Improvement of the Calibration Process for Class E_1 Weights Using an Adaptive Subdivision Method

Adriana Vâlcu/ National Institute of Metrology
Mass Laboratory
INM
Bucharest, Romania
e-mail: adriana.valcu@inm.ro; adivaro@yahoo.com

***Abstract*— Taking into account the amount and variety of measurements involved in scientific, industrial and legal activities that need traceability to the national mass standards of each country, it can be considered that mass standards calibration is one of the most important activities of the National Metrology Institutes (NMIs). For the determination of the conventional mass, in the calibration of weights of the highest accuracy classes, the subdivision method and its variants are widely used. For the NMIs, it is very important to demonstrate and maintain their capability of applying with good results such methods. In this respect, a calibration procedure for the determination of conventional mass, called "adaptive subdivision method" was developed in the Mass Laboratory of the Romanian National Institute of Metrology, which can lead to an improvement of CMCs (Calibration and Measurement Capabilities, approved and published in the BIPM database). According to the International Recommendation OIML R 111, the weights of nominal values greater than 1 g may have a cylindrical shape with a lifting knob. Considering this kind of shape and the use of an automatic comparator, with the maximum capacity of 1 kg, the diameter of the weighing pan is too small for placing a group of weights in the range of (500…100) g; therefore, the usual subdivision method can not be applied for the calibration of weights. The "adaptive subdivision method", presented in this paper, allows the cylindrical weights with a lifting knob, having nominal values of (500…100) g, to be calibrated using an automatic comparator (which is not equipped with weight support plates). The method can be used for class E_1 weights, where the highest accuracy is required. In this case, the resulting calibration uncertainty for the unknown weights is better than that usually obtained for E_1 masses, being at the level of reference standards.**

Keywords - Subdivision method; automatic comparator; efficiency of design.

I. Introduction

In 1889, at the First Conference of Weights and Measures (CGPM), the kilograms prototypes were shared - by chance - for each country. Romania has received the "National kilogram Prototype No. 2" (NPK).

NPK is a solid cylinder of Platinum-Iridium alloy (90%, 10%), having a height equal to its diameter (39 mm). Now, it is maintained by the National Institute of Metrology and serves as a reference for the entire dissemination of the mass unit in Romania

The realization and dissemination of the unit of mass by the Mass Laboratory of the Romanian National Institute of Metrology starts from the reference stainless steel standards (a set of three 1 kg mass standards and two sets of disc weights from 500 g to 50 g), which are traceable to the International Prototype Kilogram through the mass of the Romanian Prototype Kilogram No 2.

Starting from these reference stainless steel standards, submultiples and multiples of the unit are realized to permit the masses of additional bodies to be determined with traceability to the international standard. This takes place with the aid of several weights sets E_1 of suitable grading (in most cases 1, 2, 2, 5) which are determined "in themselves" according to proper weighing designs and by using a least squares analysis (with subdivision or multiplication methods).

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

The subdivision weighing design has both advantages and disadvantages:

- advantages [2]:

a) minimizes handling (and hence wear) of standards;

b) produces a set of data providing important statistical information about the measurements and the daily performance of the individual balances;

c) offers a redundancy of data.

- disadvantages [2]:

a) requires a relatively complex algorithm to analyze the data (as compared with other methods, for example Borda [3]);

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

To apply the calibration by subdivision method on the automatic comparator, a set of disc weights (reference standards) has been used. These weights constitute both support plates and check standards.

The main objective in the search for better designs was to find a calibration scheme which can be performed considering the following factors: the automatic comparator, the diameter of the disc weights (so that a group of OIML weights can be placed over) and the efficiency of design matrix.

 ISBN: 978-1-61208-219-6

The article is divided into 6 sections as follows: introduction, equipments and standards used in calibrations, statistical tools for evaluation of the measurement process and mass determination, analysis of uncertainties, quality assessment of the calibration, conclusions.

II. EQUIPMENTS AND STANDARDS USED IN CALIBRATION

The weighing system includes a proper balance (mass comparator) with weights transporter, a monitoring system of environmental conditions and a MC Link software

The mass comparator used was an automatic one, with the following specifications:

- maximum capacity: 1011 g;
- readability: 0.001 mg;
- pooled standard deviation: (0.4 to 2) µg (for nominal masses 100 g to 1 kg, respectively).

For accurate determination of the air density an environmental conditions monitoring system was used, consisting in a precise “climate station”, model Klimet A30.

Technical parameters 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;

The mass standard used for the comparisons was an 1 kg reference standard, Ni 81, Fig 1, whose mass value was determined at BIPM.

Fig.1. Reference standard of 1 kg, Ni81

Ni 81 had been purchased by the National Institute of Metrology in 1981. This mass standard is the second in importance after the NPK. The data included in its calibration certificate are as follows:

m_{Ni81} = 1 kg + 0.130 mg, U = 0.028 mg , (k=2);

The weights involved in calibration are:

- unknown E_1 weights (from 500g to 100g, marked with A12…A9) having OIML shape, Fig 2.

Fig. 2. Weights of class E_1

- disc weights (reference weights, marked with NA), Fig 3.

Fig. 3. Reference disc weights

For all the weights, the volumes *V* and associated uncertainties *U(V)*are given in their calibration certificates.

Table I shows these values:

TABLE I. VOLUMES *V* AND ASSOCIATED UNCERTAINTIES *U(V)* OF THE WEIGHTS

Nominal mass g	**Marking**	***V*** cm^3	***U(V)*** cm^3
1000 ref	Ni	127.7398	0.0012
500	NA	62.5480	0.0007
500	A12	62.266	0.032
200	A11	24.853	0.008
200	A10	24.853	0.008
100	NA	12.5083	0.0005
100	A9	12.456	0.004

III. STATISTICAL TOOLS FOR EVALUATION OF THE MEASUREMENT PROCESS AND MASS DETERMINATION

A. *Method used to evaluate the efficiency of the weighing design*

The dissemination of the mass scale to E_1 weights, using a single reference standard, requires mass comparisons between weights and groups of weights.

A mass calibration design (or design matrix) describes the general setup of these comparisons.

For a given number of mass comparisons, a criterion for the choice of a design matrix is that, the variances of the estimates be as small as practicable [4].

For this reason, the idea of efficiency was introduced, to enable designs to be analyzed using this criterion, taking into account the variances of the weighing results.

The efficiency is very useful when comparing designs involving the same masses and balances, even if the number of mass comparisons differs. It is desirable that the efficiency of a design be large, as this would indicate that the variances are small [4].

Table II lists the mass comparisons possible for the 1 kg to 100g decade, taking into account the following elements: the automatic comparator and the diameter of the disc weights (so that a group of OIML weights can be placed over).

TABLE II. POSSIBLE MASS COMPARISONS FOR THE 1 kg TO 100g DECADE

Obs.	Mass						
No	Ni 81	500NA	500A12	200A11	200A10	100NA	100A9
1	-1	1	1	0	0	0	0
2	-1	1	0	1	1	1	0
3	-1	1	0	1	1	0	1
4	0	1	-1	0	0	0	0
5	0	1	0	-1	-1	-1	0
6	0	0	1	-1	-1	-1	0
7	0	0	0	1	-1	-1	1
8	0	0	0	-1	1	-1	1
9	0	0	0	1	-1	0	0
10	0	0	0	1	0	-1	-1
11	0	0	0	0	1	-1	-1
12	0	0	0	0	0	1	-1

To establish the design matrix „X" of the comparisons, several versions were performed, then calculating the efficiency of the design for each of them.

For example, using the notation of [4], for the design (2, 1, 1, 2, 0, 1, 1, 0, 1, 1, 2, 1) an efficiency of 0.38 was obtained, while for the design (1, 0, 1, 1, 1, 1, 1, 1, 1, 2, 2, 1) the efficiency obtained was 0.61.

Finally, the design (2, 1, 1, 2, 1, 1, 1, 1, 0, 1, 1, 1) was chosen, having 13 equations of condition, since the value for the efficiency was greater, namely 1.04.

The efficiency was calculated in the following manner. Once all weighing are completed, the first step is to form the design matrix, "X", which contains the information on the equations used (the weighing design).

Entries of the design matrix are +1, –1, and 0, according to the role played by each of the parameters (from the vector β) in each comparison. Symbols used:

X the format for matrix: $X = (x_{ij})$; i=1… n;

j = 1,…, k; x_{ij} = 1, –1 or 0;

β vector of unknown departures (β_j);

s vector containing the standard deviation of each comparison;

Y the vector of measured values "y_i", including buoyancy corrections according to (6) .

$$X=\begin{bmatrix} 1&0&0&0&0&0&0\\ -1&1&1&0&0&0&0\\ -1&1&1&0&0&0&0\\ -1&1&0&1&1&1&0\\ -1&1&0&1&1&0&1\\ 0&1&-1&0&0&0&0\\ 0&1&-1&0&0&0&0\\ 0&1&0&-1&-1&-1&0\\ 0&0&1&-1&-1&-1&0\\ 0&0&0&1&-1&-1&1\\ 0&0&0&-1&1&-1&1\\ 0&0&0&1&0&-1&-1\\ 0&0&0&0&1&-1&-1\\ 0&0&0&0&0&1&-1 \end{bmatrix} \quad s=\begin{bmatrix} \mathbf{0.016}\\ 0.0013\\ 0.0013\\ 0.0009\\ 0.0010\\ 0.0017\\ 0.0017\\ 0.0004\\ 0.0013\\ 0.0006\\ 0.0005\\ 0.0005\\ 0.0007\\ 0.0009 \end{bmatrix} mg \quad Y=\begin{bmatrix} \mathbf{-3.1583}\\ 3.1896\\ 3.1896\\ 3.0994\\ 3.0758\\ 0.1001\\ 0.1001\\ 0.1796\\ 0.0801\\ -0.0052\\ -0.0396\\ -0.0414\\ -0.0579\\ 0.0225 \end{bmatrix} mg \quad \beta=\begin{bmatrix} \mathrm{Ni81}\\ \mathrm{500NA}\\ \mathrm{500A12}\\ \mathrm{200A11}\\ \mathrm{200A10}\\ \mathrm{100NA}\\ \mathrm{100A9} \end{bmatrix} \tag{1}$$

where:

"Ni81" represents the reference kilogram standard;

"NA" the disc weights;

"A12, A11, A10, A9" OIML weights of E_1 class.

In the "Fig. 4" it can be seen a detail of the weights combination: 500NA+200A12+200A11+100A9, part of determination "y_4"

Fig. 4. The combination of the weights from the 4th determination

The observations vector Y has a diagonal variance - covariance matrix G:

$$\boldsymbol{G} = \mathrm{diag}\,(u_r^2, s_1^2, s_2^2, \ldots s_{n-1}^2) \tag{2}$$

where u_r^2, is the square of the uncertainty of reference standard, named Ni81, and s_j^2 (j= 1, . . . $\boldsymbol{n}$- 1) is the variance of the j-th comparison.

If G' is the same as $\boldsymbol{G}$ without the first row and column, the matrix $G'^{-1/2}$ can be calculated.

By denoting with $\boldsymbol{J}$ a (n-1) x (k-1) a sub-design matrix that would be used if the same mass comparisons are carried out, without the use of a reference mass, the matrix K can be defined:

$$K = G'^{-1/2} J \tag{3}$$

Calculating K^T, which is transpose of K, one can determine the inverse $(K^T \cdot K)^{-1}$:

$$\left(K^T \cdot K\right)^{-1} = \begin{bmatrix} \mathbf{0.120} & -0.011 & 0.016 & 0.019 & 0.019 & -0.001\\ -0.011 & \mathbf{0.413} & 0.009 & 0.009 & 0.005 & 0.004\\ 0.016 & 0.009 & \mathbf{0.059} & 0.001 & 0.009 & 0.014\\ 0.019 & 0.009 & 0.001 & \mathbf{0.063} & 0.009 & -0.004\\ 0.019 & 0.005 & 0.009 & 0.009 & \mathbf{0.054} & 0.003\\ -0.001 & 0.004 & 0.014 & -0.004 & 0.003 & \mathbf{0.071} \end{bmatrix} \tag{4}$$

If v_i are the diagonal elements of $(K^T \cdot K)^{-1}$ corresponding to the i-th mass, σ_m is the largest of the σ_i, then the efficiency of the design, represented by the matrix $\boldsymbol{X}$ is defined as [4]:

$$E = \sum v_i^{-1} \cdot h_i^2 \cdot \sigma_m^2 \,/\, (n-1) \tag{5}$$

n is the number of comparisons;

h_i the ratio between the nominal values of the unknown weights and the reference.

In Table III and Table IV, the calculation of the efficiency for different designs containing 13 equations of condition is presented.

TABLE III. THE CALCULATION OF EFFICENCY FOR THE DESIGN

(2, 1, 1, 2, 1, 1, 1, 1, 0, 1, 1, 1)

$1/v_i$	h	$h_i^2 \cdot 1/v_i$	n-1	σ^2_m	$(h_i^2 \cdot 1/v_i) \cdot \sigma^2_m/(n-1)$	Standard deviation (µg)
8.33	0.5	2.0819	12	2.89	0.501	0.35
2.42	0.5	0.606			0.146	0.64
16.80	0.2	0.672			0.162	0.24
15.82	0.2	0.633			0.152	0.25
18.55	0.1	0.185			0.045	0.23
14.07	0.1	0.141			0.034	0.27
					***E* = 1.04**	

TABLE IV. THE CALCULATION OF EFFICENCY FOR THE DESIGN

(2, 1, 1, 2, 0, 1, 1, 0,1, 1, 2, 1)

$1/v_i$	h	$h_i^2 \cdot 1/v_i$	n-1	σ^2_m	$(h_i^2 \cdot 1/v_i) \cdot \sigma^2_m/(n-1)$	Standard deviation (µg)
1.946	0.5	0.487	12	2,89	0,117	0,72
1.946	0.5	0.487			0,117	0,72
5.773	0.2	0.231			0,056	0,42
5.222	0.2	0.209			0,050	0,44
8.848	0.1	0.088			0,021	0,34
7.410	0.1	0.074			0,018	0,37
					***E* = 0.38**	

It can be seen that, in the first case (Table III), a higher efficiency was obtained, which indicates that the standard deviations are smaller. Therefore, this weighing design was finally chosen to calculate the mass of the unknown and uncertainty of calibration.

B. *Mass results obtained in the calibration of weights*

If it is denoted by (A) the weighing of the reference weight and (B) the weighing of the test weight, an ABBA weighing cycle represent the sequence in which the two weights are measured to determine the mass difference of a comparison in a design matrix.

The calibration data used are obtained from the weighing cycles ABBA for each y_i (which is the weighing comparison according to design matrix "*X*").

The general mathematical model for "*y*", corrected for air buoyancy is:

$$y = \Delta m + (\rho_a - \rho_o)(V_1 - V_2) \quad (6)$$

with:

Δm is the difference of balance readings;

ρ_o 1.2 kg $\cdot$m^{-3} the reference air density;

ρ_a air density at the time of the weighing;

V_1, V_2 volumes of the weights (or the total volume of each group of weights) involved in measurement.

To estimate the unknown masses of the weights, the least square method was used [4, 5, 6].

The design matrix "*X*" and the vector of observations "*Y* " are transformed (to render them of equal variance) in *U* and *W* respectively, as follows [4]:

$$\boldsymbol{U} = \boldsymbol{G}^{-1/2}\boldsymbol{X} \quad \text{and} \quad \boldsymbol{W} = \boldsymbol{G}^{-1/2}\, Y \quad (7)$$

$$U = \begin{bmatrix} 0.0625 & 0 & 0 & 0 & 0 & 0 & 0 \\ -0.769 & 0.769 & 0,769 & 0 & 0 & 0 & 0 \\ -0.769 & 0.769 & 0,769 & 0 & 0 & 0 & 0 \\ -1.111 & 1.111 & 0 & 1,111 & 1,111 & 1,111 & 0 \\ -1 & 1 & 0 & 1 & 1 & 0 & 1 \\ 0 & 0.588 & -0.588 & 0 & 0 & 0 & 0 \\ 0 & 0.588 & -0.588 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0.769 & -0.769 & -0.769 & -0.769 & 0 \\ 0 & 2.50 & 0 & -2.5 & -2.5 & -2.5 & 0 \\ 0 & 0 & 0 & 1.667 & -1.667 & -1.667 & 1.667 \\ 0 & 0 & 0 & -2 & 2 & -2 & 2 \\ 0 & 0 & 0 & 2 & 0 & -2 & -2 \\ 0 & 0 & 0 & 0 & 1.429 & -1.429 & -1.429 \\ 0 & 0 & 0 & 0 & 0 & 1.111 & -1.111 \end{bmatrix} \quad W = \begin{bmatrix} -0.1974 \\ 2.4535 \\ 2.4535 \\ 3.4438 \\ 3.0758 \\ 0.0589 \\ 0.0589 \\ 0.0616 \\ 0.4490 \\ -0.0087 \\ -0.0792 \\ -0.0828 \\ -0.0827 \\ 0.0250 \end{bmatrix} mg \quad (8)$$

The estimates β_j and their variance-covariance matrix $V_{\beta j}$ are calculated as follows:

$$\langle \beta_j \rangle = (U^T \cdot U)^{-1} \cdot U^T \cdot W = \begin{bmatrix} \textbf{Ni81} \\ 500NA \\ 500A12 \\ 200A11 \\ 200A10 \\ 100NA \\ 100A9 \end{bmatrix} = \begin{bmatrix} \textbf{-3.158} \\ 0.0615 \\ -0.0345 \\ -0.0534 \\ -0.0704 \\ 0.0053 \\ -0.0175 \end{bmatrix} mg \quad (9)$$

$$V_{\beta j} = \left(U^t \cdot U\right)^{-1} = \begin{bmatrix} \textbf{256} & 128 & 128 & 51 & 51 & 26 & 26 \\ & \textbf{64} & 64 & 26 & 26 & 13 & 13 \\ & & \textbf{64} & 26 & 26 & 13 & 13 \\ & & & \textbf{10} & 10 & 5 & 5 \\ & & & & \textbf{10} & 5 & 5 \\ & & & & & \textbf{3} & 3 \\ & & & & & & \textbf{3} \end{bmatrix} \mu g^2 \quad (10)$$

The diagonals elements V_{jj} , of the V_βj represent the variance of the weights (which includes the type A variance combined with the variance associated to reference standard).

IV. ANALYSIS OF UNCERTAINTIES

A. *Uncertainty of the weighing process, u_A*

The variance $V_{\beta j}$ can be also expressed as [4]:

$$V_{\beta j} = h\, h^T \cdot \sigma_r^2 + R \quad \text{with} \quad R = \left(0 \,\vdots\, \frac{0^T}{\left(K^T \cdot K\right)^{-1}} \right) \quad (11)$$

The diagonals elements of the $(K^T \bullet K)^{-1}$ represents the type *A* variance of the unknown weight. From here, the type *A* standard uncertainty can be obtained:

$$u_{A(\beta_j)} = \begin{bmatrix} 0.35 \\ 0.64 \\ 0.24 \\ 0.25 \\ 0.23 \\ 0.27 \end{bmatrix} \mu g \quad (12)$$

B. Type B uncertainty

The components of type B uncertainties [1,9] are:

1) Uncertainty associated with the reference standard, u_r, for each weight is given by [1]:

$$u_{\mathbf{r}(\beta_j)} = h_{\mathbf{j}} \cdot u_{\mathbf{r}} = h_{\mathbf{j}} \cdot \sqrt{u_{\mathbf{cert}}^2 + u_{\mathbf{stab}}^2} = \begin{bmatrix} 0.008 \\ 0.008 \\ 0.0032 \\ 0.0032 \\ 0.0016 \\ 0.0016 \end{bmatrix} mg \tag{13}$$

where:

u_{cert} uncertainty of the reference standard from the calibration certificate;

u_{stab} uncertainty associated with stability of reference standard.

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

$$u_{b\,(\beta j)}^2 = (V_j - V_r h_j)^2 u_{\rho a}^2 + (\rho_a - \rho_o)^2 u^2_{Vj} + [(\rho_a - \rho_o)^2 - 2(\rho_a - \rho_o)(\rho_{a1} - \rho_o)] u^2_{Vr} h_j \tag{14}$$

where:

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

ρ_a air density at the time of the weighing;

$u_{\rho a}$ uncertainty for the air density determined at the time of the weighing, calculated according to CIPM formula;

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 one of the reference standard, respectively;

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

Uncertainty associated with the air buoyancy corrections, u_b, calculated for each weight is:

$$u_{b(\beta_j)} = \begin{bmatrix} 2.6\cdot10^{-4} \\ 4.5\cdot10^{-4} \\ 1.7\cdot10^{-4} \\ 1.7\cdot10^{-4} \\ 5.3\cdot10^{-5} \\ 7.7\cdot10^{-5} \end{bmatrix} mg \tag{15}$$

3) Uncertainty due to the sensitivity of the balance

When 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]:

$$u_s^2 = \Delta m_c^2 \cdot [u_{ms}^2 / m_s^2 + u^2_{(\Delta Is)} / \Delta I_s^2] \tag{16}$$

where:

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

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

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

Usually, the term from brackets is taken from the calibration certificate of the mass comparator.

Uncertainty associated to the sensitivity of the balance is calculated, giving:

$$u_s = \begin{bmatrix} 7\cdot10^{-7} \\ 7\cdot10^{-7} \\ 2\cdot10^{-7} \\ 2\cdot10^{-7} \\ 9\cdot10^{-8} \\ 9\cdot10^{-8} \end{bmatrix} mg \tag{17}$$

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

$$u_{rez} = \left(\frac{d/2}{\sqrt{3}} \right) \times \sqrt{2} = 0.00041 mg \tag{18}$$

C. Combined standard uncertainty

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

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

D. Expanded uncertainty

The expanded uncertainty "U" of the conventional mass of the weights β_j is given by:

$$U_{(\beta j)} = 2 \cdot u_c(\beta j) = \begin{bmatrix} 500NA \\ 500E_1 \\ 200NA \\ 200E_1 \\ 100NA \\ 100E_1 \end{bmatrix} = 2 \cdot \begin{bmatrix} 0.0080 \\ 0.0080 \\ 0.0032 \\ 0.0032 \\ 0.0017 \\ 0.0017 \end{bmatrix} = \begin{bmatrix} 0.016 \\ 0.016 \\ 0.006 \\ 0.006 \\ 0.003 \\ 0.003 \end{bmatrix} mg \tag{20}$$

V. QUALITY ASSESSMENT OF THE CALIBRATION

As shown, for calibration of the E_1 weights disc weights of 500 g and 100 g were used, 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 validity of individual calibrations. A history of values on the check standard is required for this purpose [1]. Considering that for the disc weights there are no sufficient calibration data to perform a statistical control according to [1], the method of normalized error E_n was chosen, 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 [7, 9]. The differences in values are normalized using the formula [8]:

$$E_n = \frac{\delta_{subdiv} - \delta_{certif}}{\sqrt{U^2_{subdiv} + U^2_{certif}}} \tag{21}$$

where:

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

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

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

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

Using this formula, the measurement and the reported uncertainty are acceptable if the value of E_n, is between -1 and +1.

Table V presents the results obtained for the normalized errors, E_n.

TABLE V. COMPARISON OF MEASUREMENT RESULTS OF DISC WEIGHTS, OBTAINED BY SUBDIVISION METHOD AND RESULTS FROM THE CALIBRATION CERTIFICATE

Nominal mass of disc weight	**Subdivision**		**Calibration certificate**		**E_n**
g	***δ (mg)***	***U (mg)***	***δ (mg)***	***U (mg)***	
500NA	0.062	0.016	0.076	0.017	0. 6
100NA	0.005	0.003	0.008	0.004	0..5

VI. CONCLUSIONS

An evaluation procedure has been presented, used for the calibration of a set of weights by subdivision (similar considerations had been published by the author in [9]). This calibration procedure for the determination of conventional mass of the weights was developed in the Mass Laboratory of the National Institute of Metrology, and can lead to an improvement of CMCs (Calibration and Measurement Capabilities), approved and published in the BIPM database.

The main feature of this kilogram subdivision method is represented by the fact that the calibration of the weights (whose shape is in accordance with OIML R111) is performed using an automatic mass comparator. Uncertainties obtained using this method for the unknown weights are better than those usually occur for E_1 (when only manual measurements are possible): 0.060 mg for the 500 g weight, 0.03 mg for the 200 g and 0.017 mg for the 100 g, being at the level obtained for reference standards (marked with NA).

The comparison of results obtained for the disc weights by the subdivision method with those from the calibration certificate using the normalized errors E_n, confirms the consistency of the results.

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

REFERENCES

[1] OIML, International Recommendation No 111,“Weights of classes E1, E2, F1, F2, M1, M2, M3”, 2004, pp. 65-69.

[2] S Davidson, M Perkin, M Buckley, “Measurement Good Practice Guide No 71”, NPL, TW11 0LW, June 2004, pp. 8-9.

[3] L. Wiener, S. Hilohi, A.Nadolo, Lucia Lazeanu, “Tehnica Măsurării Maselor, Volumelor şi Mărimilor Analitice (“Technique of masse measurement, volumes and analytical quantities”), 1977, pp. 45-46.

[4] E.C.Morris, ”Decade design for weighings of Non-uniform Variance”, Metrologia 29, 1992, pp. 374-375.

[5] A.Valcu, “Test procedures for class E1 weights at the Romanian National Institute of Metrology. Calibration of mass standards by subdivision of the kilogram”, Bulletin OIML, Vol. XLII, No. 3, July 2001, pp. 11-16.

[6] Schwartz R ,“Guide to mass determination with high accuracy”, PTB –MA-40, 1995, pp. 54-58.

[7] Matej Grum, Matjaž Oblak, Ivan Bajsić and Mihael Perman,. “Subdivision of the unit of mass using weight support plates”, Proceedings of XVII IMEKO World Congress, Croatia, 2003, pp. 407-408.

[8] International Standard ISO 13528:2005-09 (E), “Statistical methods for use in proficiency testing by interlaboratory comparisons”, 2005, pp. 27-28.

[9] Adriana Vâlcu and Dumitru Dinu, “Subdivision method applied for OIML weights using an automatic comparator”, Proceedings of XIX IMEKO World Congres, Lisbon, 2009, pp. 281-283.