The Provision of Mass Calibrations for Micro/Nano Force Measurements

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Abstract — **The accurate force measurement is a problem of a great importance in industry, research and society because of extremely wide range of force relating applications. Over the last decade, increasing attention has been paid among the National Institutes of Metrology (NMIs) worldwide in measurement of small forces, which play a more important role in micro or nanotechnology and other significant areas (medicine, energy, environment). The equipments used to make such measurements must have metrological traceability to a realization of SI unit of force, within the required uncertainty. In this respect, the NMIs have started to study methods for completing a hierarchy of SI-traceable force metrology at low force level. As it is known, one of the ways to achieve traceability route to SI units for force measurements is through the definition of mass, length and time. For this purpose, the known method for force calibrations or measurements is the use of deadweight machines, based on masses suspended in the Earth's gravitational field (force generated by a known mass in a known gravitational field). Another way for measuring small forces is based on the comparison of a force transducer with the indication of a balance, which works on the Electromagnetic Force Compensation (EMFC) mode. Starting from these two methods, the Mass laboratory of National Institute of Metrology (INM) from Romania considered it necessary to extend the dissemination of mass unit below 1 mg, in order to meet current needs in the field of small forces measurements. In this respect, the article deals with the provision of mass calibrations for low force measurements, consisting in (1) calibration of micromass standards having nominal values between (100…500) µg, corresponding to approx. (1....5) µN, and (2) metrological characterization of a weighing instrument that works on the principle mentioned above and has a resolution of 0.1 µg (corresponding to approx. 1nN).**

Keywords – ***micromasses; low forces measurement; deadweight machines; mass comparator; traceability.***

I. INTRODUCTION

Accurate measurements play a key role in all industrial activities, from research - development to the marketing of a product, being able to say with certainty that what cannot be measured, cannot be produced.

That is the reason why "the metrologists" are continuously involved in the development of new measurement standards, new technical methods of measurement, to conceive new tools and procedures to meet the growing demands in improving accuracy, increasing trust and speed of measurements.

In this time of flourishing nanotechnology research, the measurement of micro/nano forces becomes more significant in industry, research and society because of extremely wide range of force relating applications.

Given that some tests in different fields need to measure low forces, relevant test systems must be traceable to the masses smaller than a milligram.

In this respect, the development of the technique for micro-mass measuring is highly vital for MEMS (Micro-Electro-Mechanical Systems) and NEMS (Nano-Electro-mechanical Systems) applications and also, for providing traceability to the SI units (International System of Units, international abbreviation SI) for such measurements.

The extension of dissemination of mass scale below this limit represents the basis of micro/nano force measurements being required by industries such as pharmaceutical, defense, environmental monitoring, energy production and transportation, etc.

The calibration of these micromasses was carried out for the first time in Romania, at INM.

In a first stage, were calibrated two sets of micromass standards belonging to INM (having foil shape) and after that, in a second stage, these weights were used as check standards for calibration of other micromass weights having wire shape. In the paper, only the second stage of this calibration is presented.

The article is divided into six sections as follows: introduction, a short description of the methods used for force measurements, equipments and micromass standards used in calibrations, evaluation of standard uncertainty in the calibration of the weights, assessment of ultra-microbalance used for low force measurements, and conclusions.

II. METHODS USED FOR FORCE MEASUREMENTS. A SHORT DESCRIPTION

Currently, there are three main methods used for the force measurement [1]:

a) **Mass balance**, where the unknown force is balanced against a known mass using a digital weighing instrument.

The gravimetric calibration by using mass standards is much more accurate (with two orders of magnitude) than by using force measurements based on the dependence of some electric, magnetic, acoustic or optical parameters variation with the applied load;

b) **Force balance**, i.e., balancing force via a magnet-coil arrangement, called electromagnetic force compensation, or by means of Electrostatic Force Balance (EFB);

c) **Deflection type transducers** measuring the specific deformation of an elastic element, e.g., piezoresistive cantilever as portable microforce calibration standard.

By tradition, the traceability route for force measurement is the force generated by a known mass in a known gravitational field [2]. This force is referred to by the term deadweight. Thus, a deadweight force is traceable if a mass artifact (corresponding to the deadweight) is available.

For this purpose are used deadweight force machines (considered primary force standards), based on masses suspended in the Earth's gravitational field. The traceability is established from a traceable mass artifact combined with an accurate determination of the local gravity.

Another way for developing primary standards based on deadweights is to use a balance. This principle is based on the comparison of a force transducer with the indication of an EMFC balance. The force transducer is pressed in a controlled way against the balance and, according to Newton's principle (action equals reaction), the force equivalent mass indication on the balance is taken as the reference [3]. This way, the mechanical forces applied to the transducer can be compared indirectly with the deadweights.

III. DESCRIPTION OF THE EQUIPMENTS AND MICROMASS STANDARDS USED IN CALIBRATION

A. *Weighing instrument*

There are two ways to use a weighing instrument:

- As a mass comparator whose measurable properties are its sensitivity and/or the mass value of the smallest scale interval, measurement repeatability results (determined according to measurement cycle used, ABBA or ABA [4]) and, if necessary, the effect of the loads eccentrically placed (eccentricity).

In this case, the mass comparators allow only differential weighing (the mass comparator gives the difference of mass values between the two weights, mass standard and test weight); mass comparators are used only in the dissemination of mass unit starting from national mass standard (with values derived from the International Prototype of the kilogram) to the standards of the lowest accuracy.

- As a direct weighing instrument (a common balance) whose measurable properties are repeatability of indications, the whole range of display scale, built-in weights and, if necessary, the effect of the loads eccentrically placed (eccentricity)

In this case, the balance can be used both for differential and for proportional weighing (the balance indicates the mass of the body placed on its pan, without having recourse to mass standards) [5].

The weighing instrument used in our research is an UMX 5 balance (Mettler fabrication), presented in Figure 1, which operates in an electro-magnetic force compensation (EMFC) mode, namely the mass of a sample (weighed object) is determined by measuring the force that is exerted by the sample on its support in the gravitational field of the Earth [6].

Figure 1. UMX 5 Mass comparator [7]

As was shown, the UMX 5 can be used as mass comparator (for the calibration of the weights), whereas for the next stage, in the low force measurements can be used as a direct weighing instrument.

The weighing instrument has the following specifications:

- maximum capacity: 5.1 g;
- readability: 0.0001 mg.

B. *Air Density Measurement Equipment*

The mass of an object is obtained by weighing in air.

Because the weighing instrument indicates a value that is proportional to the gravitational force on the object reduced by the buoyancy of air, the instrument's indication in general has to be corrected for the buoyancy effect. The value of this correction depends on the density of the object (depending on the material that is made) and the density of the air [8].

A schematic representation of the air buoyancy is presented in Figure 2.

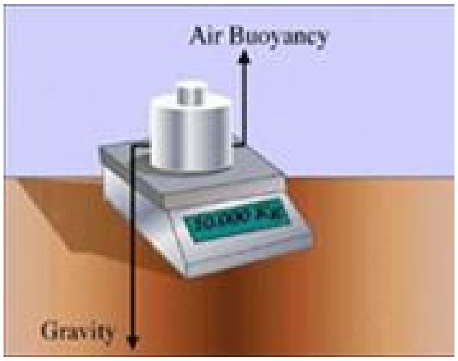

Figure 2. Representation of the air buoyancy [9]

The usual method of determining air density is to measure temperature, pressure and humidity and calculate air density using the equation recommended by the Comité International des Poids et Mesures, modified in 2007 [10]. The mass laboratory is located in a basement and the air conditions are controlled.

For accurate determination of the air density an environmental monitoring system is used, consisting in a precise "climate station", having the following technical parameters:

- 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

C. *Description of the micromasses*

The unknown weights to be calibrated are wire shaped, Figure 3, being kept in a protection box, along with a handling tool, Figure 4.

The microstandards have the nominal value between (500…100) µg with a classical sequence of (5; 2; 2; 1).

All the weights are made of aluminum alloy. At this moment, this limits the minimum mass of the standard to about 100 µg, any smaller mass being difficult to handle.

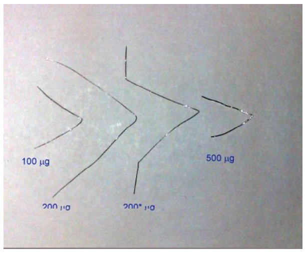

Figure 3. Micromass wire shape

Figure 4. Box containing wires shape

Micromass standards belonging to INM are foil shaped, Figure 5, being kept in a protection box, with a (5; 2; 1) sequence.

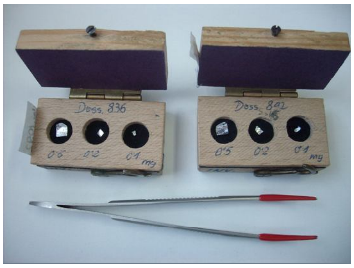

Figure 5. Boxes containing micromass foil shape

D. *Measurement model*

In the calibration of mass standards, when the highest accuracy is required, the comparison by subdivision method is mainly used. In short, the comparison consists of comparing groups of the same nominal value. These groups are performed in several variants, which have the same nominal value, variants that are compared between them, allowing a control for all the achieved measurements.

With this method, only one reference weight is used; the number of weighing equations should be larger than the number of unknown weights and an appropriate adjustment calculation should be performed in order to avoid propagating errors. In the matrix design, although all the micromass are considered unknown, in the interpretation of the results, the micromass foil shape belonging to INM, constitute also, check standards for the measurement.

As is defined in [4], a check standard is used in a statistical control process to provide a "check" to ensure that standards, measurement processes and results are within acceptable statistical limits. A check standard is usually a weight, which is included in the weighing design as an 'unknown' weight. The control procedure works best with weighing designs where the check standard can easily be incorporated into the design as an unknown weight [4].

Using as reference standard a mass of 1 mg, made of aluminum alloy, seven micromass standards are calibrated arranging them in ten possible pairs; in the design, as check standards are used: 1 mg $_{\text{INM}}$, 0.5 mg $_{\text{INM}}$ and 0.1mg $_{\text{INM}}$.

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

The comparison scheme can be represented in matrix form as follow:

$$Y = X\beta + e \tag{1}$$

where

Y $(n,1)$ is the vector of the n observations (including buoyancy corrections);

β $(k, 1)$ vector of the k mass values of the standards to be determined;

X (n, k) design matrix (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;

e $(n,1)$ vector of the deviations ;

s_i $(n,1)$ is the vector containing standard deviation of the mean value of each mass difference.

$$X = \begin{bmatrix} -1 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ -1 & 0 & 1 & 1 & 0 & 0 & 0 & 0 \\ 0 & -1 & 1 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & -1 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & -1 & 0 & 1 & 1 & 1 & 0 \\ 0 & 0 & 0 & -1 & 1 & 1 & 0 & 1 \\ 0 & 0 & 0 & 0 & 1 & -1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & -1 & -1 \\ 0 & 0 & 0 & 0 & 0 & 1 & -1 & -1 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & -1 \end{bmatrix} \quad Y = \begin{bmatrix} y_1 \\ y_2 \\ y_3 \\ y_4 \\ y_5 \\ y_6 \\ y_7 \\ y_8 \\ y_9 \\ y_{10} \end{bmatrix} = \begin{bmatrix} -0.00087 \\ 0.01727 \\ 0.01786 \\ -0.01825 \\ -0.0232 \\ 0.00116 \\ 0.00042 \\ 0.00768 \\ 0.00723 \\ -0.00634 \end{bmatrix} \tag{2}$$

$$s_i = \begin{bmatrix} 0.00018 \\ 0.00029 \\ 0.00048 \\ 0.00021 \\ 0.00043 \\ 0.00031 \\ 0.00009 \\ 0.00006 \\ 0.00017 \\ 0.00039 \end{bmatrix} mg \quad \beta = \begin{bmatrix} 1_{\text{Ref}} \\ 1_{\text{INM}} \\ 0.5_{\text{INM}} \\ 0.5 \\ 0.2 \\ 0.2* \\ 0.1_{\text{INM}} \\ 0.1 \end{bmatrix} \tag{3}$$

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

$$y = \Delta m + (\rho_a - \rho_0)(V_1 - V_2) \qquad (4)$$

with:

Δm difference of balance readings between two weights;

ρ_0 1.2 kg 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 a measurement.

To estimate the unknown masses of the weights, the least square method was used [5] [11].

The design matrix "*X*" and the vector "*Y*" are transformed in *X'* and *Y'* respectively. This transformation is usually performed when the observations are of unequal accuracy (to render them of equal variance). Taking into account that such tiny micromass standards are calibrated and the scale division of the comparator is very small (0.1 µg), any influence, which can affect the results should be considered:

$$X' = G \cdot X \text{ and } Y' = G \cdot Y \qquad (5)$$

G is a diagonal matrix containing the diagonal elements:

$$g_{ii} = (\sigma_0/s_i)^2 \;, \qquad i = 1 \ldots n \qquad (6)$$

and σ_0 a normalization factor defined by [5]:

$$\sigma_0^2 = 1/\Sigma(1/s_i^2) \;, \; i = 1 \ldots n, \qquad (7)$$

The estimates of the unknown masses are calculated, giving the next results:

$$\langle\beta\rangle = \left(X'^T X'\right)^{-1} X'^T Y' = \begin{bmatrix} 0.0013 \\ 0.0005 \\ 0.0184 \\ 0.0001 \\ 0.0010 \\ 0.0006 \\ -0.0065 \\ -0.0002 \end{bmatrix} mg \qquad (8)$$

IV. EVALUATING STANDARD UNCERTAINTY IN THE CALIBRATION OF THE WEIGHTS

In evaluating standard uncertainty associated with the results of calibration, the following contributions must be taken into account:

- type *A* uncertainty: evaluation of uncertainty method by statistical analysis of series of repeated observations;

- type *B* uncertainty is evaluated by scientific judgment based on all of the available information on the possible variability of an input quantity that has not been obtained from repeated observations.

A. Type A evaluation of standard uncertainty. Uncertainty u_A of the weighing process

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

$$U_{A(\beta_j)} = s\sqrt{c_{ij}} \qquad (9)$$

where:

c_{ij} are the diagonal elements of the matrix $(X'^T \cdot X')^{-1}$;

s is the group standard deviation calculated as follows [5]:

$$s^2 = \{\textstyle\sum[s_i^2(n_i-1)\cdot g_{ii} + \langle e_i' \rangle^2]\}/f \qquad (10)$$

f are "the degrees of freedom," being equal to :

$$f = (\textstyle\sum n_i) - M \qquad (11)$$

n is the number of weighing equations;

M is the total number of the weights.

If $\langle y' \rangle = X' \langle\beta\rangle$ are the estimates of the weighted weighing results, the vector of the weighted residuals, <e'>, can be obtained from:

$$\langle e' \rangle = y' - \langle y' \rangle \qquad (12)$$

B. Type B evaluation of standard uncertainty

The components of type *B* uncertainty are:

- Reference standard, u_r;
- Resolution of the weighing instrument, u_{res};
- Sensitivity of the weighing instrument, u_s;
- Effect of the air buoyancy, u_b;
- Effect of the load eccentric placed, u_{ecc};
- Magnetic proprieties of the weights, u_{ma};
- Convection effects, u_{conv}.

All these components are calculated in the same manner as in [11] [12].

C. Combined standard uncertainty, u_c

The combined standard uncertainty of the weight β_j is given by [4]:

$$u_{c(\beta j)} = [(u_A^2(\beta_j) + u_r^2(\beta_j) + u_b^2(\beta_j) + u_s^2 + u^2_{res} + u^2_{ecc} + \\ + u^2_{ma} + u^2_{conv}]^{1/2} \qquad (13)$$

D. Expanded uncertainty

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

$$U_{(\beta_j)} = 2 \cdot u_{c(\beta_j)} = 2 \cdot \begin{bmatrix} 0.00060 \\ 0.00033 \\ 0.00033 \\ 0.00019 \\ 0.00020 \\ 0.00017 \\ 0.00017 \end{bmatrix} = \begin{bmatrix} 0.0012 \\ 0.0007 \\ 0.0007 \\ 0.0004 \\ 0.0004 \\ 0.0003 \\ 0.0003 \end{bmatrix} mg \qquad (14)$$

V. ASSESSMENT OF ULTRA - MICROBALANCE

The balance was characterized only in the range 100 µg to 1 mg corresponding to the nominal mass of the calibrated micro standards.

A. Tests performed for the calibration of ultra-microbalance

The calibration of the weighing instrument consisted in [13]:

- applying test loads to the instrument under specified conditions;

- determining the errors of the indication and uncertainty of measurement attributed to the results.

For a load *j* applied on the pan in an ascending, descending, or in combination way, the error of indication was calculated as follows:

$$E_j = I_j - m_{mmj} \qquad (15)$$

where I_j is the indication of the balance and m_{mmj} is the mass value of the micromass from the calibration certificate. The errors of indication were determined at the next loads: 1 mg, 900 µg, 700 µg, 600 µg, 500 µg, 300 µg, 200 µg and 100 µg;

- repeatability test consisted in loading the balance with the same load under repeatability conditions, namely: the same measurement procedure, same operator, same measuring system, same operating conditions and same location and replicate measurements on the same or similar objects over a short period of time [14]. Repeatability of indication was determined at the next loads: 1 mg, 0.5 mg, 0.2 mg and 0.1 mg.

- determining the effect of the loads eccentrically placed: the load L_{ecc} was applied in an arbitrary order on the pan in the positions indicated in Figure 6 (A, B, C, D, E) in order to check the influence of eccentrically placed weights on the measurement. The eccentricity was performed at 200 µg.

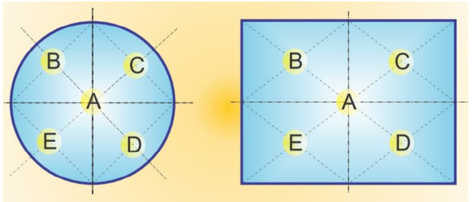

Figure 6. Positions of the load on the pan

From the indications Ii obtained for different positions of the load, is calculated the difference ΔI_{ecci}:

$$\Delta I_{ecci} = I_i - I_A \qquad (16)$$

B. *Standard uncertainty of the error of indication*

Starting from the equation (15) it can be obtained the standard uncertainty of the error:

$$u(E) = \sqrt{u^2(I) + u^2(m_{mm})} \qquad (17)$$

where:

$u(E)$ is standard uncertainty of the error of indication;

$u(I)$ is standard uncertainty of the indication;

$u(m_{mm})$ is standard uncertainty of the micromass used.

The expanded uncertainty of the error was calculated as follows [13, 15]:

$$U(E) = 2u(E) = 2\sqrt{d_0^2/12 + d_I^2/12 + s^2(I) + \hat{w}^2(\delta I_{ecc}) I^2 + u^2(m_{mm})} \qquad (18)$$

where:

- d_0 is the resolution of the balance at no-load indication;
- d_L the resolution of the balance at load ;
- $s(I)$ uncertainty due to repeatability of the indication, given by standard deviation of several weighing results;

$$u(\delta I_{rep}) = s(I_j) = \sqrt{\frac{\sum_{i=1}^{n} (I_{ji} - \overline{I_j})^2}{n-1}} \qquad (19)$$

- $\hat{w}(\delta I_{ecc})$ is uncertainty associated to the effect of the load eccentrically placed:

$$\hat{w}(\delta I_{ecc}) = \frac{|\Delta I_{ecc,i}|_{\max}}{2\sqrt{3} L_{ecc}} \qquad (20)$$

- $u(m_{mm})$ is standard uncertainty of the micromass, given by expended uncertainty from the calibration certificate, U, combined with uncertainty due to instability of the micromass, u_{instab} (or drift D).

$$u(m_{mm}) = \sqrt{\left(\frac{U}{k}\right)^2 + u_{instab}^2} = \sqrt{\left(\frac{U}{k}\right)^2 + \left(\frac{D}{2\sqrt{3}}\right)^2} \qquad (21)$$

All the uncertainty components can be graphically represented in an Ishikawa (Fishbone) diagram, as shown in Figure 7.

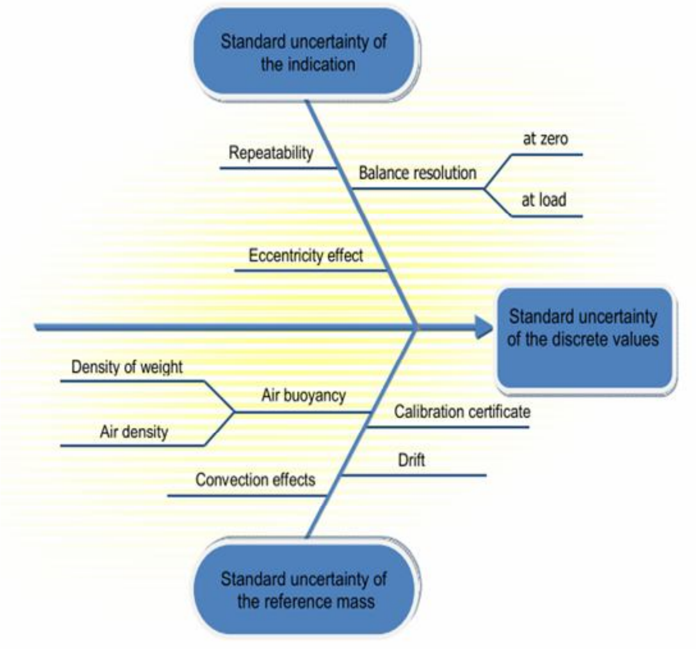

Figure 7. Ishikawa diagram of uncertainty components in the calibration of the balance

TABLE I. CENTRALIZATION OF THE RESULTS OBTAINED IN THE ULTRA-MICROBALANCE CALIBRATION

Quantity of Influence			**Results**							
Indication		in µg	100,1	200,1	300,3	500,4	600,2	701,4	901,3	1001,4
Error		in µg	-0,3	0,5	0,49	-0,26	-0,2	-0,27	0,4	-0,1
Repeatability		in µg	0,0710	0,0707	0,0707	0,0833	0,0833	0,0833	0,0833	0,0823
Digitalisat'n	$d_0/\sqrt{12}$	in µg	0,0289	0,0289	0,0289	0,0289	0,0289	0,0289	0,0289	0,0289
Digitalisat'n	$d_I/\sqrt{12}$	in µg	0,0289	0,0289	0,0289	0,0289	0,0289	0,0289	0,0289	0,0289
Eccentricity	$\hat{w}_{ecc}(I)$	in µg	0							
$u(I)$		in µg	0,0819	0,0816	0,0816	0,0928	0,0928	0,0928	0,0928	0,0919
Uncertainty of reference standard		in µg	0,350	0,390	0,740	0,660	1,010	1,050	1,440	1,200
Test loads m_N		in µg	100	200	300	500	600	700	900	1000
$u(\delta m_c)=U_{mc}/2$		in µg	0,1750	0,1950	0,3700	0,3300	0,5050	0,5250	0,7200	0,6000
$u(\delta m_D)=D/\sqrt{3}=\ U_{mc}/\sqrt{3}$		in µg	0,1010	0,1126	0,2136	0,1905	0,2916	0,3031	0,4157	0,3464
$u(\delta m_{conv})$		in µg	0							
Uncertainty of the error u**(E)**		in µg	0,2180	0,2395	0,4350	0,3922	0,5905	0,6133	0,8365	0,6989
k			2							
$U(E)=ku(E)$		in µg	0,44	0,48	0,87	0,78	1,18	1,23	1,67	1,40

Table I contains a centralization of results obtained in the calibration of the microbalance in the corresponding range of calibrated micromasses.

VI. CONCLUSION AND FUTURE WORK

The aim of the work described here is to provide mass calibrations for force measurements below 10 µN by extending the mass scale below 1 mg.

Thus, traceable force can be obtained using mass artifacts ranging from (100…500) µg to create appropriate deadweight loads.

Other applications of micromasses are in improving uncertainty in determining the indication error and sensitivity error of weighing instruments of special accuracy (micro and ultra-microbalances).

In the future, these microweights will become essential for providing traceability for new areas such as biotechnology and to meet the current requirements of various sectors of health and defense.

The paper focused on the calibration of weights below 1mg, carried out for the first time in Romania, at INM.

Also, a balance with special accuracy (ultra-micro) was characterized in a metrological manner for the first time in the low range 1 mg … 100 µg using micromass standards.

Thus, the balance can be used as was described at the Section II.

Measurement procedures and associated uncertainty obtained in the calibrations of the microweights and of the balance were presented.

Currently, the smallest mass standard used in metrology is 1 mg. Although in OIML recommendation [4], the traceability of microweights to SI units is not identified (some referring being made only in the description of the subdivision method), their calibration gives the possibility to extend the dissemination of mass scale below this limit.

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