

## MEANS FOR ENSURING SI TRACEABILITY OF MICROFORCES AND / OR MICROMASSES

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**Abstract:** Over the last period, increasing attention has been paid to measurement of small forces which play a more important role in nano-technology and other significant areas such as MEMS (Micro-Electro-Mechanical Systems). In this respect, the development of mass standards and measurement techniques below the current limit of 1 milligram is vital to provide traceability to the SI for such measurements. The lowest traceable force realized by a deadweight standard machine is typically around the level of 1 N. The national measurement institutes (NMIs), motivated by the need for small force standards, have started to explore methods for establishing a hierarchy of SI-traceable force standards at low-force level, consisting of a primary realization, a transfer standard, and methods of dissemination to instruments.

**Keywords:** traceability, microgram, transfer standard, microforce transducer, cantilever

### 1. INTRODUCTION

The three main methods used in force measurements are:

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

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. However, in the area of very low forces the electrical methods have proven their outstanding utility [1].

b) **Force balance**, i.e. balancing force via a magnet-coil arrangement, called electromagnetic force compensation (EMFC), as applied in PTB and KRISS, or by means of electrostatic force balance (ESFB), used in NIST, NPL and CMS;

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

### 2. METHODS FOR MICROFORCES AND / OR MICROMASSES TRACEABILITY

In the present paper a general scheme is proposed for the implementation of micromasses and microforces measurements traceability, starting from various achievements of the NMIs in this area. Figure 1 shows the following four possibilities:

i) The main idea [2] to develop primary standards based on deadweights is to use a mass balance. Instead of direct application of a mass artifact, force transducers (e.g. interposed piezoresistive cantilevers) are pressed against a mass comparator using a precision positioning stage. Electromagnetic forces generated by a coil and a permanent magnet in the mass comparator counteract the mechanical forces so as to maintain a constant balance position. Then these electromagnetic forces are compared with corresponding traceable deadweights by weighing calibrated mass artifacts on the mass comparator; in this manner, the mechanical forces applied to the transducer can indirectly be compared with the deadweights.

ii) The electromagnetic mass comparator could be replaced with an electrostatic force balance for the realization of micronewton forces in a manner traceable to the International System of Units [3]. The capacitance of this geometry is in principle a linear function of the overlap of the two coaxial cylindrical capacitors. For the typical balance operation, a mechanical imbalance is imposed on the counterweight side (not represented here) which is countered by an electrostatic force applied by the controller to maintain null as measured by the interferometer. Therefore, the measurement standard of microforce would be traceable to the primary standards of length and voltage instead of the mechanical artifact for the SI mass unit.

iii) As shown by Campbellova et al [4], atomic force microscope cantilevers (associated with laser and interferometer) were used as transfer standards for the calibration of the microforce transducer. They were calibrated on a Mettler Toledo mass comparator with resolution  $0.1 \mu\text{g}$  and repeatability  $0.4 \mu\text{g}$ , being traceable to their national mass standard.

iv) Instead of the optical read-out type cantilever, a piezoresistive cantilever as portable microforce calibration standard could be used, as indicated in [5].

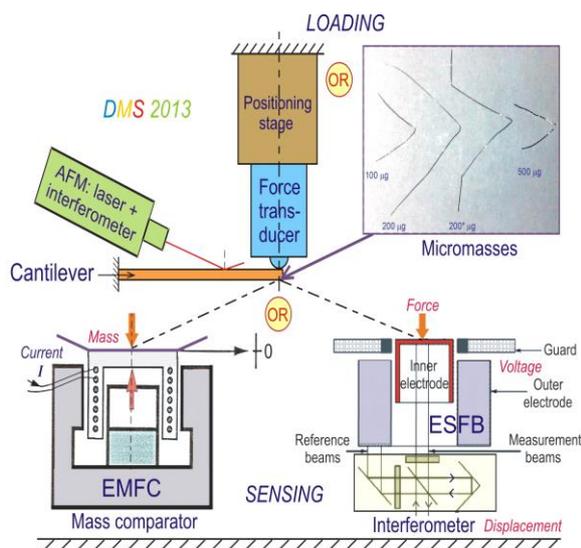
All these possibilities are schematically shown in Figure 1, in an original context of associating micromasses and microforces, under a unifying conception.

### 3. RESULTS CONCERNING MICROMASSES TRACEABILITY IN ROMANIA

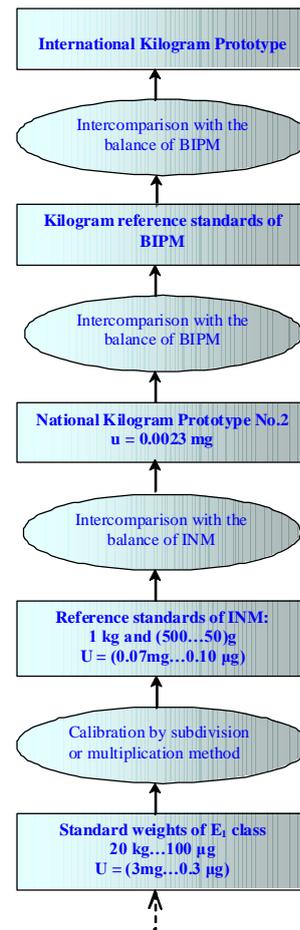
The calibration of micromass standards having nominal values between  $100 \mu\text{g}$  and  $500 \mu\text{g}$  was carried out for the first time in Romania, at INM.

In the calibration of  $E_1$  mass standards, where the highest accuracy is required, the comparison by subdivision method is mainly used (the calibration of the set weights is performed in itself).

When using only one reference standard, the weighing scheme is an overdetermined system of weighing equations and an appropriate adjustment calculation should be performed in order to avoid propagating errors.



**Fig. 1.** Means for ensuring SI traceability of microforces and/or micromasses



**Fig. 2.** Traceability chain for micromass standards

The traceability chain for micromass is shown in Figure 2.

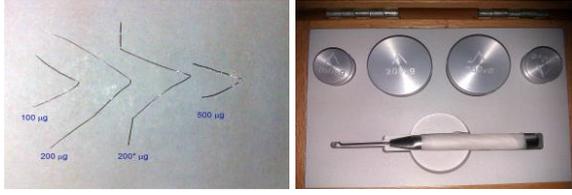
#### 3.1 Measurement instruments used for comparison

The comparator used for these measurements was an UMX 5 balance with  $0.1 \mu\text{g}$  resolution. Figure 3 shows this mass comparator.

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



**Fig. 3.** The weighing instrument, UMX 5 mass comparator, used in calibration



**Fig. 4.** a) Micromass wire shape; b) Box containing micromasses and handling tool

### 3.2 Description of the micromass

For calibration, a set of micromass standards was used, having wire shape (Fig. 4,a) kept in a protection box along with a handling tool (Fig. 4,b). All the weights are made of aluminum alloy. The set of microstandards consists of the following sequence of nominal values: (5; 2; 2; 1).

### 3.3. Measurement model

Using as reference standard a mass of 1 mg, made of stainless steel, five unknown micromass standards are calibrated according to the weighing matrix presented in the Table 1. As check standard is used a mass of 0.1 mg, made of aluminium alloy. The calibration data used are obtained from six weighing cycles ABBA for each comparison.

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

$$Y = X\beta + e$$

where

- $Y(n,1)$  is the vector of the  $n$  observations (including buoyancy corrections);
- $\beta(k,1)$  the 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
- $e(n,1)$  vector of the deviations.

**Table 1.** Weighing matrix  $X$  used for the calibration of the micromass

No.	Mass (mg)					
	1	0,5	0,2	0,2*	0,1	0,1*
1	1	-1	-1	-1	-1	0
2	1	-1	-1	-1	0	-1
3	0	1	-1	-1	-1	0
4	0	1	-1	-1	0	-1
5	0	0	1	-1	1	-1
6	0	0	1	-1	1	-1
7	0	0	1	-1	-1	1
8	0	0	1	-1	-1	1
9	0	0	1	0	-1	-1
10	0	0	1	0	-1	-1
11	0	0	0	1	-1	-1
12	0	0	0	1	-1	-1

The results “ $Y$ ” of the twelve observations, obtained from the weights combinations  $\beta$  in the matrix  $X$ , are the following:

$$Y = \begin{bmatrix} y_1 \\ y_2 \\ y_3 \\ y_4 \\ y_5 \\ y_6 \\ y_7 \\ y_8 \\ y_9 \\ y_{10} \\ y_{11} \\ y_{12} \end{bmatrix} = \begin{bmatrix} -0.00062 \\ 0.00579 \\ -0.00076 \\ 0.00556 \\ 0.00674 \\ 0.00674 \\ -0.00556 \\ -0.00556 \\ 0.00781 \\ 0.00781 \\ 0.00723 \\ 0.00723 \end{bmatrix} \text{ mg} \quad \beta = \begin{bmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \\ \beta_5 \\ \beta_6 \end{bmatrix} = \begin{bmatrix} 1 \wedge \\ 0.5 \wedge \\ 0.2 \wedge \\ 0.2 \wedge \\ 0.1 \wedge \\ 0.1 \square \end{bmatrix} \quad (1)$$

The results of the calibration are obtained with the aid of least squares adjustment which also provides the variance-covariance matrix of the calculated mass values.

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

$$\langle \beta \rangle = (X^T X)^{-1} X^T Y = \begin{bmatrix} 0.0005 \\ 0.0002 \\ 0.0009 \\ 0.0003 \\ -0.0003 \\ -0.0066 \end{bmatrix} \text{ mg} \quad (2)$$

## 4. UNCERTAINTY ESTIMATION

In order to evaluate the standard uncertainty associated with the results of calibration, the following contributions have to be taken into account:

- type A uncertainty, evaluated by statistical analysis of series of repeated observations;
- type B uncertainty, 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, such as:
  - reference standard,
  - resolution of the weighing instrument;
  - sensitivity of the weighing instrument;
  - effect of the air buoyancy.

Other contributions (magnetism or convection) in the calculation of the measurement uncertainty may be omitted during this investigation.

The uncertainty components are calculated in the same manner as in [6], [7], [8] and can be graphically represented in an Ishikawa (fishbone) diagram, as shown in Figure 5.

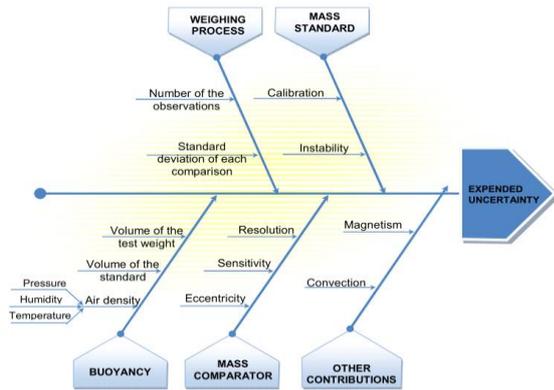


Fig. 5. Ishikawa diagram of uncertainty components in micromasses determination

### 5. UNCERTAINTY BUDGET

Based upon these experimental results, the uncertainty budget can be drawn up (Table 2). This table contains all the components described before.

Table 2. Uncertainty budget

Uncertainty component	Standard uncertainty contribution (mg)	Standard uncertainty contribution (mg)					
		1mg	500 µg	200 µg	200 µg	100 µg	100 µg
$u_{ncr} \cdot h_j$	in mg	0.0006	0.00032	0.00013	0.00013	0.00006	0.000064
$V \cdot h_i$	in cm <sup>3</sup>	0.00013	0.00006	0.000025	0.000025	0.000013	0.000013
$u_{VV} \cdot h_i$	in cm <sup>3</sup>	2E-06	0.000	0.000	0.000	0.000	0.000
$V_j$	in cm <sup>3</sup>		0.00019	0.00007	0.00007	0.00004	0.000037
$u_{Vi}$	in cm <sup>3</sup>		1.37E-06	5.49E-07	5.49E-07	2.74E-07	0.000000
$\rho_a$	mg/cm <sup>3</sup>		1.1716	1.1716	1.1716	1.1716	1.1716
$u_{\rho a}$	mg/cm <sup>3</sup>		0.0006	0.0006	0.0006	0.0006	0.000606
$(V_i \cdot V_j \cdot h_i)^2 \cdot u_i$	in mg		5.49E-15	8.79E-16	8.79E-16	2.20E-16	2.20E-16
$(\rho_a - \rho_j)^2 \cdot u_j^2$	in mg		1.52E-15	2.44E-16	2.44E-16	6.09E-17	6.09E-17
$(\rho_a - \rho_a)^2 \cdot 2(\rho_a - \rho_j) \cdot u_j \cdot u_{\rho a} + (\rho_a - \rho_j)^2 \cdot u_{\rho a}^2$			-1.62E-15	-2.60E-16	-2.60E-16	-6.50E-17	-6.50E-17
$u_b^2$	in mg		5.39E-15	8.63E-16	8.63E-16	2.16E-16	2.16E-16
$u_{re}$	in mg		0.000041	0.000041	0.000041	0.000041	0.000041
$u_s$	in mg		0	0	0	0	0
$u_{com}$	in mg		0	0	0	0	0
$u_{ecc}$	in mg		0.0001	0.0001	0.0001	0.0001	0.0001
$u_{ma}$	in mg		0	0	0	0	0
$u_{ba}$	in mg		0.000108	0.000108	0.000108	0.000108	0.000108
$u_A$	in mg		0.000035	0.000024	0.000024	0.000024	0.000026
$u_c$			0.00034	0.00017	0.00017	0.00013	0.00013
$u$			0.00068	0.00034	0.00034	0.00026	0.00026

### 6. CONCLUSIONS

In Romania, the Mass laboratory of the National Institute of Metrology has considered necessary to extend the dissemination of the mass unit below 1 mg, in order to meet current needs.

Using the subdivision method as metrological procedure and starting from the national prototype kilogram No. 2, all necessary experiments were performed for the first time in Romania to extend mass unit traceability down to 100 µg.

This extension also supports the provision of mass calibrations for low force measurements.

### 7. ACKNOWLEDGMENTS

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