New design of the automatic mass comparator with readability of 10 ng

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Measuring smaller and smaller amounts in a micro and nano scale not only requires extremely precise measuring instruments but also suitable methods of periodical weighing control. Mass standards below 1 mg can prove helpful but methods of determining their mass are crucial. Using balances or mass comparators with readability of 0.1 ug during manufacture of such standards may turn out to be insufficient, mainly in view of uncertainty of the sample mass determination. Bearing the above in mind, Radwag has developed a new design of the mass nano-comparator, NANO.AK-4.500.5Y series, with readability of d=0.01μg (10 nanograms). This publication aims to present metrological aspects of the nano-mass comparator, which may be the first step for its commercial use in scientific and research laboratories or the ones dealing with scientific and legal metrology.

1. INTRODUCTION

Metrology and weighing structures with regard to mass measurements are presently highly developed as microbalances that prove reliable in determining masses of various items with readability of 20-50 million serve as standard devices, e.g. a MYA 21.5Y microbalance. From the metrological point of view, precise determination of masses of 0.5 microgram is only part of the success as the mass measurement precision is also determined by the balance systematic error that arises from potential changes of its sensitivity. In fact the so-called internal adjustment is now a requirement, yet verification of its correct operation should be based on the external sample mass. The sample mass as a point of reference for numerous metrological inspections is defined through real mass value and determination uncertainty, while main elements of the uncertainty budget are mass comparator readability, comparator repeatability and reference weight uncertainty. The conclusion is that to manufacture 'more precise' mass standards is to use 'more precise' measuring instruments – balances, mass comparators.

Figure 1. Resolution of the UYA 2.5Y ultra-microbalance in the field of legal metrology

Similar dependencies can also be found in the legal metrology. The OIML R111-1 standard specifies the maximum permissible errors for the so-called light weights (table 1), giving the maximum extended uncertainty of determining masses of each weight (point 5.2 of OIML R 111-1). With regard to weights with a mass of 20 mg ÷ 1 mg, the maximum error for determining their mass is ± 0.003 mg, with uncertainty of 0.001 mg. While inspecting the mass of such weights, it is possible to use ultra-microbalances or mass comparators with readability of d=0.1µg, yet their repeatability is 0.15 ÷ 0.5µg, depending on the type, which may be up to ca. 50% of the uncertainty budget.

Such dependencies result in a real need to use the balance / mass comparator whose readability is smaller and value repeatability higher. With that in mind, Radwag as a leading balance and mass comparator manufacturer was the first to present the new design of the mass nano-comparator, with a maximum load of 500 mg. We can therefore hope that technological and metrological progress in terms of measuring small values will apply to the legal metrology too.

Table 1. Maximum permissible errors for weights (± δm in mg)

Nominal mass (mg)	Precision class / maximum permissible error (± mg)			
	E1	E2	F1	F2
1000	0,01	0,03	0,1	0,30
500	0,008	0,025	0,08	0,25
200	0,006	0,02	0,06	0,20
100	0,005	0,016	0,05	0,16
50	0,004	0,012	0,04	0,12
20	0,003	0,01	0,03	0,10
10	0,003	0,008	0,025	0,08
5	0,003	0,006	0,020	0,06
2	0,003	0,006	0,020	0,06
1	0,003	0,006	0,020	0,06

Please remember however that precision of each measurement is strictly dependent upon testing conditions (temperature, humidity, vibrations) and stability of the balance / comparator (Figure 2).
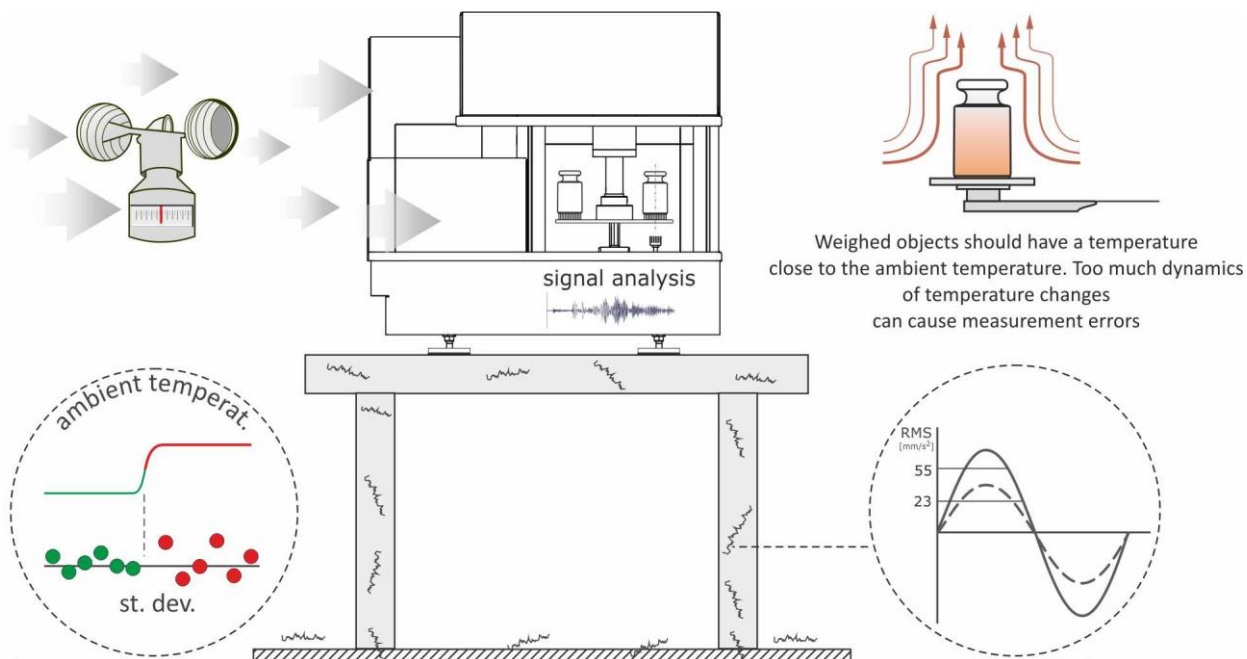

Figure 2. NANO.AK-4.500.5Y – mass-measurement-influencing factors

2. Design of the NANO.AK-4.500.5Y comparator

One of the elements that assure proper operation of the mass comparator is stability of the environment, mainly humidity and temperature. Variability of these two factors is defined in OIML R 111-1 (table 2) and applies to changes that may occur in the item in question, that is weight - thermal expansion, moisture sorption effects, etc.

Table 2. Ambient temperature while calibrating weights (recommended values)

Weight class	Temperature changes during calibration		Humidity changes during calibration	
	In 1 hour	In 12 hours	Humidity range	Max. / 4 hours
E1	± 0,3 ºC	± 0,5 ºC	from 40 % to 60 %	± 5 %
E2	± 0,7 ºC	± 1,0 ºC		± 10 %
F1	± 1,5 ºC	± 2,0 ºC		± 15 %
F2	± 2,0 ºC	± 3,5 ºC		
M1	± 3,0 ºC	± 5,0 ºC	x	x

It must be noted that variability of humidity and temperature has a negative impact on the mass comparator too. If the ambient temperature changes too rapidly, it results in linear changes to the mechanical system of the comparator while dynamic changes of humidity affects stability of values, mainly in effect of sorption ad desorption of moisture by mass comparator structural pieces. The result of these factors is a way worse precision of measurements. The design of the AK-4/500.5Y nano-comparator has been showed in the figure 3.
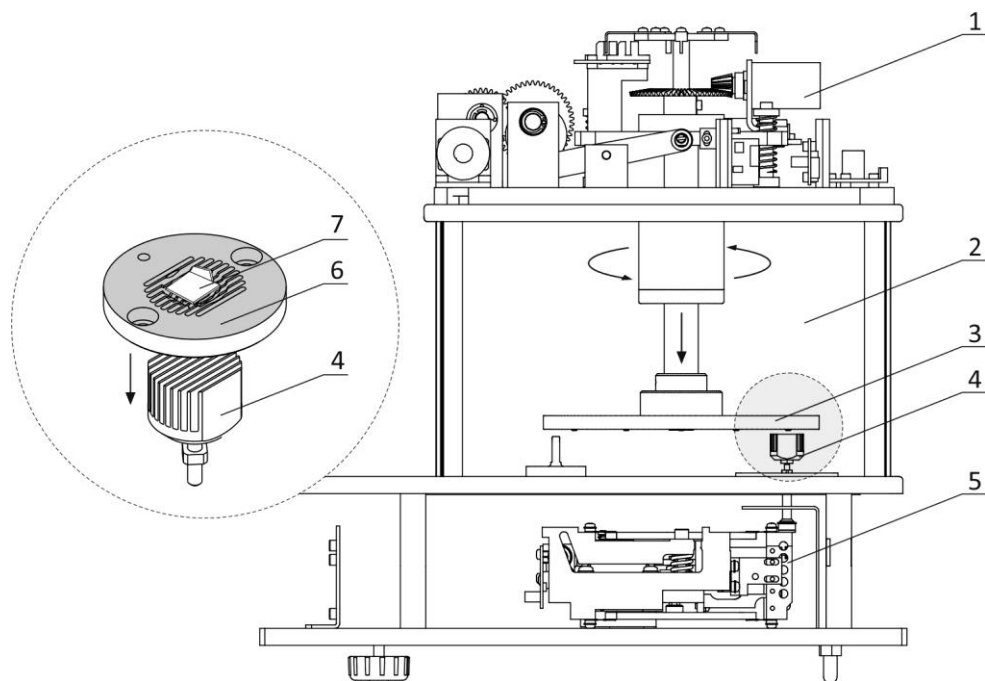

Figure 3. NANO.AK-4.500.5Y comparator sketch
Key: 1- control system, 2- weighing chamber, 3- mass standard magazine, 4- comparator weighing pan, 5-high-resolution electromagnetic measuring unit, 6-weight seat, 7-weight

The mass standard magazine (3) is controlled by control system (1), installed in the top part of the mass comparator. After placing weights/mass standards in the magazine (3), the comparison procedure starts. You can freely define the procedure with regard to the following:

- ABA, ABBA comparison method
- number of repetitions in the ABA, ABBA, e.g. 6xABBA cycle
- number of comparison cycle repetitions, e.g. 10x(6xABBA)
- comparison start time
- selection of compared weights
- other comparison-related parameters

Inside the mass standard magazine (3) are four openwork seats (6) intended for weights (7) in question. Using the operator's panel, you must define the comparison plan based on information in the database. The weighing chamber (2) of the mass comparator must be closed while testing for minimisation of negative impact of uncontrolled movement of air. As mentioned before, comparison requires very stable working conditions but the goal is to optimise the comparator design so that it can guarantee maximum stability of values, irrespective of outside factors. Once comparison is over, all information related to the test is automatically saved in the database. This information can be remotely copied via WiFi, Ethernet or transferred into an external USB memory. See below for the example of the ABBA cycle report.

```
-------------- Comparator --------------
User                        Admin
First and last name         SJ
Report no.                  C/01/11/22/14/48
Start date                  2023.04.20 14:48:49
End date                    2023.04.20 15:42:57
Test weight                 50mg
Order number                A-654
Test weight number          x
Test weight position        B2
Reference weight            500mg
Mass                        50.000012 g
Reference weight class      E1
Reference weight position   A1
```

n	|A	|B	|B	|A	D
1	| -0.00000180	| 0.00001390	| 0.00001380	| -0.00000180	|0.00001565
2	|-0.00000180	|0.00001400	| 0.00001400	| -0.00000160	|0.0000157
3	| -0.00000160	|0.00001400	| 0.00001400	| -0.00000160	|0.0000156
4	|-0.00000170	|0.00001400	| 0.00001410	| -0.00000160	|0.0000157
5	| -0.00000160	|0.00001420	| 0.00001430	| -0.00000130	|0.0000157
6	| -0.00000120	|0.00001450	| 0.00001450	| -0.00000110	|0.00001565

```
Mean difference        0.000015666667 g
Standard deviation     0.000000040825 g
Number of cycles       6

Method                 ABBA
Min. temperature       22.13°C
```

Max. temperature	22.31°C
Min. humidity	44.8%
Max. humidity	45.9%
Min. pressure	998.2hPa
Max. pressure	998.2hPa

Figure 4. Recording and visualising environmental parameters during comparison (22.04.2023)

One of the key design elements of the mass comparator is an electromagnetic actuator with a coil. In view of its design and location in the mechanical system, the coil is highly susceptible to changes of the relative humidity. Bearing that in mind, the NANO.AK-4.500.5Y comparator is supplied with an innovative coil (Figure 5) and a cover so that sorption and desorption of moisture can be substantially limited. Coil mass stability tests performed in humid conditions have been showed further in the work.
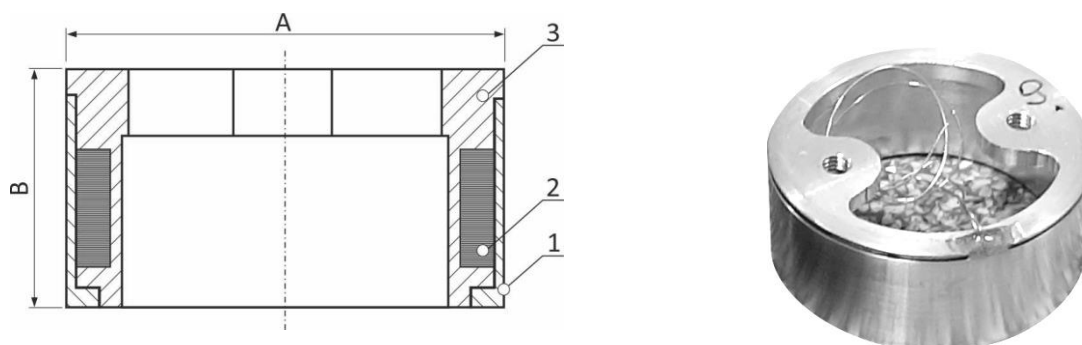

Figure 5. View and design of the coil
1- protective ring; 2 – coils; 3 – coil housing
(Coil S10: A=16.5 mm, B=9 mm, S20: A=30 mm, B=12 mm)

2.2. Actuator mechanical design

The exact composition of the material used to produce the actuator magnet remains the manufacturer's secret. RADWAG has tested numerous magnets and the one used in the NANO.AK-

4.500.5Y comparator meets magnetic field stability requirements in the face of variable temperature. Why is it so important?

If the magnet force changes dramatically while the temperature is growing or dropping, it will change the result of the mass of the item tested on the balance or comparator.

3. DESIGN OPTIMISATION – MOISTURE SORPTION TESTING

Changes to the coil mass as a result of changes to relative humidity were tested for coils used in typical laboratory balances in comparison to the coil used in the automatic NANO.AK-4.500.5Y nano-comparator. In stable temperature and humidity conditions, the coil mass was specified and then the relative humidity was increased by 20%. In increased humidity conditions, items in question were conditioned for over 24 hours. Based on the coil mass measurements after conditioning and before humidity rise, the increase in mass was calculated to measure the susceptibility of the coil to moisture absorption. Test results have been showed in the table 3.

Table 3. Mass change result from various coils, depending on protection

Coil type	Mass change during testing (mg/20%)		
	S20	S10	S10 AK-4/500.5Y
No. / protection system	ring	None	ring
1.	0.15	0.346	0.064
2.	0.16	0.346	0.057
3.	0.16	0.399	0.061
4.	0.16	0.390	0.055
5.	0.15	0.312	0.052
6.	0.15	0.331	0.048
7.	0.15	0.374	0.060
8.	0.15	0.378	0.045
9.	0.14	0.362	0.066
10.	0.12	0.348	0.057
$\acute{x}$	0.15	0.36	0.06
S	0.012	0.027	0.007

The data received clearly show that the best mass stabilisation applied to the S10 coil when it was secured with an additional sealing ring. Thanks to this, the impact of the variable humidity was about 6 times smaller when compared to the same coil without protection.

4. METROLOGICAL TESTING OF THE NANO.AK-4.500.5Y COMPARATOR

The most important metrological parameter of every mass comparator is the measurement precision which through the standard deviation shows the degree of measurement dispersion around

the mean value. The lower the standard deviation value, the higher the precision. Precision was assessed for all mass standards/weights that could be compared.

4.1. TEST METHOD

The test procedure was concerned with determination of standard deviation for 6 ABBA cycles, repeated five times. Every reference weight and every weight were weighed 60 times for objective assessment of stability and precision of comparator mass measurement with various loads. The mean difference r_i for ABBA method for every x series of measurements was determined on the basis of the equation (1) and (2). Based on differences, the standard deviation for differences was determined in accordance with the equation (3).

$$\acute{r} = \frac{1}{n} \times \sum_{i-1}^{n} r_i \tag{1}$$

where: r_i mean difference (B-A) for x measurement
     $\acute{r}$ arithmetic mean of differences for n measurements

$$r_i = \frac{\left[\left(B_i^{AB} - A_i^{AB}\right) + \left(B_i^{BA} - A_i^{BA}\right)\right]}{2} \tag{2}$$

where: $B_i^{AB} - A_i^{AB}$ – mass difference in AB sequence
        $B_i^{BA} - A_i^{BA}$ – mass difference in BA sequence

$$S = \sqrt{\frac{\sum_{i=1}^{n}(r_i - \acute{r})^2}{n-1}} \tag{3}$$

where: n – number of measurements in ABBA method

4.2. MEASURING POSITION

The test was carried out in the Radwag Research Metrology and Certification Centre. The climatic conditions during the test, temperature of ~ 21.3 °, relative humidity of 46 % ÷ 48 %. The dynamics of changes of temperature and humidity was compliant with values specified in the OIML R-111-1 standard for weights in E1 precision class. All testing rooms were equipped with an automatic temperature and humidity adjustment system, recordable online. The laboratory in which measurements were made is situated in the '-1' level. The base of the concrete console with a weight of ca. 3000 kg on which the NANO.AK-4.500.5Y comparator was positioned was separated from foundation of the building. The work stand and general view of the laboratory have been presented in the figure 6.

Figure 6. Testing stand for mass comparators/ general view of the laboratory

4.3. RESULTS

Each load was compared on the basis of the ABBA method in stable outside conditions – variability of humidity 1.20%, of temperature 0.14ºC in 24 hours. The results obtained in the test have been showed in the table 4.
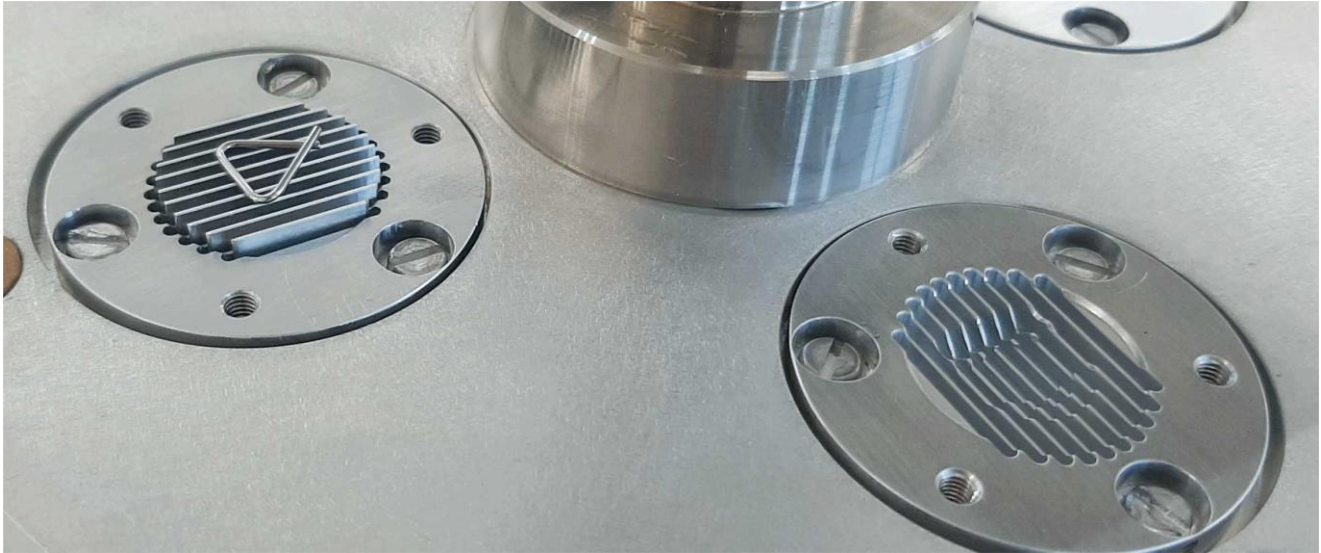

Figure 7. NANO.AK-4.500.5Y – mass standard weighing process

Table 4. Precision of measurements of the NANO.AK-4.500.5Y mass comparator for various test loads.

Nominal sample mass	Standard deviation S $\bar{x}$ (5)	Precision of determination of mean value $\bar{x}$
500	0.06 µg	± 0.02 µg
200	0.06 µg	± 0.02 µg
100	0.04 µg	± 0.03 µg
50	0.04 µg	± 0.01 µg
20	0.07 µg	± 0.03 µg
10	0.04 µg	± 0.01 µg
5	0.06 µg	± 0.02 µg
2	0.05 µg	± 0.02 µg
1	0.05 µg	± 0.01 µg

The NANO-AK-4.500.5Y comparator provides the so-called full-range balancing, that is measures the sample mass in the following range: 0 ÷ 500 mg. Metrological experiences show that precision of measuring low-mass items is determined only by precision (standard deviation). With regard to NANO-AK.4.500.5Y, it was found that precision in the series of measurements was constant with minor variability that was likely to result from environmental factors and partly from mass comparator drifts. Dispersion of standard deviation results for all series of the ABBA method has been showed in the figure 8.
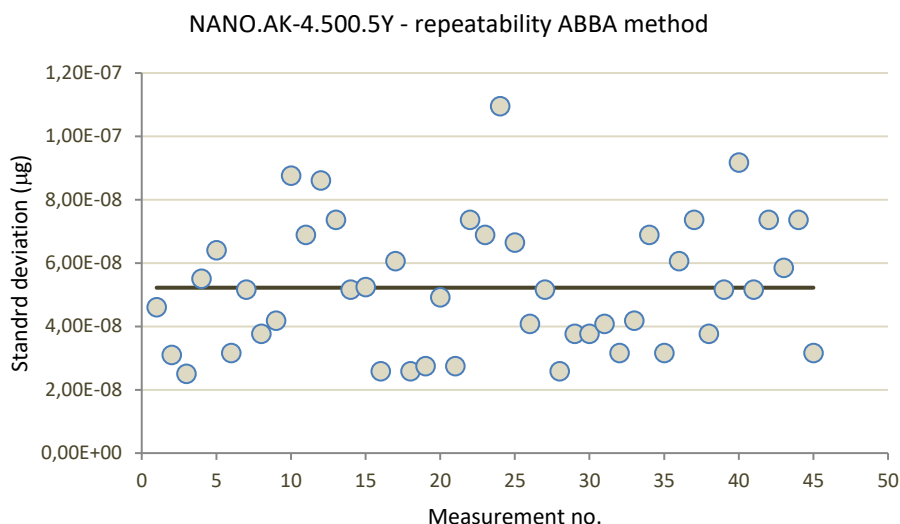
NANO.AK-4.500.5Y - repeatability ABBA method

Figure 8. Dispersion of standard deviation results for all series of the ABBA method

Additionally, for the standard mass of 1 mg, 10 repetitions in 6xABBA method were conducted in order to compare precision of mass measurement provided by the NANO.AK-4.500.5Y comparator with other method adopting the automatic UMA-5 comparator with readability of d=0.1µg. Basic metrological parameters of mass comparators have been showed in the table 5.

Table 5. Basic metrological parameters of UMA / NANO mass comparators

	UMA 5.5Y	NANO.AK-4.500.5Y
Precision class E1	1 mg ÷ 5 g	0.05 mg ÷ 500 mg
Precision class E2	1 mg ÷ 5 g	0.05 mg ÷ 500 mg
Precision class F1	1 mg ÷ 5 g	0.05 mg ÷ 500 mg
Precision class F2	1 mg ÷ 5 g	0.05 mg ÷ 500 mg
Maximum load (Max)	6.1 g	510 mg
Readability (d)	0.1 µg	0.01 µg
Standard repeatability (5% Max)	0.2 µg	0.04 µg
Standard repeatability (Max)	0.4 µg	0.06 µg
Adjustment	internal	external
Balancing range	0 g ÷ 6.1 g	0 g ÷ 510 mg
Weight/mass standard store	36 pcs.	4 pcs.

The results of comparing measurements for ABBA method have been showed in the table 6,7.

Table 6. Results for the NANO.AK-4.500.5Y mass comparator, d=0.01 µg, coil S10.

No.	1	2	3	4	5	6	7	8	9	10	$\bar{S}$
S (µg)	0.046	0.031	0.025	0.055	0.064	0.044	0.039	0.058	0.045	0.056	**0.046**

Table 7. Results for the UMA-5 mass comparator, d=0.1 µg, coil S20

No.	1	2	3	4	5	6	7	8	9	10	$\bar{S}$
S (µg)	0.124	0.098	0.163	0.181	0.013	0.189	0.128	0.144	0.176	0.190	**0.141**
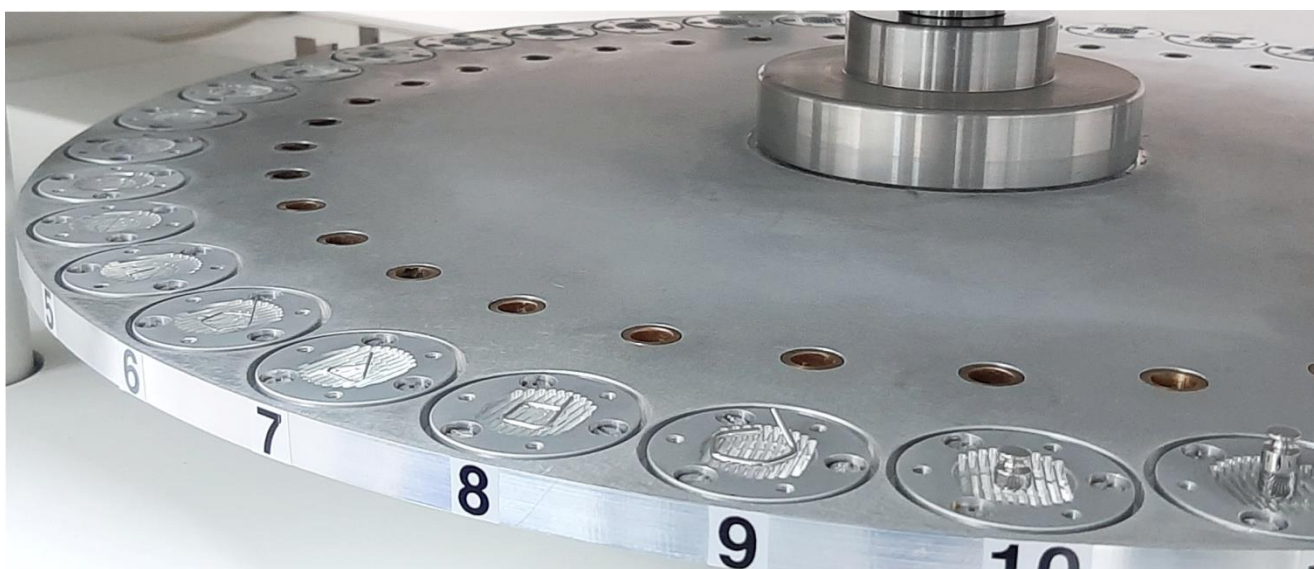
Figure 9. View of the UMA 5.5Y comparator store with mass standards

The standard deviation serving as a measure of precision from the series of measurements is around 4 times smaller for the NANO.AK-4.500.5Y comparator. The precision for both mass comparators was tested at the same time and in the same conditions, in the automatic cycle. Therefore the conclusion is that the only essential factor affecting the comparison result was different mechanical design of the NANO.AK-4.500.5Y comparator (engineering innovation).

The performance features of NANO.AK-4.500.5Y can be considerably improved by using the magazine for a larger number of weights. This will entail larger mechanical design of the device but the mass measurement unit will retain its metrological properties.

5. CONCLUSIONS

The comparison, that is determination of difference between the mass standard and test item requires very high stability of the weighing unit. It can be guaranteed with the NANO.AK-4.500.5Y comparator, mainly in view of its design optimisation. Thanks to this, the mass of the test weight can be determined with lower uncertainty, which may be essential for development of measuring techniques adopted in legal and scientific metrology.

On the other hand comparison is universal and applies to other fields, e.g. automotive industry, too. This being the case the testing is concerned with determination of amounts of solids emitted by combustion engines through differential measurement of the filter mass. It is a common knowledge that exhaust fume emission standards are getting more and more restrictive so the amount of emitted solids is lower. This in turn requires the use of more and more precise measuring equipment, automatic at best (elimination of human error).

LIST OF FIGURES

LIST OF TABLES

6. REFERENCES

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