METROLOGY IN THE LABORATORY

WEIGHING SYSTEMS IN THE SCIENTIFIC RESEARCH AND INDUSTRY

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1. Introduction

The science started growing rapidly as early as in the 1960s and it primarily resulted from automation of numerous physical and chemical processes. Back then, measurements made in milli scale, that is 10^{-3}, proved sufficient to describe phenomena and processes in question. The 1980s witnessed the emergence of measurements in a micro scale, that is 10^{-6}, and presently nano scale, that is 10^{-9}, and more accurately. It is estimated that our environment is home to over 65 million chemical compounds that are likely to grow in quantity in view of the use of artificial intelligence that substantially boosts testing processes. Some of these compounds may be hazardous to us in terms of their toxicity or presence in a nano scale. In all probability we encounter around 100 thousand chemical compounds throughout our lives, most of which of anthropogenic origin. It can be stated that the use of analytical methods suitable for designation of mg/L are no longer adequate to trace- or ultra-trace-based analyses. The definition for these terms is evolving, and what was once considered as traces is now defined as macro. Such an approach entails adoption of new design and IT solutions for measuring instruments, including the ones related to mass measurements. The majority of advanced weighing systems are complicated hybrid mechanical units in which mass measurement is processed by complex computer-electronic measurement tracks. It is a completely new quality, provided by professional high-resolution balances, such as UYA, MYA microbalances by Radwag.

At this point one can ask the following question: is it even possible to operate such advanced measuring instruments if you are unaware of how such a measuring unit works and with no basic knowledge of metrology, gravimetry, thermo-gravimetry, etc.? The answer is yes, you can operate them, but not necessarily understand how they work and how to use them efficiently. Please note that most modern measuring instruments, including weighing systems, are intuitive, which is desired on one hand but may lead to the so-called „black box" effect on the other → you know the input data and analysis result, yet being unaware of how it was obtained. Is therefore the analysis result precise and can it be used further in the process? They say that faith moves mountains. However this saying does not apply to science and metrology.

2. Scientific metrology

The metrology as a scientific study of measurements is one of the significant aspects assuring proper operation of economies. Such fields as trade, taxes, customs, settlements, imposing fees and penalties, are under control of statutory bodies that temporarily supervise all measuring instruments used for the aforesaid operations. Such a solution secures recipients of these services against fraud through clearly defined requirements concerning how measuring devices are to work and what tolerance limits they are assigned with. It also applies to mass measurements that are common in the direct business trade and remain the integral part of testing in the pharmaceutical industry and R&D laboratories.

Historically, development of every civilisation evolving from the ancient times until the present day has been strictly dependent upon measurements. It must be noted that the dynamics related to changes as well as qualitative and quantitative requirements in the contemporary world requires flexibility, also in the field of scientific metrology. This field of science is focused on development and supervision of units of measure, specifying new requirements for the measuring equipment. And it applies to mass measurements too. To illustrate it, let's consider the redefinition of a 1-kg mass standard in the form of a Watt current balance, as the highest in the rank of transferring the unit of measure.

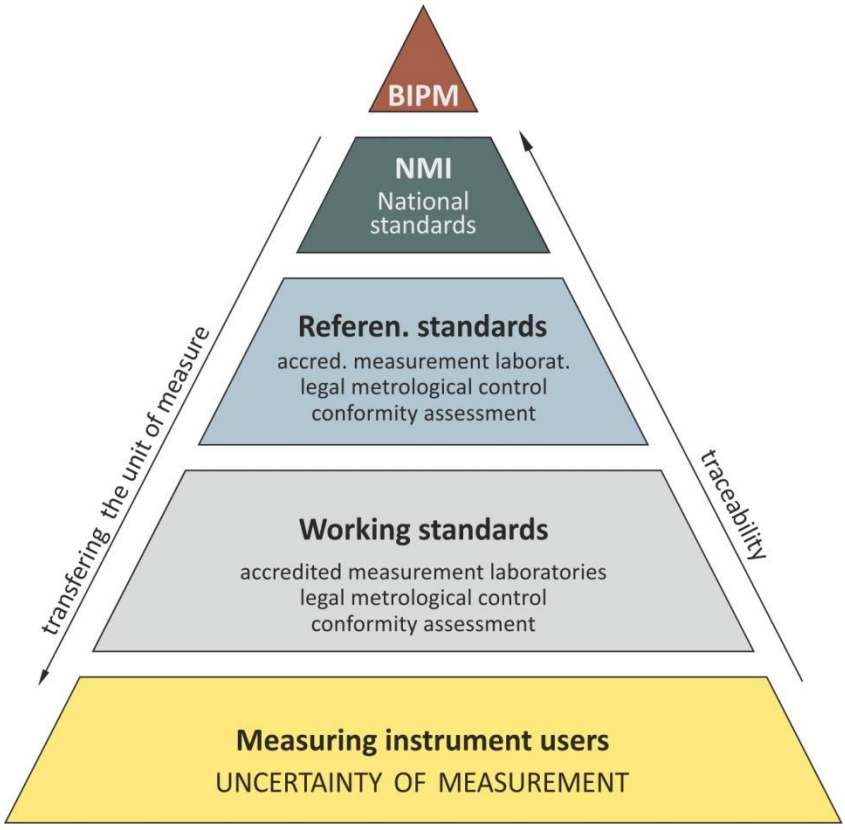

Figure 1. Metrological traceability chain

Mass comparators come as one of the most advanced mass measurement solutions adopted by the scientific metrology. Generally speaking comparison is concerned with comparing two weights, i.e. a reference and test weight, in order to determine the test weight mass. This process entails the use of comparators of various design and weighing range, but mass comparators with the so-called limited balancing range are the most popular. Considering their structure, the weight mass represents the so-called preload of the comparator, the best example is AKM comparators.

Figure 2. AKM comparator 2.20.5Y,d= 0,1 mg

Maximum load 20,5 kg, electric balancing range - 500g to + 500 g
Internal ballast weights, with semi-automatic control, comparison range from 1 kg to 20 kg (depending on accuracy class)

Thanks to mass comparators, it is possible to test both single mass standards and defined sets in the manual or automatic cycle, with permanent pressure or in the vacuum. The most advanced product is AVK 1000.5Y comparator used to compare weights whose mass is 1 kg with a reading unit of 10 billion units (10^{-9} g), which apparently is the upper limit for differential mass measurements. This device is intended for national notified bodies that using engineering solutions adopted in this comparator are able to transfer the unit of measure, simultaneously maintaining a minor measuring uncertainty. The testing cycle can be performed in the vacuum or with permanent pressure. The main metrological parameters of the AVK-1000.5Y comparator are showed in the table 1.

Table 1. Metrological parameters of the AVK-1000.5Y comparator

E1 ÷ F2 weight accuracy class	0,1 kg ÷ 1 kg
Maximum load	1002g
Elementary reading unit	0,1µg
Standard repeatability	0,5µg
Permissible repeatability	1µg
Electric balancing range	-1 g ÷ +2 g
Stabilisation time	60s
Weight/standard magazine	6
Pressure in vacuum chamber	$10(^{-6})$ mbar
Comparable unit dimensions	cylindrical ø (22-95)x110; spherical ø (40-100) mm

It must be emphasised that the quality of comparison depends not only on the resolution of the comparator but also measuring conditions. It often happens that comparison is completely automatic and therefore the so-called human factor does not influence the analysis result. Sadly the stable working environment at ±0.1^{o}C /24 hours is required to obtain acceptable results, which is relatively difficult, even in notified bodies' laboratories. The design of the AVK comparator has been depicted in the figure 3.

Figure 3. AVK 1000.5Y vacuum comparator
Maximum load 1000 g, electric balancing range - 1g to + 2 g
Internal ballast weights, automatic control,

The AVK-1000.5Y comparator is supplied with the so-called limited electric balancing range, which means that its weighing pan is always loaded with the reference or test weight. Therefore, throughout the comparison, it is required to monitor weighing result discrepancies in relation to the starting (zero) position of the comparator. AVK is capable of testing 6 weights at the most, relying on the ABA or ABBA method, as per the so-called programmable comparison plans that define the number of cycles, location of weights in the magazine, their accuracy class, etc. It is possible to control the comparison process remotely/Ethernet/ or using the operator's panel (11). The design of the AVK-1000.5Y comparator can be seen in the figure 4.

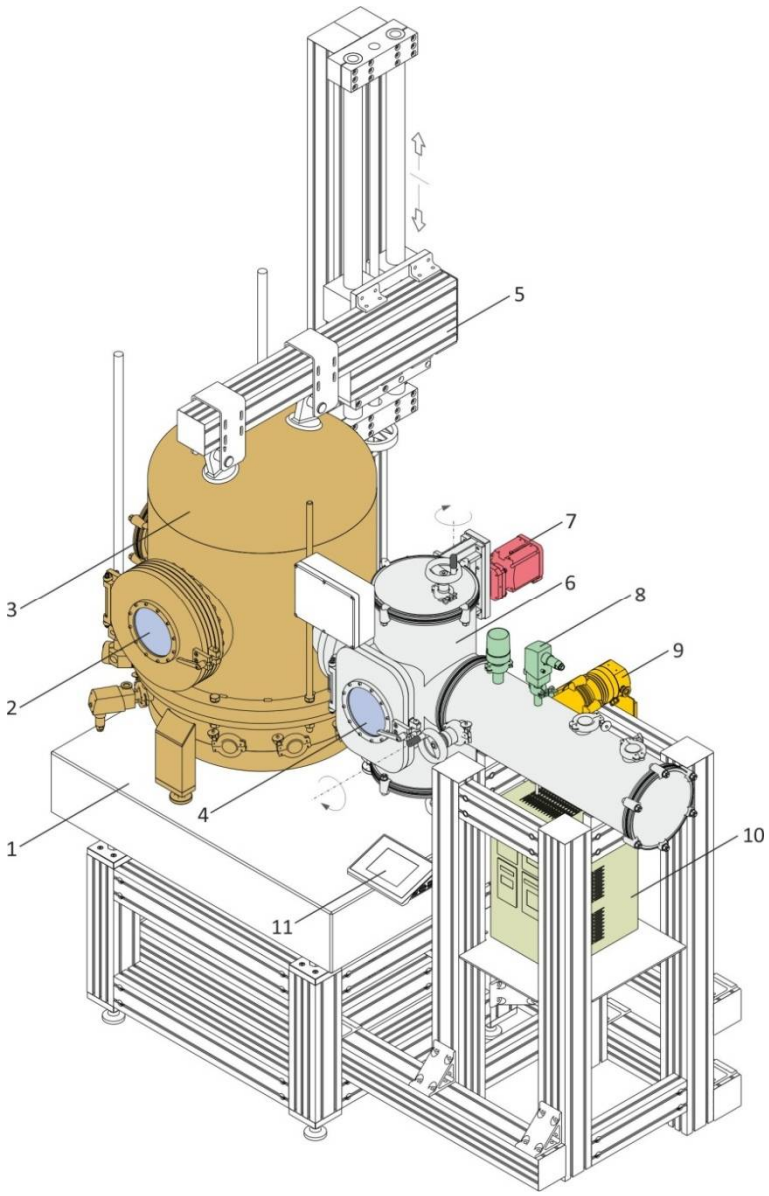

Figure 4. AVK-1000 vacuum comparator design

Key: 1 – weighing table, 2 – inspection window, 3 – comparator, 4 – Load Lock inspection window, 5 – maintenance lift, 6 – Load Lock, 7 – cut-off valve, 8 – sensors, 9 – molecular pump, 10 – control unit, 11 – operator's panel

It is no secret that the so-called „good handling", understood as a good weighing practice, is restricted in terms of generation of strokes while putting the weight on the weighing pan of the mass comparator. For this reason, when it is necessary to obtain very „precise" measurements, the measuring procedure is automated → automatic, robotic comparators are installed. Radwag provides such solutions not only to mass measuring laboratories but also entities dealing with environmental protection, where weighing efficiency and accurate measurement of minor quantities, e.g. dust PM 2.5, PM10, amounts of PM fixed particles (automotive industry), are essential.

Figure 5. Manual WAY mass comparator

Maximum load 500 g, electric balancing range - 10g to + 20 g
Internal ballast weights, automatic control

The comparison results are used to specify the weight class, mass standards and their real mass using as low measuring uncertainty as possible. The standard limiting error values for weights can be accessed in the OIML R111-1 document entitled ,,Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3. Part 1: Metrological and technical requirements". The abbreviated list is showed in the table 2.

Table 2. Maximum permissible weight errors (± δm in mg)

	Accuracy class / maximum permissible error (± mg)			
Nominal mass (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

As mentioned before, the conditions under which the procedure is performed determine the comparison accuracy. Recommendations in this respect are given by OIML R111-1, and showed in table 3. Yet only the operational qualification, that is real-condition tests, provide a real picture of how the comparison result is dependent upon temperature and humidity fluctuations in the working area.

Table 3. Ambient temperature during weight calibration

Weight class	Temperature fluctuations during calibration		Humidity fluctuations during calibration	
	within 1 hour	within 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

Preparing for testing, please remember that floor vibrations substantially disturb the mass measurement. The aforesaid source of disturbance may be complex, starting from mechanical vibrations, through free movements of the earth's crust as a result of natural physical phenomena, and ending up with earthquakes. Bearing these in mind, Radwag's balances and mass comparators have been equipped with sensors that detect these phenomena, which is one of the aspects of the Digital Weighing Auditor (**DWA**) application.

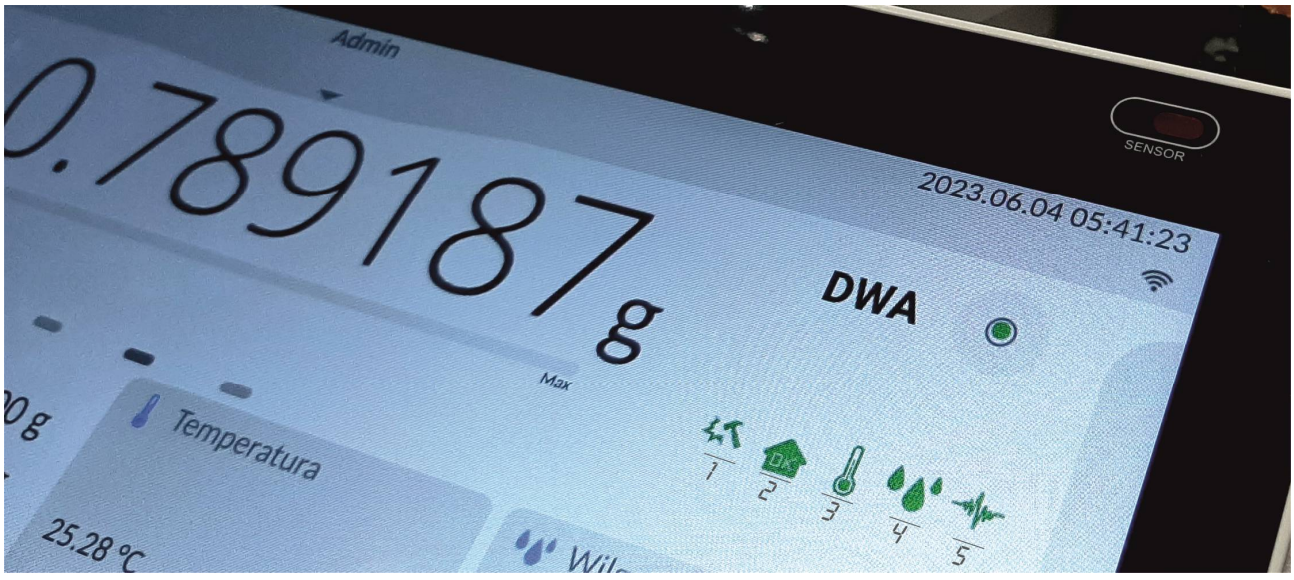

Figure 6. Digital Weighing Auditor application

Key: 1 – weighing quality monitor (strokes), 2 – Digital Weighing Auditor status, 3 – temperature control, 4 – humidity control, 5 – floor vibration detection.

Aside from being precise, comparison must be efficient, especially when it is commercially used - „time is money”. Such features are offered by the RMC 1000.5Y mass comparator that adopts an extra draft shield and suspended weighing pan unit. Thanks to these, it assures a perfectly central position of the weight in relation to the weighing unit, thus eliminating the impact of air movement on the result of the mass measurement (fig. 7).

Figure 7. RMC 1000.5Y mass comparator

Key: 1 – comparator outer cover, 2 – mass standards, 3 – mass standard linear magazine, 4 –comparator control robotic unit, 5 – comparator weighing unit (mass measurement)
6 – operator’s panel.

Weights of various masses can be compared thanks to inner ballast loads whose mass is automatically adapted to the mass of the test weight. The parameters of the RMC 1000.5Y comparator are showed in the table 4.

Table 4. Metrological parameters of the RMC 1000.5Y comparator

Comparison range - E1, E2, F1, F2 class weights	10g ÷ 1kg
Maximum load (Max)	1020g
Elementary reading unit (d)	1µg
Standard repeatability for 5% Max *)	1.2µg
Standard repeatability for Max *)	2µg
Electric balancing range	-1 g ÷ +20 g
Adjustment	internal
Relative humidity	40% ÷ 60%
Weights/mass standard magazine	36 pcs
*) Repeatability is expressed as a standard deviation established for 6 ABBA cycles	

It is possible to control the RMC 1000.5Y comparator remotely, via the RMCS application that manages the entire procedure, starting from the order receipt, through comparison, ending up with issuance of the calibration certificate. The comparator is optionally equipped with a camera connected to the client's unique environment (supervising robotic unit operation).

As part of own testing, the precision of measurements made by the RMC 1000.5Y comparator in the entire measuring range, from 10 g to 10 kg, has been evaluated. The tests have been carried out in the Radwag Research Metrology and Certification Centre. The reference weight in the ABBA method for each load was the E_1 class weight, and the unit tested was the F_1 class weight as per OIML R111-1.

For every load, a standard deviation was determined out of 6 series of ABBA method weighing. The series was repeated 8 times in order to determine the stability of the comparator in the long term. The average difference r_i for the ABBA method for each "i" series of measurements was determined on the basis of the equation (1) and (2).

Basing on the differences, a standard deviation was determined for differences as per the following equation (3):

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

where: r_i average difference (B-A) for "i" measurement

$\acute{r}$ arithmetic mean for "n" measurements

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

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

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

where: n – number of measurements in ABBA method

Table 5. RMC 1000.5Y comparator measuring precision

standard deviation out of 6 series under ABBA method $\bar{x}$ (μg)						
10 g	20 g	50g	100 g	200 g	500 g	1000 g
0.96 ± 0.29	1.00 ± 0.19	1.01 ± 0.18	1.18 ± 0.37	1.20 ± 0.24	1.35 ± 0.29	1.43 ± 0.36

Having analysed the test results, the conclusion is that precision of measurements in the ABBA comparison cycle ranges from 0.96 to 1.43 μg. The best precision of measurements is for standards whose mass is up to 50 g, about 1 microgram. The higher the mass of the weight in comparison, the higher requirements for the mechanical unit, at least from a physical point of view, which eventually leads to a deteriorated precision of measurements, as applies to the weight with a mass of 1 kg.

2.1. Dissemination

The traditional manual method of determining the weight / standard mass through direct comparison usually entails a large number of repetitions. Such a way of determining masses is subject to errors coming from the so-called deviating values that are hard to diagnose correctly. In effect the test weight mass determination uncertainty may be too high. Automation and robotisation of mass measurements have substantially changed the reality as the number of measurements is no longer so important.

Another problem is concerned with weight mass determination uncertainty and applies primarily to weights with an accuracy class of E_1 and whose mass is lower than 1g. A considerable impact is exerted by reference weight mass determination uncertainty, but you cannot forget about the influence of external factors, especially when the surface of the weight is large. Bearing these restrictions in mind, more and more studies are adopting the so-called dissemination method. Generally speaking the process requires comparing a set of weights in relation to one or more reference weights. The dissemination method requires several or dozen weighing cycles with various combinations of weights of the same total nominal mass, using adjustment calculations for the purposes of limiting the error propagation. Using robotisation, the RMC 1000.5Y robotic comparator is not problematic as all calculations are made by the RMCS software that cooperates with the robotic unit.

Figure 8. RMC 1000.5Y – weights subjected to dissemination

Maximum load 1000 g, electric balancing range - 10g to + 20 g
Possible to compare mass standards 10 g ÷ 1kg class: E0 –F2,
Internal ballast weights, automatic control

2.2. Nano-scale measurements

Measuring out smaller and smaller amounts in the micro- and nano scale requires not only extremely precise measuring instruments but also suitable methods for periodical inspection of the weighing procedure. Mass standards below 1 mg may prove helpful yet the method of determining their mass is crucial. The use of balances/scales or mass comparators whose elementary reading unit is 0.1 ug during production of such standards may be insufficient, mainly in view of standard mass determination uncertainty. Considering the above, research works have been initiated in Radwag. The early result of these works is the first nano mass comparator named NANO.AK-4.500.5Y with a reading unit of d=0.01μg (10 nanograms).

Figure 9. NANO-Comparator AK/4-500.5Y

Maximum load 500 mg, 4 positions for standards, automatic control
External adjustment, elementary reading unit of 0,00001 mg,

Please note that the legal metrology referring to weights clearly specifies the maximum permissible error values and maximum extended uncertainty of the mass determination (point 5.2 of OIML R 111-1). With regard to weights whose mass range is 20 mg ÷ 1 mg, the maximum permissible error for determination of their mass is ±0.003 mg while the extended uncertainty is 0.001 mg (table 7). While inspecting these weights, it is possible to use ultra-microbalances or mass comparators with an elementary reading unit of d=0.1μg, but their repeatability falls within the following range: 0.15 ÷ 0.5μg, which may represent ca. 50% of the uncertainty budget. It is a true problem, particularly when you look for methods of producing and controlling weights with a mass lower than 1mg.

Table 6. OIML R111-1, maximum permissible errors for weights (± δm in mg)

	Accuracy class / Maximum permissible error (± mg)			
Nominal mass (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

Testing in the Radwag Research Metrology and Certification Centre focused on checking correct operation of the NANO.AK-4.500.5Y comparator. The comparison was performed for every load using the ABBA method in stable ambient conditions – humidity variability was 1.20%, temperature fluctuations 0.14^{o}C within 24 hours. The test result are showed in the table 7.

Table 7. Precision of measurements in the NANO.AK-4.500.5Y mass comparator for various test loads.

Standard nominal mass	**Standard deviation *S* $\bar{x}$ (5)**	**Precision of determining the 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

Our experience proves that precision of measuring low-mass items is determined only through measuring precision. As for NANO-AK-4.500.5Y, precision in the series of measurement was found to be permanent with a minor variability of 0.02μg , regardless of the mass of the test weight.

3. Industrial metrology

You may not realise that majority of items or processes that surround you used to be or are strictly related to the metrology. Measurements are used to determine the mass of single-packaged products, people measure out the amount of fuel at the petrol station, or distance covered, vehicle speed, substance volume in the mixture, weight of products they purchase, etc. All of them are nearly intuitive, using measuring instruments, including the ones that focus on quantity, volume or mass. It must be highlighted that some fields of industry, such as trade, health protection, are subject to legal metrology regulations with a view to securing service recipients against potential fraud. Whereas the legal metrology is concentrated on the quality of measuring instruments, the industrial metrology is aimed at obtaining the acceptable quality of products or processes. It is attainable through permanent monitoring of essential quality indicators, measurement of production series mass → the so-called automatic control scales, through inspection of the final product mass → balances in the QC Department, etc.

Figure 10. PS 1000.R2 balance – measuring paint and lacquer mass

Maximum load 1000g, reading unit of d=0.001g
automatic adjustment, OIML certificate

Regardless of the variant adopted, the best solution is the QbD (Quality by Design) concept as per which the quality is embedded in the product.

When speaking of mass measurements, such an approach is concerned with determining mass variability limits that guarantee the quality and stability of the product, at the same time keeping correct economic dependencies, regulated by competitors and market. It is difficult because a real accuracy of mass measurement may be established only during the operating qualification in the workplace, while a comprehensive picture requires proper connection of numerous factors. Here comes the following question: what are the weighing system selection criteria? We know that precise measurement is expected, but...

Precision (of analysis, measurement) is a qualitative notion and therefore cannot be expressed in numbers. The precision of measurement is defined by two parameters: trueness → systematic error and precision → random error. Trueness of the measurement is a correspondence between the mean of the unlimited number of repeated values of measured masses, and the value of the reference mass. The test can be carried out only with the use of certified mass standards, as conducted in the Radwag's QC department (fig. 11).

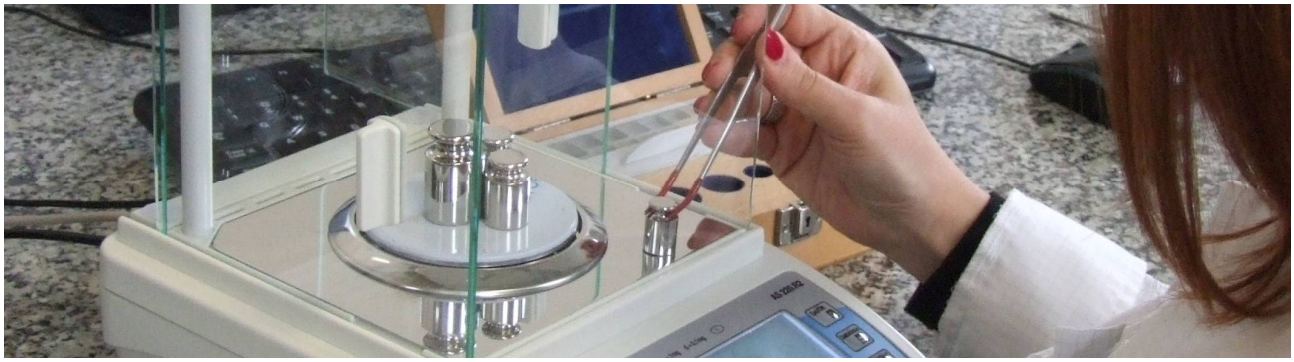

Figure 11. Quality Control Department – AS 220.R2 balance, evaluation of correct values
AS 220.R2, Maximum load 220g, reading unit of d=0.1mg
automatic adjustment, OIML certificate

The precision of measurement is correspondence between indications or values of measured items, received for repeatability of measurements with the same or similar items under specific conditions. High precision is achievable when the measured values are close to one another. This parameter is widely dependent upon testing conditions (temperature, humidity, air movements, vibrations) and weighing skills.

The measurement can be precise only when the value of the systematic and random errors is acceptable (fig. 12). Such a comprehensive approach is adopted when the mass of the weighed item is higher than 25% of the maximum load.

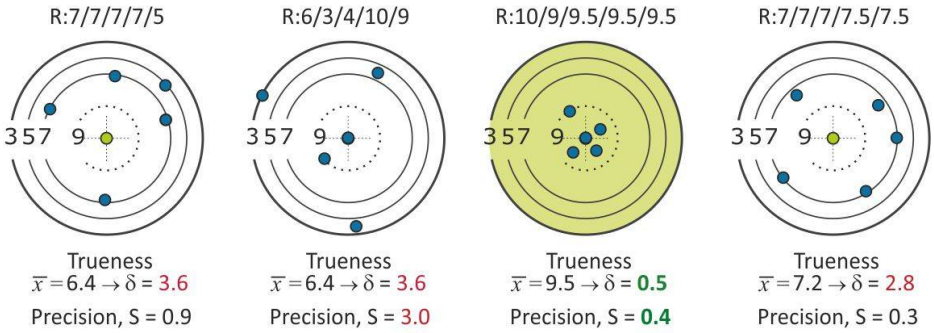

Figure 12. Trueness and precision in mass measurement

In the process of weighing low-mass samples, e.g. measuring powder quantities, mass assessment of the suspended dust emission, only the random error substantially contributes to precision of measurement. Such an assessment of the mass measurement quality is also showed in the documents of the American pharmacopoeia USP-NF (United States Pharmacopeia National Formulary, Chapter 41) and European Pharmacopoeia (Ph. Eur. Annex 10.7, Chapter 2.1.7). In order to determine the value for the random error, it is necessary to perform a series of at least 10 measurements, and then calculate the standard deviation value on the basis of results. The standard deviation value is further used to calculate the starting point of the weighing range → the so-called MSW (Minimum Sample Weight).

Figure 13. Testing changes to filter mass after filtering (wastewater treatment plant), measuring the amount of substance (pharmacy)

MYA 5.5Y.FA microbalance – filter mass measurement, maximum load 5g, reading unit of d=0.001mg
XA 82/220.5Y analytical balance, maximum load 220g, elementary reading unit of d=0.01mg

According to USP / Ph.Eur., the precision of weighing the so-called „low masses" is assured when the condition (4) is met. During the test, the load of up to 5% of the maximum loading capacity of the balance is used. However it can be a weight whose mass is similar to the mass of weighed samples.

Based on our experience, it can be concluded that the precision of measurement for the so-called „low masses" has a fixed value, providing the testing conditions are stable.

$$R = \frac{2 \cdot S}{m} \leq 0.10\% \rightarrow \frac{2 \cdot S}{m} \leq 0.001 \tag{4}$$

where: S – standard deviation for values (e.g. in grams);
m – the lowest net mass of the sample that will be weighed.

The starting point of the weighing range, the so-called MSW, is determined in the relationship (5) when the requirement described through the relationship (4) is met.

$$MSW = 2000 \cdot S \tag{5}$$

The lowest possible standard deviation from the series of measurement is 0.41d, so the lowest MSW value, depending on the elementary reading unit of the balance (d), may take a value given in the table 8.

Table 8. Minimum MSW values, depending on the value of the reading unit

Element. reading unit (d)	Formula	MSW	Type of balance
1 mg	0.41 · 1mg ·2000	820 mg	PS 1000.X2
0.1 mg	0.41 · 0.1mg ·2000	82 mg	AS 220.5Y
0.01 mg	0.41 · 0.01mg ·2000	8.2 mg	XA 82/220.5Y
0.001 mg	0.41 · 0.001mg ·2000	0.82 mg	MYA 5.5Y
0.0001 mg	0.41 · 0.0001mg·2000	0.082 mg	UYA 2.5Y

Figure 14. MYA 5.5Y microbalance with an active MSW function

MYA 5.5Y, maximum load 5g, elementary reading unit of d=0.001mg
Internal adjustment, OIML certificate

In the industrial metrology, the standard deviation value (S) can be used to specify the range at which the measurement result is likely to be → there are no ideal measurements. Here comes the so-called three-sigma rule applies. According to the rule, the measurement result is in the range:

- ±/– 1S with a likelihood of 65 %
- ±/– 2S with a likelihood of 95,5 %
- ±/– 3S with a likelihood of 99,7 %

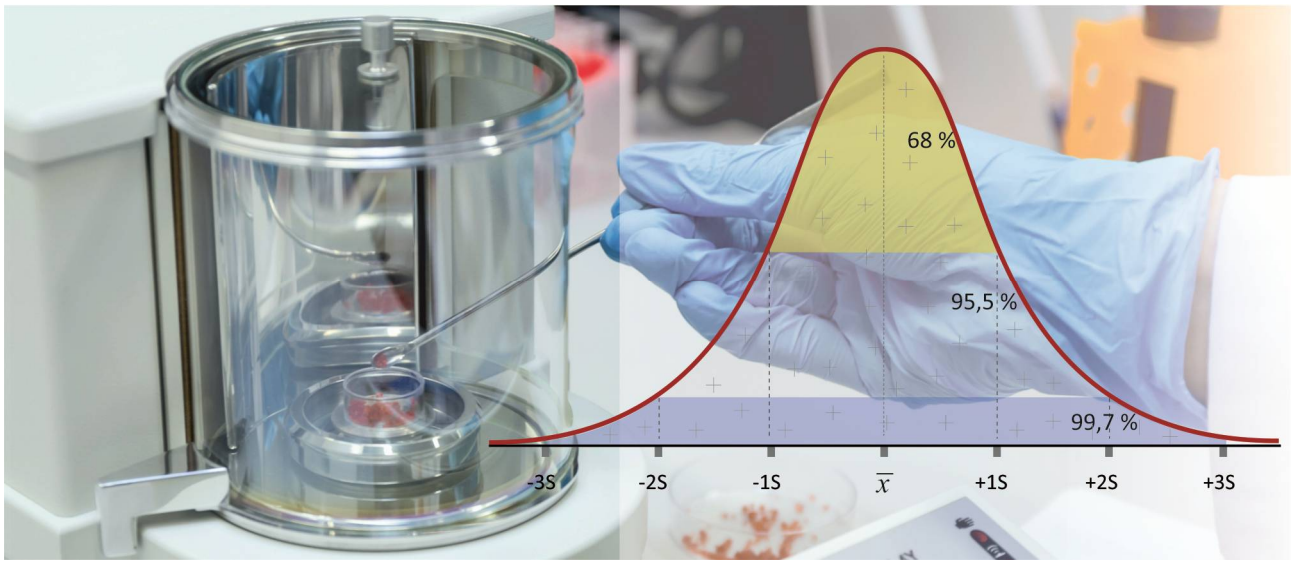

Figure 15. MYA 2.4Y microbalance – weighing powder portions

MYA 2.4Y, maximum load 2g, elementary reading unit of d=0.001mg
Internal adjustment, OIML certificate

While estimating the measurement uncertainty, the standard deviation value from the series of measurements is the so-called A-type measurement uncertainty that is used to estimate the extended uncertainty.

3.1. Adjustment

The mass measurement in the industry must be fast and reliable, especially when the measuring result is used in the production control. It is therefore expected that weighing systems are always precise, irrespective of variable ambient conditions. Such an assumption applies to balances whose elementary reading unit takes a relatively high value → d ≥ 1g. In such a case we refer to the so-called technical measurements usually made with the use of weighing cells. More precise mass measuring in the industry or laboratory requires the use of more complicated weighing systems → magnetoelectric transducers. Their precision is guaranteed thanks to periodical adjustment. The examples of weighing systems used in Radwag-manufactured balances are showed in the figure 16.

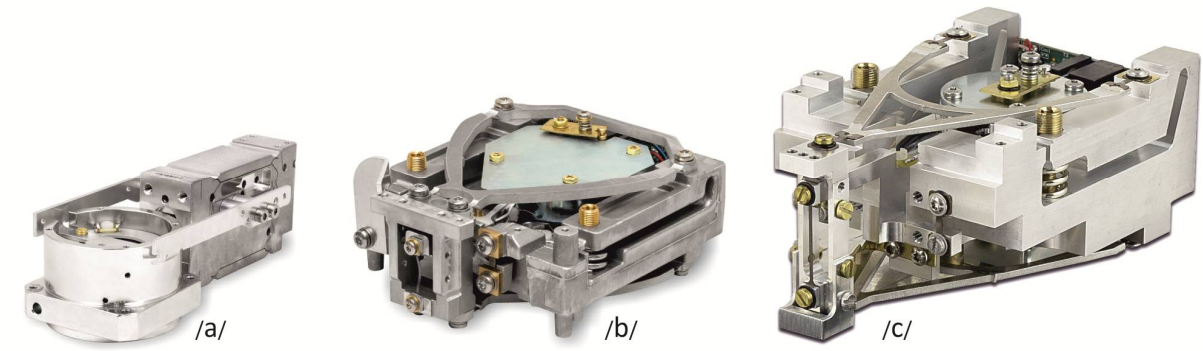

Figure 16. Weighing systems with electromagnetic processing
a. electromagnetic unit, monolithic, load range 6 ÷ 300 kg
b. electromagnetic unit, load range 50 g ÷ 1000 g
c. electromagnetic unit, load range 2 g ÷ 50 g

The task of adjustment is to correct balance indications, and this can be obtained as a result of comparing the standard weighing result (the so-called adjustment mass) with the known value. These comparisons are automatic → change of temperature, passing time, or semi-automatic before initiation of testing → interference of the balance operator. The adjustment mass is not calibrated, so are external standards. The calibration as a procedure that demonstrates „precision" of indications is performed for the balance, so in fact also with reference to the internal value of the adjustment mass. The adjustment rule has been depicted in the figure 17. It is identical to all balances, and correct operation of the unit is verified in Radwag, by the QC Department.

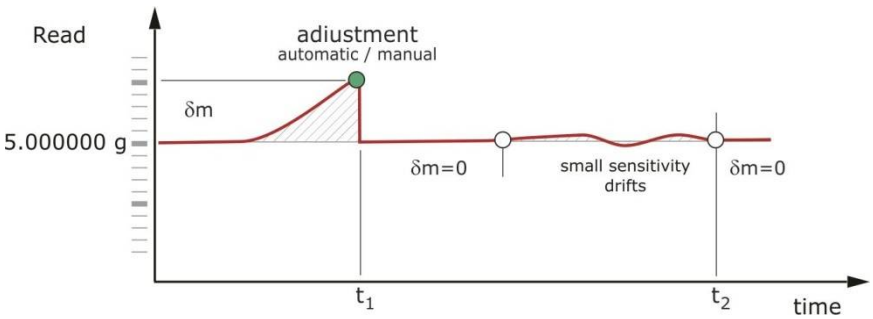

Figure 17. General electronic balance adjustment rule

The balance adjustment is a typical load weighing so from the metrological point of view the quality of this process can be defined through the precision of measurement. It is a common knowledge that the precision of measuring is widely dependent upon measuring conditions and operator's skills. It must be emphasised that adjustment automation and installation of the adjustment mass inside the balance contributes to substantial improvement of measuring precision when compared to manual measurements. It is highly important and desired because the adjustment mass weighing result determines the balance sensitivity correction.

There is no universal technical solution for adjustment as the latter is always an integral part of the balance in terms of shape, location, dimensions and mode of operation. The example of laboratory balance adjustment is demonstrated in the figure 18.

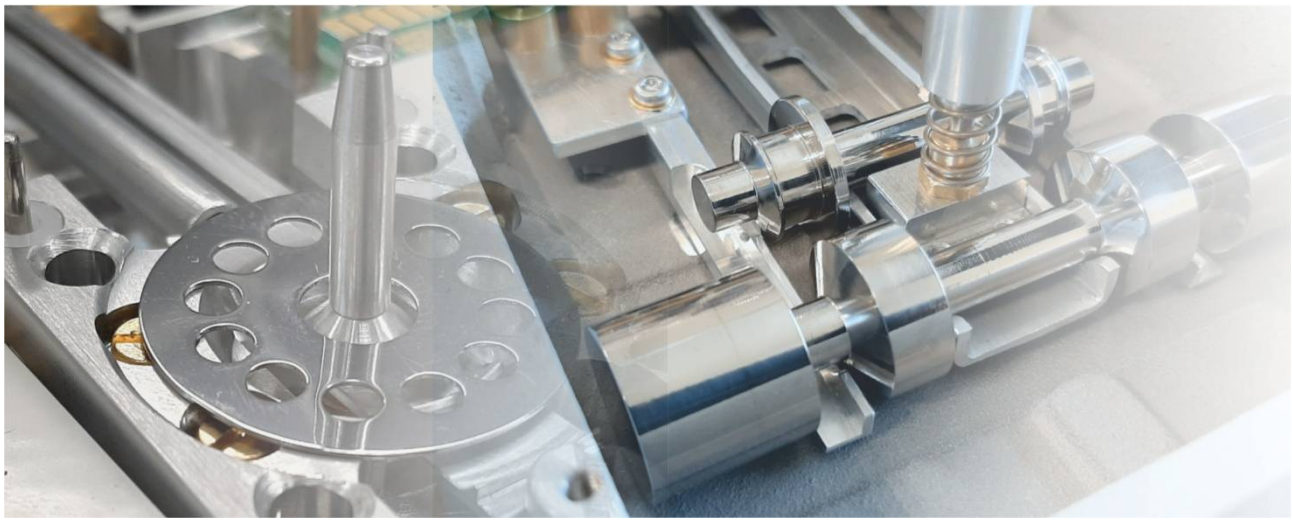

Figure 18. View of adjustment mass of the MYA microbalance weighing system and XA analytical balance

As mentioned before, the qualitative parameter of adjustment is the precision of measurement that can be determined through the GLP Autotest diagnostic function. It is available in the menu of most laboratory balances by Radwag. The mode of operation of the GLP Autotest is concerned with determination of the standard deviation → measure of measuring precision, based on a series of 10-fold weighing of internal adjustment mass. The standard deviation value is usually lower than the value of the balance elementary reading unit.

The adjustment mass in .4Y and .5Y balances is also used as a diagnostic tool during balance production and control. Periodical weighing of the adjustment mass in stable and variable environmental conditions allows determining optimal factors that correct the impact of the environment on precision of mass measurement. It is the original solution adopted in professional laboratory balances, such as MYA microbalances and XA analytical balances. On the other hand possibility of observing and recording changes in the balance is the first step to optimise the balance/scale and high-resolution mass comparator designs → product improvement. The analysis of test results focuses on comparing quality indicators with permissible values. It is also possible with regard to the balances that are already operated, but requires remote interference of the service technician. Following the measurement data, it is possible to specify the dynamics of changes of the working environment which the balance is operating in. The operator's belief in stable working conditions does not always reflect the reality. The example of the diagram with data from the XA 82/220.5Y analytical balance autotest is showed in the figure 19.

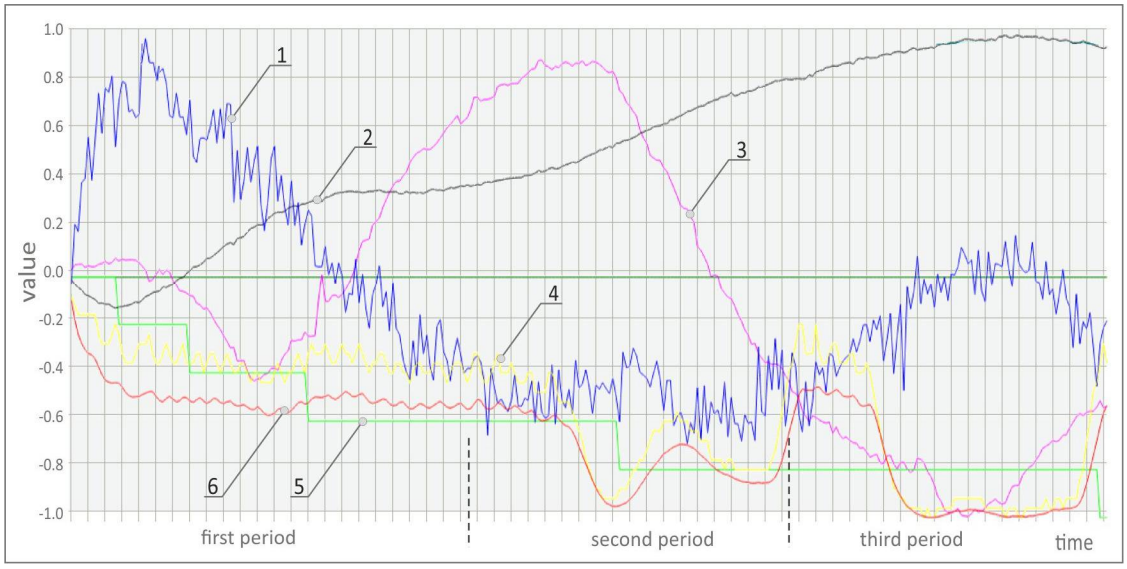

Figure 19. Autotest – XA 82/220.5Y balance

XA 82/220.5Y, maximum load 220g, elementary reading unit of d=0.01mg
1 – sensitivity shift, 2 – balance zero drift, 3 – atmospheric pressure, 4 – balance internal temperature,
5 – relative humidity in workplace, 6 – balance transducer temperature

The first testing period demonstrates stability of the transducer temperature (6) and balance internal temperature (4). In spite of this, the balance sensitivity change is recorded, see the curve number (1), as also thermal stability of the mechanical unit is required for proper operation of the balance. Briefly speaking, the first period can be compared to the balance self-warming time which ends with the assumed precision of measurement. The balance sensitivity stabilises at the outset of the second period and remains constant even when the periodical change of the external temperature is forced, which results in changes to the transducer temperature (6) and internal temperature (4). The essential change of the balance sensitivity is recorded only when the change of the external temperature is durable – the third testing period.

The high-resolution balance acclimatisation time is virtually 24 hours as it is primarily due to mechanical elements related to detection and transmission of gravitational forces that require time to stabilise thermally. For this reason the balance zero point change curve (2) proves stable only after the third period of testing. It must be noted that the balance zero point change is not critical when the measuring time is relatively short, as in XA balances. Evaluating curves from the service Autotest, operator's real needs related to precision of mass measurements must always be taken into account.

As mentioned before, the development of weighing systems with the aim to improve their measuring precision is not feasible without detailed tests. The effect of such testing is engineering and design-related modifications and further research. It is a pretty difficult task as operations performed concern IT, electronics and mechanics of weighing systems.

4. Micro-scale measurements

The task of the electronic balance is to permanently keep the weighing pan balanced and to compensate the deviation of the weighing pan if it has been relocated when it is loaded with any weight, see: figure 20. The signal that compensates the gravitational force is scaled in relation to certified mass standards, which allows you to express the weighing result in the following units of measure: gram, milligram, kilogram, etc.

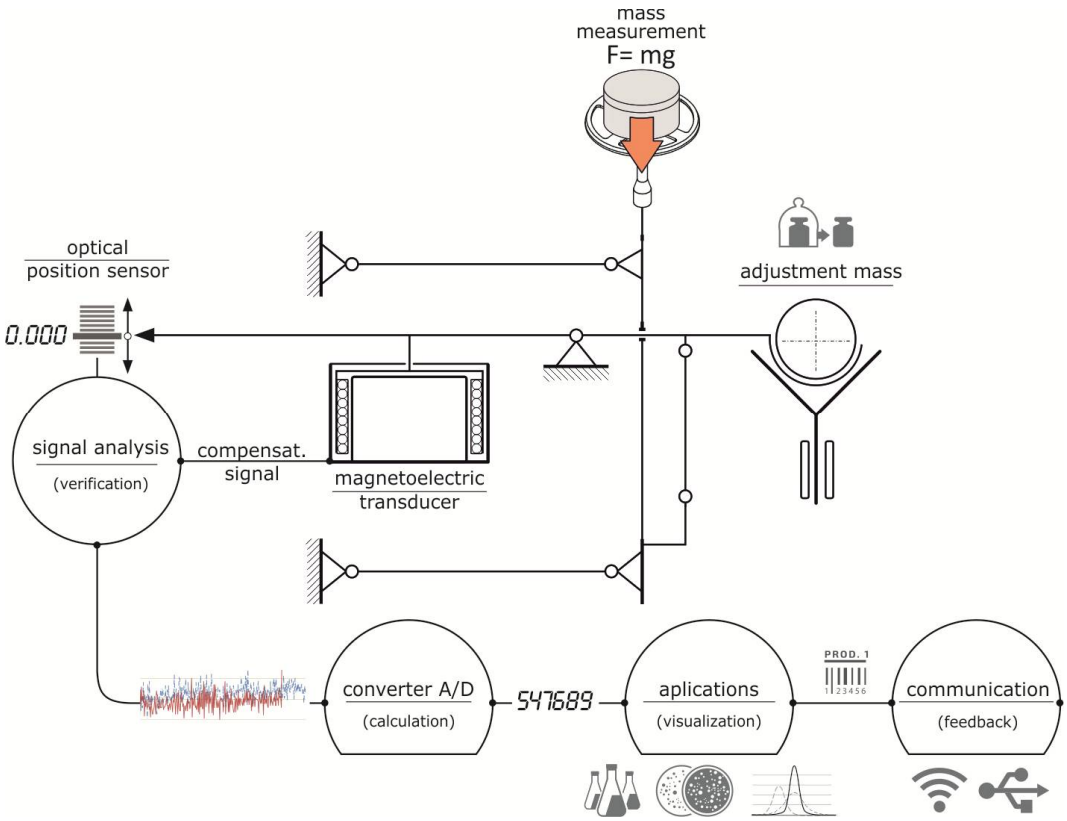

Figure 20. General principle of balance operation with magnetoelectric transducer

The bigger the mass of the sample, the easier it is to measure the force as the impact of other physical factors that accompany the weighing procedure (unstable environment, operator's error, balance sensitivity) is negligible. Correct detection of minor values requires optimisation of the weighing design with regard to mechanics as well as electronics and IT solutions, as in mass comparators, microbalances and ultra-microbalances by Radwag. Thanks to this, a long-term stability and precision of measurement have been achieved. They are desired in R&D and during numerous control processes, e.g. in the pharmacy.

Careful measuring quantities out must be treated as a process whose final result depends on several factors. They derive from the working environment, device, sample weighed as well as balance operator's knowledge and skills, see: figure 21. When the weighing result goes out of the required tolerance range, a risk analysis must be carried out. The aforesaid analysis should specify mass measurement fields that have the biggest influence on the measuring error.

It may be difficult but it is one of the improvement stages → control circle (Shewhart cycle) that allows achieving the required process/product quality level.

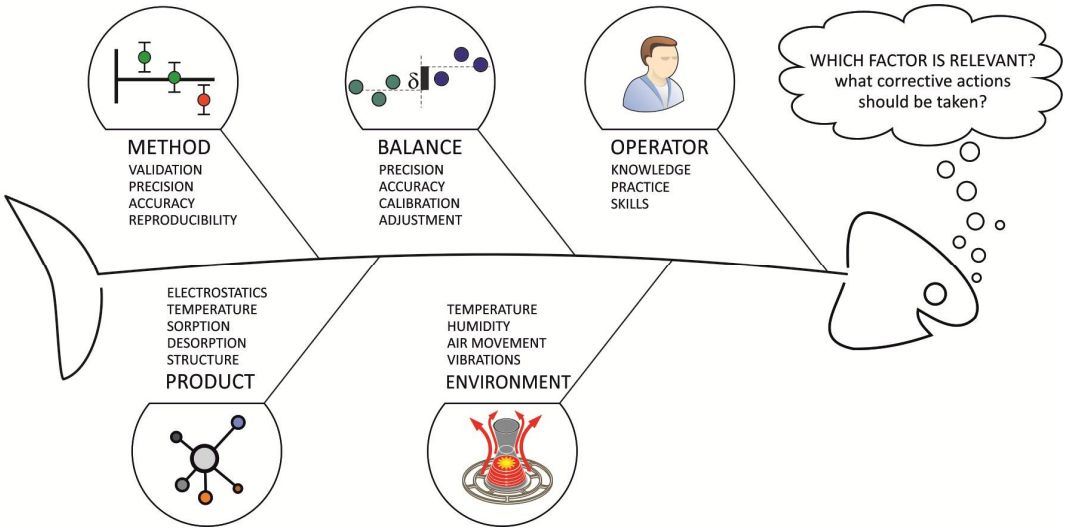

Figure 21. Sources of errors in mass measurement

As a rule the weighing method is defined through PN-EN standards, ASTM, ISO guidebooks or other renowned and acknowledged trade documents, both internal or external. The requirements may be related to sample mass, sample collection method, storage and weighing tolerance. It must be remembered that samples are not neutral and may prove unstable in view of moisture sorption, unbalanced electrostatic charges and thermal instability. The working environment may also influence the weighing procedure as its variability with regard to temperature, humidity, air movement and vibrations is too high.

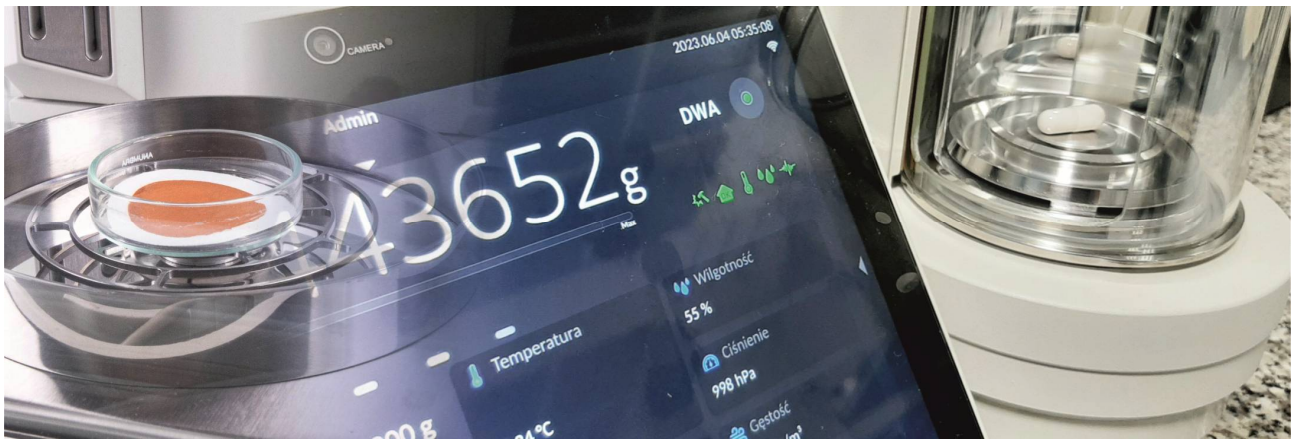

Figure 22. XA 82/220.5Y balance, filter mass measurement,
MYA 2.5Y microbalance, capsule mass measurement – homogeneous mass of medical preparations (USP)

XA 82/220.5Y analytical balance, maximum load 220g, elementary reading unit of d=0.01mg, OIML certificate
MYA 2.5Y microbalance, maximum load 2g, elementary reading unit of d=0.001mg, OIML certificate

Variability of the working environment may be monitored by internal sensors installed in 5Y balances → green icons in the screen, figure 22. It is a professional solution for any laboratory dealing with supervision of working environment variability.

It is also possible to connect additional external temperature and humidity sensors to the USB port of the balance → calibration. Their task will be to monitor the working environment.

Figure 23. XA 210.5Y.A balance with an additional environmental conditions sensor

XA 210.5Y.A analytical balance, maximum load 210g, elementary reading unit of d=0.01mg, OIML certificate
Interfaces: USB-A ×2, USB-C, HDMI, Ethernet, Wi-Fi®, Hotspot

The information on values measured, that is temperature, humidity, vibrations, air density, is displayed in the balance screen or directly on the control panel of the THB-Pro sensor (figure 24).

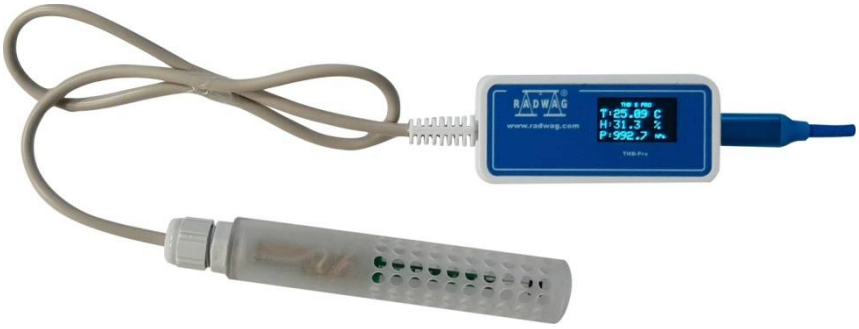

Figure 24. THB-Pro sensor

Elementary reading unit of temperature 0.01°C, humidity 0.1%, atmospheric pressure 0.1hPa
Temperature measurement precision ±0.05 °C, humidity ±0.2%
Sensor power supply – via USB or power adapter, standardised C-type port

It is a common knowledge that measuring a certain amount out in the micro scale requires gross weighing. Therefore the first step is to choose the suitable weighing vessel.

It must be made of materials that are neutral and have no tendency to accumulate static charges on their surfaces.

Bearing in mind the above-stated dependencies, it can be stated that the precision of measurement in the micro scale can be disturbed by numerous factors that influence one another. Despite that, a potential precision of mass measurement can be specified by checking the precision of measurement in the place of using the balance. The test is usually performed with the use of a mass standard whose mass is similar to the mass of samples weighed. The standard deviation (S) from a series of measurements is a measure of inaccuracy. The lower the value (S), the better the correspondence of the results in the series → better precision of measurements. As we know, the precision of measurement is a random error, and the precision of measurement is affected by a systematic error too → sensitivity error. What does it mean?

If the balance sensitivity is a linear dependency between load and indication → $\Delta R/\Delta m$, then the sensitivity correction will be effective when the adjustment mass is at least 75% ÷ 85% of the maximum loading capacity of the balance, see: figure 25. Sensitivity error → systematic error can be noticed only when the mass of weighed samples is higher than 10% of the maximum load of the balance. When the mass of the weighed samples is very low, the systematic error is negligible, and the main component of the measurement error is the random error.

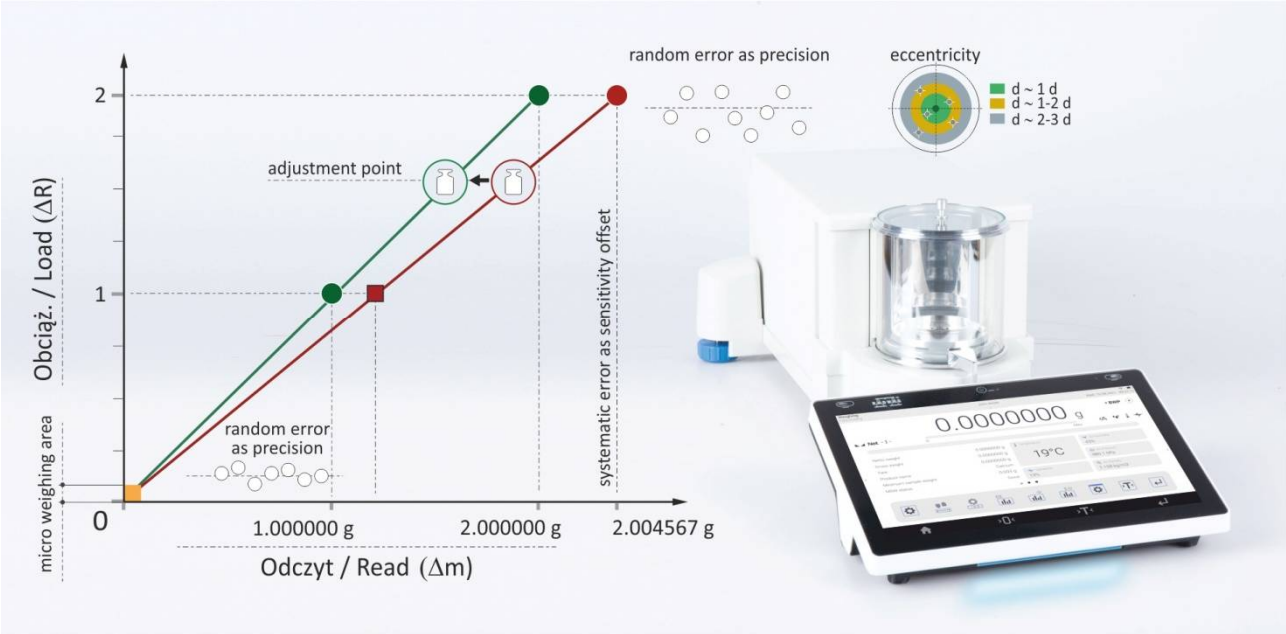

Figure 25. MYA 21.5Y microbalance – random and systematic errors in micro measurements

MYA 21.5Y microbalance, maximum load 21g, elementary reading unit of d=0.001mg, OIML certificate
Interfaces: USB-A ×2, USB-C, HDMI, Ethernet, Wi-Fi®, Hotspot

The value of the random error for the so-called small mass/micro weighing area (fig. 25)/ can be quickly determined by experiments → reproducibility, or using values given by the manufacturer. The weighed item is not always in a suitable shape and it is not always a liquid. There are therefore special weighing holders that allow weighing flasks, micro-vessels, beakers, stents, and other pieces of irregular shape, etc.

5. Automation in mass measurement

The main feature of every measurement must be its usefulness, that is possibility of using the obtained information for the purposes of assessing the physical and chemical condition of the sample, confirming that it meets requirements, and possibility of its further processing, etc. Unit measurements can be quickly performed by the operator who can then draw conclusions only from own observations. Human work is becoming unprofitable and inefficient, and when you need to process a large amount of information in short time, you opt for automation. This is used in automated production lines which involve no operators but allow checking essential features of the product, including its mass → dynamic balances. Essentially efficiency of the production line (number of pieces per minute), product segregation and possibility of marking them. The example of such a solution is the DWR dynamic balance by Radwag (fig. 26).

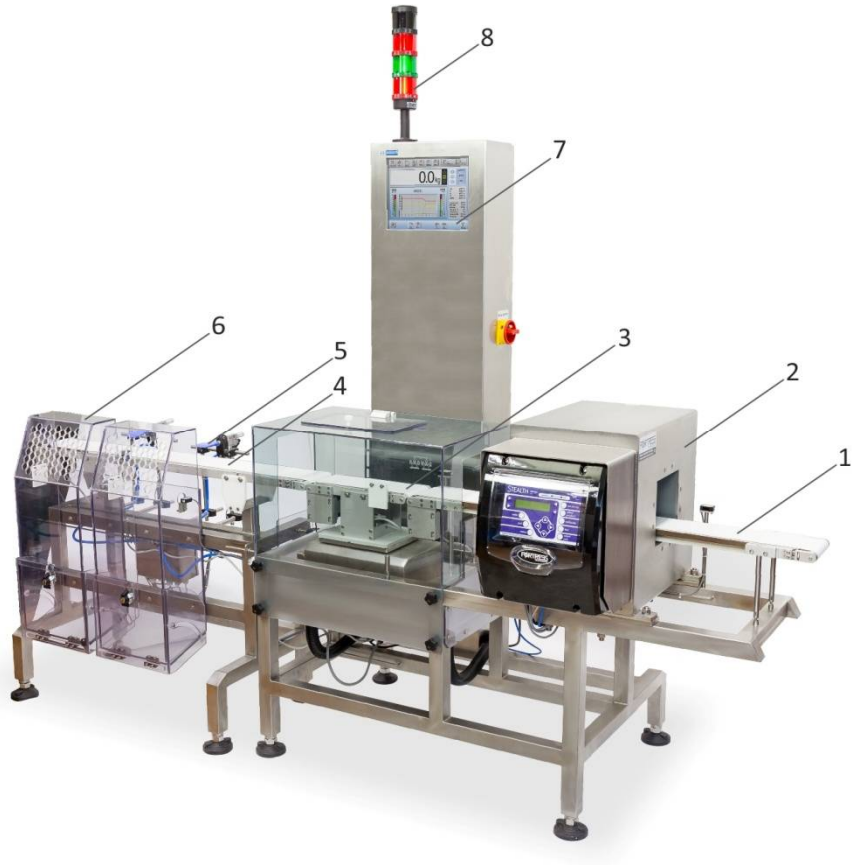

Figure 26. DWM 7500 automatic balance

DWM 7500, maximum load 7500g, elementary reading unit of d=0.1g, accuracy class XIII (1), Y (a)
Efficiency Max 500szt./min. Key: 1 – input feeder, 2 – metal detector,
3 – weighing feeder (mass measurement), 4 – output feeder, 5 – pneumatic selection,
6 – segregated piece basket, 7 – balance screen, 8 – light signals.

The design and mode of operation of automatic balances is always optimised for specific application → engineering lines and environment work → IT system.

The mass measurement automation is also possible in micro scale but requires more subtle technical and IT solutions. The example of such a solution is the automatic system intended for checking and calibrating multi-channel pipettes. The mode of operation of the piston pipette is always identical, regardless of the design. The manual or automatic pressure against the piston causes the liquid to be drawn or discharged from the pipette hole for dosing purposes. To control the precision of the pipette, it is necessary to measure the mass of the liquid dosed through the pipette, which allows determination of its density, equal to the pipette volume, provided the liquid density is known. Such a process has been depicted in the figure 27.

$$V = \frac{m}{\rho} \tag{6}$$

where: V volume of liquid discharged from pipette (cm^3)
m liquid mass (g)
ρ liquid density (g/cm^3)

Figure 27. Single-channel pipette volume control

MYA 21.5Y.P microbalance maximum load 21g, elementary reading unit of d=0.001mg, OIML certificate
Pipette control equipment: steam curtain, weighing vessel, operator's application, compliance with ISO 8655-6.
Interfaces: USB-A ×2, USB-C, HDMI, Ethernet, Wi-Fi®, Hotspot

Verification of the pipette operation applies to at least several volumes and is time-consuming when involving multi-channel pipettes. Only automation may boost this process. AP 12.5Y is able to quickly and smoothly perform such a task. When piston pipettes are tested on the basis of the gravimetric method, the mass of the discharged liquid is always recorded. Therefore weighing results are always to be converted into volume. It can be done in two ways. Using the first method, it is necessary to adopt a general equation (7) that allows calculating the liquid volume, with special regard to essential environmental factors.

$$V_{i,ref} = \left(m\middle|L - m_E + m_{evap}\right) \times \frac{1}{\rho_w - \rho_a} \times \left(1 - \frac{\rho_a}{\rho_b}\right) \times \left[1 - \gamma\left(t_w - t_{ref}\right)\right] \quad (7)$$

where: $V_{i,\mathrm{ref}}$ liquid volume at rated temperature in ml,

m_L weight for the weighing vessel after giving liquid value in g,

m_E balance indication for weighing vessel before giving liquid value in g (m_{mi} = 0 for balance tarring with weighing vessel)

m_{evap} estimated evaporated mass in the testing cycle in g,

ρ_A air density in g/ml during test, ρ_B – mass standard density (8 g/ml),

ρ_W water density at testing temperature (in °C) in g/ml,

γ thermal cubical expansion rate for pipette (°C−1),

t_W pipette temperature = test liquid temp. at °C; t_{ref} – pipette rated temp. (20°C or 27°C).

Figure 28. AP-12.1.5Y automatic system

Maximum load 21g, elementary reading unit of d=0.001mg, Max number of channels: 12.
Operator's application, compliance with ISO 8655-6, Interfaces: USB-A ×2, USB-C, HDMI, Ethernet, Wi-Fi®, Hotspot

The second method is simpler as all aforementioned factors have been included in the so-called Z corrective factor (equation 3) whose value allows for water density, atmospheric pressure and temperature of the test.

$$V_i = m_i \times Z \quad (8)$$

The automatic mass measurement units are also used in environmental protection tests → standard gravimetric measuring method for determination of fraction mass concentrations PM 10, PM 2.5 of the suspended dust. As we know, the dust mass is determined on the basis of difference of masses of filters before exposure and filters after exposure. The essential element in this process is filter conditioning at specific temperature and humidity. Assurance of required filter conditioning conditions is always easy and feasible, especially when it requires reconstruction of the existing infrastructure. If this is the case, robotic weighing systems are used, for example RB 2.5Y or RMC 2.5.Y.FC, fig. 29.

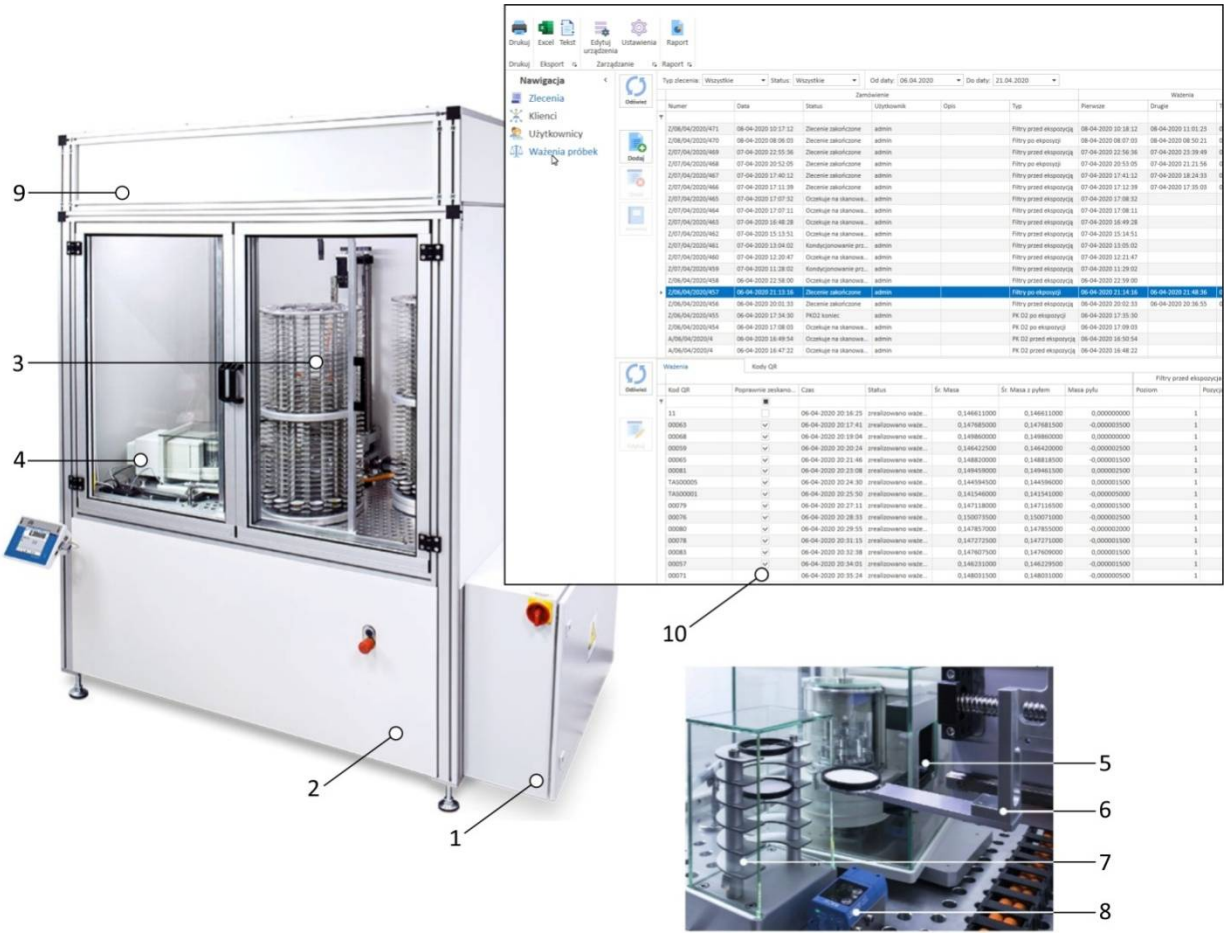

Figure 29. RB 2.5Y – filter conditioning and mass measurement
Maximum load 2g, elementary reading unit of d=0.001mg, Max. number of filters: 1000
Filter conditioning as per EN 12341:2024, control: PC application, 1- control unit, 2 – housing
3 – filter magazine, 4 – microbalance (mass measurement), 5 – deioniser, 6 – robotic arm,
7 – mass standard and reference filter magazine, 8 – QR code scanner, 9 – HEPA filters, 10 – PC application

Every filter is marked with a QR code that allows its clear identification, regardless of the place in the magazine. Thanks to a large capacity of the magazine, it is possible to simultaneously condition → prepare filters for measurements in the field and weigh filters after exposure → specify the dust mass concentration PM. The mass standard and reference filter magazine (7) is used to establish stability of the weighing unit and potential impact of environmental conditions on variability of the test filter mass.

A similar automatic weighing unit is used in the automotive industry to assess the mass emission of particles by combustion engines. The description and requirements for the measuring method that is applied in the automotive industry have been referred to in the EU resolution no. 2017/1151 on test approval of combustion-engine vehicles with reference to emission of pollutants emitted by light passenger vehicles and commercial vehicles, and the document drawn up by the United States Environmental Protection Agency 40 CFR Part 1065 - Engine-Testing Procedures. In the context of mass measurements, the essential metrological requirement is a need to use a balance or weighing system with an elementary reading unit of 0.1μg, with acceptable precision of measurement. Obtaining such a precision of measurement requires the use of other weighing method in which every filter is placed in a special container, as in the UMA 2.5Y.FC and RMC 2.5Y.FC automatic units (figure 30).

Figure 30. Chassis dynamometer and filter mass measurement automatic unit.
UMA 2.4Y.F, maximum load 2g, elementary reading unit of d=0.0001mg, Max. number of filters 24
Filter conditioning in the laboratory, control: PC application
Source: Instytut Badań i Rozwoju Motoryzacji BOSMAL Sp. z o.o. – research under the BOS/0779/BH/21 project

By limiting the space which the filter is weighed in and by eliminating the so-called human factor, it is possible to obtain the precision of measurement of 0.2 ÷ 0.3 μg, which is unavailable to manual measurements. The filter container diagram is showed in the figure 31.

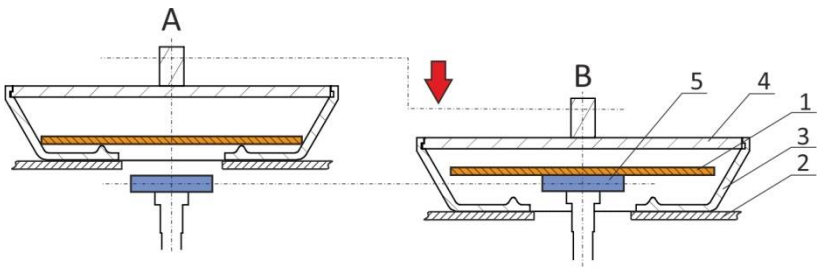

Figure 31. Filter container

1 – filter, 2- filter magazine, 3 – container housing,
4 – upper container housing, 5 – weighing pan of the weighing unit

It is a common knowledge that measurement result is widely dependent upon filter conditioning conditions, which requires the temperature of 22°C ± 1°C and relative humidity of 45% ± 8% in the laboratory (source: EU Resolution 2017/1151, point 4.2.2). It may be expensive and hard to achieve but such conditions are guaranteed by the internal space of automatic units equipped with own air-conditioner → UMA 5.5Y.FC and RMC 2.5Y.FC. When the number of filters for testing is high, the dedicated solution is the RMC robotic unit that aside from air conditioning has also an internal filter magazines for 136 items. Such a number allows simultaneous conditioning and weighing of filters in any configuration. The figure 32 illustrates the aforesaid weighing unit.

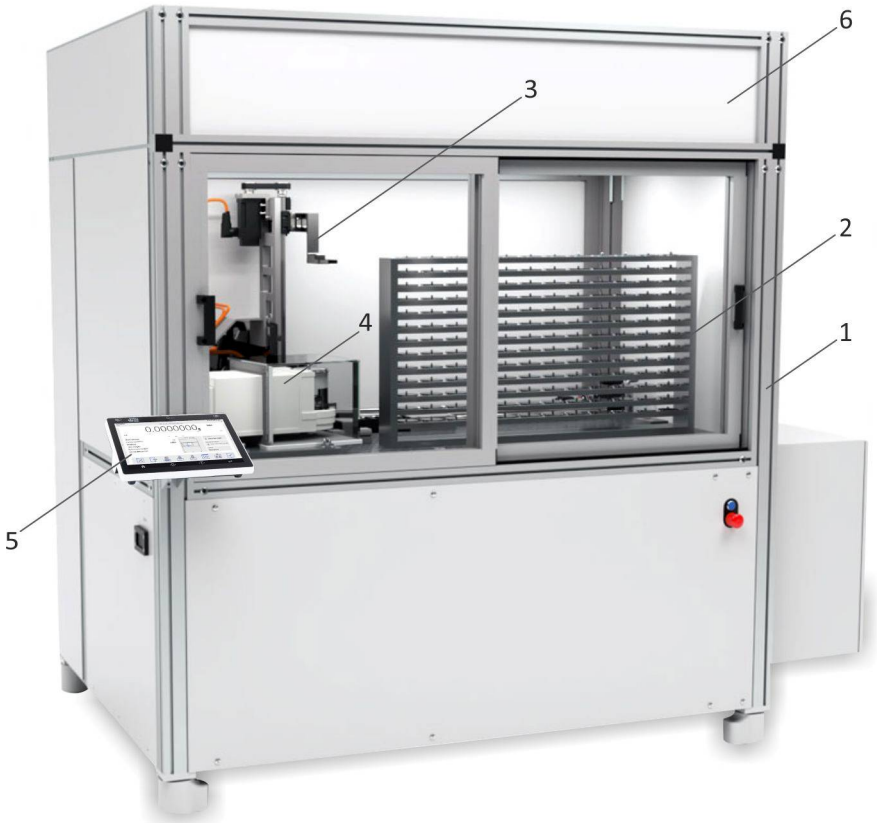

Figure 32. RMC 2.5Y.FC – filter mass measurement in the automatic cycle

Max load 2g, elementary reading unit of 0.001 mg or 0.001 mg, Compliance with EN 12341:2024,
1 – housing 2- filter magazine, 3 – robotic unit, 4 – microbalance (mass measurement), 5 – operator's panel, 6 – HEPA filter

At present particulate matter emission standards for combustion engines are 4.5mg/km (Euro 6c,d; China 6a) and are to be reduced by about 30% in the future. Emission limitation requires a suitable testing stand that must allow more precise measurements → better measuring precision. As we know, the precision of measurement is a key metrological parameter that proves widely dependent upon testing conditions, environmental factors and human imperfections. It seems that the only reasonable direction for developing measuring methods dedicated to the automotive industry is automation.

6. Metrology in scientific research

The metrology as a science and mass measurement practice has always been widely associated with scientific research. We always measure something in a macro or micro scale, compare products to reference items, specify safety, particularly in the pharmacy, as this is how the modern world works. In the process of improving our reality, scientific attainments are priceless as they are the only ones that set directions for growth and provide solutions to important social problems. It is obviously possible to rely on the empirical discovery of new solutions but it is time-consuming, costly and cannot guarantee success.

6.1. Filter mass stability in time

Testing the quality of atmospheric air has always been significant, and its importance grew when environmental changes, such as storms, hurricanes, floods, have become common in some parts of the world, and scientific data on human death rate as a result of air pollution confirm a substantial cause-and-effect relationship. One operation is not sufficient to improve this situation, it is all about comprehensive research, starting from industry and ending up with laboratory, that is required. One of the elements of these tests was the research project marked as C2-001/2020/NP-I ,,Reference method testing PN-EN 12341:2014 of emission of suspended dust PM with the use of the RB 2.4Y.F robotic weighing system" that was completed in the Institute of Environmental Protection of the Polish Academy of Science in Zabrze.

It must be noted that the gravimetric method is considered as the most precise method of measuring the mass of suspended dust. This method entails the use of the so-called dust samplers which usually hold 14 filter holders. The mass of these filters have been previously determined in the gravimetric weighing process. Every filter is then automatically placed in the regular-air-flow tunnel (fig. 34) for 24 hours. At this time the filter structure accumulates PM dust particles typical of a specific place and collection time.

Figure 33. Method of testing suspended dust quantities as per EN 12341:2024

After the end of exposure, the filter with PM particles is instantly removed from the tunnel and the sample installs another holder with a pure filter in the place of the old filter, simultaneously initiating another measuring cycle. The example of the sampler diagram is showed in the figure 34.

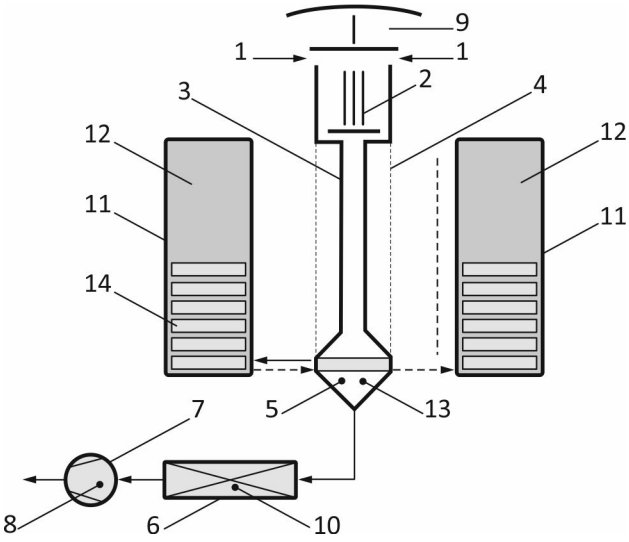

Figure 34. Sampler diagram

1 – air sample (Ta, Pa), 2 – impactor inlet, 3 – sampler housing, 4 – air inlet, 5 – filter holder, 6 – air flow measurement, 7 – pump, 8 – flow control unit, 9 – pressure and temperature measurement, 10 – pressure and temperature measurement (option), 11 – filter magazine, 12 – filter storage temperature measurement, 13 – temperature measurement near filter during absorption, 14 – filter

The essential element of the gravimetric method that substantially contributes to precision of the analysis is filter conditioning before and after exposure. In this process, the filter mass is determined a few times at stable temperature and humidity. The purpose is to determine the average filter mass before and after exposure, to be further used in suspended dust concentration calculations. According to the standard[1], the filter conditioning temperature must range from 19 to 21^{o}C, while relative humidity from 45 to 50%. Therefore it is unlikely to maintain the perfectly stable conditions in the laboratory for a long time, so they are basically variable. If this is the case, the question is how variable the filter mass is going to be when the humidity and temperature fall within limits defined by the standard. The problem is serious as to determine the amount of collected dust, the filter mass is measured twice $\rightarrow$ differential weighing, and testing may involve diverse filters: quartz, glass fibre, Teflon, nylon, polycarbonate, etc.

The view of essential elements of the RB 2.4Y measuring unit is showed in the figure 35. Before testing was initiated, all test filters had been marked with a QR code to assure their clear identification during testing. The testing cycle was performed according to requirements of the EN 12341 standard with regard to ambient conditions and permissible filter mass change tolerances. The conditioning and filter mass measurement cycle was controlled by the external PC software.

1 EN 12341:2014 ,, Ambient air - Standard gravimetric measurement method for the determination of the PM10 or PM2,5 mass concentration of suspended particulate matter”,

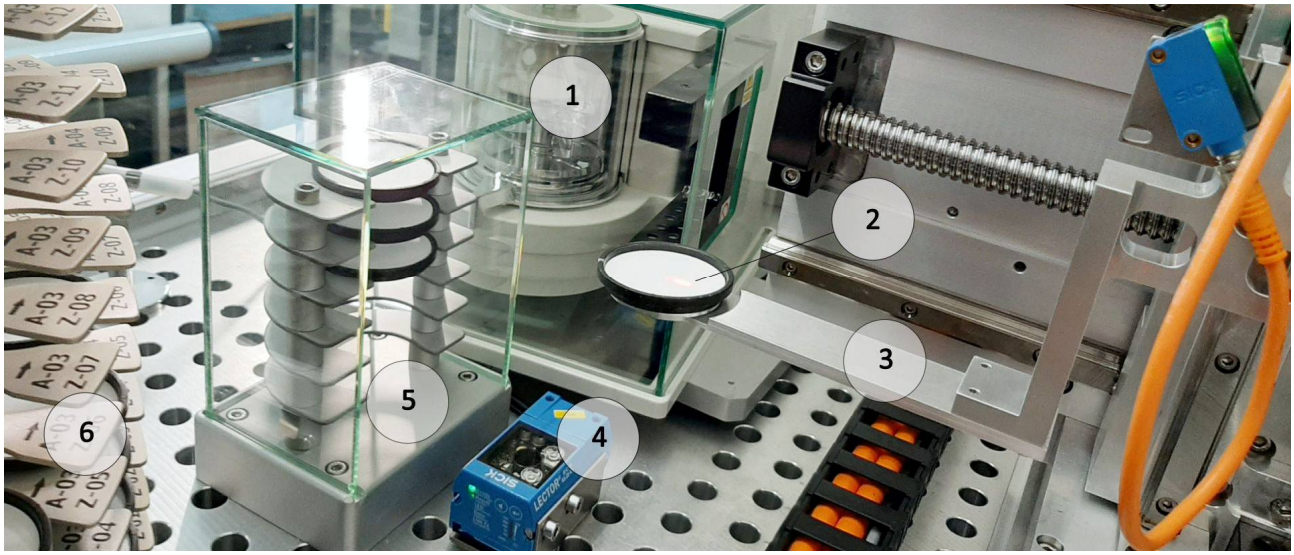

Figure 35. Filter verification and weighing - RB 2.4Y robotic unit

In the magazine (6), each of the filters was placed in the anti-static holder. Next the robotic arm (3) collected the filter (2) and carried it to the microbalance weighing chamber (1). The filter moved above the QR code scanner (4) and was recorded in the superior system as a currently weighed sample. The microbalance weighing chamber (1) opened automatically and the robotic arm put the filter on the microbalance weighing pan. Next the weighing chamber closed to carefully specify the filter mass. After the end of the weighing process, the microbalance chamber opened and the robotic arm put the filter in the magazine again. The reference filter and mass standard magazine (5) was used periodically to test potential sensitivity drifts of the robotic unit and the impact of ambient conditions on variability of reference filter mass.

The elementary reading unit for the mass measurement during testing was 1 microgram (10^{-6}g), but as mentioned before, the precision of the low-mass items weighing depends on the value of the random error only. During initial testing, it was found that the precision of measuring the mass of the QMA filter with a diameter of 47 mm ranged from 1μg to 2.1 μg. Before weighing, PTFE filters required deionisation in view of unbalanced static charges. The denionisation procedure must be adapted to the number of charges that are to be neutralised.

Throughout tests, it was found that variable conditioning (19.5 to 20.5^{o}C; air relative humidity: 40-45 %RH) had no negative influence on stability of mass measurement of filters used to collect suspended dust, regardless of the type of the filtering material. Variability of quartz fibre filter mass ranged from 148.84 to 150.34mg, and Teflon (PTFE) filters from 134.38 to 136.25mg.

With regard to glass fibre filters, the variability of masses was 89.61 ÷ 92.55mg, and polyamide membranes (nylon filters) was 73.16 ÷ 75.31mg. The filters made of polyester membrane (the so-called polycarbonate) were found to be variable in terms of mass, from 34.13 to 36.03mg. Based on the measurements, linear regression equations were estimated for test filters[2], and they demonstrate the variability of filter masses, depending on changes to temperature and humidity.

$$
\begin{aligned}
m_{QMA} &= m_{QMA} + 0.003RH + 0.015T \\
m_{GF/A} &= m_{GF/A} + 0.018RH + 0.055T \\
m_{PTFE} &= m_{PTFE} + 0.186RH - 1.473T \\
m_{NL} &= m_{GNL} + 2.242RH - 2.907T \\
m_{PC} &= m_{PC} + 0.004RH - 0.046T
\end{aligned}
\tag{9}
$$

Key:
RH – relative humidity (factor with rise of RH by 1%)
T – air temperature (factor with rise of T by 1°C)
PTFE – PTFE (polytetrafluoroethylene), PM2.5 PTFE W/PP
NL – polyamide membrane (nylon), NL 16
Cyclopore PC – polyester membrane (polycarbonate) CycloporeTM Polycarbonate
QMA 4.7CM 100/PK – quartz fibre QMA 4.7CM 100/PK
GF/A 4.7CM 100/PK – glass fibre GF/A 4.7CM 100/PK

The figure 36, 37, 38 show graphic interpretation of changes to masses of PTFE, NL, PC, QMA, GF filters with special regard to variability of conditioning.

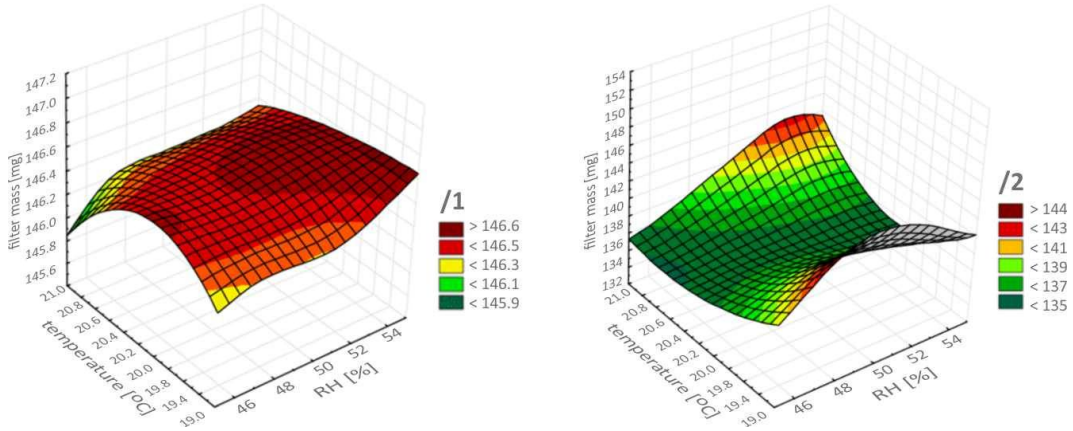

Figure 36. Dependency of changes to quartz fibre filter masses /1, PFTE /2 in relation to variable conditioning at humidity of 50±5% and temperature of 20±1°C

[2]Widziewicz-Rzońca K, Janas S, Błaszczak B et al. Advancing the understanding of pm filter mass stability: unveiling the influence of humidity and temperature. Scientific Reports of Fire University. (2023);1(88):7-26. https://doi.org/10.5604/01.3001.0053.9741.

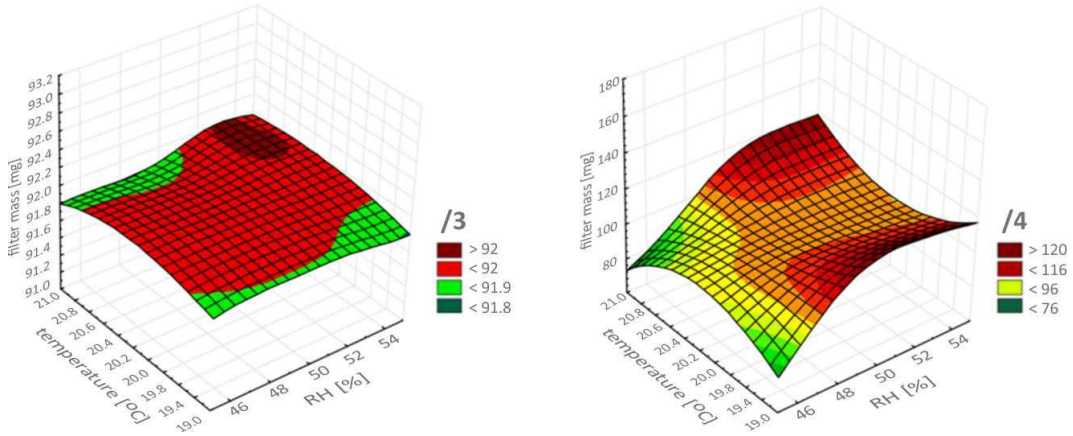

Figure 37. Dependency of changes to glass fibre filter mass /3, nylon /4 in relation to variable conditioning at humidity of 50±5% and temperature of 20±1°C

The best mass stability in variable ambient conditions was recorded for the polyester membrane filters (polycarbonate). Mass result deviation at 0.43 mg and quartz fibre, mass measurement deviation at 0.47 mg.

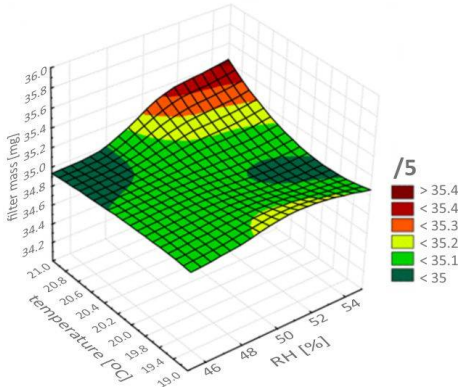

Figure 38. Dependency of polycarbonate filter mass changes /5, in relation to variable conditioning at humidity of 50±5% and temperature of 20±1°

To illustrate, for quartz fibre filters and permanent temperature of 20°C and relative humidity of 50%, the filter mass is on average 146.01 + 0.003 * 50 + 0.015 * 20 = 146.01 + 0.15 + 0.3 = **146.460 mg**. When the relative humidity rises by 5% at the fixed temperature of 20°C, the filter mass will be 146.01 + 0.003 * 55 + 0.015 * 20 = 146.01 + 0.165 + 0.3 = **146.475 mg.** The mass gain for the quartz filter is therefore 0.015 mg. At the same temperature, the air relative humidity gain by 5% will cause the filter mass to rise in the following way:

- PTFE by 0.93 mg (93 µg);
- glass fibre by 0.09 mg (9 µg);
- polyamide membrane by 11.21 mg (112 µg) and
- polyester membrane (polycarbonate) by 0.02 mg (20 µg).

The directional factors for regression dependencies show that raise in the temperature from 19°C to 21°C, with regard to quartz fibre, glass fibre and polycarbonate filters, has a bigger impact on precision of filter mass measurements than air relative humidity rise from 45% to 55%. Analysing likelihood values specified for directional factors in regression equations ($p < 0.05$), the conclusion is that the error deriving from variability of the filter mass as a result of changes to conditioning may be essential for calculation of suspended dust concentrations.

The second stage of scientific research focused on assessing stability of filter masses is 2021/42/E/ST10/00209 project, ID 526286, entitled: ,, Water – high-importance matter for aerosol mass measurement uncertainty”[3]. The mass measurement experience demonstrates that the larger the surface of the weighed item, the bigger the impact of the air movement → disturbance, affecting measuring result. It has been confirmed through a series of own research done with the use of weighed filters and various weighing systems, be it automatic and manual. For this reason the research project adopted the UMA 2.5Y.FC automatic system with a built-in air-conditioner, and filters are weighed in steel containers (fig. 31). The test filter mass is usually relatively low, so precision of measurement depends on the random error only.

Figure 39. UMA 2.5Y.FC – Filter mass variability testing automatic system

UMA 2.5Y.FC, maximum load 2g, elementary reading unit d=0.001mg, Max. number of filters 24
Compliance with EN 12341:2014, Testing under 2021/42/E/ST10/00209 project.

[3] Chyzhykov, D., Widziewicz-Rzońca, K., Błaszczak, M. et al. Automatic weighing system vs. manual weighing precision comparison in PM-loaded filter measurements under different humidity conditions. Environ Monit Assess 195, 1393 (2023). https://doi.org/10.1007/s10661-023-11939-7

The UMA 2.5Y.FC automatic unit diagram is showed in the figure 38. The filters were located in containers (6.a,b) that were then placed in the magazine. The set humidity and temperature are maintained inside the weighing space (8) by the moisturisation system that operates in the feedback with ambient conditions sensors. The rotational movement of the filter magazine (7) is allowed through the automatics system (4) that aside from rotation is responsible for up and down movements, if the filter is in the „weighing" position. Air purity inside the weighing chamber is assured by HEPA filters (5) that are situated at the air inlet and outlet from the weighing chamber.

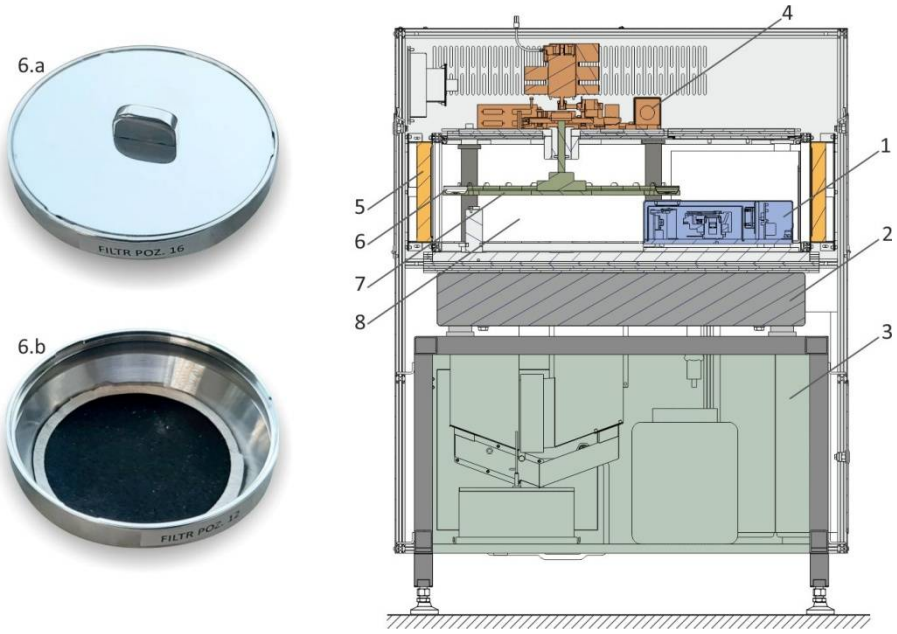

Figure 40. Measuring unit diagram

1 – weighing module (mass measurement), 2- anti-vibrating base of measuring unit, 3 – moisturisation and temperature control unit, 4 – automatics unit, 5 – HEPA filter, 6 – filter container (a – container view, b – filter inside container), 7 – filter magazine, 8 – filter conditioning chamber.

The weighing module (1) together with the entire structure is positioned on the anti-vibrating stone base (2). The measuring cycle in the testing process is programmable in the computer application, where it is necessary to define the number of cycles and filter position in the magazine. The mass of these filters is to be verified. The research project number 2021/42/E/ST10/00209, ID 526286, entitled ,, Water – high-importance matter for aerosol mass measurement uncertainty" is presently in the testing phase, and the above-stated tests will be presented in the form of a scientific publication afterwards.

6.2. Magnetic susceptibility of alloys in medical implants

The magnetic susceptibility is one of the basic physical quantities describing magnetic properties of the matter. Generally speaking it is an ability of the medium to become magnetised under the influence of external magnetic field. This value is directly proportional to the content of magnetic particles that are available in the test sample. The magnetic susceptibility (κ) is defined as a ratio of volumetric magnetisation (M) induced in the material with susceptibility (κ) to the intensity of the magnetic field H, causing the magnetisation:

$$\kappa = \frac{M}{H} \tag{10}$$

where: κ - volumetric magnetic susceptibility
M – volumetric magnetisation of medium [A/m]
H – intensity of magnetic field [A/m]

When it comes to medical application, excessive magnetic susceptibility of implants made of metal alloys poses a true threat during tests that adopt imaging techniques, e.g. magnetic resonance imaging (MRI). It is one of the basic imaging diagnostic techniques that unlike traditional X-ray radiography does not expose the organism to potentially harmful impact of X-ray radiation. MRI is highly useful for detection of pathological changes in tissues, particularly those shielded by bones. The image in this method is generated by placing the subject (patient) in a strong magnetic field, and the very signal is produced by exciting the sample via radio waves.

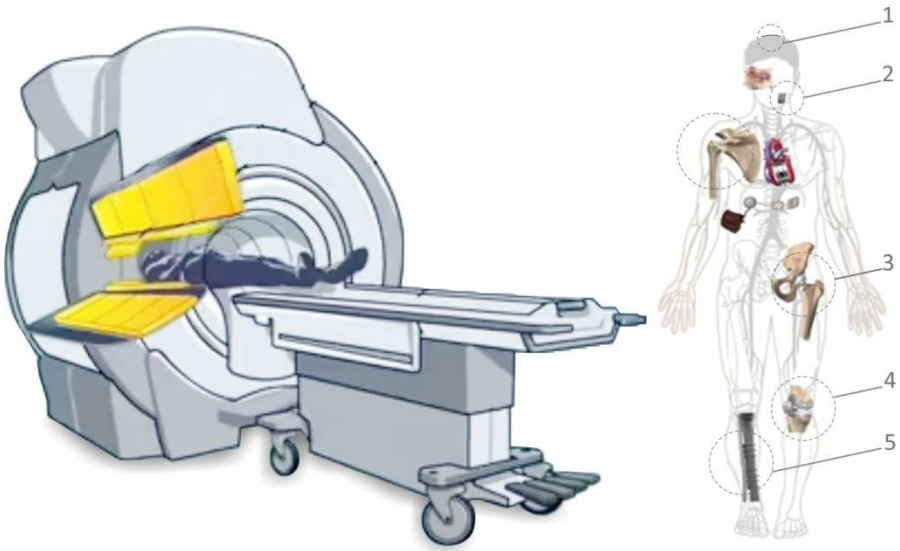

Figure 41. Magnetic resonance – medical implants

1 – stainless steel 316L, Ti / Ti alloys, 2 – Co/Cr/Mo alloys, stainless steel 316L, 3 – stainless steel 316L, Co/Cr/Mo alloys, Ti / Ti alloys, 4 – stainless steel 316L, Co/Cr/Mo alloys, 5 – stainless steel 316L, Ti-Al-V alloys
Source: https://science.howstuffworks.com/mri.htm; http://doi.org/10.13140/RG.2.1.3461.1921

As mentioned before, the MRI examination using standard medical equipment entails the impact of a strong magnetic field on the subject, ~ 1.5T. The precision and detail of the image widely depends on intensity of the magnetic field generated inside the body; thus the stronger the field, the better the image. For this reason new-generation resonances operate at the intensity of ca. 3T.

One of few contraindications for MRI testing may be possession of various metal implants or other medical equipment by the patient. It is because of a high magnetic susceptibility of metal elements, which in case of implants may lead to distorted MRI image near such an implant, or may cause the implant to heat up during tests, or in extreme situations may cause the implant to relocate. Currently metal elements of implants mainly involve stainless steel, CoCr alloys, titanium alloys, zirconium alloys. The material of the implant must be highly resistant to corrosion in the bodily fluids, highly bio-compliant, known for Young modulus and density similar to the replaced part of the bone and, following the previous statement, low magnetic susceptibility.

Considering the current metallurgical technique advancement, the aforesaid requirements are best met by titanium and zirconium alloys. The CoCr alloys or iron alloys are much heavier, demonstrate certain cytotoxicity and have a high Young modules, which may lead to weak bones in the long term, as per the Wollf's law. Their production is however expensive in view of high affinity to oxygen of the aforesaid elements and very high melting temperature. Additionally, to lower the magnetic susceptibility, the alloy must have a suitable phase structure, e.g. in the case of Zr, ω-Zr phase should exist.

One of the methods of lowering costs of producing such alloys is powder metallurgy with the use of SPS or HPHT method, where it is possible to form moulded pieces out of these metals at temperatures below 1200 °C. Such tests are currently carried out by the Department of Non-Ferrous Metals at the AGH in Cracow.

The measurement of the magnetic susceptibility of the substance used to produce the medical implant can be made in two ways, magnetometrically or gravimetrically. The magnetometric measurement using the SQUID magnetometer (superconducting quantum interference device) requires the measurement of the sample magnetisation, depending on the magnetic field applied. At present one of the most precise devices used to test magnetic properties of materials is the MPMS-SQUID-VSM magnetometer. In this magnetometer, the principal element is the SQUID superconducting quantum interference device that adopts the quantum interference of current carriers to detect and measure minor changes of magnetic induction.

Speaking of weighing methods, the force of magnetic field influencing the sample is measured → Gouy's, Faraday's or Evans balances. For the purposes of calibrating weighing methods, compounds of a stable and well-known magnetic susceptibility are used, such as $HgCo(NCS)_4$ susceptibility 16.44×10−6 (±0.5%) CGS at 20 °C $[Ni(en)_3]S_2O_3$ susceptibility: 1.104 x 10−5 erg G^{-2} cm^{-3}.

The diagram of the modified weighing method adopting the MYA microbalance by Radwag is showed in the figure 40. The weighing unit design allows specifying the mass of the test sample and determining its magnetic susceptibility as per the OIML R111-1[4] standard. Before putting the load on the weighing pan, the measuring unit is balanced. **Mass measurement idea:**

While the load is positioned on the weighing pan (A), the location sensor is knocked out of the previous stable condition as a result of the gravitational force impacting the load as per the relation: F=mg. The measuring system analyses the measuring signal and generates the compensating signal in order to return the measuring unit into its original position of balance. The factory adjustment of measuring transducer indications in relation to certified mass standards allows presenting the generated compensating signal as a result of weighing, expressed in mass units, in grams. Thanks to such a method, it is possible to determine the exact mass of the test sample, both net and gross.

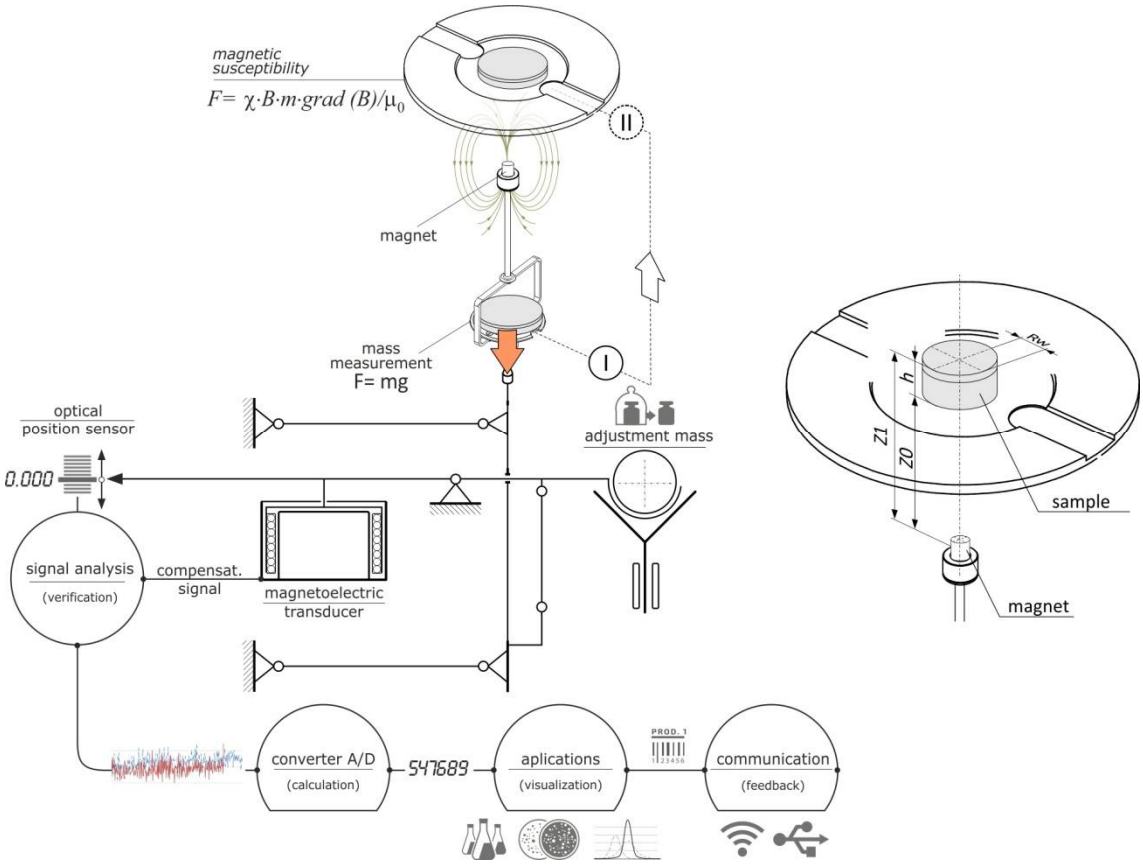

Figure 42. Magnetic susceptibility testing weighing unit diagram

[4] Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3. Part 1: Metrological and technical requirements

Magnetic susceptibility measurement idea:

When the sample is put into the position (B), the measuring unit indication changes as a result of the interplay of magnetic particles in the sample structure and the permanent magnet field that is located in the upper holder of the weighing pan, as per the relationship (11).

$$F = \chi \cdot B \cdot m \cdot grad\,(B)/\mu_0 \tag{11}$$

The view of the microbalance with a special holder of the weighing pan and magnet is showed in the figure 43. Magnetic susceptibility of samples of various structure may be measured in accordance with the methods referred to in OIML R111-1 or may be referred to other reference methods in which the magnetic susceptibility is defined.

Figure 43. MYA 5.5Y microbalance – magnetic susceptibility measurement

MYA 5.5Y microbalance, maximum load 5g, elementary reading unit d=0.001mg, OIML certificate

Interfaces: USB-A ×2, USB-C, HDMI, Ethernet, Wi-Fi®, Hotspot

7. Innovativeness as a source of technological progress

Innovation encompasses any actions related to preparation and production of new or improved products that clearly stand out in relation to other equivalents available on the market. Innovativeness can be understood as higher quality, impressive use ergonomics, long-term stability, accuracy and precision of measurements, as regards 5Y-series balances. It is not easy to prepare a better product as you need to find the balance between market expectations and project economics → profitability. Considering its long-lasting experience in producing weighing systems, Radwag has always prioritised innovativeness, although the term was not so popular in the past. In some cases innovativeness pertains to operation and design of devices, which is conspicuous and clear to any recipient, and otherwise innovation may be hidden inside the balance, yet giving substantial metrological benefits. Below are two examples of innovations that come as protected patents.

7.1. Automatic mass comparator, Pat. 228368

Comparison has always been obvious as various products would be compared in order to emphasise similarities or differences. In retrospect, such a procedure was performed manually, but then turned automatic as the technology grew → meaning efficiency. Analogical examples apply to mass measurement metrology, especially when highly precise measurements are required, while maintaining process efficiency. Such an example is the automatic mass comparator used to compare mass standards. Radwag has elaborated a special design of the device to automatically compare mass standards with higher-rank standards, fig. 44.

Figure 44. UMA 100 mass comparator
UMA 100.5Y, maximum load 110g, elementary reading unit d=0.001mg, Max. number of standards 36

Test and reference weights are initially positioned on the comparator feeder (2), integrated in the form of a circle situated and rotated horizontally and lifted using the vertical-motion electric-motor drive and transmission with a cam (19) on the output axis. During rotation, the cam (19) directly influences the lever (20) which the feeder circle (2) is suspended on, the circle is lowered gravitationally vertically so that its angular position in the horizontal plane is carefully designated using the positioning mandrel (11), which allows using the ribbed bases under weights (8) taken by the weighing pan ribs, that go between base ribs under the weight (8) while lowering the feeder circle (2).

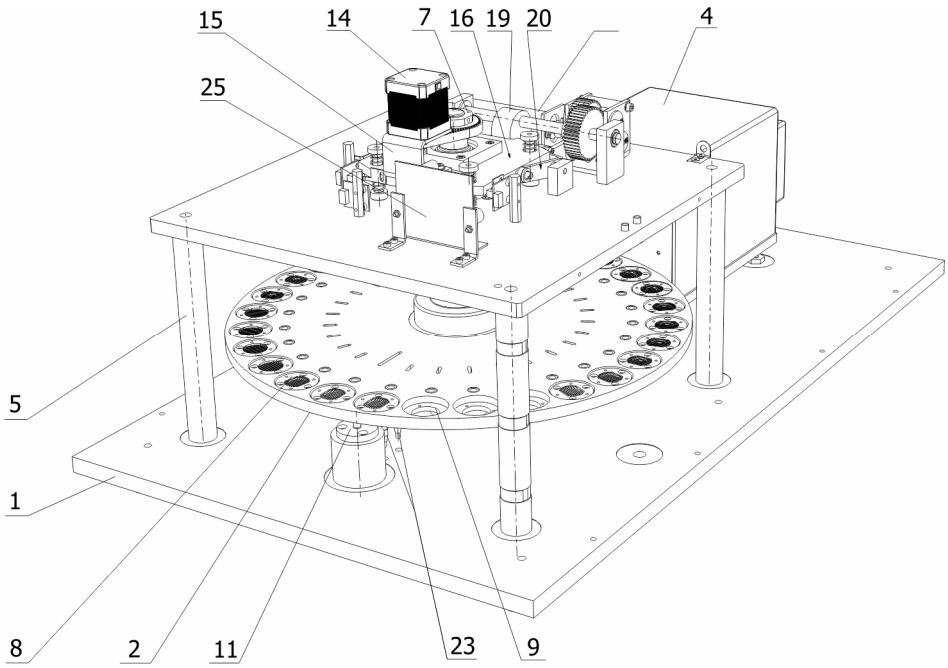

Figure 45. UMA mass comparator functional diagram

The UMA automatic comparator is used in the Radwag Metrology Research and Certification Centre. Thanks to this solution, the testing time has been substantially shortened, and precision of measurements has considerably improved in relation to manual measurements. The comparison process is initiated and supervised remotely, which is followed by a report containing all important data. The example of the comparison report (partially) is showed below.

---------- Comparator ----------

Operator	AK
Full name	AK
Report no.	C/05/02/24/01/47
Start date	2024.02.05 01:47:39
End date	2024.02.05 02:01:21
Test weight	5 g SN:30151 GBD
Order number	559/24
Test weight number	30151
Test weight position	B18
Reference weight	5 g E1 G0601916 SN:G0601916
Mass	4.999955 g
Reference weight class	E1
Reference weight position	A12

N	A	B	B	A	D
1	0,0000004	0,0000596	0,0000597	0,0000004	0,00005925
2	-0,0000001	0,0000594	0,0000595	-0,0000002	0,00005960
3	-0,0000007	0,0000592	0,0000592	-0,0000005	0,00005980
4	0,0000003	0,0000602	0,0000598	0,0000003	0,00005970
5	-0,0000001	0,0000595	0,0000596	-0,0000001	0,00005965
6	-0,0000005	0,0000593	0,0000593	-0,0000004	0,00005975

Average difference	0,0000596250 g
Standard deviation	0,00000020 g
Number of cycles	6
Method	ABBA
Min. temperature	21.45 °C
Max. temperature	21.48 °C
Min. humidity	33.3 %
Max. humidity	33.4 %
Min. pressure	985.3 hPa
Max. pressure	985.4 hPa

Signature

7.2. Calibration with internal weight for electronic balance - Pat. 226501

Proper operation of each balance would not be possible if not for periodical adjustment whose major goal is to check and correct the balance sensitivity by comparing the adjustment mass weighing result with its known value. Speaking of professional balances, the adjustment mechanism is installed inside the balance, forming its integral part, to assure the precision of measurements, regardless of the ambient conditions. The design of such adjustment units can be protected by the patent law, as in PS and AS balances by Radwag.

Figure 46. View of PS 8100.5Y.M balance

PS 8100.5Y.M, maximum load 8100g, elementary reading unit d=0.01mg, verification unit e=0,1g
OIML certificate, Interfaces: USB-A ×2, USB-C, HDMI, Ethernet, Wi-Fi®, Hotspot

From the metrological point of view, the adjustment mass is not calibrated, as opposed to periodical calibrations of mass standards. The calibration procedure applies to the balance, its indications and this is the measure of correct operation of the adjustment mechanism in terms of mechanics, IT and metrology. It must be stressed that procedures that describe the operations of the adjustment system are relatively complex and sometimes used in combination with other mechanisms to evaluate the impact of the working environment on precision of balance indications → Autotest. It is applicable with regard to high-resolution balances, such as MYA microbalances and mass comparators.

The size of the adjustment mass differs, depending on the maximum loading capacity of the balance. Whether such a system works correctly can be assessed using the GLP Autotest that determines the adjustment mass weighing precision in the automatic cycle. The view of the PS laboratory balance adjustment mechanism is showed in the figure 47.

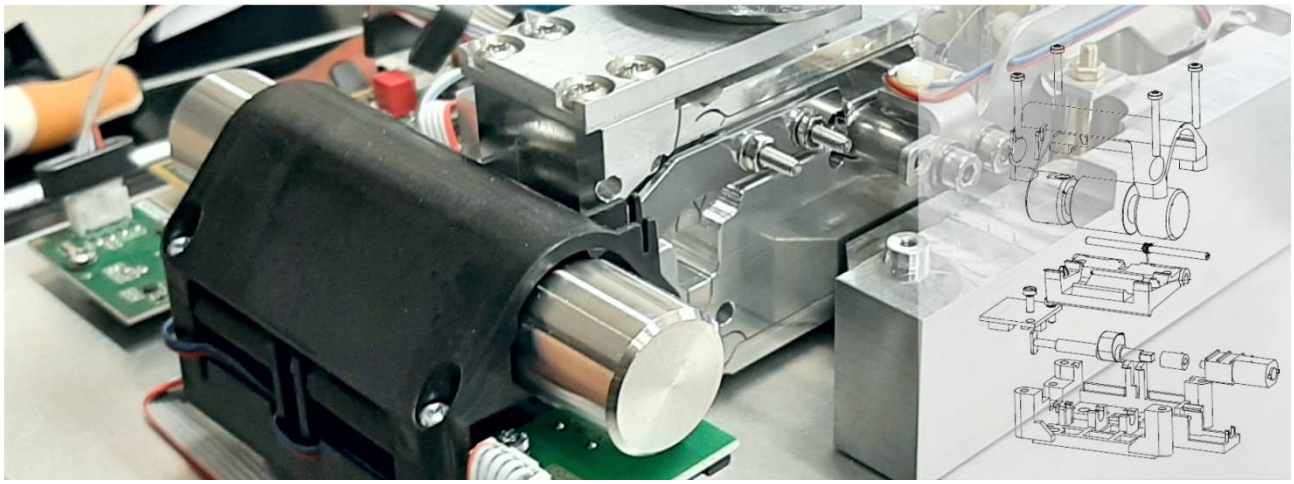

Figure 47. PS balance adjustment mechanism in the balance with a monolithic measuring unit

During production and inspection, correct indications of the balance are compared to certified external mass standards. It also confirms correct operation of adjustment. The schematic diagram of the adjustment mechanism is showed in the figure 48.

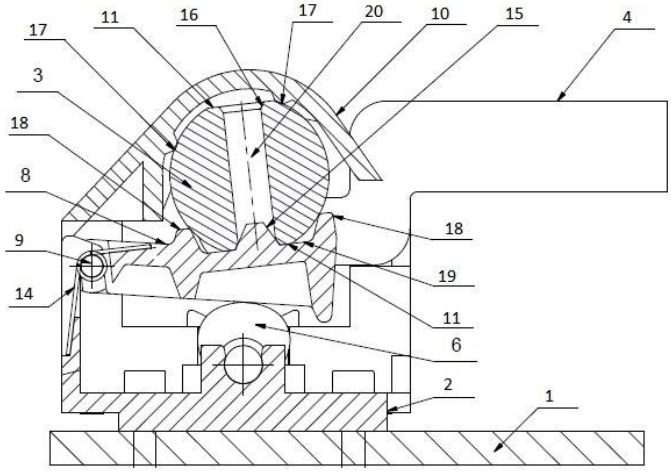

Figure 48. PS/AS balance adjustment mechanism functional diagram

1 – balance base, 2 – adjustment unit housing, 3 – adjustment mass, 4 – gravitational force transmission arm, 6 – adjustment mass lifting unit, 10 – housing, 15,20 – adjustment mass positioning

8. Weighing system certification as a guarantee of quality and reliability

Reliable manufacture of technologically advanced measuring equipment, such as XA 5Y balances, must be performed in line with specific processes and methods. It is assumed that the final product should be of high quality and good metrological parameters. It is not a secret that every production process entails a certain permissible manufacture variability that can still guarantee the expected quality of the final product. Such a Quality Management System seems to be the optimal solution in terms of economics (costs) and metrology (quality). Many times the manufacturer is subject to supervision through periodic audits conducted by independent inspecting units. Their task is to confirm that the manufacturing process is carried out in accordance with specific procedures → ATEX safety that eventually assures a high-quality product → balances, weighing systems, dynamic balances, etc.

On the other hand development of weighing systems entails introduction of innovative solutions to the existing structures → MYA microbalance 5Y terminal, or creation of new balance models → X7 series of AS analytical balances. As we know some weighing systems are used in the field of legal metrology that requires certification by the Notified Body. The legal requirements related to this process can be accessed in the OIML R 76-1 document entitled,, Non-automatic weighing instruments Part 1: Metrological and technical requirements – Tests", or EN 45501 standard entitled ,,Metrological aspects of non-automatic weighing instruments". To comply with OIML (The International Organization of Legal Metrology) requirements, it is necessary to carry out own tests → Research Laboratory, that are then verified by the Notified Body Laboratory. As part of these tests, mass measurement precision tests and electromagnetic compatibility tests are performed.

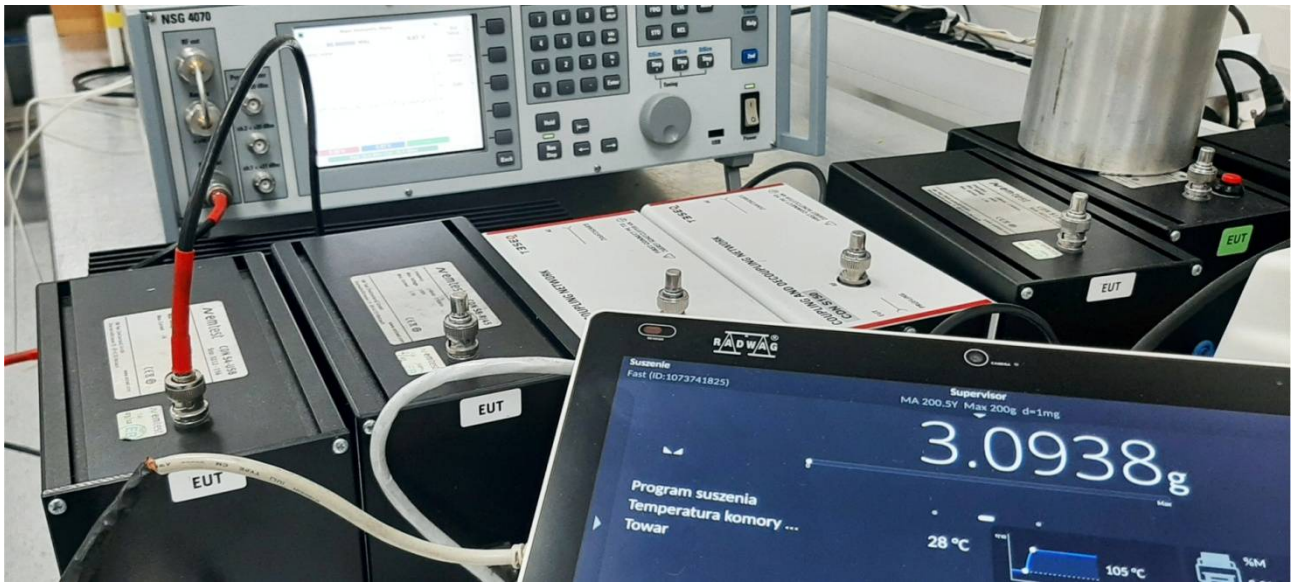

Figure 49. Testing the electromagnetic compatibility of the balance with 5Y terminal Test: resistance to radio signals

It is possible to put forward a claim that certification is not obligatory but is surely able to confirm high quality of balances and weighing systems. Such an approach is a standard policy in Radwag that certifies its products in European bodies, e.g. the Czech Metrology Institute (CMI), GUM (Central Office of Measures), in the past in the Nederlands Meet Institute (NMI), and also out of the European Union, e.g. in Brazil, Morocco, China, the United States of America, etc.

The tests performed by the Radwag Research Laboratory are used not only for the purposes of balance certification but primarily provide suggestion as to further modifications in order to increase quality and ergonomics of balances produced. Eventually the measured value is always the mass measurement, but from the structural point of view there are also other factors that matter, for example stability in time, resistance to external factors, external condition change signalling, etc. All of these matters are subject to verification and periodical re-certification, when changes are significant, as in the case of X2 balances supplied with a much larger balance screen, see fig. 50.

Figure 50. PS 8100.X7.M balance – availability of a larger (7”) screen

PS 8100.X.M, maximum load 8100g, elementary reading unit d=0.01mg, verification unit e=0,1g
Touch-screen display, OIML certificate, Interfaces: USB-A ×2, Ethernet, Wi-Fi®, Hotspot

9. Cobot in mass measurement

Automation in mass measurements can be provided in several ways but it is usually dedicated to a specific goal in terms of efficiency, ergonomics, precision of measurement in the X-Y-Z plane → RMC 1000.5Y robotic system. This solution proves ideal when positions of test items are fixed in the defined space. Please note that the working space is always isolated so that there is no way the operator can interfere → security.

A virtually unrestricted freedom with regard to relocation of test items is obtained when the robotic arm called „Cobot – collaborative robot" is used. The task of the arm is to cooperate with the operator in a safe and effective way. In this respect the working environment may be shared by the operator and cobot, as defined. The example of cobot installed in the balance work stand is showed in the figure 51.

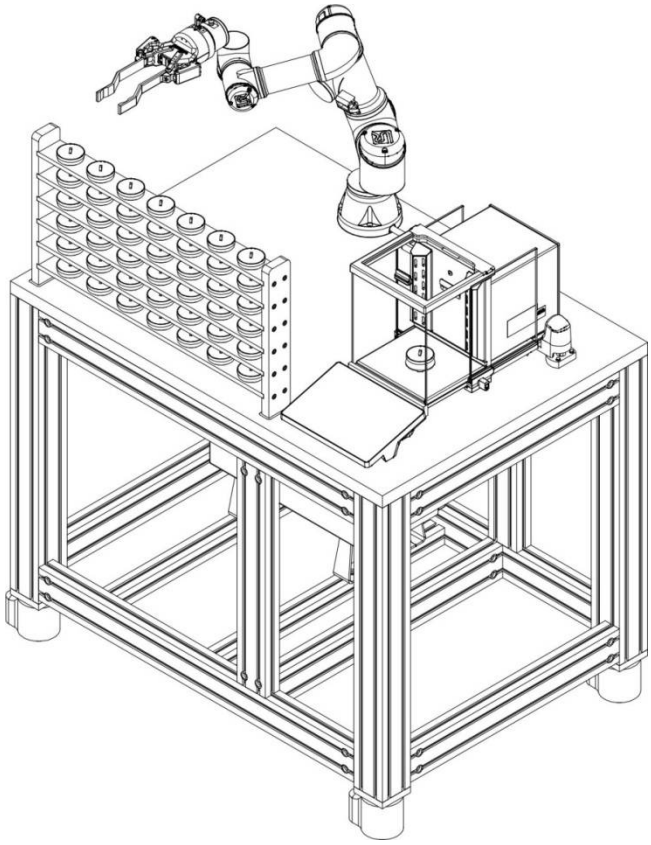

Figure 51. „Cobot" robotic unit in mass measurement

At present Radwag has initiated research works whose aim is to specify the mass-measurement-related fields in which it is possible to use the Cobot automatic units.

Following our experience, we may put forward a claim that the use of measuring units coupled with Cobot is possible to a broad extent but each situation must be considered on a case-by-case basis. It applies to the shape of the transferred item, its mass and Cobot operation algorithm.

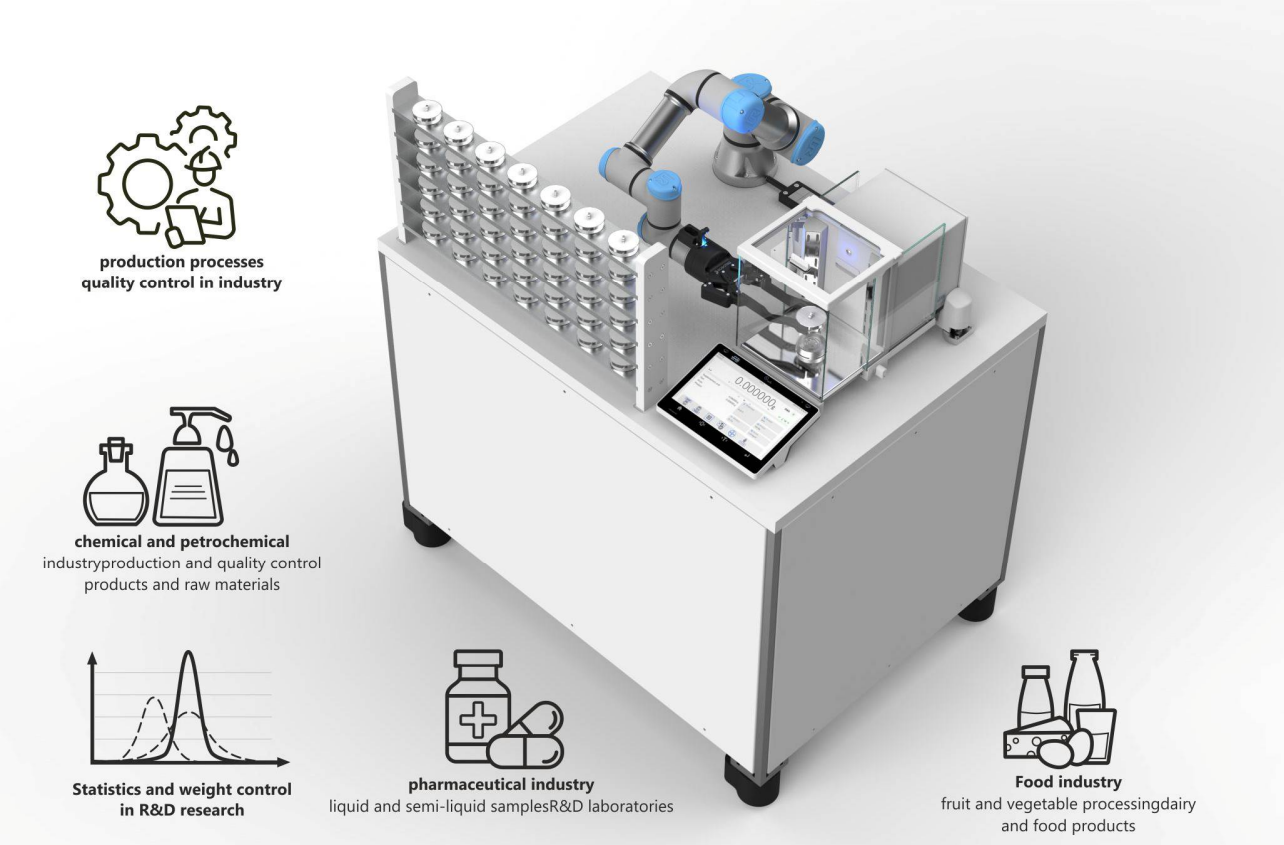

Figure 52. Fields in which Cobot may be used for mass measurement

10. Balance and weighing system metrological verification

Looking from the metrological and normative[5] point of view, there is no doubt that every measuring device, including balances, must be periodically checked as per the schedule. When it comes to balances, the above-stated procedure is based on one or several mass standards and requires the comparison of balance indications with a known certified value of the standard. As a rule the permissible tolerance +/- is established and the standard weighing result must correspond to the tolerance said.

[5] ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories, EN ISO 9001:2015 Quality management systems – Requirements)

The metrological verification entails the use of mass standards for the purposes of assessing the precision of measurement → systematic error. It is a normative approach recommended by OIML with reference to all non-automatic balances. It must be stressed that in reality the mass measurement is made for completely different items than steel weights. For this reason you can assume that precision of measurement will be different, particularly when the item you weigh has a much larger surface → filter.

Figure 53. Non-automatic balance metrological verification

Automatic units clearly differ in terms of design when compared to standard hand-operated balances and scales. Sometimes, during periodical inspection, it is not possible to use typical mass standards in view of the shape of the weighing pan or location of the weighing module. If this is the case, it is necessary to use dedicated mass standards whose shapes are suited to the design of the weighing unit weighing pan, as in RB 2.5Y.

Figure 54. RB 2.5Y automatic unit – mass standard and filter magazine

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