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SUBDIVISION METHOD APPLIED FOR OIML WEIGHTS USING AN AUTOMATIC COMPARATOR

Adriana Vâlcu [1], *Dumitru Dinu* [2]

[1] National Institute of Metrology, Bucharest, Romania, email: adriana.valcu@inm.ro
[2] Romanian Bureau of Legal Metrology, Bucharest, Romania, email: dinudumitru@brml.ro

Abstract – According to International Recommendation OIML R 111, [1], the weights of nominal values greater than 1 g may have a cylindrical shape with a lifting knob.

Taking into account this kind of shape and that in the case of an automatic comparator, with the maximum capacity of 1 kg, the diameter of weighing pan is quite small for placing a group of weights from 500g to 100g, the subdivision method can't be applied for the calibration of weights.

By using the subdivision method presented in this paper, the cylindrical weights with a lifting knob, having nominal values from 500g to 100 g are calibrated using an automatic comparator (which is not foreseen with weight support plates).

The method can be used for E_1 weights, when the highest accuracy is required.

Uncertainty obtained in this case for the unknowns weights is better than that obtained usually for E_1, being at the level acquired for reference standards.

Keywords: subdivision of the kilogram, mass calibration, automatic comparator.

1. INTRODUCTION

The realization and dissemination of the unit of mass by the INM is ensured with the aid of reference stainless steel standards of kilogram, which are traceable to the IPK (International Prototype Kilogram) through the mass of the Romanian Prototype Kilogram No2.

Since March 2002, an automated mass comparator was available for the dissemination of mass unit from the National Prototype kilogram No.2 to a set of three 1 kg stainless steel mass standards (which are the reference standards in the Romanian hierarchy of mass).

As reference standards are also used two sets of disc weights from 500g to 50g that were purchased in 2006.

In the calibration of class E_1 weights, when the highest accuracy is required, the subdivision method is mainly used.

The sub-division weighing scheme has both advantages and disadvantages.

Advantages [2]:

a) it minimizes use on (and hence wear on) standards;

b) it produces a set of data which provides important statistical information about the measurements and the day to day performance of the individual balances;

c) there is a redundancy of data .

Disadvantages [2**]**:

a) it requires a relatively complex algorithm to analyze the data;

b) it necessitates placing groups of weights on balance pans (this can cause problems for instruments with poor eccentricity characteristics or automatic comparators designed to compare single weights).

In the procedure, to achieve the calibration by subdivision method on the automatic comparator, a set of disc weights (reference standards) is used.

These weights constitute both support plates and check standards.

The criterion used in finding the weighing design wasn't the orthogonality because the weights are used individually.

The objective in the search for better designs was to find a calibration scheme which can be performed taking into account the two elements: the automatic comparator and the diameter of the disc weights (in terms of that a group of OIML weights can be disposed).

2. MEASUREMENT SYSTEM

The measurement system consists in: an automatic mass comparator, "Figure1"; a precise "climate station" system Klimet A30 (for accurate determination of air density) "Figure 2"; the unknown E_1 weights are OIML shape (from 500g to 100g) and a set of disc weights (reference weights, marked with NA), "Figure 3".

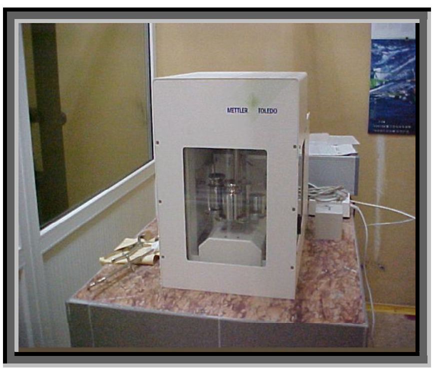

Fig.1 Automatic mass comparator

Fig.2 Precise "climate station" system

Fig.3 The cylindrical and the disc weights [7]

The measurements were performed on the Mettler AT 1006 comparator with a scale division of 1 µg and a pooled standard deviation from 0,4 µg to 2µg for nominal masses from 100 g to 1 kg, respectively.

A precise "climate station" system Klimet A30 is used for accurate determination of air density. Technical requirements for Klimet A30 are:

Temperature: Readability: 0,001°C
U (k=2) : 0,03°C
Dew Point: Resolution : 0,01°C
U (k=2) : 0,05°C
Barometric pressure: Resolution : 0,01 hPa
U (k=2) : 0,03 hPa

3. CALIBRATION PROCEDURE

The least square method was used to estimate unknown masses of the weights [3].

The system of equations is given below:

-(1000 Ref)+(500NA) + (500E$_1$) = y_1
-(1000 Ref)+(500NA)+(200NA)+(200E$_1$)+(100NA) = y_2
-(1000 Ref)+(500NA)+(200NA)+(200E$_1$)+(100E$_1$) = y_3
(500NA) – (500E$_1$) = y_4
(500NA) – (200NA) – (200E$_1$) – (100NA) = y_5
(500E$_1$) – (200NA) – (200E$_1$) – (100E$_1$) = y_6
(200NA)–(200E$_1$)–(100NA)+(100E$_1$) = y_7
200NA - 200E$_1$ = y_8
200NA - 200E$_1$ = y_9
(200NA)–(100NA)–(100E$_1$) = y_{10}
(200NA) – (100NA) – (100E$_1$) = y_{11}
(200E$_1$) – (100NA) – (100E$_1$) = y_{12}
(200E$_1$) – (100NA) – (100E$_1$) = y_{13}
100NA-100E$_1$ = y_{14}

Where:

"Ref" represents the reference kilogram standard
"NA" are the disc weights.
"E$_1$" are the OIML weights of E$_1$ class.

For all the weights that are calibrated, the volumes are known from the calibration certificates [4].

TABLE 1. Volumes and standard uncertainties of the weights

Nominal mass g	Marking	*V* cm3	*U*(*V*) cm3
1000 ref	Ni	127,7398	0,0012
500	NA	62,546	0,031
500	E$_1$	62,266	0,032
200	NA	25,017	0,028
200	E$_1$	24,853	0,008
100	NA	12,509	0,027
100	E$_1$	12,456	0,004

For the calibration, as the known mass is used 1 kg reference standard Ni81, having the mass value determined at BIPM.

The results of this comparison (the mass) from the calibration certificate [4] are:

m_{Ni81} = 1 kg+ 0,13mg U = 0,028 mg (k=2)

The certificate gives also for this reference standard:

V = 127,7398 cm^3 U_v = 0,0012 cm^3 (k=2).

In the calculation, for the reference standard was used the conventional mass.

Once all weighing are completed, the first step consists in the formation of the design matrix.

Matrix "X" contains the information on the equations used (the weighing scheme). Entries of the design matrix are +1, –1, 0, according to the role played by each of the parameters in each comparison.

Denote: X = (x_{ij}); i=1, …, n; j= 1, …, k; x_{ij} = 1, –1 or 0;

β is vector of unknown departures (β_j); and

Y is a vector of measured values (y_i), including buoyancy corrections:

$$X=\begin{bmatrix} -1 & 1 & 1 & 0 & 0 & 0 & 0 \\ -1 & 1 & 0 & 1 & 1 & 1 & 0 \\ -1 & 1 & 0 & 1 & 1 & 0 & 1 \\ 0 & 1 & -1 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & -1 & -1 & -1 & 0 \\ 0 & 0 & 1 & -1 & -1 & 0 & -1 \\ 0 & 0 & 0 & 1 & -1 & -1 & 1 \\ 0 & 0 & 0 & 1 & -1 & 0 & 0 \\ 0 & 0 & 0 & 1 & -1 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & -1 & -1 \\ 0 & 0 & 0 & 1 & 0 & -1 & -1 \\ 0 & 0 & 0 & 0 & 1 & -1 & -1 \\ 0 & 0 & 0 & 0 & 1 & -1 & -1 \\ 0 & 0 & 0 & 0 & 0 & 1 & -1 \end{bmatrix} \quad Y = \begin{bmatrix} 3,2586 \\ 3,2709 \\ 3,2539 \\ 0,0718 \\ 0,0692 \\ 0,0106 \\ 0,0946 \\ 0,1038 \\ 0,1038 \\ 0,0486 \\ 0,0486 \\ -0,0551 \\ -0,0551 \\ 0,0131 \end{bmatrix} \quad \beta = \begin{bmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \\ \beta_5 \\ \beta_6 \\ \beta_7 \end{bmatrix} \qquad (1)$$

The least squares solutions having the well known form:

$$\langle \beta \rangle = (X^T \cdot X)^{-1} X^T \cdot Y \qquad (2)$$

(where X^T is transpose of X and $(X^T \cdot X)^{-1}$ is termed the inverse of $(X^T \cdot X)$), gives the next results:

$$\langle\beta\rangle = \begin{bmatrix} Ni \\ 500NA \\ 500E_1 \\ 200NA \\ 200E_1 \\ 100NA \\ 100E_1 \end{bmatrix} = \begin{bmatrix} -3{,}158 \\ 0{,}089 \\ 0{,}016 \\ 0{,}057 \\ -0{,}047 \\ 0{,}011 \\ -0{,}002 \end{bmatrix} mg \qquad (3)$$

4. ANALYSIS OF UNCERTAINTIES

4.1 Type A uncertainty

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

$$s = \sqrt{\frac{1}{v}\sum_{i=1}^{n} res_i^2} \qquad (4)$$

The residuals "res." are the elements of the vector $\langle e\rangle$; "v" = n – k represents the degrees of freedom ("n – k" is the difference between the number of performed observations and the number of unknowns).

$$\langle e\rangle = res_i = Y - \langle Y\rangle = \begin{bmatrix} 3{,}2622 \\ 3{,}2671 \\ 3{,}2541 \\ 0{,}0729 \\ 0{,}0681 \\ 0{,}0081 \\ 0{,}0918 \\ 0{,}1047 \\ 0{,}1047 \\ 0{,}0491 \\ 0{,}0491 \\ -0{,}0556 \\ -0{,}0556 \\ 0{,}0130 \end{bmatrix} \qquad (5)$$

with the adjusted mass difference of the weighing equations:

$$\langle Y\rangle = X\cdot\langle\beta\rangle \qquad (6)$$

With the group standard deviation "s" of the observations $s = 0{,}0024$ mg and the inverse matrix $(X^T\cdot X)^{-1}$, the variance – covariance matrix V_β can be calculated [3].

The diagonals elements V_{jj}, of the V_β represents the type A uncertainty of the unknown weight[3]:

$$V_\beta = \begin{bmatrix} 0 & 0 & 0 & 0 & 0 & 0 & 0 & 6\cdot10^{-6} \\ 0 & 1{,}3\cdot10^{-6} & -2\cdot10^{-7} & -1\cdot10^{-7} & -2\cdot10^{-7} & 6\cdot10^{-8} & -2\cdot10^{-7} & 3\cdot10^{-6} \\ 0 & -2\cdot10^{-7} & 2{,}3\cdot10^{-6} & 2\cdot10^{-7} & 3\cdot10^{-7} & -10\cdot10^{-8} & 3\cdot10^{-7} & 3\cdot10^{-6} \\ 0 & -1\cdot10^{-7} & 2\cdot10^{-7} & 7{,}3\cdot10^{-7} & -7\cdot10^{-8} & 1\cdot10^{-7} & -9\cdot10^{-8} & 1\cdot10^{-6} \\ 0 & -2\cdot10^{-7} & 3\cdot10^{-7} & -7\cdot10^{-8} & 7{,}6\cdot10^{-7} & -2\cdot10^{-7} & 2\cdot10^{-7} & 1\cdot10^{-6} \\ 0 & 6\cdot10^{-8} & -10\cdot10^{-8} & 1\cdot10^{-7} & -2\cdot10^{-7} & 8{,}5\cdot10^{-7} & -3\cdot10^{-7} & 6\cdot10^{-7} \\ 0 & -2\cdot10^{-7} & 3\cdot10^{-7} & -9\cdot10^{-8} & 2\cdot10^{-7} & -3\cdot10^{-7} & 9{,}2\cdot10^{-7} & 6\cdot10^{-7} \\ 6\cdot10^{-6} & 3\cdot10^{-6} & 3\cdot10^{-6} & 1\cdot10^{-6} & 1\cdot10^{-6} & 6\cdot10^{-7} & 6\cdot10^{-7} & 0 \end{bmatrix} \qquad (7)$$

$$u_{A(\beta_j)} = (V_{jj})^{1/2} = \begin{bmatrix} 0{,}0011 \\ 0{,}0015 \\ 0{,}0009 \\ 0{,}0009 \\ 0{,}0009 \\ 0{,}0010 \end{bmatrix} \qquad (8)$$

4.2 Type B uncertainty

The components of type B uncertainties are:

4.2.1 uncertainty associated with the reference standard, u_r, for each weight is given by:

$$u_{r\,(\beta j)} = h_j\cdot u_{ref} \qquad (9)$$

where h_j is the ratios between the nominal values of the unknown weights β_j and one of the reference m_r.

Uncertainty of the reference standard comprises a component from calibration certificate (u_{cert}) and another one from its drift (u_{stab}) (stability of standard) [1].

$$u_{ref} = \sqrt{u_{cert}^2 + u_{stab}^2} \qquad (10)$$

The calculation of the uncertainty associated with the stability of the standard (u_{stab}) has to take into account a change in value between calibrations, assumed that a rectangular distribution. This component would be equivalent to the change between calibrations divided by $\sqrt{3}$:

$$u_{stab} = \frac{D_{max}}{\sqrt{3}} \qquad (11)$$

where D_{max} represents the drift determined from the previous calibrations. Uncertainty associated with the reference standard will be:

$$u_{r(\beta_j)} = h_j\cdot u_{ref} = \begin{bmatrix} 0{,}0081 \\ 0{,}0081 \\ 0{,}0032 \\ 0{,}0032 \\ 0{,}0016 \\ 0{,}0016 \end{bmatrix} mg \qquad (12)$$

4.2.2 *Uncertainty associated with the air buoyancy corrections*, u_b is given by [1]:

$$u_{b\,(\beta j)}^2 = (V_j - V_r h_j)^2 u_{\rho a}^2 + (\rho_a - \rho_o)^2 u^2_{Vj} + [(\rho_a - \rho_o)^2 - 2(\rho_a - \rho_o)(\rho_{a1} - \rho_o)]u^2_{Vr} hj \qquad (13)$$

where:

V_j, V_r represents the volume of test weight and reference standard, respectively;

$u_{\rho a}$ - uncertainty for the air density, calculated according to CIPM formula;

$\rho_0 = 1{,}2$ 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;

ρ_{a1} - air density determined from the previous calibration of the standard.

The variances associated with the air buoyancy corrections are:

$$u^2_{b(\beta_j)} = \begin{bmatrix} 7\cdot10^{-6} \\ 1\cdot10^{-5} \\ 1\cdot10^{-6} \\ 2\cdot10^{-6} \\ 4\cdot10^{-7} \\ 4\cdot10^{-7} \end{bmatrix} mg \qquad (14)$$

4.2.3 *Uncertainty due to the sensibility of the balance*

If the balance is calibrated with a sensitivity weight (or weights) of mass, m_s, and standard uncertainty, $u_{(ms)}$, the uncertainty contribution due to sensitivity is [1]:

$$u_s^2 = \Delta m_c^2 \cdot [u_{ms}^2/m_s^2 + u^2_{(\Delta Is)}/\ \Delta I_s^2] \qquad (15)$$

where:

ΔI_s the change in the indication of the balance due to the sensitivity weight;

$u(\Delta I_s)$ the uncertainty of ΔI_s;

Δm_c the average mass difference between the test weight and the reference weight.

$$u_s = \begin{bmatrix} 9{,}8\cdot10^{-7} \\ 9{,}8\cdot10^{-7} \\ 4{,}6\cdot10^{-7} \\ 4{,}6\cdot10^{-7} \\ 3{,}1\cdot10^{-7} \\ 3{,}1\cdot10^{-7} \end{bmatrix} mg \qquad (16)$$

4.2.4 Uncertainty due to the display resolution of the balance, u_{rez}, (for electronic balances) is calculated according to the formula [1]:

$$u_{rez} = \left(\frac{d/2}{\sqrt{3}}\right) \times \sqrt{2} = 0{,}00041 mg \qquad (17)$$

4.2.5 Uncertainty due to eccentric loading

The indication difference ΔI_s between two weights (when the positions are interchanged) was calculated. This may be interpreted as an eccentric loading error and the corresponding uncertainty was estimated using equation below [1]:

$$u_{ex} = \frac{|\Delta I_1 - \Delta I_2|}{\sqrt{3}} = 0{,}001 mg \qquad (18)$$

4.3 Combined standard uncertainty

The combined standard uncertainty of the conventional mass of the weight β_j is given by [1]:

$$u_{c(\beta j)} = [(u_A^2(\beta j) + u_r^2(\beta j) + u_b^2(\beta j) + u_{rez}^2 + u_s^2\ ]^{1/2} \qquad (19)$$

$$u_c(\beta j) = \begin{bmatrix} 500NA \\ 500E_1 \\ 200NA \\ 200E_1 \\ 100NA \\ 100E_1 \end{bmatrix} = \begin{bmatrix} 0{,}0087 \\ 0{,}0089 \\ 0{,}0037 \\ 0{,}0038 \\ 0{,}0023 \\ 0{,}0023 \end{bmatrix} mg \qquad (20)$$

4.4 Expanded uncertainty

The expanded uncertainty "U" (with k=2) of the conventional mass of the weights βj is given by:

$$U_{(\beta j)} = 2\cdot u_c(\beta j) = \begin{bmatrix} 500NA \\ 500E_1 \\ 200NA \\ 200E_1 \\ 100NA \\ 100E_1 \end{bmatrix} = \begin{bmatrix} 0{,}017 \\ 0{,}018 \\ 0{,}007 \\ 0{,}008 \\ 0{,}005 \\ 0{,}005 \end{bmatrix} mg \qquad (21)$$

4.5. Uncertainty budget

The table 2 shows all the uncertainty components described above and the standard uncertainty contributions for all of them.

5. DISCUSSION OF THE RESULTS

As is shown, for the calibration of E_1 weights were used disc weights from 500g to 100 g, having both the role of check standards and weight support plates for the whole determination.

To see if the mass values obtained for disc weights are consistent with previous values, it is necessary to perform a statistical control. The purpose of the check standard is to assure the goodness of individual calibrations. A history of values on the check standard is required for this purpose [1].

Taking into account that for the disc weights do not have sufficient calibration data to perform a statistical control according to [1], it has chosen the method of normalized error E_n, which takes into account the result and its uncertainty from the last calibration.

The results obtained for the disc weights in this subdivision procedure are compared with data from their calibration certificates [4,5]. The differences in values are normalized using the formula [6]:

$$E_n = \frac{\delta_{subdiv} - \delta_{certif}}{\sqrt{U^2_{subdiv} + U^2_{certif}}} \qquad (22)$$

where:

δ_{subdiv} represents the mass error of the disc weight obtained by subdivision method;

δ_{certif}, - the mass error from the calibration certificate of the disc weight;

U_{subdiv} - the expanded uncertainty of the disc weight obtained in subdivision method;

U_{certif}, - the expanded uncertainty of the disc weight from the calibration certificate.

Using this formula, an acceptable measurement and reported uncertainty would result in an E_n, value of between -1 and +1. The table 3 presents the results obtained for the normalized errors, E_n.

Table 3. Comparison of measurement results of disc weights, obtained by subdivision method and results from the calibration certificate

Nominal mass of disc weight	**Subdivision**		Calibartion certificate		E_n
g	δ mg	U mg	δ mg	U mg	
500 NA	0,089	0,017	0,074	0,017	0,1
200NA	0,057	0,007	0,050	0,007	0,7
100 NA	0,011	0,005	0,015	0,004	0,7

6. CONCLUSIONS

The feature of this kilogram subdivision is represented by the fact that the calibration of the weights (whose shape is in accordance to OIML R111) is performed using an automatic mass comparator.

Uncertainty obtained in this case for the unknowns weights is better than that obtained usually for E_1, being at the level acquired for reference standards (see table 2).

The comparison of results obtained for the disc weights by subdivision method to those from the calibration certificate shows the consistency of the results.

The method described for calibration of E_1 weights, can be used when the highest accuracy is required.

Table 2 The uncertainty budget

Uncertainty component			**Standard uncertainty contributions (mg)**						
			1kg Ni	**500g NA**	**500 g E_1**	**200 g NA**	**200 g E_1**	**100gNA**	**100g E_1**
	$u_{mr} \cdot hj$	in mg	**0,0162**	0,00808	0,00808	0,00323	0,00323	0,00162	0,00162
	$V_r \cdot h_j$	in cm^3	**127,7398**	63,8699	63,8699	25,5480	25,5480	12,7740	12,7740
	$u_{Vr} \cdot h_j$	in cm^3	**0,0006**	0,0003	0,0003	0,0001	0,0001	0,0001	0,0001
	V_j	in cm^3		62,5460	62,2660	25,0170	24,8530	12,5090	12,4560
	u_{Vj}	in cm^3		0,0155	0,0160	0,0140	0,0040	0,0135	0,0020
	ρ_a	mg/cm^3		1,1700	1,1800	1,1743	1,1743	1,1708	1,1708
	$u_{\rho a}$	mg/cm^3		0,002	0,002	0,002	0,002	0,002	0,002
	$(V_j - V_r h_j)^2 u$	in mg		7,019E-06	1,030E-05	1,129E-06	1,934E-06	2,812E-07	4,049E-07
	$(\rho_a - \rho_o)^2 u^2_V$	in mg		2,16E-07	1,02E-07	1,29E-07	1,05E-08	1,55E-07	3,41E-09
$[(\rho_a - \rho_o)^2 - 2(\rho_a - \rho_o)(\rho_{a1} - \rho_o)]u^2_{Vr} h_j$				-2,700E-11	-3,600E-11	-5,296E-12	-5,296E-12	-1,136E-12	-1,136E-12
	u_b^2	in mg		0,0000072	0,0000104	0,0000013	0,0000019	0,0000004	0,0000004
	u_{rez}	in mg		0,00041	0,00041	0,00041	0,00041	0,00041	0,00041
	u_d	in mg		9,805E-07	9,805E-07	4,637E-07	4,609E-07	3,133E-07	3,133E-07
	u_{ma}	in mg		0	0	0	0	0	0
	u_{ex}	in mg		0,001	0,001	0,001	0,001	0,001	0,001
	u_{ba}	in mg		0,00108	0,00108	0,00108	0,00108	0,00108	0,00108
	u_A	in mg		0,001138	0,001506	0,000854	0,000875	0,000921	0,000959
			uc=	**0,00866**	**0,00890**	**0,00369**	**0,00379**	**0,00225**	**0,00226**
			U=	**0,017**	**0,018**	**0,007**	**0,008**	**0,005**	**0,005**

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