

MASS AND DENSITY DETERMINATION OF OIML E1 WEIGHT SET IN CZECH METROLOGY INSTITUTE

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Abstract – In this article we introduce the results of density and volume measurements of sub-gram and 1 kg weighs. At first the theoretical principles of such experiments are described. These include the uncertainty calculations. Sub-gram weights were measured in volume and mass comparators. The results are shown in the last part of this paper. The result is, that our primary mass laboratory can measure the density of milligram weight with uncertainty below 2000 kg/m³ (k=2).

Keywords – mass determination, density determination, E1 weighs

1. INTRODUCTION

Measuring of mass is one of the most important processes in this world, so it is important to measure it precisely. Due to the fact that the international prototype is made of Platinum – Iridium compound and working etalons are made of stainless steel, we have to take in account the effect of buoyancy forces if we measure the mass in the air. We can determine the effect of such forces if we know the volume of weighed body.

The volume determination were not held in Czech Metrology institute until last year so we did not have any density measurements to prove the class of our weighs. The OIML recommendation R111 [1] describes several procedures for measuring the density of weighed body. All methods are based on direct measurement using comparison methods but these are not suitable for our laboratory because only one weight has known volume. One of the solutions is method from [1], the other one is based on the system used for the realization of the mass scale.

2. THEORETICAL BACKGROUND

Equation (1) describes the weighing process in air or any other environment. One of its parameters is the volume of the tested body.

$$\Delta m = (m_T - V_T \rho_a) - (m_E - V_E \rho_a) \quad (1)$$

If we look more deeply into this equation, we can see that other parameter is density of air during the measurement. But we can measure in other environments, for example in water or other special liquids or in vacuum. Finally we have two equations with measurements in two different environments and two unknown parameters (mass and

volume of the tested body). This system is solvable and the solution is

$$m_T = m_E + \Delta m_a + \frac{\Delta m_l - \Delta m_a}{\rho_a - \rho_l} \rho_a \quad (2)$$

$$V_T = V_E + \frac{\Delta m_a - \Delta m_l}{\rho_l - \rho_a} \quad (3)$$

In real measurements we have to use conventional values so the equations will be more difficult. But the main idea of such calculations is clear.

In our case we know the parameters of only one weight exactly so we have to use different method then typical comparison. The method is based on the well known system of 12, 14 or 16 equations in one decade. System of such equations is said to be orthogonal and the covariance matrix is diagonal if we omit the uncertainty of the reference mass.

The estimation of result of such system is

$$\bar{m} = (A^T H_{\xi}^{-1} A)^{-1} A^T H_{\xi}^{-1} W \quad (4)$$

where W is the vector of the results from the comparator, A is the matrix of the weighing scheme and H is known matrix of the weights of each measurement.

The estimation of the standard deviation of the result (4) is

$$\sigma^2 = \frac{(W - A\bar{m})^T H_{\xi}^{-1} (W - A\bar{m})}{n - k} \quad (5)$$

with associated covariance matrix

$$\overline{U}_m = \sigma^2 (A^T H_{\xi}^{-1} A)^{-1} + Q U_{\xi} Q^T \quad (6)$$

where Q is any matrix and U_{ξ} is vector with uncertainties of reference masses. In case of the mass measurement and typical set of weighs the matrix $Q = \frac{1}{10} (5, 2, 2, 1, 1)^T$ [4].

Another approach is to use different criteria to find the optimal weighing scheme. One of them shall be the lowest sum of relative variances [3] which came from the least square model. This coefficient is useful when we did not need the covariances between the weighs for further calculations which is the case for mass laboratory of Czech Metrology Institute.

Interesting attribute of this criteria is that we can obtain lower sum using lower number of equations. This can be

explained by simple realization that equations where there are more than one weigh in each positions provide us same information as another equations with one weigh on one side, but larger uncertainty. For example, the typical system of 14 equations is described by the matrix

$$\begin{pmatrix} 1 & 1 & 1 & 1 & 0 \\ 1 & 1 & 1 & 0 & 1 \\ -1 & 1 & 1 & 1 & 0 \\ -1 & 1 & 1 & 0 & 1 \\ 0 & -1 & 1 & -1 & 1 \\ 0 & -1 & 1 & 1 & -1 \\ 0 & -1 & 1 & 0 & 0 \\ 0 & -1 & 1 & 0 & 0 \\ 0 & -1 & 0 & 1 & 1 \\ 0 & -1 & 0 & 1 & 1 \\ 0 & 0 & -1 & 1 & 1 \\ 0 & 0 & -1 & 1 & 1 \\ 0 & 0 & 0 & -1 & 1 \\ 0 & 0 & 0 & -1 & 1 \end{pmatrix} \quad (7)$$

and its coefficient is (for 10 times repeated ABA) 0,73. Another matrix, where the 5th and 6th equations are missing and the number of the equations is lower is for example

$$\begin{pmatrix} 1 & 1 & 1 & 1 & 0 \\ 1 & 1 & 1 & 1 & 0 \\ 1 & 1 & 1 & 1 & 0 \\ 1 & 1 & 1 & 0 & 1 \\ 1 & 1 & 1 & 0 & 1 \\ -1 & 1 & 1 & 1 & 0 \\ -1 & 1 & 1 & 0 & 1 \\ 0 & -1 & 1 & 0 & 0 \\ 0 & -1 & 0 & 1 & 1 \\ 0 & 0 & -1 & 1 & 1 \\ 0 & 0 & 0 & -1 & 1 \\ 0 & 0 & 0 & -1 & 1 \end{pmatrix} \quad (8)$$

where the coefficient is 0,55. The systems with lower coefficients exist but we have to take in account the necessary time for these measurements.

We prepare the comparisons in air and in the special liquid. If we know the volume of one weight we can calculate its apparent mass in the liquid and use it as standard. After all comparisons we have one system of equations from air, the other one from liquid, we calculate the apparent masses of each weight and using simple equations (9) we are able to compute the mass and volume of each tested body.

$$m_T = \frac{\Delta m_1 \alpha_{\Delta m_1}}{\alpha_T} \quad (9)$$

$$\rho_T = \frac{\rho_2 \Delta m_1 \alpha_{\Delta m_1} - \rho_1 \Delta m_2 \alpha_{\Delta m_2}}{\Delta m_1 \alpha_{\Delta m_1} - \Delta m_2 \alpha_{\Delta m_2}}$$

These equations (9) depend on the densities of the atmosphere or used liquid and on the density of the weight which was used for the calibration of the comparator.

3. EXPERIMENTAL SETUP

Our equipment consists of mass comparators Mettler Toledo AT 10005, AT 1006, AT 106 and UMT 5. The masses were measured only at UMT 5. The volume was measured at volume comparator Mettler Toledo VC 1005.

The mass comparator Mettler Toledo UMT 5 has the maximum capacity of 5,1 g. Readability of this comparator is 100 ng and typical repeatability is about 400 ng. This comparator is manual which increase the total time of the measurement.

The volume comparator Mettler Toledo VC1005 has maximum load of 1109 g with 100 g scale. The readability is 10 µg and repeatability 40 µg. This comparator is automatic.

As the standard was taken the 1 gram weight which parameters were measured as a part of diploma thesis [2]. Its mass is 1 g – 0,006 mg ± 0,006 mg and density 7970 kg/m³ ± 290 kg/m³. The method we used to determine its parameters is similar to the method A 3 of density determination in [1].

In the air and also FC-40, which is used in our laboratory, the system of equations in decades was used. The systems of 12, 14 and 16 equations were used and in this extended abstract the results of 14-equations system are to be presented.

4. UNCERTAINTY CALCULATIONS

The uncertainty of each measurement was determined. We made 10 cycles ABBA in liquid and 5 cycles ABA in the air. Then the parameters for uncertainty of type A are fully determined.

The uncertainty of type B consists of parameters such as uncertainty of difference between support discs used in the liquid, uncertainty of the reference weight or uncertainty of the comparator itself (linearity and scale interval). Typical uncertainty obtained in the air was about 20 µg, in the liquid about 0,2 mg. It is possible to achieve better uncertainty and the experiment to prove this will be realized in summer 2009.

5. RESULTS

Tab 1: Results of calibration

m [mg]	Δm [mg]	u _m [mg]	ρ [kg/m ³]	u _ρ [kg/m ³]
500	0,007	0,041	7993	16
200	0,005	0,016	7994	21
200	-0,002	0,016	7988	24
100	0,003	0,009	7998	42
50	0,000	0,034	7990	150
20	0,001	0,014	7950	210
20	0,001	0,014	7930	250
10	0,007	0,007	7780	440
5	0,003	0,030	7800	1300
2	-0,001	0,013	7800	1800
2	0,000	0,013	7800	2100
1	-0,001	0,008	6600	3000

6. CONCLUSION

The results are presented in the short form in Tab. 1. The results of each comparison are not presented here due to the lack of place. Also the more detailed uncertainty calculations are not presented.

The results are reasonable and prove the fact that quality of our laboratory increases. In some cases the uncertainty is higher than expected but this is due to the unstable temperature in the days of measurement. This instability was caused by the revision of the air condition system which was held in our laboratory in the same time. If we eliminate the temperature instability then the results should be with lower uncertainty.

The theory has shown that orthogonal designs which are widely used can be substituted by other systems which are not orthogonal but their sum of relative uncertainties is lower.

These results are very promising for the forthcoming realization of vacuum comparator. The ambient conditions shall be more stable than nowadays and the better readability and associated repeatability will cause the better uncertainty of the results.

In this paper the progress in the field of density measurements in Czech Metrological Institute is presented. The method using system of equations was used for measurements in FC-40, a special fluorocarbon liquid. The obtained results are acceptable and should be improved in better conditions. In the following months the complete determination of masses and volumes of the weights will be realized.

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