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MASS AND VOLUME CALIBRATION OF WEIGHTS UNDER VACUUM CONDITIONS

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Abstract - Primary task of the mass calibration laboratory is dissemination of mass unit to its multiples and submultiplies. Due to the buyoancy force the volume of the mass standards have to be known prior to the measurements. The method presented in this paper allows determination of mass and volume separately. Using right devices it is possible to perform all measurements in one experiment.

Keywords: Mass calibration, volume calibration, vacuum mass measurement

1. INTRODUCTION

Primary task of the mass calibration laboratory which operates on national level is to maintain the mass unit and the mass scale of the country. This is done by periodical mass calibrations of the national primary mass standard and using its value to dissemination of the mass unit to the multiplies and submultiplies. The national mass standard used in Czech Metrology Institute is Platinum-Iridium prototype and is calibrated every 10 years at BIPM.

Calibration to submultiplies is performed with stainless steel mass standard of nominal mass $1\,\mathrm{kg}$ which is directly traceable to the national mass standard. It is often expected that the density of the weights in the same set is equal which results in neglecting the buyoancy effect. The volume of the weights is determined by another comparison in distilled water or other liquid.

The method proposed by authors in this paper uses the apparent mass determination under different conditions which can be obtained in vacuum mass comparator such as high vacuum or ambient air conditions. The method is applicable to any other environment or any other device. The advantage of using the vacuum mass comparator is in less handling with the weights for higher number of results.

Authors expect the method leads to lower uncertainties of the primary mass weight set and more reliable and precise results of the calibrations for the customers of the laboratory. The method will be used as a primary procedure after redefinition of the kilogram.

2. DISSEMINATION OF THE UNIT OF MASS

The mass of the weight is determined according to the fundamental weighing equation

$$m_T = m_R + \rho_a \left(V_T - V_R\right) + \Delta U \left(1 - \frac{\rho_{aj}}{\rho_j}\right) \qquad (1)$$

where m_T and V_T are mass and volume of the test weight, m_R and V_R mass and volume of the reference weight, ρ_a density of air or other environment, ρ_{aj} density of air at the adjustment of the weighing range of the comparator and ρ_j density of the weight used for the adjustment. The adjustment term is ommited in the following equations to reduce neccesary space.

2.1. Air density evaluation

Important part of the buyoancy correction factor is air density. This can be determined using CIPM formula or by set of special artefacts with known volume difference.

CIPM formula

Air density can be calulated using pressure, temperature, tempreature of the dew point and CO_2 content. The equation is based on the equation of state of a non-ideal gas

$$\rho_a = \frac{pM}{ZRT} \qquad (2)$$

where p is air pressure, M molar mass of moist air, Z coefficient of compressibility, R molar gas constant and T thermodynamic temperature. Details on determination of unknown quantities can be found in 1.

Air buyoancy artefacts

If we measure mass difference in vacuum where we expect density of $0\,\mathrm{kg/m^3}$ and air of density ρ_a we obtain set of two equations

$$m_1 - m_2 = \Delta V \rho_a + \Delta U_a, \qquad (3)$$
$$m_1 - m_2 = \Delta U_v. \qquad (4)$$

This leads to equation

$$\rho_a = \frac{\Delta U_v - \Delta U_a}{\Delta V}. \qquad (5)$$

If we have two artefacts with different volumes and the value of this difference is known we can easily determine the air density using two measurements where one of them is done in vacuum. In general, we can measure density of any environment using this equation.

2.2. Mass and volume determination

The equation 1 applies to any mass measurement in any environment. Using two environments we obtain a set of equations

$$m_T = m_R + \rho_1 (V_T - V_R) + \Delta I_1 \qquad (6)$$

$$m_T = m_R + \rho_2 (V_T - V_R) + \Delta I_2. \qquad (7)$$

We use I_1 and I_2 in these equations for mass differences of the weights to distinguish them from mass differences of buyoancy artefacts U_i.

If we combine those two equations together and use equation 5 for the air density evaluation we receive the result for the volume of the unknown weight

$$V_T = V_R + \frac{\Delta I_2 - \Delta I_1}{\Delta U_2 - \Delta U_1} \Delta V. \qquad (8)$$

In general we need two volume reference values, one of which is absolute, the other one is difference of volumes between two air buyoancy artefacts. The measurement in vacuum is not necessary for the volume calibration.

Using the volume of the unknown weight we obtain for mass

$$m_T = m_R + \frac{\Delta I_1 (\Delta U_2 - \Delta U_v) - \Delta I_2 (\Delta U_1 - \Delta U_v)}{\Delta U_2 - \Delta U_1}. \qquad (9)$$

The volume of reference nor test weight is not needed for the mass determination if we use buyoancy artefacts. Also density of the environment is not needed. On the other hand we have to determine mass difference of the artefacts in vacuum or we can use value determined in other measurement.

Important result of the equations for mass and volume of the unknown weight is that we need mass reference for mass measurements and volume reference for the volume determination. The volume of the mass standard or mass of the volume standard are not needed. Therefore we can use national mass standard such as Pt-Ir prototype and volume standard such as silicon sphere.

2.3. Mass and volume calibration

The masses and volumes of the unknown weights are calculated using explicit multivariete measurement model as described in 4. In our case we have a model $\mathbf{X} = \mathbf{f}(\mathbf{W})$ with $\mathbf{W} = (m_R, \Delta I_1, \Delta I_2, \Delta U_1, \Delta U_2, \Delta U_v)^T$ or $\mathbf{W} = (V_R, \Delta I_1, \Delta I_2, \Delta U_1, \Delta U_2, \Delta V)^T$.

The mass and volume are both determined using the same general equation

$$\mathbf{y} = \mathbf{U_y} \mathbf{A}^T \mathbf{U_x}^{-1} \mathbf{x} \qquad (10)$$

where $\mathbf{A}$ is matrix representing used system of equations, $\mathbf{U_x}$ is covariance matrix associated with $\mathbf{x}$ and $\mathbf{U_y}$ is covariance matrix associated with $\mathbf{y}$.

For each environment we measure mass differences for each possible equation. If we have 3 different environments and 3 cycles in each of them we have in total 9 results for each combination. Both equations 8 and 9 require two mass differences so in total we have 36 possible combinations of two environments. We take weighed mean of all these results as an input vector $\mathbf{x}$.

3. LABORATORY EQUIPMENT

3.1. Mass comparator

The mass is determined in vacuum mass comparator Mettler Toledo M-One. The maximum capacity is $1\,\mathrm{kg}$ and weighing range $2\,\mathrm{g}$. Resolution of the comparator is $10^{-1}\,\mu\mathrm{g}$. The exchange mechanism has 6 positions for weights or other object. It is possible to place silicon spheres with diameter $94\,\mathrm{mm}$ on each position.

The minimum pressure achieved inside main chamber is $10^{-4}\,\mathrm{Pa}$ which is considered as absolute vacuum for purposes of mass measurements. It is possible to fill the main chamber with different gases.

The load-lock system is connected to the main chamber for easier exchange of the weights inside the chamber mainly in experiments where long term vacuum stability is necessary. The load-lock is equipped with independent vacuum pumping system.

3.2. Weights

Laboratory used several stainless steel weights of different producers with nominal mass of $1\,\mathrm{kg}$ and classess E_1 and E_2.

The buyoancy artefacts used for air density determination have volume difference $211,462\,\mathrm{cm}^3$. Mass difference in vacuum is $248,682\,\mathrm{mg}$. Both artefacts are made of stainless steel which is used for weights of class E1.

The reference stainless steel weight is E1 class weight directly traceable to national prototype of kilogram. Its mass is $1\,\mathrm{kg} + 0,66\,\mathrm{mg} \pm 0,05\,\mathrm{mg}$ and volume $125,435\,\mathrm{cm}^3 \pm 0,001\,\mathrm{cm}^3$.

The volume reference used in the laboratory is the silicon sphere of nominal mass $1\,\mathrm{kg}$ and diameter of $93,62\,\mathrm{mm}$. Therefore the volume is $429,64\,\mathrm{cm}^3 \pm 0,005\,\mathrm{cm}^3$.

4. CALIBRATION PROCEDURE

The first measurement is performed in ambient air conditions after $24\,\mathrm{h}$ for stabilization of conditions. Each weight is compared to other with exception to air density artefacts which are compared only to themselves. After first air measurement the vacuum chamber is evacuated to approximately 50 % of the ambient pressure. The next measurement starts after $24\,\mathrm{h}$ of stabilization under vacuum

Table 1. Comparison of air density determination

	1	2	3
p[mbar]	973,45	981,30	980,00
h	54	50	59
t	20,6	20,5	21,0
ρ_{CIPM}	1,1491	1,1592	1,1545
ΔU	5,76	3,51	4,33
$\rho_{artefacts}$	1,1489	1,1595	1,1556

conditions. The last ambient air measurement is started after $48\,\mathrm{h}$ of stabilization on ambient pressure.

In some cycles the step with the high vacuum is used between the lower pressure and second ambient measurement.

The calibration then consists of determining the mass differences of every combination of the weigths. Between every combination the air buyoancy artefacts mass difference is measured. For every mass difference the air buyoancy correction can be calculated as the average of the two artefacts measurements before and after respective calibration step. If we measure with 4 weights and 2 artefacts we have 13 mass differences in total where 6 of them are for the weights under calibration and 7 are used for air buyoancy correction.

Each mass difference is determined as an average of 3 ABBA differences. The whole cycle is repeated 3 times for every atmosphere inside the weighing chamber.

5. RESULTS

5.1. Air density evaluation

The first series of measurements are used to verify the results of the air buyoancy artefacts used for the mass calibrations. The results were obtained only in ambient air conditions where the CIPM formula is valid.

The air buyoancy artefacts used in this validation have known volume difference of $211,462\,\mathrm{cm}^3$. Pressure was measured by Fluke RPM4 pressure monitor with readibility of $0,1\,\mathrm{Pa}$ and uncertainty $1,5\,\mathrm{Pa}$. Temperature was measured with Hart Scientific TS8504 with readibility $0,0001\,^\circ\mathrm{C}$ and uncertainty $0,01\,^\circ\mathrm{C}$. Temperature of dew point was measured with Michell Instrument S8000. Its readibility is $0,01\,^\circ\mathrm{C}$ and uncertainty $0,15\,^\circ\mathrm{C}$.

The results for three different air conditions are shown in Table 1. We can see that the air density determined by air buyoancy artefacts is close to the results of the CIPM formula. The difference in the third measurement is caused by instability of the air condition system at the time of measurement.

5.2. Mass and volume calibration with stainless steel mass standard

The first measurements uses one reference weight for both mass and volume calibration as usual in the standard calibration procedures. All weights are made of stainless steel.

Table 2. Calibration of stainless steel weights in different environments

ABE	ABD	E2	m [kg]	V [cm^3]
1	0	0	1,000003196	127,2189
0	1	0	1,000003138	127,2081
0	0	1	1,000000845	125,5781
-1	1	0	$-5,79 \times 10^{-8}$	-0,0106
-1	0	1	$-2,35 \times 10^{-6}$	-1,6399
0	-1	1	$-2,29 \times 10^{-6}$	-1,6299

Table 3. Results of calibration of the stainless steel weights

Weight	m [kg]	V [cm^3]
ABE	$1\,\mathrm{kg} + 3,196\,\mathrm{mg}$	$127,219\,\mathrm{cm}^3$
ABD	$1\,\mathrm{kg} + 3,138\,\mathrm{mg}$	$127,208\,\mathrm{cm}^3$
E2	$1\,\mathrm{kg} + 0,845\,\mathrm{mg}$	$125,578\,\mathrm{cm}^3$

The first comparison was made in ambient conditions. The second one was performed in lower pressure at the density of approximately $35\,\%$. The last measurement was at minimum pressure achievable in the vacuum mass comparator which was $2 \times 10^{-4}\,\mathrm{Pa}$. We assume that the air density is $0\,\mathrm{kg/m}^3$.

In each environment we repeated full comparison 3 times. For each equation we have 9 mass indication differences. The input for the matrix was calculated from all possible combinations even from the same environment as the weighed arithmetic mean. The weights used for the mean are based on standard deviations of mass indication differences.

The calibration scheme and respective results for mass and volume are given in Table 2.

Masses and volumes of the unknown weights are shown in Table 3.

The difference in the volumes between two stainless steel weights is caused by different types of the steel from two different producers. We selected ABD and E2 for other measurements with silicon sphere to check reproducibility of the results. The volumes of ABD and ABE were measured in volume mass comparator Mettler Toledo VC1005 with the same results.

Uncertainty evaluation

We have standard deviation of each measurement. Due to the usage of very stable conditions inside the main chamber of the vacuum mass comparator the standard deviations are much smaller than the uncertainty of the standard weight used for measurements. Type A uncertainty of the first equation is $0,032\,\mu\mathrm{g}$ or $0,056\,\mathrm{mm}^3$.

Table 4. Calibration of mass, Stainless steel reference

Si	ABD	E2	m [kg]	u_A [mg]
1	0	0	1,000079949	7,5
0	1	0	1,000003128	1,8
0	0	1	1,000000850	1,8
-1	1	0	$-7,68 \times 10^{-5}$	7,2
-1	0	1	$-7,91 \times 10^{-5}$	7,8
0	-1	1	$-2,28 \times 10^{-6}$	2,4

Table 5. Calibration of volume, Silicon sphere reference

51701	ABD	E2	V [cm^3]	u_A [cm^3]
1	0	0	125,438	0,010
0	1	0	127,206	0,009
0	0	1	125,583	0,009
-1	1	0	1,769	0,002
-1	0	1	0,146	0,002
0	-1	1	-1,622	0,003

We can say that in the case of calibration of the weights of same nominal massess of $1\,\mathrm{kg}$ the uncertainty of all weigths is almost the same as for the standard weight. The situation might change if we use standard weight of different material such as national prototype of kilogram made of platinum-iridium.

5.3. Volume calibration with silicon sphere

The second measurements uses stainless steel weight as mass standard and silicon sphere as volume standard. With these measurements we check the possibility to have different standards for mass and volume and use both in one experiment.

The calibration procedure was same as in the previous case with one major difference. In this measurement we did not measure the mass differences at the minimum pressure. Since we know the mass difference of the air buyoancy artefacts from the previous measurements we did not have to measure it again.

The calibration schemes for mass and volume are given in Tables 4 and 5.

The large standard deviation is caused by the large mass difference between respective weigths.

The results for the mass and volumes for all the weights are presented in Tables 6 and 7.

We can see that both mass and volume measurements are in good equivalence with the previous method where we used the same reference for both mass and volume evaulation.

Uncertainty evaluation

The largest source of uncertainty in bioth cases is the uncertainty of the reference. We expect to lower these values with national mass prototype of platinum-iridium and with better calibration of the volume of the silicon sphere.

Table 6. Results of mass calibration with stainless steel reference

Weight	m [kg]	u_m [mg]
Si	$1\,\mathrm{kg} + 79,949\,\mathrm{mg}$	0,051
ABD	$1\,\mathrm{kg} + 3,127\,\mathrm{mg}$	0,051
E2	$1\,\mathrm{kg} + 0,850\,\mathrm{mg}$	0,051

Table 7. Results of volume calibration with silicon sphere reference

Weight	V [cm^3]	u_V [cm^3]
51701	125,4376	0,0054
ABD	127,2063	0,0054
E2	125,5837	0,0054

6. CONCLUSIONS

The method proposed by the authors introduces measurement of both mass and volume in one time and under vacuum conditions. The calibration scheme is used for the first time under vacuum in Czech Metrology Institute. Authors expect that the values of mass and volume and respective uncertainties will allow improvement of the CMC tables in KCDB database of CIPM.

The advantage of the method presented is in independent evaluation of mass and volume using different standards. We can use national prototype of the kilogram for the mass calibration without precise knowledge of its volume and at the same time silicon sphere for the volume calibration.

The study of the process will continue in the following months. The authors expect to have more results that will prove the advantage of the method and better uncertainties especially after introduction of the national mass prototype of the Czech Republic.

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