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Nanoscale mass measurement

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Abstract
The paper addresses issue of the calibration of small masses, 2 mg and less. Since there are no traceable mass references for the masses below 1 mg, the proposed solution of the comparator is novel and unique. The main challenge for such small masses was to reduce sorption-induced variation of mass of the elements involved into the measurement. Application of aluminium alloy PA13 and special coil protection enabled reducing the abovementioned mass variation from 0.3586 mg down to 0.0565 mg, which made it reasonable to increase readability up to 10 ng. Further reduction of the measurement errors was obtained through application of advanced-type Sm–Co magnet in the actuator design. Remnant magnetization of this material reveals exceptional thermal stability (the relative change not greater than 10^{-5} per one Kelvin), which made negligible the error generated by magnetic force variation due to temperature changes. To eliminate appraiser variation of the measurement results, the comparator system was fully automated. As a result, standard uncertainties for the measurements of masses 0.2, 1, and 2 mg were 0.0359, 0.0386, and 0.0373 µg, respectively. Thus, with expanded uncertainty below 100 ng, the proposed solution provided possibility of nanoscale mass measurement.

Keywords: Mass comparator, Mass, Uncertainty, Accuracy

1. Introduction

Readability of te balances available on the market can be as good as 0.1 µg. However, their uncertainty is much higher, since there are no traceable mass references for the masses below 1 mg [1], which makes impossible legalization for a resolution below 1 mg. Moreover, readability of 0.1 µg is insufficient for calibration of 0.05 mg masses.

Since it is impossible to estimate sensitivity parameter of high-resolution comparators without measurement of higher accuracy, the increasing number of researchers are busy to solve the abovementioned problems. For instance, at the Estonian national metrology institute, uncertainty for 1 g is ca. 0.4 µg [2]. There are papers describing the calibration of mass with a nominal of 0.1 mg, and the standard uncertainty was reported 0.075 µg [3]. Works performed at NIST enabled to reach standard uncertainties of 9.0 µg and 6.7 µg for masses of a nominally 5 g and 1 g mass, respectively, in the case of a tabletop Kibbele balance [4]. For the measurement of a 20 g mass performed at PTB, a combined relative uncertainty of 2.5×10^{-6} (k = 1) has been achieved [5]. For the calibration carried out with respect to the international prototype of the kilogram (IPK) in anticipation of the redefinition of the kilogram, the uncertainty in terms of the general least squares fit was estimated as 0.7 µg [6].

In the present paper, results for a novel automatic comparator AK-4 are presented. The device id shown in Figure 1, and its main features were described in [7]. There were two main constructional tasks to be solved. First of all, the measurement errors from the temperature change and subsequent variation of magnetic force had to be reduced, so that the range of temperature variations in the laboratory would have no effect on the work of mass comparator. It is well known that magnetic systems have immence impact on the uncertainty of balances [8]. The advanced-type Sm–Co magnets were chosen because even in the case of standard Sm–Co magnets, the effect of a temperature increase of 100°C on their residual magnetic flux density fis usually below 3% [9]. And secondly, it was important to reduce sorption-induced mass variation, which may have dramatic effect on the mass measurement results [10].

2. Sorption-induced mass variation

The coil mass can be largerly by humidity dynamics, because a coil has large sorption surface. The problem can not be definitely solved solely by fine polishing of the surfaces, since they always retain small pores able to absorb some humidity. The initial researches indicated that in a mass comparator, the coil prevents from the measurement accuracy increase due to its sorption-induced mass variation. Re-design of the coil was proposed with special seal, and it was tested after stabilization in the air for 24 hours at a temperature of 25°C and humidity of 50%. Then, the mass m_1 was measured and air humidity was increased up to 70%, but the temperature remained 25°C. After stabilization in new conditions for 23 hours, the second measurement was made, denoted m_2. The procedure was repeated 10 times.

The differences $\Delta m_i = m_{1i} - m_{2i}$ (for i=1 to 10) demonstrate the degree of sorption-induced mass variation for the novel coil applied in AK-4 device and the standard one. From the histograms of the obtained of Δm_i values presented in Figure 1, it can be seen that not only average Δm was reduced by 85%, but also the standard deviation of the results decreased in the similar degree from 0.02709 mg down to 0.00672 mg.

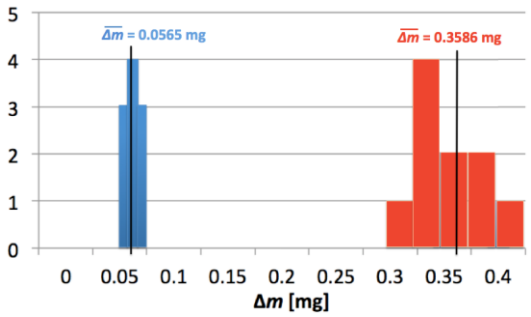

Figure 1. Results of sorption-induced mass variation measurement (red histogram corresponds with a standard coil, blue one with a novel protected coil design)

3. Type A uncertainty

To keep repeatability conditions, the Type A uncertainty check included 20 repetitions of full measurement cycles. Each cycle consisted of six ABBA sequences, where each sequence measured a difference between the mass of a test mass (B) and a reference mass standard (A) [11]. The Shapiro–Wilk statistical test confirmed normal distribution, and standard deviations s_d of each repetition were analysed. In Figure 2, there are results for the reference mass of 0.2 mg. Figure 3 contains the respective histograms of the results obtained by AK-4 novel mass comparator and the ones from UMA-5 device with the best achievable readability of 0.1 µg.

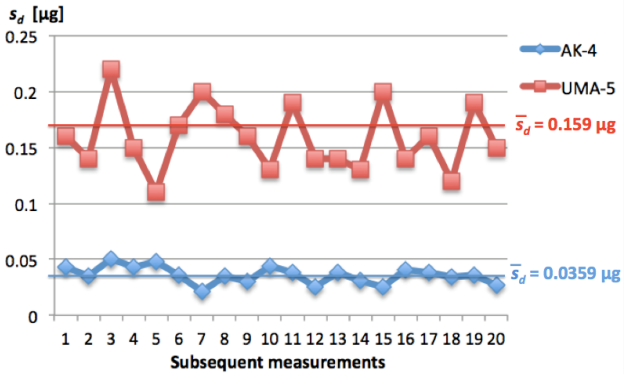

Figure 2. Comparison of the measurement results for the novel AK-4 mass comparator with readability of 0.01 µg and UMA-5 with readability of 0.1 µg

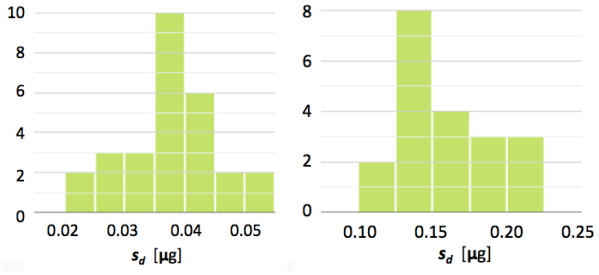

Figure 3. Histograms of the results obtained from 20 repetitions with AK-4 novel mass comparator (left) and UMA-5 mass comparator (right)

Standard uncertainty for AK-4 measurement of the mass 0.2 mg was $u(x)$ = 0.0359 µg. Compared with the value of $u(x)$ = 0.159 µg obtained for the best available device UMA-5, it is by 77% smaller. These results demonstrated that not only resolution of 10 ng is possible to be achieved, but also the standard uncertainty $u(x)$ = 35.9 ng reasonably corresponds with readability of the novel mass comparator shown in Figure 4.

Figure 4. View of the novel AK-4 mass comparator with readability of 10 ng

4. Conclusions

The results of mass measurement with the novel AK-4 mass comparator of readability 10 ng, compared to the most accurate available comparator UMA-5, demonstrated substantially better performance. Improvements of actuator material, less sensitive to the temperature variations, and redesigned coil minimized the effects of ambient conditions on the measurement results. In particular, sorptio-induced mass variation Δm for the new coil protected with a special seal, was reduced by 85%.

The standard uncertainty of mass measurement was reduced, too. The novel design AK-4 measured the mass of 0.2 mg with the expanded uncertainty U_{95} = 0.070 µg (level of confidecne p=95%, k=1.96), which was by 77% smaller than that of UMA-5. The experiments proved that the proposed desigh ensured nanoscale mass measurement, unreacheble before.

References

[1] Hu MH, Wang J, Zhang Y, Cai CQ, Zhong RL, Yao H and Ding JA 2012 Research on micro-gram weights standards below 1 mg in NIM, XX IMEKO World Congress, September, 2012, Busan, South Korea.

[2] Vabson V, Kübarsepp T, Vendt R and Noorma M 2010 *Measurement* **43** 1127-33.

[3] Ota Y, Ueki M and Kuramoto N 2021 *Measurement* **172** 108841.

[4] Chao L, Seifert F, Haddad D, Pratt J, Newell D and Schlamminger S 2020 *Metrologia* **57** 035014

[5] Vasilyan S, Rogge N, Rothleitner Ch, Lin Sh, Poroskun I, Knopf D, Härtig F and Fröhlich Th 2021 *tm – Technisches Messen* **88(12)**, 731–756

[6] Stock M, Barat P, Davis RS, Picard A and Milton MJT 2015 *Metrologia* **52** 310

[7] Solecki M, Szumiata T and Rucki M 2021 *Precision Engineering* **72**, 576-582.

[8] Krishnan R 2010 *Permanent Magnet Synchronous and Brushless DC Motor Drives*. Boca Raton: CRC Press.

[9] Jones FE and Schoonover RM 2002 *Handbook of Mass Measurement*. Boca Raton: CRC Press.

[10] Li S and Schlamminger S 2022 *Metrologia* **59** 022001

[11] Szumiata T and Hantz A 2017 Optimization of mass standards and weights calibration (1 mg – 1 kg) using rotational automatic mass comparator, Proceedings of IMEKO 23rd TC3, 13th TC5 and 4th TC22 International Conference, June, 2017, Helsinki, Finland.