \begin{vmatrix} 3.780 \\ 3.3911 \\ -0.04 \\ -0.05 \\ 0.01 \\ 0.01 \\ 0.025 \\ 0.028 \\ 0.017 \\ 0.017 \\ 0.020 \\ 0.022 \\ -3.109 \end{vmatrix}$$

The vector $\langle\beta\rangle$ with the unknown masses, according to equation (3) above, gives:

$$\langle\beta\rangle = \begin{vmatrix} 1000\text{ g} - 3.109\text{ mg} \\ 500\text{ g} + 0.115\text{ mg} \\ 200\text{ g} + 0.075\text{ mg} \\ 200\text{ g} + 0.061\text{ mg} \\ 100\text{ g} + 0.020\text{ mg} \\ \Sigma\ 100\text{ g} + 0.029\text{ mg} \end{vmatrix}$$

The value assigned to the summation Σ 100 g by the first decade constitutes the restraint for the second decade with the individual weights in the summation being calibrated separately in the second series. The summation of weights Σ 10 g becomes the restraint for the third decade. Then, the same procedure is used for the second and the last decades.

4 Analysis of uncertainties

4.1 Type A uncertainty

If the adjusted mass difference of the weighing equations is $\langle Y \rangle = X \cdot \langle \beta \rangle$, the residual for each equation is calculated as follows:

$$\langle e \rangle = Y - \langle Y \rangle \quad (4)$$

The calculation of $\langle e \rangle$ for the example gives the results:

$$\langle e \rangle = \begin{vmatrix} -2 \cdot 10^{-3} \\ 2.1 \cdot 10^{-3} \\ 10 \cdot 10^{-4} \\ 0 \\ 5 \cdot 10^{-3} \\ 5 \cdot 10^{-3} \\ 2 \cdot 10^{-3} \\ 5 \cdot 10^{-3} \\ -9 \cdot 10^{-3} \\ -9 \cdot 10^{-3} \\ 8 \cdot 10^{-3} \\ 8 \cdot 10^{-3} \end{vmatrix}$$

The standard deviation "s" of the observations is calculated by:

$$s = \sqrt{\frac{1}{v} \sum_{i=1}^{n} res_i^2} \quad (5)$$

The residuals "res." are the elements of the vector $\langle e \rangle$; "v" = n – k + 1 represents the degrees of freedom ("n – k" is the difference between the number of performed observations and the number of unknown weights; "1" is the number of the restraints). According to this equation the standard deviation is:

s = 0.007 mg

$$V_\beta = \begin{vmatrix} 0 & 0 & 0 & 0 & 0 & 0 & 1 \\ 0 & 1/4 & 0 & 0 & 0 & 0 & 1/2 \\ 0 & 0 & 1/10 & 0 & 0 & 0 & 1/5 \\ 0 & 0 & 0 & 1/10 & 0 & 0 & 1/5 \\ 0 & 0 & 0 & 0 & 1/10 & 0 & 1/10 \\ 0 & 0 & 0 & 0 & 0 & 1/10 & 1/10 \\ 1 & 1/2 & 1/5 & 1/5 & 1/10 & 1/10 & 0 \end{vmatrix} \cdot 0.000049$$

The variance – covariance matrix for $\langle \beta \rangle$ is given by:

$$V_\beta = s^2 (X^T \cdot X)^{-1} \quad (6)$$

where the variances on the values of the solutions $\langle \beta \rangle$ are given by the diagonal elements of the matrix $(X^T \cdot X)^{-1}$ denoted by c_{ij}. The off-diagonal elements of the matrix give the covariance between the weights.

The standard deviation (uncertainty of type A) of a particular unknown weight is:

$$u_{A(\beta j)} = s\sqrt{c_{ij}} = \begin{vmatrix} 0 \\ 0.0035 \\ 0.0022 \\ 0.0022 \\ 0.0022 \\ 0.0022 \end{vmatrix} \text{mg}$$

The random uncertainty $u_{A(\beta j)}$ has a "local" component arising from measurements in the current decade and after the first decade, a propagated component arising from random uncertainty in the intermediate standards.

4.2 Type B uncertainty

The components of type B uncertainties are:

4.2.1 Uncertainty associated with the reference standard

$$u_{r(\beta j)} = h_j \cdot u_{mcr} = \begin{vmatrix} 0.0220 \\ 0.0110 \\ 0.0044 \\ 0.0044 \\ 0.0022 \\ 0.0022 \end{vmatrix} \text{mg} \quad (7)$$

where h_j is described above.

4.2.2 Uncertainty associated with the air buoyancy corrections

$$u^2_{b(\beta j)} = (V_j - h_j V_r)^2 \cdot u^2_{\rho a} + (\rho_a - \rho_o)^2 (u^2_{Vj} + h_j u^2_{Vr}) \qquad (8)$$

where:

V_j, V_r = volume of test weight and reference standard, respectively;
$u^2_{\rho a}$ = uncertainty for the air density;
ρ_o = 1.2 kg·m^{-3} is the reference air density;
u^2_{Vj}, u^2_{Vr} = uncertainty of the volume of test weight and reference standard, respectively.

$$u_{b(\beta j)} = \begin{vmatrix} 0 \\ 0.0030 \\ 0.0011 \\ 0.0011 \\ 0.0006 \\ 0.0006 \end{vmatrix} \text{ mg}$$

4.2.3 Uncertainty due to the display resolution of a digital balance

For the first decade where a digital balance with the scale interval of d = 0.01 mg is used, the uncertainty due to resolution is [1]:

$$u_d = \left(\frac{d/2}{\sqrt{3}}\right) \times \sqrt{2} = 0.0041 \text{ mg} \qquad (9)$$

4.3 Combined standard uncertainty

The combined standard uncertainty of the conventional mass of the weight βj is given by:

$$u_{c(\beta j)} = [u^2_{A(\beta j)} + u^2_{r(\beta j)} + u^2_{b(\beta j)} + u^2_d]^{1/2} \qquad (10)$$

The summation contains all the contributions described above.

$$u_{c(\beta j)} = \begin{vmatrix} 0.0220 \\ 0.0126 \\ 0.0064 \\ 0.0064 \\ 0.0051 \\ 0.0051 \end{vmatrix} \text{ mg}$$

4.4 Expanded uncertainty

The expanded uncertainty "U" (with k = 2) of the conventional mass of the weights βj is as follows [8]:

$$U = k\, u_{c(\beta j)} = \begin{vmatrix} 0.0440 \\ 0.0252 \\ 0.0128 \\ 0.0128 \\ 0.0102 \\ 0.0102 \end{vmatrix} = \begin{vmatrix} 0.044 \\ 0.03 \\ 0.01 \\ 0.01 \\ 0.01 \\ 0.01 \end{vmatrix} \text{ mg} \qquad (11)$$

5 Uncertainty budget for the first decade

Table 1 on page 16 shows the results obtained from the least squares analysis of the weighing data and their associated uncertainties. It also lists the contribution due to the uncertainty in the value of the standard, in the buoyancy correction and in the balance.

6 Conclusions

A calibration scheme for mass standards below 1 kg has been described. The whole set of masses is calibrated, decade by decade, in terms of a 1 kg standard.

The test procedure described leads to an efficient calibration of sets of class E_1 weights, also used to calibrate laboratory standards with lower uncertainty.

The subdivision weighing scheme and the electronic mass comparator used lead to an appreciable reduction in uncertainty in each mass value, compared with previous calibrations.

One way to reduce the uncertainty and to obtain better results is to use balances of much greater accuracy and in near perfect environmental conditions. ■

Table 1 and References on page 16

Weights:		1 kg	500 g	200 g	200* g	100 g	Σ 100 g
$u_{mr} \cdot h_j$	mg	0.022	0.011	0.0044	0.0044	0.0022	0.0022
$V_r \cdot h_j$	cm^3	127.7398	63.8699	25.5480	25.5480	12.7740	12.7740
$u_{Vr} \cdot h_j$	cm^3	0.0012	0.0006	0.0002	0.0002	0.0001	0.0001
V_j	cm^3	-	62.428	24.975	24.976	12.485	12.506
u_{Vj}	cm^3	-	0.014	0.004	0.004	0.002	0.001
ρ_a	mg/cm^3	1.196					
$u_{\rho a}$	mg/cm^3	0.002					
$(V_j-V_r h_j)u_{\rho a}$	mg	-	$2.9 \cdot 10^{-3}$	$1.15 \cdot 10^{-3}$	$1.15 \cdot 10^{-3}$	$5.8 \cdot 10^{-4}$	$5.4 \cdot 10^{-4}$
$(\rho_a-\rho_o)(u_{Vj}^2+u_{Vr}^2)^{1/2}$	mg	-	$5.6 \cdot 10^{-5}$	$1.61 \cdot 10^{-5}$	$1.61 \cdot 10^{-5}$	$8 \cdot 10^{-6}$	$4.3 \cdot 10^{-6}$
u_b	mg	-	0.003	0.0011	0.0011	0.0006	0.0005
u_d	mg	0.004					
u_A	mg		0.0035	0.0022	0.0022	0.0022	0.0022
u_c	mg	0.022	0.0126	0.0064	0.0064	0.0051	0.0051
k		2					
U	mg	0.04	0.03	0.01	0.01	0.01	0.01

Table 1 Uncertainty budget for the first decade

References

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