Micro scale measurements

Practical guidelines for users of MYA / XA series microbalances - ed. II/2022

dr Sławomir Janas

Radwag metrology Research and Certification Center, Poland

e-mail: radom@radwag.pl; http://radwag.com,

The paper presents the basic issues concerning the installation and use of microbalances manufactured by Radwag Wagi Elektroniczne. In the first part of the study, attention was paid to the installation site and environmental conditions in order to indicate potential threats that may negatively affect the mass measurement process. In the following, the basic procedures used during the metrological evaluation of microbalances, such as the repeatability test, accuracy test and built-in diagnostic tools that can be used during such tests, are shown. The most common processes in mass measurements are also discussed, with an indication of the factors related to the microbalance and the environment, which are important for quality reasons. The last section addresses the compatibility issue with legal requirements and those applicable under GMP, GLP.

Keywords:
mass measurement, mass, microbalance, pharmacy, adjustment, weight analysis, accuracy, measurement precision, validation, weighing, GMP, GLP.

Radom 2022

Edition II

RADWAG Wagi Elektroniczne
26-600 Radom, Toruńska 5
Phone: +48 48 3848800, fax: +48 48 3850010
e-mail: radom@radwag.pl
http://www.radwag.pl
NIP: 796-000-03-27

TABLE OF CONTENTS

Author’s note

Contemporarily manufactured equipment provides intuitive operation holding the users back from thorough analysis of its operation. This phenomenon is evident and at the same time desired, owing to rapid and dynamic technological development. New inventions and PC software stand for ergonomics of use and simplicity, both of which are a result of advanced know-how involving group of engineers. When it comes to determination of sample weight by means of high resolution balance, the case is likewise. The balance seems to be unsophisticated measuring device, whereas in practice it comprises extremely precise transducer, an outcome of hours of work of R&D departments.

Quite simple, when approached from a manual perspective, weighing process, depends on several factors powerfully effecting a weighing result. Both, the affecting processes and relation between them are not commonly known to a regular user. This document provides relevant guidance that may turn out to be useful.

dr Sławomir Janas
Research Laboratory Manager
RADWAG Wagi Elektroniczne

1. Introduction

Measurement usually lacks accuracy which is mainly caused by imprecision of measuring equipment and applied methods. Such situation concerns electronic balances too, regardless of their design or resolution. Satisfying results do not depend on the construction exclusively, the ambient conditions and used methods are of the great importance here as well. The users practically do not notice any problems while using low resolution balances (< 2 000 000 d). In contrast, for measurement performed by means of high resolution equipment, it happens that user expectations collide reality, i.e. not good enough measuring capacity possible to be achieved for particular ambient conditions. Being able to understand the mechanism and processes occurring in-course of measurement, is a key factor allowing to analyse and design such weighing systems and methods that may satisfy even the most demanding requirements.

2. Installation at the workstation

Installation is a process in course of which balance operation parameters and working conditions parameters are optimized in order to provide possibility of obtaining satisfying results, i.e. results within specified tolerance. The respective tolerance limits shall be determined with regard to actual requirements resulting from analysis of sample weight variation, accuracy of technological processes, etc. Taking the above into account it usually happens that the reading unit for a particular balance is one order of magnitude lower when compared to the demanded measuring accuracy. Such technique must be applied due to an effect of repeatability and linearity errors and due to errors being a result of an applied method.

If the said above requirements have not been defined then conformity with parameters declared by a manufacturer shall be tested. Assuming that it is possible to obtain exactly the same results is quite an optimistic approach. One of the most decisive factors when it comes to the test result are ambient conditions of the workplace, it is almost impossible to provide exactly the same conditions twice. Being aware of relation between ambient conditions and the test result is a starting point for discussion on optimization of the following: balance, working conditions, methods. The said three factors must be optimized in order to assure the most precise results. Exchange of information and support of the manufacturer are basis of optimization.

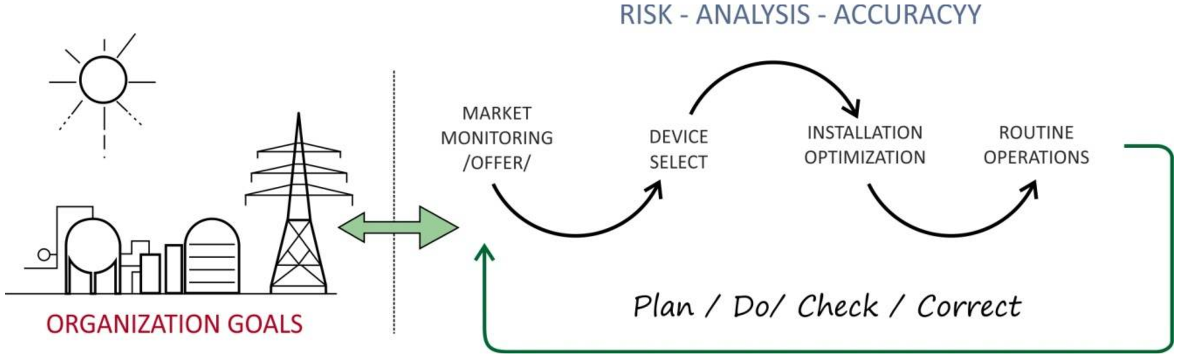

Fig. 1. The process of adapting the balance to the requirements of the weighing process

2.1. Workstation

Workstation localization is usually determined in advance, it is a rare treatment that a user may decide himself/herself where to place it. It is more frequent to optimize the workplace using draft shields rather than to move the balance elsewhere. While selecting a respective place for the balance, microbalance especially, it is necessary to take the following into account:

- The workstation cannot be located near air conditioning devices being a source of air drafts. A solution to strong drafts might be air stream dispersion, performed by means of more than one discharge channels.

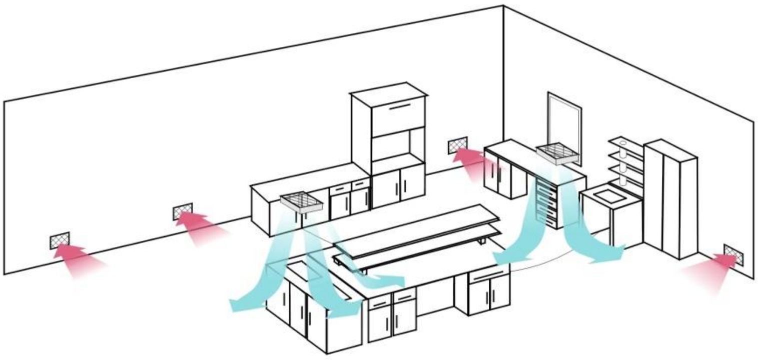

Fig. 2. Laboratory – air conditioning system

- The balance workstation must be stable, the device shall rest on a ground-supported counter rather than on a counter fixed to the wall.
- The device cannot be located in drafts neighbourhood.
- It is not recommended to place the balance next to windows.

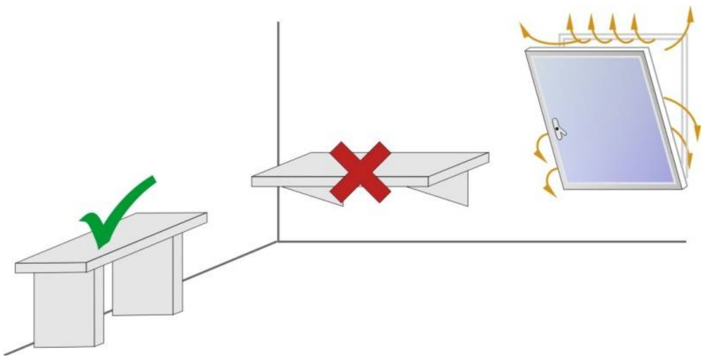

Fig. 3. Workstation location

- The laboratory room size shall be respective to number of employees and amount of carried out works.
- Ground floor is more recommended than other locations.

2.2. Acclimatization

In course of installation balance indications and working conditions are observed. One of the most significant issues is the amount of time needed for balance acclimatization. The said time is a period within which balance temperature becomes stable for a particular place of operation. The greater the difference between balance temperature and workplace temperature, the longer the temperature stabilization period. Actually the period about few hours long, therefore PRECISE weighing tests are possible to be performed on the next day. The below figure presents balance temperature variation in relation to repeatability of indications.

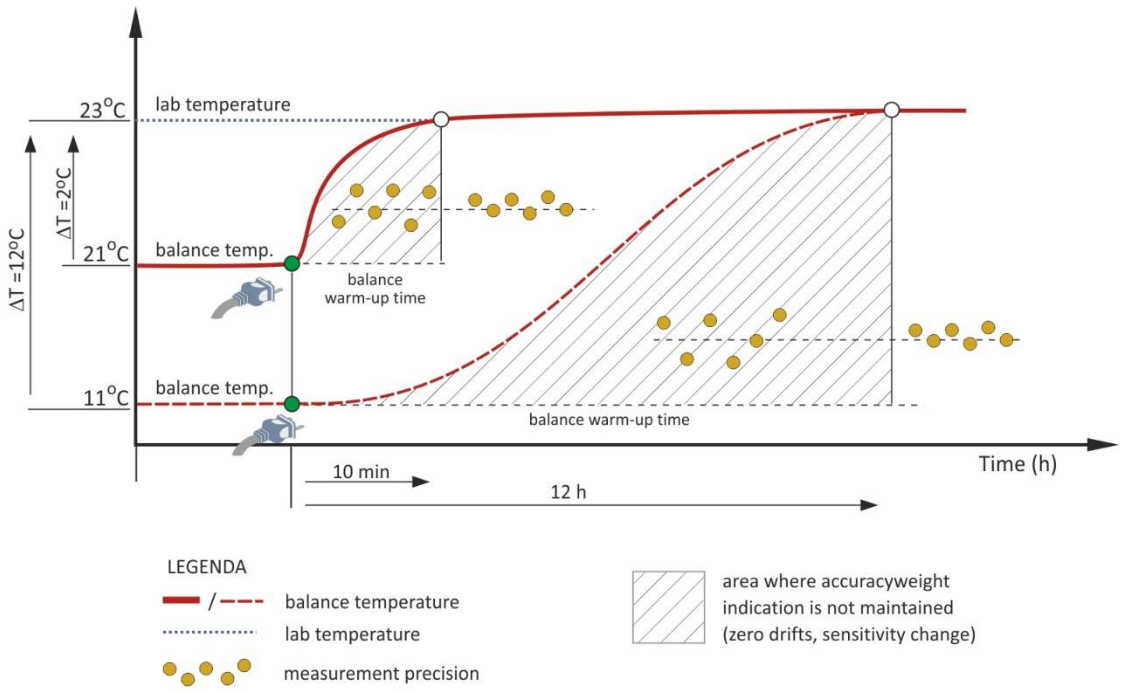

Fig. 4. Balance parameters change over acclimatization period

Correct balance operation requires thermal stabilization of the whole design. Such thermal stabilization is reached within considerably long period of time, for microbalances it is even more than 10 hours. It is possible to use balances during the stabilization period, nevertheless one shall be aware of the fact that for such a case, metrological important parameters may significantly differ from what the manufacturer declares.

After plugging the balance to mains, its user is certainly interested in its look, but evidently it is its operation that matters the most. The first assessment usually concerns two parameters, namely:

- Stability of indication,
- Ability of getting back to zero.

Throughout acclimatization both of these parameters may not be comprised within permissible limits. Certain instability of indication may be expected, its degree dependent on temporary drifts. Upon taking the load of a weighing pan the balance indication does not have to equal zero. The noticed deviations are not too high, few reading units usually. While operating the balance during acclimatization it is recommended to use zeroing button more often. More frequent adjustment is as much required, this owing to possible change of balance sensitivity (internal heating process).

It is assumed that for stable conditions the acclimatization process shall end upon reaching stable temperature. What if working conditions are not stable, e.g. considerable temperature difference during day and night?

It is definite that internal temperature shall oscillate like ambient temperature does, with certain delay. External microbalance design isolates its weighing module from the working environment, therefore even significant temperature variations may be of marginal importance. Due to divergent dynamicity of this phenomenon it is difficult to estimate the effect it exerts on the measurement result. Therefore it is better to maintain stable ambient conditions.

3. Operating conditions

By operating conditions one shall understand any factors relating to the balance working environment, these are:

- Temperature variation dynamics,
- Moisture content variation dynamics,
- Speed and direction of air flow,
- Possibility of vibrations occurrence,
- Unbalanced electrostatic charges around balance and a sample.

Some technical documents contain information on the above listed factors, unfortunately not many users are able to record dynamics of their variations. Such detailed control is not necessary for most measuring devices. This influences the adopted approach.

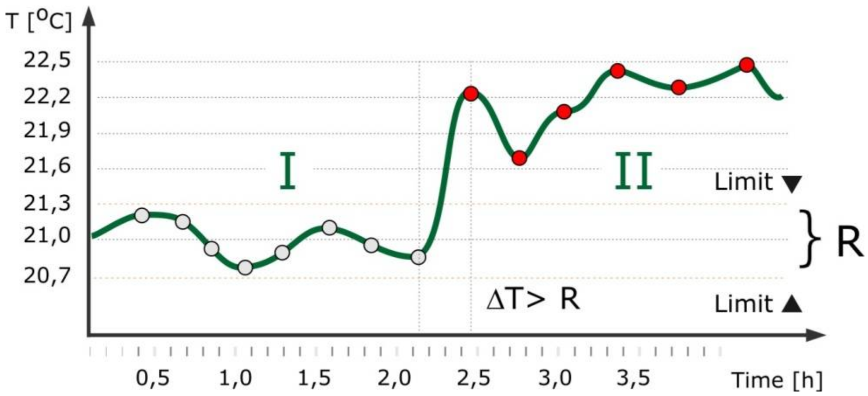

Fig. 5. Temperature variation dynamics (I- stability period, II – dynamic changes period)

Providing working conditions that allow the best possible measuring performance is a standard operation. It is always difficult to estimate how much does variation dynamics of ambient conditions influence the measurement. The most basic test concerns balance indications repeatability, the said test means determination of standard deviation being an outcome of series of measurements. Knowing the standard deviation value, one may consider whether it is possible to obtain even lower rate. It is worth to mention that obtained standard deviation is a random value and several measuring series could provide more information on this parameter.

In practice it turns out that trying to make the workplace an ideal one is nothing more than just a theory, most users simply do not care. Therefore it is advisable to familiarize section on “Ambient conditions effect” which shall help one gain at least elementary level of knowledge on influence of ambient conditions on measurement indication.

It is worth to be mentioned that correct operating conditions for microbalances are a result of used design solutions (air conditioning, tables) which add to infrastructure of the room intended for weighing performance. The room size is a key factor here.

4. Measuring capacity – how to obtain precise measurement

Measuring capability is an indicator by means of which the metrological quality of a balance can be assessed, thus whether it can be used in the mass measurement process. The measure of the measurement capability is the expanded uncertainty related to the limit value, e.g. to the maximum error that may occur in the mass measurement process (critical limit).

$$Q_{MS} = \frac{U_{MS}}{E_{MAX}} \cdot 100\ \% \qquad (1)$$

where: U_{MS} – expanded uncertainty (k=2)
E_{MAX} – maximum permisible error (limit)

A seemingly simple topic can be a bit confusing for some people, mainly due to the need to calculate expanded uncertainty. As you know, in this process, all relevant factors must be taken into account, including those related to the accuracy and precision of the balance.

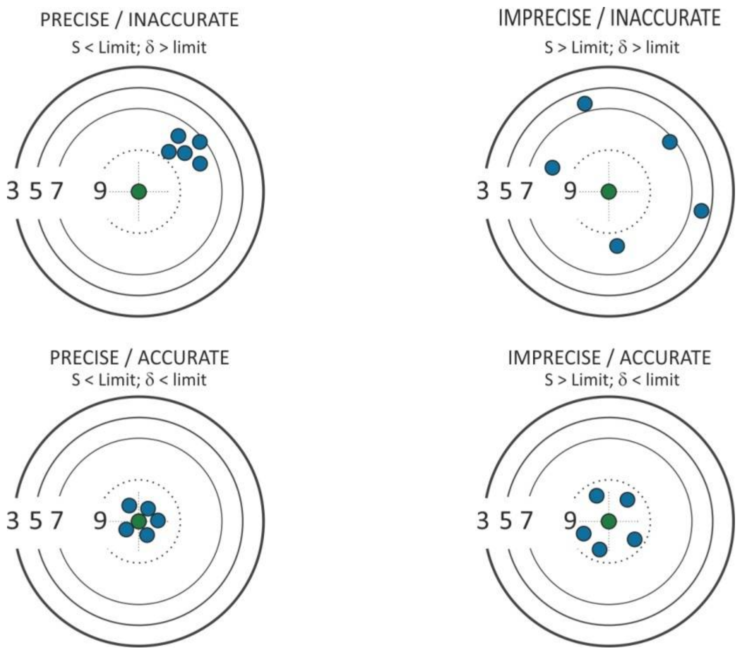

Fig. 6. Accuracy and precision measurement

When determining the weight of a sample, it is assumed that the result of weighing the sample is accurate. Confirmation of this fact requires checking balance indications with mass standards. The procedure is quite simple. Adjust the balance and then place a mass standard on the pan, whose mass is within the weighing range of the actual sample mass. If the balance

indicates the correct mass of the standard (taking into account its error), then the mass of the sample will also be determined correctly.
Even a significant deviation from the expected value displayed by the balance during standard weighing can be used to determine the actual sample mass. This deviation should be taken into account as the so-called systematic error.

In the case of checking the accuracy of balance indications, the main problem is the need to have a standard of the appropriate accuracy class with the appropriate mass (close to the mass of the sample). This is an economic problem due to the costs of purchasing and maintaining the pattern. It is assumed that as a result of factory scaling, the characteristics of the balance have a linear dependence, thus a single standard test is sufficient. Its weight can be any in the range from ½ Max ÷ Max weight.

The above solution is used quite often during every day, very simple balance checks. Despite the theoretically linear dependence of the balance indication on the applied load, it is recommended to check the accuracy of the balance indications with standards of the same mass as the weighed samples. By doing so, it can be demonstrated that the accuracy of the balance is actually correct within the range in which it is used.

Evaluating the precision of a measurement is much simpler because the measure of this value is usually the standard deviation of a series of measurements. The measurement is precise when we achieve the same or slightly different results during the test. This test is performed with mass standards, but can be performed with any object whose mass is constant over time. This is a good solution for those who want to determine the repeatability of indications for the packaging, glass flask, initial sample weight (differential weighing), clean filter (reference weighing), etc. Usually 6 ÷ 10 measurements are made to calculate the mean value and standard deviation.

Mean value

$$\bar{x} = \frac{1}{n}\sum x_i \qquad 2)$$

Standard deviation estimator $S_x = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$

$$3)$$

Estimator of the standard deviation of the mean

$$S_{\bar{x}} = \frac{S_x}{\sqrt{n}} \qquad 4)$$

where x_i – measurement

$\bar{x}$ – arithmetic mean of a series of measurements

n – number of repetitions in a series of measurements

During the test, there may be a special case when all measurement results will have the same values. Mathematically, this results in a standard deviation value of zero. Unfortunately, this value cannot be used, because it does not take into account the phenomenon of rounding of the measurement result of devices with a digital indication. The idea of rounding is shown in Figure 7.

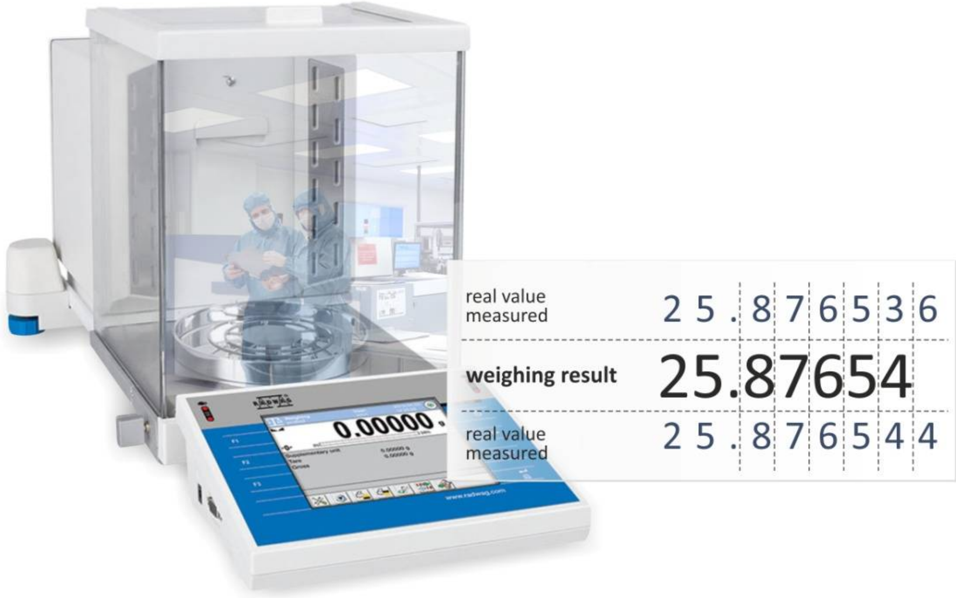

Fig. 7. Rounding of the measurement result of devices with digital indication

When the obtained measurement results have the same values or only slightly differ from each other, the value of the standard deviation is determined from the relationship:

$$S = 0.41 \cdot d \tag{5}$$

where: d - value of the elementary division of the balance

Measurement precision (indication repeatability) depends on such factors as:

- climatic conditions in which the test is performed
- operator ability (knowledge, skills, weighing without impacts)
- duration of the measurement,
- method of checking - measurement is not always performed on the weighing pan, sometimes by suspending the load,

It is generally assumed that the repeatability of indications of each balance is a constant value, which results from the construction of the balance. In own tests, the manufacturer determines this value in conditions close to ideal. In real conditions, the repeatability of indications is disturbed by external factors (mentioned above). It is therefore reasonable to choose a balance for the weighing procedure in such a way that the weighing precision and accuracy requirements are met with a certain margin.

4.1. Scales measuring capability analysis

Evaluation of the measurement capability of a balance requires calculation of the expanded uncertainty and determination of the largest permissible error that may be affected by the measurement. Measurement uncertainty can be calculated using the A method, where the value of repeatability of balance indications or the so-called method B, where all information related to the weighing process is taken into account. For an electronic balance, the components of type B uncertainty are:

- value of scale interval d,
- repeatability of balance indications (standard deviation),
- weight indication error (max deviation, or at the control point),
- uncertainty of determining the indication error at a given coverage factor k

The expanded uncertainty is obtained by multiplying the value of the standard uncertainty by the coverage factor k, (k = 2, 95 %).

Type A standard uncertainty

$$u(x) = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n(n-1)}} \tag{6}$$

Expanded uncertainty

$$U = k \cdot u(x) \tag{7}$$

Information on repeatability of indications is contained in the manufacturer's documentation, and the value of the so-called balance error. On the basis of these two values, the measuring capacity of the balance can be estimated, however, taking into account that in real use conditions, a slightly higher test result can be obtained.

The influence of the potential balance centricity error can be neglected, the mass standard should always be placed in the middle of the pan. It is assumed that the centricity error is small, of the order of a few scale divisions. Deviations due to undefined drifts in the zero indication can be minimized by zeroing before each measurement. The impact of the variability of the weighing result coming from sensitivity drifts can be considered insignificant when the measurement time is short, about 6 - 15 seconds, and the balance is adjusted periodically.

Table 1. Technical parameters of balances

Name	St. deviation (S)	Linearity error (E_{max})
Microbalances MYA 5.4Y.A PLUS	0.0006 mg	0.005 mg
XA 82/220.4Y.A PLUS	0.005 mg	0.06 mg

The measuring capacity of the balances Q was calculated according to equation 1, and the value of the expanded uncertainty U was calculated according to equation 7.

Fig. 8. Microbalance MYA 5.4Y.A PLUS

Microbalance MYA 5.4Y PLUS

- Maximum capacity 5 g
- Readability 0.001 mg
- Repeatability (S) 0.0006 mg
- Linearity (E_{MAX}) 0.06 mg
- Expanded uncertainty U 0.0012 mg
- Measuring capability Q 0.02 mg (2 %)

Fig. 9. Analytical balance XA 52 4Y.A PLUS

XA 82/220.4Y.A PLUS

- Maximum capacity 220 g
- Readability 0.01/0.1 mg
- Repeatability (S) 0.005 mg
- Linearity (E_{MAX}) 0.06 mg
- Expanded uncertainty U 0.01 mg (typu A)
- Measuring capability Q 0.16 mg (16 %)

Measuring ability in this approach is the greatest uncertainty of measurement that can be associated with a balance. This parameter can be used to evaluate comparable weighing systems, i.e. those that offer measurement at the same scale interval. Calculations of the measuring capacity of the scales were made using only the manufacturer's data - hence the choice of the type A method. Having full knowledge of the balance and its errors (calibration certificate), one can also use the method of determining the uncertainty using the type B method.
In practice, the limit of the required accuracy of E_{MAX} is much greater than the so-called linearity error given by the manufacturer. As mentioned earlier, this limit should be related to the required accuracy of mass measurement in a given real process.

Accuracy of sample mass measurement is assessed by comparing the result of weighing a standard with a known mass with the value shown on the balance display. This is important information for those who need to measure, determine the mass of the sample accurately. For processes in which the change of the sample mass as a result of sorption, desorption, absorption, drying, etc. is important, repeatability of balance indications is a more important parameter. Here, special attention should be paid to factors interfering with the measurement, such as the thermal instability of the sample, the phenomenon of electrostatics.

Regardless of the type of processes carried out, it should be remembered that giving the mass measurement result without measurement uncertainty is inappropriate - incomplete. Deviations in the accuracy of measurements should not be confused with the uncertainty of determining these values.

5. Ambient conditions effect

Regular microbalance user believes that the place he or she has appointed for the balance is the perfect one, if not 100 percent perfect then at least enough adequate. When problems start to occur, his or her first thought is "the balance operates incorrectly – it must be damaged". Trying to find source of faulty operation, may turn out to be too difficult for the user, this is mainly due to the fact that the problem may concern the following:

- balance (damages),
- working environment (extreme variation), or
- samples (e.g. absorption, electrostatics).

Only observation and analysis of both, weighing process and type of a sample may make one conclude on the problems source. This section provides information on typical problems, nevertheless it does not supply the reader with complete list of possibilities.

5.1. Workstation and its location

Size of the room where the microbalance is to be operated shall be optimal with regard to number of employees working there and scope of preset operations. Theoretically simple and clear dependence may turn out to be quite difficult for interpretation, especially when combination of information on workplace and of knowledge on weight measurement is required, and when dependence between these two has to be transferred to accuracy of weight analysis. One may go for simplifications as follows:

a. small room = small number of employees
b. small room = problems with maintaining the conditions stability when air conditioning operates in an automatic mode (air drafts, overshoot)

The said dependences may be verified and adjusted in course of microbalance installation. Then and only then it may be observed if room size influences measurement result significantly or not. Prior installation process it is advisable to collect as much information on the workplace as possible. Based on the collected information some user prompts shall be provided, the instruction must inform about operation and additional equipment to be used (supplementary draft shield, anti-vibration table, THB-R module for ambient conditions monitoring purposes). If this is only possible the balance shall be located in a place free from regular air flows, source of which are vents, windows or moving around personnel.

5.2. Personnel

Operating microbalance requires no more skills than operation of any other weighing equipment. The main difference is the microbalance design, it is characterized by a narrow weighing range (small Max value) and by small reading unit (d=1µg or 0,1µg).

Fig. 10. Mikrcobalance – powder weighing

With regard to the above it shall be noticed that any weighing pan shocks may cause weight measurement errors therefore used weighing technique is an important factor. Gaining respective skills when it comes to weighing, is possible only by practice, knowledge is not enough. Awareness of how other factors, relating to the working environment and to the sample, effect the process of weight measurement is really useful, especially in case of balances with high resolution.

5.3. Workplace temperature

Workplace temperature issues shall be approached from two different perspectives. The first one concerns the room and its temperature, more precisely dynamics of changes occurring in course of 24-hour-long cycle. The second in turn concerns balance construction, more specifically its internal temperature. Temperature stability depends on changes that take place within the whole laboratory space.

Fig. 11. Chart of changes in environmental conditions from December 21-24, 2021

Fig. 12. Detailed analysis of environmental data for the selected period

Optimal solution shall be such one that guarantees constant temperature value for a room intended for weighing (independently from the room size). Stable temperature shall be understood here rather as insignificant temperature oscillations around the specified value. Quite important concern arises from the above, namely how much shall the temperature oscillate. Trying to find the best possible answer one needs to know that:

- migration of heat from the working environment to the microbalance weighing module is significantly slower owing to use of internal and external draft shields,
- extreme temperature variation causes small indication drift, which may occur as lack of zero indication upon taking a load of the weighing pan,
- sample measurement takes little time, about 5 – 12 seconds, this means that drift of indication resulting from temperature variation may be of marginal importance.

Being aware of the above, it has to be concluded that dynamics of air temperature changes in Laboratory of the order of 1 ÷ 2°C / hour, do not affect weight measurement. As mentioned earlier, the migration of heat from the environment to the interior of the weighing module is limited by the construction of external covers for microbalances and analytical balances. The dynamics of temperature changes inside the balance is also controlled by sensors installed inside the balance, and the recorded values are used to initiate internal adjustment and to visualize working conditions. Such monitoring is presented in Figure 13.

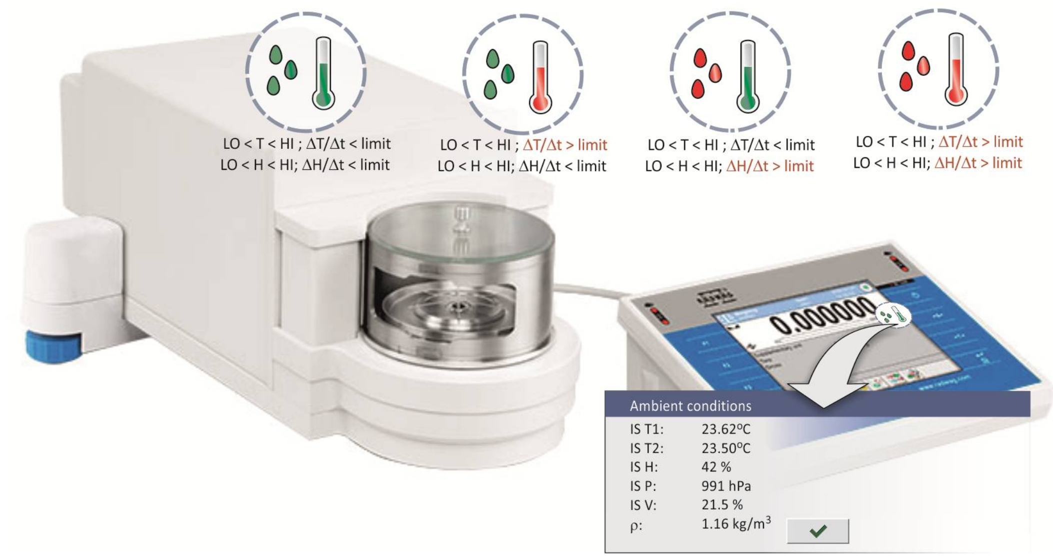

Fig. 13. Environmental conditions module for ultra-microbalances and microbalances

Despite mechanical protections in the form of thermal shields of the weighing module and on-line measurement of the dynamics of temperature and humidity changes, it is recommended to maintain high stability of environmental conditions for the most accurate analyses. Such requirements apply to UYA series ultra microbalances and are given in the technical data (Table 2).

Table 2. Technical parameters for ultra-microbalance UYA 2.4Y.A PLUS

Maximum capacity	2.1 g
Readability	0.1 µg
Standard repeatability *)	0.15 µg
Standard repeatability **)	0.35 µg
Environmental conditions	
Working temperature	+10 ÷ + 40 °C
Rate of change of working temperature changes	± 0.3 °C / 1 h (±1 °C /8 h)
Humidity	40% — 80%
Rate of change of relative air humidity	± 1 % / h (± 4 % / 8 h)

*) – repeatability as St. dev. for 5% Max
**) – repeatability as St. dev. for Max

5.3.1. Self-heating

Microbalance temperature stability is an effect of both external temperature and balance self-heating. This process is a result of systems of electronics operating inside the balance. Practically each microbalance comprises two main components. One of them is the weighing module equipped with an electromagnetic transducer, the other one is an LCD display featuring communication interface. These two components have to be separate in order to provide stable temperature of a weighing module. This requirement does not apply to the other component, see the thermal images below.

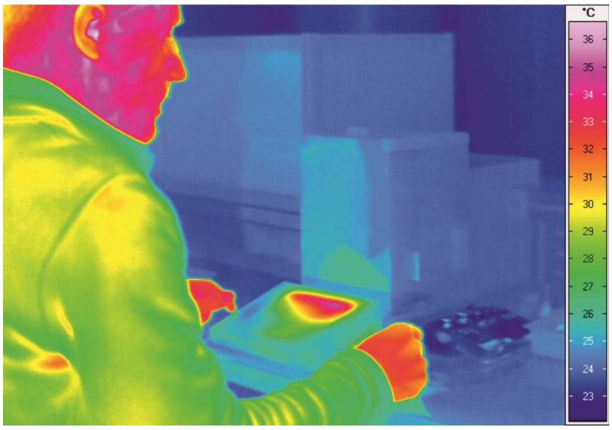

Fig. 14. Thermovision - Analytical balance

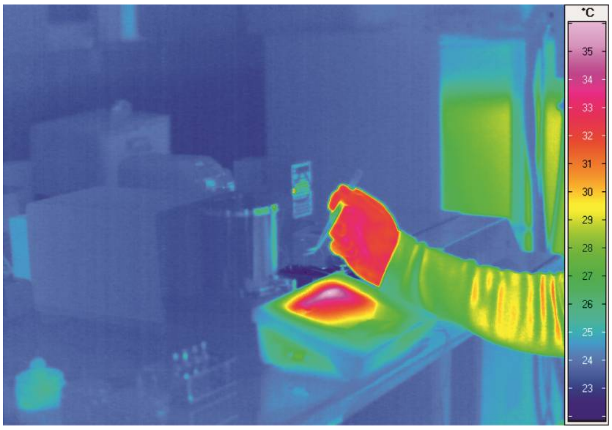

Fig. 15. Thermovision - microbalance

On plugging balance to mains one may observe difference between workplace temperature and weighing module temperature. This shall be particularly noticeable for balance transported from a place where the temperature was dissimilar. In order to ensure repeatable measurements one shall wait a respective amount of time, i.e. as long as it takes to reach temperature stability of the weighing module. One may ask "How long shall I wait?". To such posed question there are two answers:

1. If the user wants to be 100 percent sure that his or her device is stable, the tests shall be performed 24 hours after plugging the balance to mains (providing that laboratory temperature is stable.

2. Microbalance may be used after 3-hour-long or 4-hour-long period of time, but then zeroing button must be used more frequently, automatic adjustment performed more often and it must be remembered that repeatability of indications may vary from what has been declared by the manufacturer.

5.3.2. Systems for temperature control

Some standards specify the temperature range that should be maintained in the Laboratory, e.g. 20 ^{o}C ± 1 ^{o}C acc. EN 12341,,Air quality. Determination of PM 10 fraction of particulate matter". The method of implementing this requirement is basically free, however, one should remember not to introduce excessive air movement into the Laboratory at the expense of temperature stabilization. This is quite a difficult task. Existing premises and economic (price) constraints mean that the simplest solutions in the field of air conditioning are usually used. Unfortunately, they almost always generate significant air movement, which makes it impossible to perform a correct measurement. In this case, an additional cover of the microbalance should be used, which is a buffer limiting the movement of air near the weighing chamber. Such solutions are commonly used not only in the case of microbalances. The target solution in the field of laminar air conditioning is shown in Figure 16. It is a solution developed and verified by Radwag, dedicated to those processes where very high accuracy of weighing, microbalances and ultra-microbalances manufactured by Radwag is required.

Fig. 16. An example of dispersed air conditioning.

Note

Temperature stability can also be achieved through natural air circulation in the room. Obtaining temperature stability at any level, eg 25^{o}C, is sufficient for accurate measurements.

5.3.3. Accuracy and variable temperatures influence

Thermal stability for the workplace and for the balance is a need. Unfortunately it is sometimes impossible to provide stable temperature which results from external conditions. What do temperature variations mean for measurement performance?

It is worth to remember that each weighing device is a system of mechanical components. As it is commonly known, according to the laws of physics the components may shrink or extend as an effect of temperature variations. The said processes may show the following results:

- Drift of indication
  Dynamics of measuring system variations noticeable as slightly decreased or increased indication for an unloaded weighing pan. Each modification of the state, e.g. from 0,000 to 0,003, is zeroed prior weighment, this means that each weighment starts with so called "precise" zero value. Since this process applies also to the measurement result, therefore it must be remembered that the said result may as well be affected by drift errors. This explains why repeatability for unstable conditions is worse that repeatability obtained for stable conditions.

- Modification of balance accuracy
  As it has been presented in the graph (Fig.13), balance adjustment can provide respective accuracy. Between adjustment processes, insignificant drifts of accuracy may occur, nevertheless their detection is impossible since the said drifts are comprised within balance repeatability range. Noticeable deviations of accuracy, being an effect of dynamic ambient conditions changes, are eliminated by means of automatic adjustment (green colour).

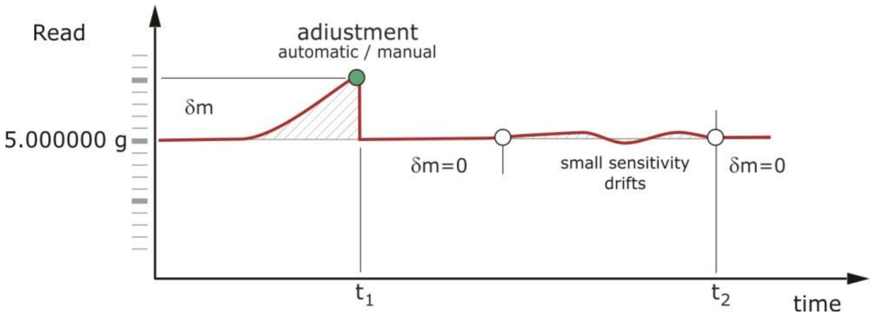

Fig. 17. Correction of accuracy of indications by means of automatic adjustment

NOTE
A change in temperature, apart from changes in the geometrical dimensions of the weighing module, may cause changes in the characteristics of the magnetoelectric converter and electronic systems. As a result of these phenomena, the balance may lose its accuracy, the sample may be weighed with a greater error. For this reason, most balances use automatic adjustment, which eliminates this error. This solution is standard in all Radwag laboratory balances.

5.4. Moisture content and the weighing process

Moisture content of the laboratory may be controlled and adjusted by means of humidifiers and dehumidifiers. Increasing humidity of spacious areas is not a challenging or complicated process. Quite contrary to humidification, the drying process is much more time-consuming one. It is much less sophisticated to generate a required amount of moisture content than to filter the particular amount of air in order to provide decrease of its humidity. Commonly available literature informs that low humidity favours electrostatic charges formation. Occurrence of electrostatic charges may interrupt the weighing process. This makes manufacturers provide specification and guidelines for working conditions: humidity range of 40 % ÷ 85 % (non-condensing conditions).

For balances with high resolution, microbalances, ultra-microbalances, it is recommended to maintain stable level of humidity. This is due to balance design mostly and the way it operates. The input state of 0,000000 g is a result of weighing balance components such as weighing pan, levers, coil etc. If the said components weight is constant over time then balance indication for an unloaded weighing pan shall be 0,000000 g almost continuously. If the amount of moisture content increases then humidity absorption process may occur, this means that the moisture is absorbed by balance components which adds to growth of their weight. The initial steady state gets disturbed and one may notice insignificant drift of indication. This is quite slow process nevertheless it does influence the weighing result (especially when it comes to balances with d=0,1 μg). The described issue is almost meaningless for typical analytical balances with reading unit d=0,1 mg.

Fig. 18. Microbalance MYA 2.4Y.F.A PLUS – filter mass measurement, XA 82/220.4Y – powder weighing

The same phenomenon also applies to samples such as powders and cellulose filters. When weighing such objects, their mass increases quite quickly, which an inexperienced balance operator may perceive as damage to the scale. Detection of the source of these problems is quite simple, weigh any object of constant mass. If, as a result of the test, correct stabilization of the weighing result is observed, it clearly indicates that the problem is related to the structure of the sample. Two phenomena are possible, moisture sorption/desorption or unbalanced static charges. The first defect can be minimized by weighing the sample in a container or acclimating it.

Equalization of the electrostatic potential requires the use of an ionizer, e.g. DJ-04, manufactured by Radwag.

5.4.1. Disturbed humidity and the measurement

Maintaining stable level of humidity, e.g. 50% ± 5%, requires use of humidifying and dehumidifying system. These two should be coupled but even though the problem of nominal value readjustment would not be solved. The described system is a highly complicated one and it must be adopted to the room size, intensity of its use, ventilation system etc. In practice, for humidifying purposes humidifiers are used, without the need of applying dehumidifiers. This means that as a result of change of atmospheric air parameters one may observe humidity increase in the laboratory. Does it affect weight measurement?

The answer depends on :

- Used balance type
- Weighed samples
- How accurate the sample weight must be

If the reading unit of your balance is not smaller than 0,01 mg then the laboratory humidity level may vary dynamically. Variable humidity, 20% increase/decrease over a specified time period, shall not influence metrological important parameters of the balance.

For balances with reading unit smaller than 0,01 mg, the metrological important parameters will worsen if the balance is operated in a room where the humidity level changes dynamically. One may expect a little bit higher value for indications repeatability, this is to be observed for tests performed by means of mass standard. What shall be remembered is the fact that increase of humidity influences real weight of a sample.

When sample weight does not have to be precise, i.e. when it is enough to determine its value with less than 99 percent accuracy, then any parameters changes become insignificant.

NOTE

1. Trying to conclude how much variable humidity influences weight measurement, one has to take into account the following: reading unit, measuring capacity needed for weighing and a particular sample type. Negative effects of dynamic humidity variations are as follows:
   - drift of balance indication (humidity adsorption by balance components),
   - humidity adsorption by samples, sample weight variation,
   - electrostatics process occurrence when the humidity level is too low.

5.4.2. Humidity influence on sample weight measurement

There are two aspects of humidity influence on a sample. The sample weight may vary due to absorption or desorption processes. Each of these adds to user dissatisfaction. It is clear that the user expects to get a stable result rather than observe drift of indication making him/her realize that sample weight determination is not possible. In order to conclude on source of such instability it is necessary to deposit a load characterized by constant weight on a weighing pan, e.g. mass standard. If the obtained result is unstable, then it is certain that measurement instability is an effect of processes occurring within the sample.

Example processes where there are problems with sample weight instability:

a. **differential weighing of cellulose filters, measurement of dustiness**
   Use glass fibre, quartz or Teflon (PTFE) filters, these do not absorb humidity therefore it is the absorbed medium that is being measured, not moisture. Electrostatics process may occur in case of PTFE filters. If this is so then +/- charges ionization is recommended.

b. **not acclimatized sample weight determination after prior thermal processing (heating, burning)**
   Stabilize sample weight, using desiccator provide stable temperature and humidity. If the sample is to be weighted when it is still warm then the measurement must be performed quickly (optimization), the indication is certain to decrease. Such is the result of hot air convection currents formation occurring in a weighing chamber.

c. **dry powders weighing when the humidity level is high**
   The sample shall be kept in a closed container, it must be mixed prior weighing, weighing time shall be optimized (as short time as possible), the first stable indication shall be taken for analysis (successive measurements will be increased – process of absorption)

d. **determining weight of cardboard sample, absorption of humidity**
   Samples comprising cellulose are hygroscopic samples which means that their weight changes over time. Sample weight increase depends on humidity level of a particular room and on the sample surface size. Balance reading unit is important too (possibility of variation detection).
   GUIDELINES:
   Isolation of a sample from the environment (weighing packaged samples), acclimatizing a sample to ambient conditions of a room.

SUMMARY
Processes relating to humidity interactions are physical processes and they concern every single balance. Possibility to observe their effect depends on the given process scope, balance resolution and sample susceptibility. For low humidity level required, electrostatics processes may occur. Electrostatics process may be eliminated by particular balance design, e.g. antistatic shields, such one that is used for MYA series microbalances.

5.5. Air flow

Air flows in a balance neighbouring area almost always disturb weight measurements. Air flow is a process of a physical nature – the weighing pan is left up or pushed down depending on the air flow direction. The effect of air flows may be visible or not which in turn depends on weighing pan size and speed of air masses moves.
Too strong air flow is a result of used devices which serve to adjust the temperature. In order to assure stability, air masses need to be mixed, unfortunately such process is sometimes unwanted.

Preventative measures shall concern two aspects. First of all, balance design must enable operation of the device in extremely different conditions. It is the manufacturer who shall provide respective solutions based on experience, research and users' comments. With respect to the already mentioned, RADWAG has come up with numerous changes of the analytical balances and microbalances design.

As for the second aspect, these are user-performed preventative measures:

- isolation of the balance by means of supplementary draft shields,
- relocation of the workstation.

Fig. 19. Microbalance in an anti-draft chamber

When it comes to weight measurement, air flow is a cause of greater indication dispersion and temporary indications instability. The corrective actions taken usually help to obtain correct results.

5.6. Unbalanced electrostatic charges

Sample weight measurement means determination of the gravitational force applied to a particular sample. For regular working conditions, the air, balance and the sample are characterized by the same amount of positive and negative charges. This allows undisturbed weighing process. This neutral state change may be a result of air ionization but usually this is an effect of:

- Transfer of charges from the operator to a sample
- Electrification of sample being a consequence of rubbing the sample

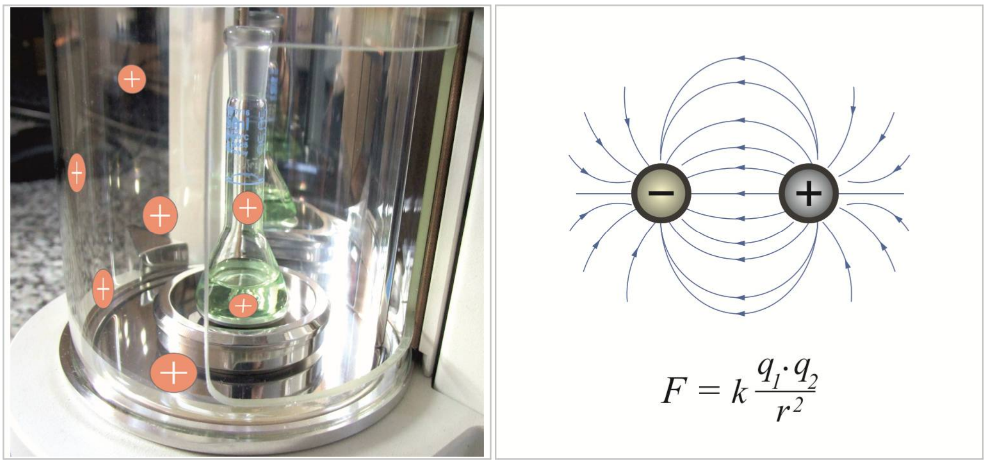

Fig. 20. Unbalanced electrostatic charges, Coulomb law

As a result, the weighing process (gravitational force) is deformed. FG gravitational force value is increased or decreased depending on the dominant charge type. Degree of the force interaction depends on value of accumulated charges, distance between the charges and on the neighbouring area where the interaction takes place. Taking the above into consideration it may be easily concluded that the process is random in its nature. If this is so then the question is how to recognize it. Unquestionable effect of this process is an indication drift. Similar symptoms occur for humidity absorption therefore it is necessary to evaluate the process correctly. Diagnosis and controls shall proceed as follows:

a. Carry out test weighing using a mass standard
   - if the indication is stable the problem applies to a sample,
   - if the indication is not stable the problem applies to a balance.

b. Carry out sample weighing
   - if the sample is a powdered substance and the balance indication increases, it is certain that the sample absorbs humidity,
   - if the sample is a powdered substance and the balance indication decreases, then probably sample temperature is diametrically opposed to the ambient temperature ,
   - the measurement result may be influenced by sample packaging, plastic components may electrify, place the sample in a metal container.

c. while weighing Teflon-made filters (PTFE), charges may accumulate on their surface, in such a case deionization shall be carried out prior weighing (e.g. DJ-02 ionizer)

d. one of the solutions may be weighing a sample in packaging or use of antistatic shields (MYA microbalances), this is due to the fact that electrostatic interference occurs between sample surface and static balance components.

Electrostatic interactions are not very common in the work environment. Their occurrence is usually the result of the properties of the sample and low humidity. To effectively counteract them, you should know that:

- If a negatively charged body is brought into contact with an electrically neutral body, the excess electrons will split into two bodies. As a result, both bodies are charged with the same sign.

- if a positively charged body is brought into contact with a non-electrically charged body, some of the electrons from the electrically neutral body will transfer to the charged body and the positive charge will be reduced on it. At the same time, a neutral body becomes positively charged as a result of the loss of some electrons.

In the phenomenon of electrifying by body touch, they electrify themselves. Therefore, an inert body cannot be obtained. The assessment of phenomena related to electrostatics is quite embarrassing, because only the effect of the occurrence of the phenomenon is visible, not itself. Since it is impossible to remove the cause, eliminating or compensating unwanted electrostatic charges are used. The solution offered by Radwag is the ionizer, which is the so -called Electrostatic charges neutralizer.

Fig. 21. Ionizer DJ-04 type

5.7. Ground vibrations

Historically, the first weights were probably isosceles levers, in which the loads placed on the edges of the lever were comparison. Most modern magnetoelectric processes have a similar structure. On the one hand, there is a weignig pan on which the load is placed and on the other side there is a coil suspended in a magnetic field (magnetoelectric transducer). The transducer's task is to compensate for the gravity force that is subject to the load. This is done by observing changes in the position sensor and generating a force compensating for gravitational impact that works on the load.

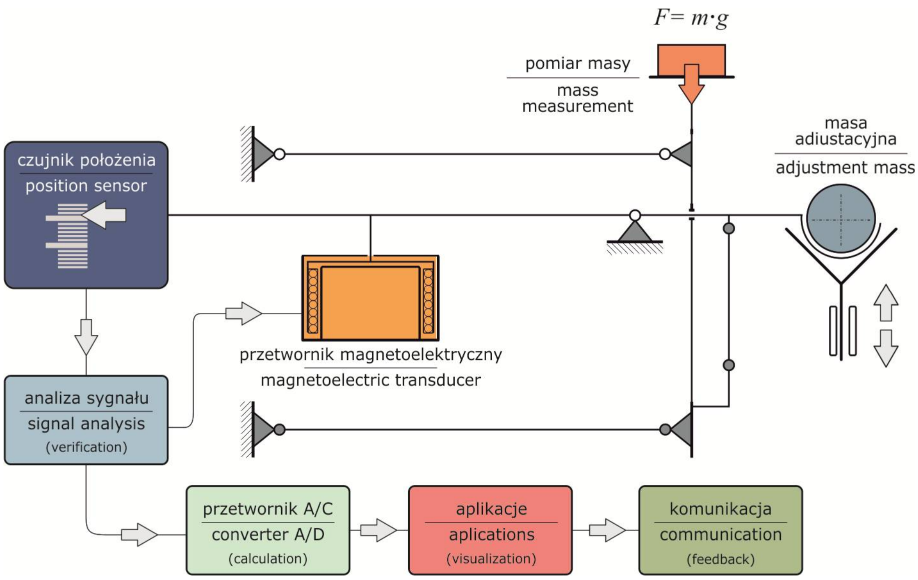

Fig. 22. Scheme of the balance of the magnetoelectric processing system

The consequence of such construction is the susceptibility of the measuring system to shocks and vibrations. It is not unequivocal that all substrate vibrations are a factor that interferes with the measurement. Whether it is in fact depends on the weight, size and variability of the vibration and the size of the sample. For mass measurements, the main symptom of significant vibrations is instability of indications. The observed result only achieves temporary stable conditions, so determining the weight of the sample is impossible. What corrective actions can therefore be implemented in such cases?

a. When machines and devices are the source of vibration, one may try to limit their operation time
b. Balance relocation might help to reduce vibration effect since different grounds transfer different amounts of vibrations
c. Workstation design shall comprise components dampening vibrations, SAL/M anti-vibration table is an example of such workstation

Fig. 23. Anti-vibration tables

d. For vibrations of geological origin it is better to locate workstations on ground floors of a particular building. The higher the workstation is placed the more vibrations it absorbs.

e. In extreme situations the only one possible solution is electronic optimization of a weighing module. Typical balance is characterized by settings allowing to define relation between measuring signal and its duration. In case of serious disturbances it is necessary to apply different criteria for variation dampening and measuring signal verification. This may be carried out only after analysis of workplace processes. RADWAG has designed an automatic method for testing dependence of metrological important parameters in real working conditions, the method is called Service Autotest.

Substrate vibrations are also recorded by internal or external microwaga sensors. Under normal working conditions, the vibration icon is green, detecting vibrations larger than the permissible limit causes a change in the color of the icon - red. The value of vibrations is also presented in the Info field of environmental conditions.

Fig. 24. Detection of substrate vibrations - a sensor of environmental conditions

6. Optimization as a balanced process

Optimization shall be understood as a process in course of which any factors decisive for ambient conditions are verified, among the said factors there is temperature, moisture content, intensity of operation (number of operators working simultaneously) and internal balance set points. Optimization is a bi-directional process and it aims to provide such adjustment that allows to minimize measurement error as much as possible. Judging by experience the said adjustment usually concern balance settings.

Target of the performed corrective actions and measures is to obtain better parameters that are metrological important, especially the repeatability. The said actions may also increase speed of weighing. In order to be successful while working on improvement of both, repeatability and speed of weighing, one needs to acquire comprehensive knowledge. A manufacturer or distributor may turn out to be supportive and helpful.

6.1. Optimization – effect on metrological performance

Balance settings are appropriate for measurements performed in stable external conditions. These are such conditions for which the following features apply: temperature insignificantly oscillates around the specified value, humidity value is constant, air flow is dispersed to a laminar form. It happens that upon balance installation onto its workplace the above listed conditions are deviated, sometimes even considerably. The said deviation may be a reason for measurement divergence.
Scope of corrective actions that shall be applied for a particular balance is dependent from the particular process and balance performance.

So what is the regulation scope?

The user menu contains three elements that change the weighing process. Their short description is below:

WEIGHING PROFILE
Describes all parameters related to the weighing process such as the currently used filter, method of defining stable measurement, type of work environment, weight excitability, the speed of measuring signal growth, etc. 4 weighing profiles are available:

- fast, profile that allows you to quickly weigh any masses, the measurement result is achieved as soon as possible,
- Fast Dosing, the profile is dedicated to dispensing, high weight excitability
- Precision, profile dedicated to obtaining results with the best precision,
- User, a basic profile for which filter settings are selected so that the weighing is relatively fast and precise.

Only after selecting the user profile, it is possible to change the filter value and the type of approved the result. For other weighing profiles, the filter and confirmation of the result are not available.

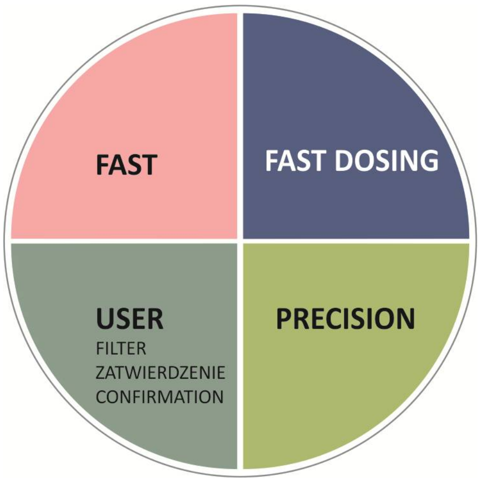

Fig. 25. Weighnig profile in laboratory balances

FILTER
Decides on amount of information that is to be analysed. The higher filter value, the more data analysed. In case of single disturbance, its effect on measurement is less significant when the analysis concern greater amount of information. The following filters are available:

- very fast
- fast
- average
- slow (* - recommended value)
- very slow

Access path in microbalance program structure: SETUP/PROFILE/HOME/READOUT/

VALUE RELEASE
Describes conditions necessary to provide stable measurement. This is ensured by specifying changeability of indication over a particular time. Respective parameters are comprised within balance service menu, but a regular user has simplified facility for such specification at his or her disposal. There are three options of Value Release available:

- fast
- fast + reliable (* - recommended value)
- reliable

Access path in microbalance program structure: SETUP/PROFILE/HOME/READOUT/

AMBIENT CONDITIONS

Gives general information on current ambient conditions for balance operation. Stable ambient conditions option stands for very good working environment, as a result the balance automatically performs less intense result damping in course of weighing. The measurement takes a little bit less time. Unstable ambient conditions option means more intense damping of a measuring signal which aims to minimize ambient conditions influence. Time needed for weighing may lengthen when this option is selected. To make sure which option shall be chosen, one may perform a short test using any object of constant mass.

a**. An indication of the weight oscillates around the stable result**
ACTIONS
Change the filter value by one level (e.g. from average value ⍰ free; free ⍰ very slow), approval of the result should have a fast + accurate value.

Comment
When the deviations are insignificant (several elementary plots) can be eliminated by using other weight parameters. For much larger oscillations, the source of interference, which causes them to form and then correct the weight parameters.

b. stable result changes by several units from one state to another
ACTIONS
Change the value of the appointment by one level from the value quickly to the value quickly + exactly or quickly + exactly to exactly the value exactly

Comment
A similar effect will be visible when the moisture absorption process occurs through the sample. In this case, you can observe a continuous increase in sample weight - corrective actions may be ineffective.

If the variability of the result is the result of moisture absorption, the sample should first be acclimatized to the environment or accept the first initial stable measurement for analysis. All subsequent ones reflect moisture absorption. A certain rarely used solution is weighing the sample in a tight packaging, the mass of the sample should be corrected by a lot of packaging.

c. Weighing result is constantly changing in one direction
ACTIONS
Changing weight parameters in this case is ineffective.
For hygroscopic samples, actions are possible: stabilization in the work environment, reducing the sample surface or weighing in a tight packaging. A certain solution is to reduce humidity in the workplace, but as you know it is not an easy process, especially in the context of maintaining its stability.

The occurrence of electrostatic charges can be reduced by using the volume of sample ionization. You can then get a significant improvement in the measurement stability. An effective tool is the DJ ionizer DJ-04 or DJ-05 (Fig. 21).

Comment

The state in which we observe a constant change in one direction may result
from two phenomena. The first of them is moisture absorption through the sample. Such a phenomenon applies to all hygroscopic samples that can take moisture from the environment (mass increase) or moisture can evaporate from their surface (weight loss). Such samples are tissue paper, cellulose filters, powders, cardboard elements and those containing cellulose and fibrous materials. The second phenomenon is the occurrence of unbalanced electrostatic charges. Depending on their type, intensity and distance between them, there will be growing or decreasing indications.

d. The dispersion of indications when weighing the same sample is significant
ACTIONS

They should be adequate for observation. If the result shows the variability of several elementary plots with the stability sign displayed, change the value of the result of the result by one level:

- from value quickly to value quickly + thoroughly,
- From quickly + exactly to the value exactly.

Check the value for the environment parameter - the recommended setting of the unstable environment. If you have doubts, make measurements using the mass pattern.

Comment

Before taking action, it should be remembered that the repetition of the indications quite strongly depends on the conditions in which it is performed. First of all, a diagnosis of the working environment and weight in terms of its temperature stability should be carried out. Divergences in the results may also be the result of phenomena occurring in the sample (evaporation, absorption, electrostatics).

e. After removing the load, the weight indication does not return to zero.
ACTIONS

Remove the samples from the scarf without causing strokes, while closing the weight cabinet, gently move the weighing ventricle windows, check the option approval option - active value can quickly cause displaying stable results. If you use a quick or dosing profile, the weight is automatically configured for detection of very small weight changes. This may be the reason for the slight instability of the indication.

Comment

After removing the load from the scales, the conditions are stabilized inside the weighing chamber. It can be disturbed through air movement (air conditioning), substrate vibrations, no weight acclimatization. To measure the mass when the scarf is not burdened, the same criteria are used as when measuring the mass weight.

6.2. Optimization of speed

Correction of balance settings can be implemented to increase the weighing speed. It is true here that by increasing the weighting rate, metrological parameters such as the repetition of indications deteriorate. It should be noted here that to assess the speed of weighing it is crucial to define the weighing time. The following terms can be found in the available literature:

- weighing time, time calculated from the moment the load is placed on the scales of the weight to the moment the stable result is obtained,

- weighing time, time calculated from the moment the load is placed on the scales of the weight to the moment the result is in the assumed limit,

- Weighing time, time needed to carry out all operations related to the weighing process, i.e. opening the weighing chamber, placing the load on the shawl, obtaining a stable result and a photo of the cargo load.

The lack of one normative definition for weighing results in the fact that in the technical documentation of manufacturers you can find different values for this parameter. Shorter weighing time can be obtained by changing two settings. Use a smaller filter level, e.g. a filter as a very fast, and select the approval of the result as quickly. The measurement time will then be shortened by around 25-30 % compared to standard settings. Even with such quick measurement, you can get stable results and quite good repetition, but only if the test conditions are stable.

7. SOP - monitoring of metrological important parameters

In most cases information on metrological important parameters is provided by means of adjustment procedure. The said procedure is performed periodically, once a year or in two-year-long intervals. Between the adjustment procedures balance parameters shall be monitored to the required extent.

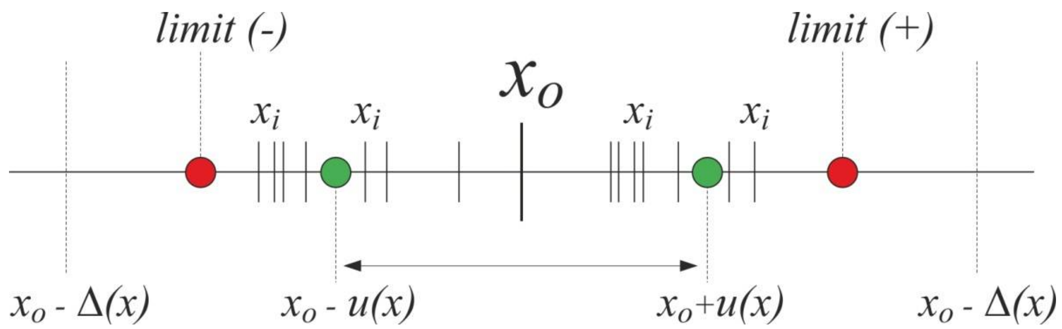

Fig. 26. Weighing limits using measurement uncertainty

An additional parameter that can be helpful when assessing the quality of electronic scales is the uncertainty of measurement. As you know, each measurement is characterized by a certain inaccuracy, which results from the measurement capabilities of scales and the research methods used.

These factors make up the so -called Measurement error. It is worth noting here that the measurement result is a random variable - you can practically get some differences when weighing the same load. The spread of weighing the same sample is characterized by standard deviation. In some cases, it is used to estimate the standard uncertainty, i.e. the parameter, which describes where it can find the measurement with some probability.

7.1. Repeatability of indications

It is tested by weighing the same weight for several times and by calculation of Max - Min difference or standard deviation out of series of measurements. Depending on adopted or regulated requirements 6 – 10 measurements are performed.

Testing methodology is clear and it shall not be a subject of these guidelines, nevertheless it is worth to mention once again that repeatability of indications depends on ambient conditions existing in a room where the measurement is carried out.

Repeatability can be tested for the whole weighing range, e.g. 5% Max, 50% Max, 100% Max or for a weighing range within which the balance is operated. For some branch industries (pharmacy – USP 1251), it is possible to determine MINIMUM WEIGHT value, so called MSW, using the result of repeatability.

Standard deviation - interpretation

One shall realize that measurement cannot be 100 percent reliable, it can only be concluded with a great dose of probability what the measured value is. By calculating average value out of measurement series first, and standard deviation next, it can be stated that the measured value is included within a particular range

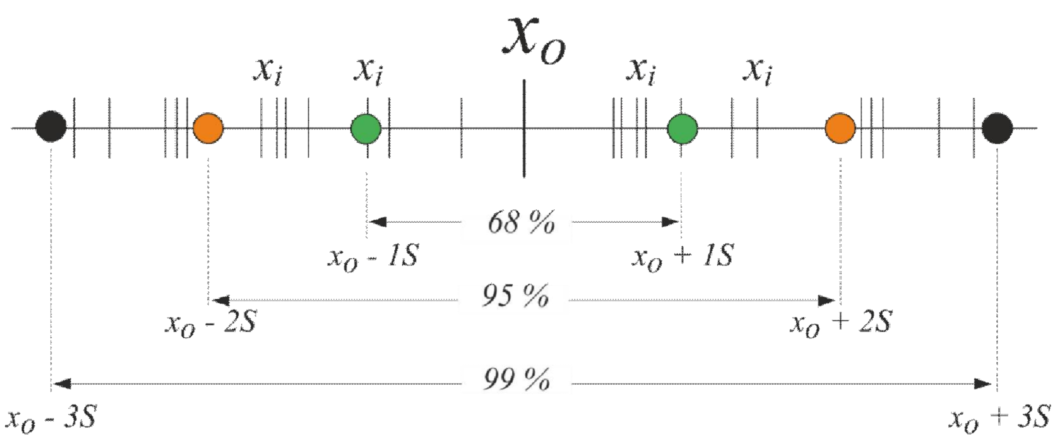

Fig. 27. Interpretation of the standard deviation

where x_0 – mean value

x_i – measurement result

S – standard deviation

The lower the standard deviation (S), the better the precision of the measurement, the better focus of results around the mean value. Measurement precision should not be confused with the accuracy of the measurement. Accuracy is a deviation of the value measured relative to the expected value (mass pattern value).

Example of checking the repetition of the MYA 5.4Y Plus microbalance. Metrological parameters:

- maximum load Max = 5 g
- Readability d = 1μg
- Standard repeatability 0.6 μg (for ~ 5 % Max)
- Permissible repeatability 1.2 μg (for ~ 5 % Max)
- Standard repeatability 1.6 μg (for Maximum capacity)
- Permissible repeatability 2.4 μg (for for Maximum capacity)

Table 1. Microbalance MYA 5.4Y PLUS repeatability

No.	0.5 g	2 g	5 g
1	0.000501	1.999997	5.000009
3	0.000501	1.999998	5.000008
4	0.000500	1.999998	5.000010
5	0.000499	1.999996	5.000009
6	0.000500	1.999997	5.000009
7	0.000501	1.999999	5.000010
8	0.000501	1.999997	5.000008
9	0.000501	1.999998	5.000007
10	0.000500	1.999997	5.000011
S	0.73 μg	0.88 μg	1.22 μg
Max - Min (R 76)	2 μg	3 μg	4 μg

COMMENT

The repetition of indications determined by standard deviation (S) for a weight of 0.5 g was in the range between the standard and permissible value. For a load of 2 g and 5 g, the precision of measurements was less than the value of standard repetition - the weight meets the metrological requirements.

7.2. Eccentricity

Eccentricity is usually tested using load of 1/3 Max capacity weight, wherein the load is placed anywhere but the weighing pan centre. Testing points are defined by respective standards, e.g. EN 45501 or EURAMET, they are presented in the picture below. No other methods are used as far as this test is concerned.

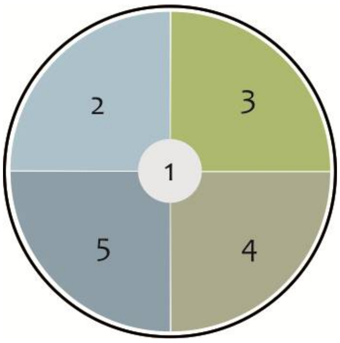

Fig. 28. Weighing pan – eccentricity testing points (acc. to EN 45501 test shall be performed for points 2-5, acc. to EURAMET for points 1-5)

Differential error of eccentricity – deviation between weighing result for weighing of mass standard, wherein the mass standard is placed in points 2-5, and a weighing result obtained by weighing of the same mass standard, wherein it is placed in a central position 1. Testing point no. 2 is calculated according to the following formula:

$$\delta_{(i)} = m_{(i)} - m_1 \tag{8}$$

where $m_{(i)}$ – indication when pattern is placed in 2 ÷ 5 position
$m_{(1)}$ – indication when pattern is placed in Centre (1) of the weighing pan

Determination of eccentricity deviations is carried out in course of microbalance validation procedure. The deviation values shall not be greater than few reading units. Eccentricity value is constant therefore it does not have to be tested too often. There are cases when the test result is of no importance, e.g. when the sample is really light (differential weighing of the filters).

NOTE

Testing eccentricity in accordance with EN 45010 (legal metrological control) makes no sense. This is because of MPE described by respective requirements. The MPE equals 0,5 of verification scale interval e, the lowest value of which is 1 mg. This means that the maximum permissible error is 0,000500 g, therefore the test is nothing more than just a formal confirmation of compliance with the requirements.

Adjustment prior test is not required if the test concerns differential error of eccentricity. According to regulations the test shall be carried out using mass standard, wherein it should be characterized by a compact structure. It is better to use one mass standard of a bit greater nominal than 1/3 Max capacity then few mass standards. Example:

MYA 5.4Y PLUS
Max = 5 g
⅓ Max = 1,6 g (required mass standards: 1 g, 500 mg, 100 mg)
Test mass standard = 2 g

MYA 21.4Y PLUS
Max 21 g
⅓Max = 7 g (required mass standards: 5 g, 2 g)
Test mass standard = 10 g

Fig. 29. Microbalance MYA 5.4Y. PLUS – eccentricity error

Evaluation of centricity deviation

m_1 = 2.000008 g
m_2 = 2.000006 g δ_2 = 2.000006 g – 2.000008 g= - 0.000002 g
m_3 = 2.000005 g δ_3 = 2.000005 g – 2.000008 g= - 0.000003 g
m_4 = 2.000009 g δ_4 = 2.000009 g – 2.000008 g= 0.000001 g
m_5 = 2.000010 g δ_5 = 2.000010 g – 2.000008 g = 0.000002 g

7.3. Linearity

Linearity parameter determines difference between the weighing result and a reference value of mass standard. The parameter can be tested for the whole weighing range or for a partial weighing range of a microbalance. Example of the later might be measurement of filters, weight of which is about 50 mg – 500 mg. If the measurement is performed by means of microbalance with 5 g capacity, then linearity of its upper range is not important contrary to cost of used mass standards. Both, eccentricity and repeatability do not require "accurate" mass standards but linearity does. For linearity tests, mass standards deviations and their uncertainty shall be as minimal as possible (OIML accuracy class).

For test performed by means of microbalances and ultra-microbalances only one mass standard and one ballast weights set is used. If balance linearity is correctly defined then independently from the load (ballast size), measurement of the same mass standard shall provide the same results.

METHOD

a. A particular number of ballast weights is required. The greater the number of ballast weights, the more precisely tested weighing range of a balance. The weights mass shall be constant throughout the test but it is not required to know the exact value of their mass.

b. Prior test it is necessary to carry out balance adjustment procedure. Next, mass standard of specified weight is to be put on the weighing pan (m_{REF}), readout performed. Replace the mass standard with the first ballast weight (m_{t1}). Wait until the indication stabilizes and tare it. Place mass standard (m_{REF}) on the weighing pan next to the ballast weight (M_{t1}) and perform the readout again. Take the mass standard (m_{REF}) off the weighing pan, add another ballast weight (m_{t2}), tare the balance. Put the mass standard (m_{REF}) on the weighing pan and perform the readout for the third time. Such weighing cycle shall be carried out for the whole weighing range.

Fig. 30. Balance linearity – testing method
1 linearity points only 500 mg mass standard,
(ballast weight is at the bottom of a draft shield)

Fig. 31. Balance linearity – testing method
2 linearity points 500 mg ballast weight (B) and 500 mg mass standard

c. Method for testing linearity with use of ballast weights is practically unlimited when it comes to number of measuring points. Of course this manually carried out method is not recommended for hundreds of measurements to be performed. The greatest advantage of the said method is the fact that it does not require use of extremely precise mass standards (economy). There are two essential conditions under which the method is successful: balance stabilization (time, thermal) and stable ambient conditions.

d. If the balance is used within a partial weighing range then linearity test may be limited to the range (narrower range, narrower interval).

e. Repeatability influences each measurement, even linearity testing process. This influence may be partially limited by averaging the results obtained for tested points. The test result is also affected by sensitivity variation over time therefore it shall be performed quickly. Practically it takes few minutes.

NOTE

The balance linearity testing for the whole weighing range does not have to be performed frequently. Linearity is a parameter characterized by a stable value which means that it may be determined once at balance activation, usually in course of measuring system validation.

Each weighing result, for any sample, partially features error resulting from repeatability, linearity and balance sensitivity. All these combined together decide on measurement reliability. In order to specify deviation for linearity only, it is necessary to determine balance precision for a particular measuring point (SD calculation) first and take it into account when analysing the linearity. The measurement shall be performed right after balance adjustment, thus any deviation relating to balance sensitivity is eliminated.

It is worth to note that most technical guidelines provide information on the fact that linearity deviation is slightly greater than standard deviation. Dispersion of indication determines how much one can adjust the balance.

An example of a microbalance linearity test using the ballast weights method is shown below. The following assumption was made:

if the balance is perfectly linear over the whole measuring range, testing any point of its characteristics with the use of the same standard should give the same results, regardless of the applied ballast load.

Technical parameters of microbalance

– Type MYA 11.4Y PLUS
– Maximum capacity 11 g
– readability d= 1μg

Table 3. Microbalance linearity test – ballast method

Nominal mass	Tare	Real paterm mass	Indication	Linearity
(N)	(T)	(m_{ref})	$I_{(i)}$	$I_{(i)} – m_{ref}$
1 g	0 g	1.000005 g	1.000007	+ 0.000002
1 g	2 g		1.000003	- 0.000002
1 g	4 g		1.000009	+ 0.000004
1 g	6 g		1.000008	+ 0.000003
1 g	8 g		1.000004	- 0.000001
1 g	10 g		1.000001	- 0.000004

COMMENT

By default, it is assumed that the linearity deviation shows how accurately a certain amount of substance can be measured. However, it should be remembered that the value of the linearity deviation is affected by the precision of weighing the mass standard and the accuracy of determining its mass. As mentioned earlier, the precision of the measurement depends on the test conditions (temperature / humidity / vibrations), weighing skills (no impacts), thermal stability of the balance (indication drifts) and to a small extent on the centricity error.

The accuracy of determining the standard mass is specified in the calibration certificate along with the uncertainty of determining this value. Another factor that has a significant impact on determining the linearity deviation is the adjustment of the balance. Here, too, we have the influence of two factors, the precision of weighing the internal adjustment mass and the accuracy of its determination. There are quite a lot of significant factors, therefore the process of determining the linearity of a microbalance should be looked at a little more broadly than in the case of testing the linearity of balances with much lower resolution (when d ≥ 0.1 mg).

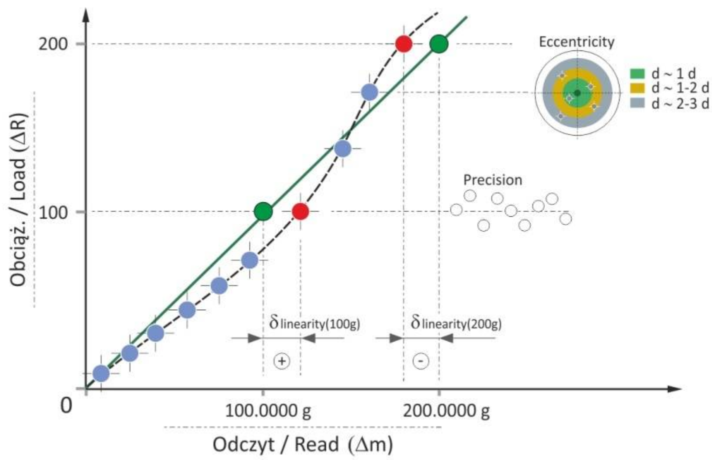

Fig. 32. Example of balance linearity

8. Diagnostic measures

Diagnostics means temporary balance control aiming at determination of its errors. Most users assume that it is nothing more than manual operation of placing mass standards on a weighing pan. Reality proves that it is not a rule. An internal adjustment weight is frequently used for balance parameters diagnostics. This provides an objective, human-fault free method. Automatic adjustment is a particular example of such method allowing balance sensitivity control and modification.

8.1. Adjustment

Adjustment procedure is designed to provide correction of balance indications. The said correction is possible in course of comparison of result obtained during mass standard (so called adjustment weight) weighment with the weighed mass standard value. The adjustment procedure completion practically assures correct balance sensitivity. This means that upon placing 200 g eight on a weighing pan, the indicator shall display 200.0000 g value. From a metrological point of view this assumption is right, providing that influence of repeatability of indications is not too significant.

Many analytical balances (d=0,1 mg) can be adjusted by means of an EXTERNAL adjustment weight, these balances are popular mostly due to their reasonably low cost. Balances with smaller reading unit (d = 0,01 ÷ 0,0001mg), among them microbalances, are always adjusted using internal adjustment weight. The characteristic feature of this adjustment system is traceability of temperature variation and time flow, this means that the adjustment process shall be performed automatically. The said mechanism is used for any RADWAG manufactured microbalance, ultra-microbalance. Means of balance adjustment operation at the moment of balance connection to mains has been presented below.

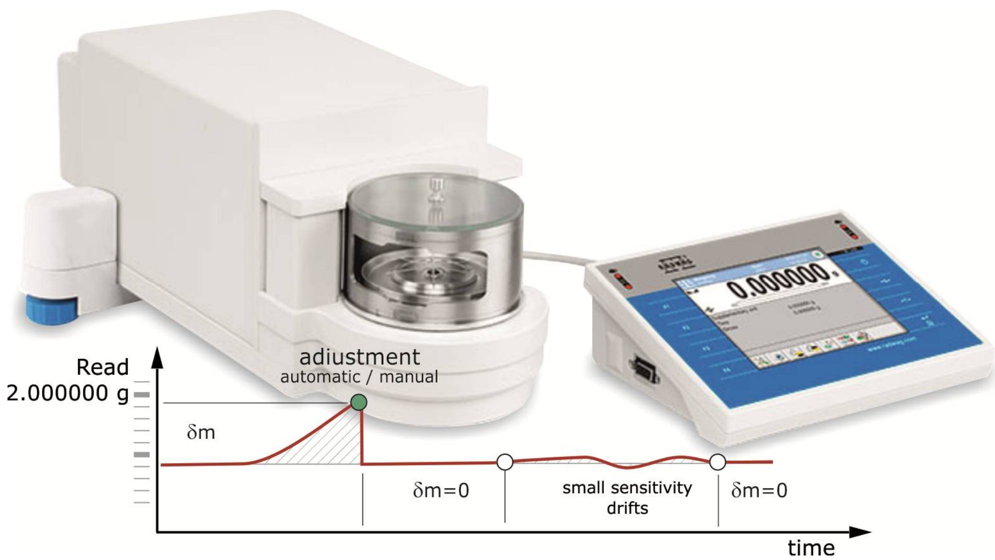

Fig. 33. Principle of balance adjustment operation

The methodology of the mass measurement process should ensure, with the greatest possible probability, that the balance will indicate correctly. It is assumed that in constant environmental conditions and for a thermally stable balance, the accuracy of balance indications after adjustment is maintained for a long period of time. However, for processes in which the weight of the product must be rigorously monitored, it is recommended to make adjustments before measurements, or at least to check the accuracy of the balance's indications with a mass standard.

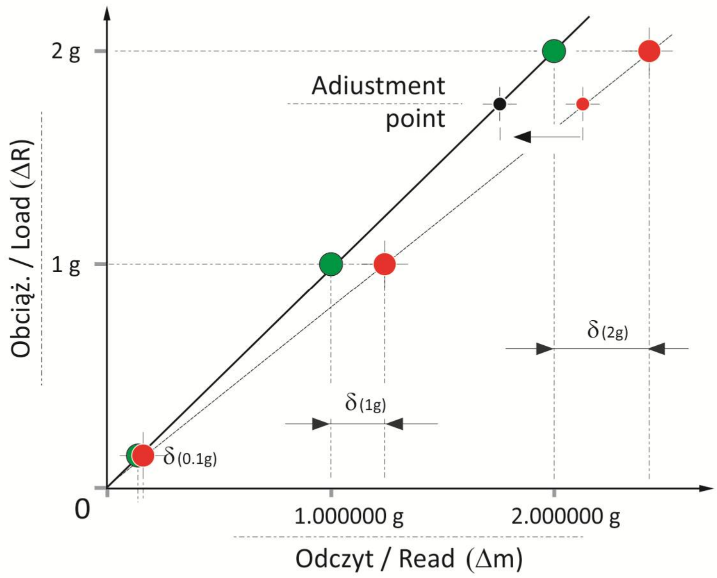

Fig. 34. Adjustment as sensitivity correction of the microbalance

Figure 34 shows that the possible problem of weighing accuracy is important when the sample weight is in the range ½ max ÷ Max. For samples with smaller masses, a more important parameter is the repeatability of indications, which determines the accuracy of the measurement. After each adjustment, a report is generated as a GLP procedure.

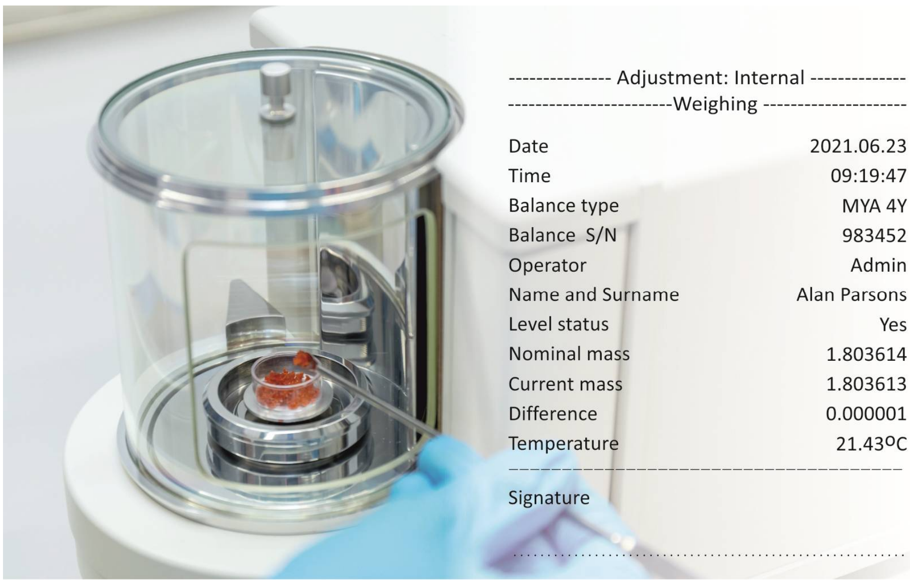

Fig. 35. Adjustment report

8.2. Autotest GLP

Undoubtedly it is repeatability of indications that is considered to be the most metrological important parameter. To check repeatability it is necessary to perform a specified number of measurements using a particular load of constant weight. Internal adjustment weight might be such a load, it is measured:

- automatically
- repeatedly
- when the same conditions are maintained

The test is performed when Filter and Value Release parameters have been selected. Depending on setup configuration of these parameters, it is possible to obtain some result variation. Final Autotest GLP report includes a lot of crucial information, the most important one is standard deviation.

Fig. 36. Microbalance XA 6/21.4Y.M.A PLUS series

The value of the adjustment mass is almost always in the range of ¾ Max ÷ Max. The repeatability result therefore always applies to the upper measuring range. How can this value relate to weighing in the lower scale range?

COMMENT

At the beginning of the measuring range, the repeatability is constant. This initial point is defined as a load of approximately 1000 d - reducing the load will not improve repeatability. However, with increasing load, the value of this parameter increases slightly. Having determined the value of the standard deviation through the GLP Autotest, you can expect slightly better repeatability when weighing lower weight samples. The dispersion of indications is slightly affected by the test method (manual is worse) and the properties of the sample.

8.3. Autotest FILTER

Most modern scales have a whole range of filters and other auxiliary settings that allow you to optimize the operation of the scale to the conditions of use. Effective use of these possibilities, however, requires some knowledge and a lot of practical tests. For this reason, a significant part of users is not able to apply these mechanisms in practice. In this case, the manufacturer's technical support is helpful - the Autotest Filter diagnostic function. The principle of operation of this function is to automatically test the repeatability of indications and determine the measurement time for all possible combinations of settings: filter $\rightarrow$ confirm the result. The user has 5 levels of filtering and 3 levels to confirm the result. As you can see, the assumption that the entire study can be done manually is rather wrong; even if it's time consuming. During the test, we use the mechanism of automatic application of the internal adjustment mass.

Weighing time is calculated as the average value of 10 internal mass weighing measurements. This parameter is a certain approximation of real time because the time counting algorithm assumes the moment of generating the "put weight" command as the beginning of the measurement. The end of the measurement is a stable state, reached according to the set criteria (user menu).

At the end of the procedure, the display shows a summary of the results for all tested settings. The filter sequence that was selected as active was marked with a tag. By analyzing the results from the report, you can choose the optimal setting for a given working environment of the balance, taking into account the duration of the measurement or the designated repeatability.

Fig. 37. Autotest FILTER - results

First part of the report contains data on a balance and working conditions parameters. The other part includes results obtained within the test. Current balance settings are marked with a tick. In order to swap to other settings one shall click a respective option.

----------- Autotest FILTR Report -----------

Balance type	XA 4Y
Balance ID	876573
Operator	Admin
Application revision	LL 1.S
Date	2021.05.27
Time	10:14:23
-----------------------------	----------------------
Reading unit	0.0001 g
Internal weight mass	209.65432 g
Temperature: Start	24.27 °C
Temperature: Stop	24.39 °C
Humidity: Start	51 %
Humidity: Stop	53 %

----------- Autotest FILTR Report -----------

Filter	Fast
Value release	Fast
Repeatability	0.00007 g
Stabilization time	1.688 s
Filter	Fast
Value release	Fast and reliable
Repeatability	0.00007 g
Stabilization time	2.255 s
Filter	Fast
Value release	Reliable
Repeatability	0.00007 g
Stabilization time	2.760 s
Filter	Normal
Value release	Fast
Repeatability	0.00007 g
Stabilization time	1.894 s
Filter	Normal
Value release	Fast and reliable
Repeatability	0.00004 g
Stabilization time	2.423 s
Filter	Slow
Value release	Fast
Repeatability	0.00006 g
Stabilization time	2.533 s

Fig. 38. Autotest GLP report

Legend:

- the shortest weighing time

- current balance settings in terms of filters

- the best measurement precision

- optimal solution for time and precision of measurements

8.4. Ambient conditions module

Control of main ambient parameters (temperature, humidity) may be performed automatically by means of microbalance mechanisms. Software allowing to specify both, limit values and dynamics for their variation and providing visualization is an ergonomic tool. Each microbalance monitors humidity, temperature and air pressure using internal sensors.

Fig. 39. Ambient conditions module

The microbalance has 2 internal temperature sensors (T1, T2), humidity, pressure and ground vibration sensors. All these sensors are built inside the microbalance. In addition, an external THB sensor can be connected to the USB port of the microbalance, which will record the variability of environmental conditions in the vicinity of the balance. After pressing the button (fig. 39), the environmental parameters of the balance and the workplace are visible.

Each change of environmental conditions beyond the set limits changes the color of the icons from green to red. The correctness of the indications of all sensors can be confirmed by a calibration certificate (Accredited Laboratory).

8.5. Weighing quality monitoring

Under normal operating conditions, the accuracy and precision of the weight measurement is constant. Unfortunately, environmental conditions usually show some oscillation or drift, the weighing method is not perfect and the operator's knowledge and capabilities may be limited. The sum of these factors determines the accuracy with which a substance can be weighed.

As mentioned earlier, it is possible to monitor environmental conditions and modify the weighing method, which allows to limit the impact of these factors on the mass measurement process. Thus, only the human factor remains as a potential source of errors. However, there is a clear distinction between knowledge and skills, the fact that I know does not mean that I can.

The principle of operation of each scale is to measure the force with which the Earth attracts the load being weighed, thus you should not generate too large impacts when placing the load on the weighing pan. Excessive impact is transferred to the mechanical system of the balance, which introduces some disturbance to the analysis of the compensating signal. As a consequence, the measurement result may have a slight error. With this in mind, Radwag microbalances introduce quality control of the weighing process. This is done by observing the dynamics that occur when placing a load on the pan. When the process is running correctly, the icon is green. (fig. 40).

Fig. 40. Quality control of the weighing process.

The red colour of the icon means that the load was placed on the pan with too much dynamics, which may result in a slightly longer measurement time and the measurement precision and accuracy may be disturbed. The analysis and visualization of the way of placing the load on the pan is also educational because it allows you to improve the manual skills of the operator. It is an important parameter of each weighing method.

9. Weighing applications

Balance as a universal weighing device may be used for determination of different objects weight. In order to obtain precise measurement one has to apply a particular methodology. In most cases it is the methodology that decides on the final measurement result. Therefore knowledge on balance operation, sample structure, disturbance and possibilities of settings modifications is beneficial when it comes to performance of precise measurement. Prior taking any actions one shall specify how precise his or her measurement has to be.

Some important facts concerning weighing have been described in this section. This information brings used methodology issues closer only partially. Advice on ambient conditions are contained within previous sections. In practice different combinations of metrological important factors and ambient conditions, uneasy to be foreseen, may take place.

9.1. Filters weight measurement

Filter mass determination almost always requires differential measurement, i.e. such measurement wherein the filter is weighed at least twice. This is crucial information number one and it shall be remembered by a user. Clean filter weight and weight of a filter subjected to absorption is insignificantly different, around tens of hundreds of milligrams. The measurement applies to low extend of the weighing range. This is crucial information number two. Regular filter size may vary between ϕ 20 mm and ϕ 100 mm or even between 210 x 254 mm. Balance design shall account for that. To what then one shall pay attention when performing these measurements?

a. Repeatability of indication

Decisive factor when it comes to measurement reliability is repeatable determination of a sample weight. Since there are at least two measurements, therefore repeatability influences the measurement at least twice. Repeatability result depends on various factors, those that relate to ambient conditions and to a sample, they have been already described before.

b. Sample stability

For a differential weighing sample is defined as anything that has been left in a filter structure after a performed weighing process. This could be dust, some solid particles, sediment etc. Independently from an amount of a particular sample it is obvious that it interacts with humidity influencing the sample weight variation. Both, sample and filter acclimatization is required. Acclimatization shall be a part of any methodology applied. Sample weight loss occurring during sample relocation or weighment may be a problem too.

c. Filter weight stability over time

Structure and material used for filter shall guarantee its weight stability over time. In course of testing this is controlled through so called blank tests (zero tests). This provides certainty or objective information on how much filter weight change influences the result of sample measurement. Any filters but cellulose ones shall be used since these absorb humidity. When humidity absorption occurs, drift of indication may be observed which might make one come to wrong conclusions either on balance quality or sample weights. Glass fibre or quartz filters are recommended in order to prevent described above situation. Some standards guide the operators to use Teflon filters (PTFE), nevertheless they do drag ones attention to the fact that Teflon filters may favour accumulation of unbalanced electrostatic charges. These charges shall be eliminated via ionization process. DJ-03 ionizer of Radwag production might be found helpful with regard to the above. The picture below presents ionizer and balance. The sample is subjected to ionization prior being put on a weighing pan.

9.2. Light loads net measurement

Light samples are such ones weight of which is not greater than 10% of maximum balance capacity. In practice it is possible to weight even loads of mass equal 0,02% of maximum load and lighter. What is substantial for measurement of such loads?

a. Firstly, indication stability.
   Lack of stability may be an effect of humidity impact exerted on a sample or of incorrect balance operation

b. Secondly, unloaded balance stability.
   Most balances feature zero indication, it is displayed when zero deviation is not greater than 0,25e. The operator reads displayed value of 0.000000 g, nevertheless it is necessary to be aware of the fact that the real value may change insignificantly within an extremely narrow range. It is possible to disable so called autozero function thus making any indication changes visible.

c. Thirdly, stable working conditions. For stable conditions, the measurement reliability depends on repeatability of indications. Balance settings optimization shall be discussed here, such optimization is understood as selection of respective parameters providing the best possible repeatability of indications.

d. Weighing skills, i.e. the ability to place loads on the weighing pan without impacts

e. sample stability - hygroscopicity of the sample (powders with low humidity), influence of electrostatic charges (plastic samples, PTFE filters), sample temperature close to ambient temperature (sample conditioning)

f. measurement methodology

9.3. Liquids checkweighing and control

Weighing procedure designed for liquids shall respect not only issues concerning weighed substance but also the weighing process itself. Evaporation processes shall be minimized through sample acclimatization and use of particular weighing vessels. The whole process comprising tarring, filling shall be optimally short.

Fig. 41. Weighing liquid by means of vessel with a tapered inlet, liquid evaporation in course of weighing limited

Fig. 42. Weighing liquid by means of so called evaporation ring, designed to check piston pipettes using balance

Photo 1 – MYA 11.3, Used vessel has to account for amount of measured liquid, the vessel shape shall limit liquid evaporation. Weighing process comprises flask tarring and filling. Weighing the liquid takes about 6 seconds.

Photo 2 – MYA 21.3Y.P,
So called evaporation ring has been used here, the ring minimizes distilled water evaporation. This is one of the parameters considerably influencing piston pipette volume determination. Sequential portions of liquid discharged from the pipette are dosed into the vessel, the balance is tarred after each cycle.

Liquid weighing processes sometimes use dosing function, wherein dosing is performed until a specified value, with a certain tolerance, is reached. Apart from dosing, checkweighing function is also used when it comes to liquid weighing processes. Checkweighing means measuring off a particular amount of liquid that shall be contained within specified limits. Slow increase of sample weight may require a different system of filtration for a measuring signal. Such solution is automatically carried out within dosing function of RADWAG balances. while using this function the user needs to pay attention to ambient conditions stability. System for filtering the signal is more prone to any weight variations, this is to ensure detection of the slightest weight increase or decrease.

9.4. Comparative weighing (comparison)

Comparative weighing application is based on presentation of difference between two objects, wherein one of them is a mass standard. Commonly comparison is associated with mass standards and ABA or ABBA methods, nevertheless it can be carried out in reference to any object, but:

a. Comparison should take little time
b. It shall control balance sensitivity if periods between weighing are long
c. Attention to sample stability should be paid (acclimatization)

Diversity of processes to which a particular sample can be subjected requires a respective methodology, among the processes there are: heating, abrasion, layers dusting, melting, sorption, burning, ashing, etc.

Comparison process for legal metrology means contrasting weights, mass standards in order to specify variation between an object and a mass standard. Two measuring methods are of use here: ABA, ABBA. The cycle may be either semi-automatic one (manual comparators) or automatically. Comparators stand in need of appropriate ambient conditions (dynamics of changes over time) allowing to provide reliability.

Exemplary report on comparison – UYA 3Y.KO comparator

```
-------------- Comparator --------------
User            Radwag
Start date      2014.07.03 15:15:13
End date        2014.07.03 15:23:47

n  |A          |B          |A          |D
1  |0.1000674  |0.0999239  |0.1000681  |-0.00014385
2  |0.1000684  |0.0999239  |0.1000696  |-0.0001451
3  |0.1000685  |0.0999238  |0.1000688  |-0.00014485
4  |0.1000690  |0.0999242  |0.1000690  |-0.0001448
5  |0.1000695  |0.0999243  |0.1000693  |-0.0001451
6  |0.1000693  |0.0999248  |0.1000691  |-0.0001444

Mean average          -0.000144683333 g
Standard deviation    0.000000482355 g
Number of cycles      6
Method                ABA
----------------------------------------
```

Cycle of comparison may be monitored by RMSC PC software. Module responsible for calculation cooperates with comparators and ambient condition recorders. The software manages the whole process of comparison from the moment of order placement, through calibration, to calibration certificate issuing.

9.5. Measurement of heavy weight samples

One may observe different approaches to definition of heavy weight samples. It is very difficult to provide a specified value of such weight therefore referring to a weighing range of a particular balance is much better solution. Weight contained within ¾ of max. capacity and max. capacity (Max) may be considered to be heavy. Such measurements reliability depends on described below factors.

a. Sensitivity variation over time

Each balance is adjusted on the basis of comparison wherein the weighing result is compared with mass standard value – such comparison is part of a production cycle. Through balance lifetime and its operation a periodical control and verification of indications correctness is carried out in course of adjustment. Length of an interval between successive adjustments depends on time and temperature variations. The adjustment is automatic and right after its completion the balance indication is an optimal one. However after some time unspecified drifts of indication may occur, these can be a result of sudden changes of external conditions causing measurements inaccuracy. In order to eliminate measurement inaccuracy it is advised to use a respective button and perform adjustment.

b. Repeatability of indications

Repeatability is a metrological important parameter. It takes its value dependently from conditions for which it is tested. For typical laboratory practice, a single weighment of a particular sample is carried out, therefore it is not possible to observe how repeatability influences the measurement result. Such relation is determined in course of individually performed tests (validation), it is also possible to refer to manufacturer's declaration.

While weighing heavy weight samples it is necessary to:

- minimize effect of ambient conditions,
- optimize balance parameters.

If precise and reliable analysis of a weighing process is needed, then repeatability of indications shall be tested by means of loads used for the process performance.

c. Balance linearity

In theory, balance characteristic is a linear relation between applied load and observed indication. In practice it happens that in course of factory adjustment process some deviations of this relation occur. The said deviations are a result of measurement methodology and characteristics of used mass standards. Linearity deviation value is constant therefore it can be taken into account. More detailed information is received during calibration, nevertheless it must remembered that for calibration, mass standards accuracy class is an important factor.

d. Ambient conditions effect

Heavier weight almost always means increased volume. This is true even when dosing small amounts of liquid into a flask of great volume. While weighing samples wherein right conditions are provided, the increased volume does not impact the weighing process. For inappropriate conditions the measurement may not be exact due to disturbance being an effect of air flows. If such is a need a supplementary draft shield shall be used.

The greater sample surface (packaging, container) the more intense electrostatic interference. Solution to such a problem might be use of ionizer providing electrostatic charges balance on a given sample surface.

Greater sample surface means greater absorption or evaporation. These require sample acclimatization prior weighing. Possible processes are similar one to another, nevertheless each of them shall be diagnosed with reference to required accuracy and particular balance capabilities.

SUMMARY

The accuracy of mass measurement can be affected by many factors, the sum of which can lead to significant measurement errors. Typically, only one of the areas and one of the factors is relevant. Reducing the impact of this negative factor on the mass measurement process is usually sufficient.

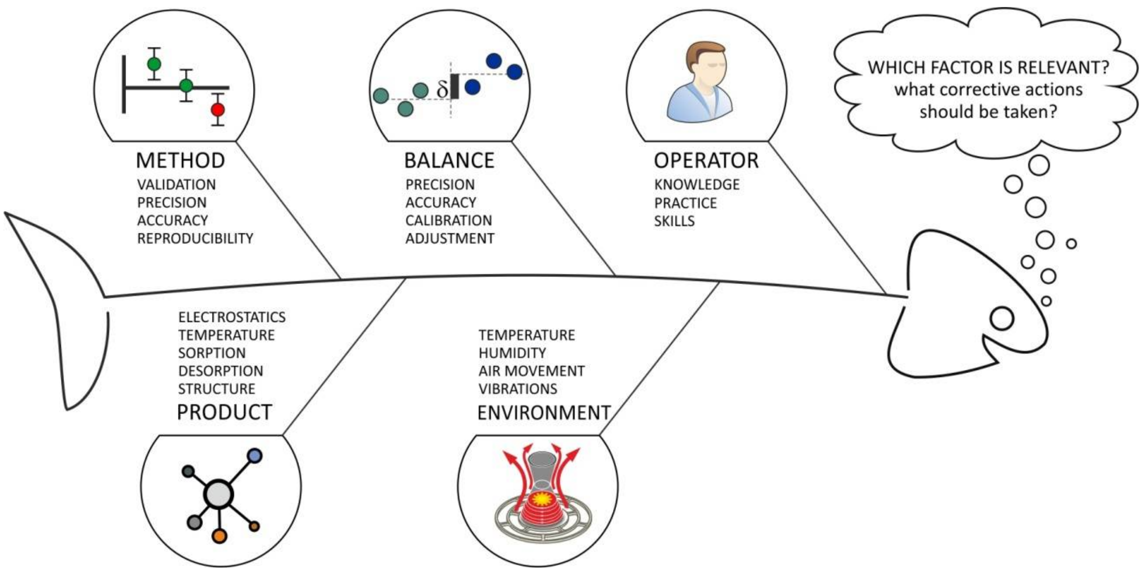

Fig. 43. Ishikawa diagram - sources of errors in mass measurements.

10. Conformity with regulations

When it comes to a balance, there are various areas of use as far as branch is concerned (pharmacy, petrochemistry, environmental protection, etc.), not to mention geographical location. For any possible branch there are respective requirements which on one hand specify balance design, and on the other hand define limit values for the balance metrological important parameters.

Fig. 44. Microbalance and standard mass

It can be said that the basis for most control procedures is the methodology given in the OIML R76 documents. It contains a list of metrological tests, classification of weights and values of maximum permissible errors. However, it should be noted that only some of the scales are subject to legal metrology requirements. Many users who are not subject to legal metrology have their own requirements for weighing accuracy, but continue to use the OIML procedures, adding to or limiting them to those tests that are relevant. A separate issue is the measurement uncertainty, which is a necessary supplement to the mass measurement process.

10.1. Legal metrology and practice

Legal regulations resulting from WELMEC law acts, OIML guidelines and drafts are commonly known mainly due to the fact that WELMEC is a globally operating organization. WELMEC-approved methods and procedures are world-wide spread and used in course of individually performed testing. The said methods and measurements demand defining limit values (MPE). The limit values always refer to verification scale interval e, they include used load m. Example for balances of I accuracy class:

Table 4. Maxium permisible error for balances I class accuracy

Load	0 e ≤ m ≤ 50 000 e	50 000 e < m ≤ 200 000 e	200 000 e < m
MPE	0.5 e	1 e	1.5 e

Stating that balance complies to legal regulations means that errors occurring while measurement performed by its means are comprised within specified thresholds. This is just a general information giving no details on reliability of any weighing system. Knowing that the narrowest verification scale interval is 1 mg and that reading units and MPE values are as follows:

- d = 0.1 mg MPE = 5 d e.g. AS 220.R2 PLUS
- d = 0.01 mg MPE = 50 d e.g. XA 82/220.4Y PLUS
- d = 0.001 mg MPE = 500 d (microbalance) e.g. MYA 5.4Y PLUS
- d = 0.0001 mg MPE = 5 000 d (ultra-microbalance) e.g. UYA 2.4Y PLUS

Then comment informing that deviation of balance indication is not greater than MPE in no significant fact at all. This is often the reason why most balance users obliged to apply the devices in accordance with legal regulations decide to perform adjustment procedure. This provides information on errors of the particular balance. It may be concluded that legal metrology regulations are not relevant when it comes to high resolution balances. Still, the testing methodology is of importance and use.

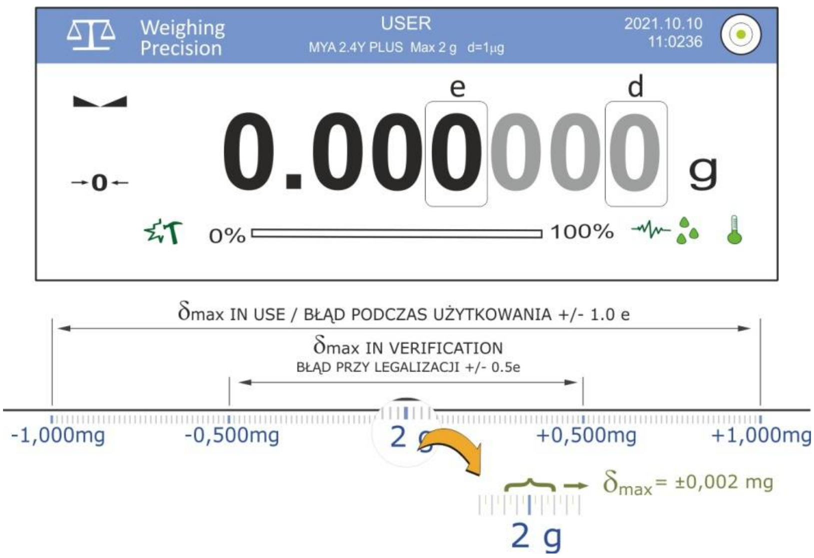

Fig. 45. Microbalance – legal metrology

10.2. GMP – metrology for industry

As for the methodology perspective, industrial metrology is based on the same schemes that legal metrology is. The main difference when comparing the former one to the later, is specifying required measurement reliability first, next checking if the requirement has been met. Two problems may be noticed here.

As for the first one, it may be difficult to determine how big the greatest deviation shall be in relation to the real value. It is worth to memorize that for some cases this parameter may be determined only by tests performed by means of a mass standard. Test of eccentricity or repeatability may be carried out using any load of mass constant over time.

The second problem concerns testing methodology, more specifically choice of the most appropriate tests, such ones that:

- are appropriate for the performed measurements range, i.e. only necessary respective tests are selected, this is described in Risk Analysis documentation – QRM process production, formality and documentation shall be adequate to level of risk and it shall be based on knowledge,

- are quick and simple, test of the balance shall not interrupt operating cycle,

- provide basic information allowing to decide on post testing balance use, adjustment or ruling it out

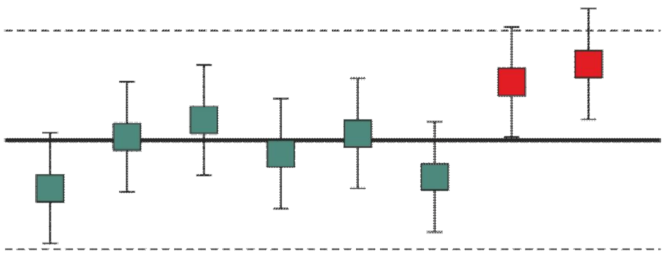

Fig. 46. Limits in weighing process

A great help for testing results interpretation is establishment of Warning Limits and Critical Limits.

10.3. Pharmacy

Extraordinary requirements for this branch are mainly a result of the fact that these measuring devices are used for medicine production. Their quality may affect particular drugs quality and as a result exert an impact on patient's health. Apart from legal regulations valid for a particular region also pharmacopoeia guidelines are adhered to. Pharmacopoeia are documents binding for a particular continent, e.g. American , Japanese, European, Russian pharmacopoeia, etc. American pharmacopoeia is the one of the greatest priority.

There are two chapters devoted to requirements concerning balances, <General Chapters, Apparatus for Tests and Assays <41 ,,BALANCES"> and <General Information, <1251 ,,WEIGHING ON AN ANALYTICAL BALANCE">. The former one describes specification for repeatability and accuracy, the description is as follows:

Repeatability is satisfactory if two times the standard deviation of the weighed value, divided by the nominal value of the weight used, does not exceed 0.10%. If the standard deviation obtained is less than 0.41d , where d is the scale interval, replace this standard deviation with 0.41d

$$R = \frac{2 \cdot S}{m} \leq 0.10\% \quad \rightarrow \frac{2 \cdot S}{m} \leq 0.001 \tag{9}$$

where: *S* – standard deviation (10 repetition)
m – weight of the standard with which the balance was tested

Assuming that the repeatability of balance indications for small loads has a constant value, then knowing the value of the standard deviation (S) it is possible to indicate the limit value at which the condition required by USP 41 and Ph Eur 1.27 is met. (equation 9).

Table 5. MYA 5.4Y PLUS – MSW evaluation USP 41

(mg)	mass (mg)	USP 41
S = 0,0006	0.1	0.0120
Limit = 0,001	0.2	0.0060
	0.5	0.0024
	1	0.0012
	1.2	0.0010
	1.4	0.0009
	1.6	0.0008
	1.8	0.0007
	2	0.0006

Requirements for balance indication repeatability, sample weighing precision are met when the sample weight is greater than/equal to 1.2 mg.

The requirements for measurement accuracy according to USP 41, PH Eur 1.2.7 is:

The accuracy of a balance is satisfactory if its weighing value, when tested with a suitable weight(s), is within 0.10% of the test weight value. A test weight is suitable if it has a mass between 5% and 100% of the balance's capacity.

According to the above description, the MYA 5.4Y PLYS microbalance accuracy test should be performed in the range of:

- 0.25 g (10% Max) to
- 5 g (Max – scale load).

The measurement error of the reference mass in each tested point cannot be greater than 0.10% of its mass. Of course, the mass of the standard should be known before the test, its value is given in the calibration certificate

Chapter USP 1251 is not mandatory, it contains explanations and definitions of terms that are important for electronic balances: sensitivity, accuracy, linearity, centricity and the so-called. minimum weight. The minimum weight value determines the beginning of the weighing range for the balance. The value of the minimum mass is determined from the relationship (10), which is a transformation of the relationship (9).

$$MSW = 2000 \cdot S \tag{10}$$

In order to comply to regulations of pharmacy industry it is necessary to meet chapter 41 requirements, especially those intended for repeatability of indications. As it is commonly know, it is repeatability that decides on measuring accuracy, i.e. balance reliability. For microbalances this parameter is used for MSW determination.

HOW TO DETERMINE MINIMAL SAMPLE WEIGHT?

In order to determine minimal sample weight one shall:

- perform a series of 10 repetitions using a weight standard
- the mass of the standard used for the test should be much greater than the expected MSW value (recommendations USP 1251, Ph Eur 1.2.7)
- the standard deviation should be calculated from the obtained results
- calculate the MSW value according to equation (10)

NOTE

Having in mind the above information, it may be concluded that in order to obtain as low MSW value as possible it is necessary to provide proper working conditions. The greater resolution, the more demanding requirements for ambient conditions stability.

For balances with lower resolution, it is possible to obtain all the same results during the repeatability test, which means that mathematically the value of the standard deviation would be zero. In this case, the USP 41 order should be used and the value of the standard deviation should be calculated from the relationship:

$$S = 0.41 \cdot d \tag{11}$$

where: d – value of the elementary division of the balance

Assuming that the value of the standard deviation is less than 0.41d, the so-called Minimum mass in relation to the scale interval (table 7). The values shown in Table 7 are the smallest possible MSW values that are potentially possible.

$$MSW = 2000 \ \cdot \ 0.41 \cdot d = 820 \ \cdot d \tag{12}$$

Table 6. The MSW value depending on the scale division

d (mg)	Type of Radwag balance	Standard deviation S (mg)	MSW value (mg)
10	PS 6000.R2 PLUS	4.1	8200
1	PS 1000.X2	0.41	820
0.1	AS 220.X2 PLUS	0.041	82
0.01	XA 82/220.4Y PLUS	0.0041	8.2
0.001	MYA 5.4Y PLUS	0.00041	0.82
0.0001	UYA 2.4Y PLUS	0.000041	0.082

In addition to the metrological aspect, functional requirements for the use and safety of scales are also important. These topics include, for example:

- several-level authorization system
- user login mechanism
- register of changes to settings, etc.

Detailed requirements are contained in documents, e.g. 21 CFR Parts 11.

SUMMARY

From January 2022, the requirements for electronic scales are the same in the American (USP) and European (Ph. Eur) pharmacopoeias. This is one of the elements of unifying the quality requirements for the global drug manufacturing market.

10.4. Environmental protection

The use of balances in processes related to environmental protection requires compliance with the legal requirements applicable in a given area and with the normative requirements. As previously shown, the legal requirements contain quite high MPE values, so metrological most balances meet them with a large margin. Legal metrology requires periodic verification of balances by an authorized entity, which is not always sensible from the point of view of metrology, example of a microbalance. For this reason, most users of microbalances decide to carry out the calibration procedure, thanks to which they obtain certain information about potential weighing errors.

An example of a calibration certificate is presented in the further part of this document.

RADWAG Wagi Elektroniczne Witold Lewandowski
26-600 Radom, ul. Toruńska 5

CENTRUM METROLOGII, BADAŃ I CERTYFIKACJI - LABORATORIUM POMIAROWE
26-600 Radom, ul. Starowiejska 17A
tel. /38/ 386 64 70; fax /48/ 385 00 11

PCA POLSKIE CENTRUM AKREDYTACJI >o< WZORCOWANIE
AP 069
ilac-MRA

Calibration laboratory accredited by
Polish Centre for Accreditation, a signatory to EA MLA and ILAC MRA
that include recognition of calibration certificates.
Accreditation No AP 069.

CALIBRATION CERTIFICATE

Date of issue: 17 June 2021 | Certificate No: 6076/2252/21 | Page: 1 / 2

OBJECT OF CALIBRATION	Non-automatic electronic weighing instrument - single range Manufacturer: RADWAG Wagi Elektronicze Type / symbol: MYA 5.4Y Serial No: 702517 Capacity *Max*: 5,1 g Scale interval *d*: 1 mg
APPLICANT	RADWAG Wagi Elektronicze ul. Toruńska 5, 26-600 Radom
USER	
PLACE OF CALIBRATION	RADWAG Wagi Elektronicze Laboratorium Pomiarowe ul. Starowiejska 17A, 26-600 Radom
CALIBRATION METHOD	Calibration Procedure: PW 01 rev. XIII of 28 February 2018
ENVIRONMENTAL CONDITIONS	Air temperature: (22,59 ÷ 22,94) ± 0,20 °C Relative humidity: (54,3 ÷ 56,3) ± 1,1 %
DATE OF CALIBRATION	17 June 2021
TRACEABILITY	This certificate is issued under the agreement EA MLA in the field of calibration and provides treacebility of measurement results to the International System of Units (SI)
CALIBRATION RESULTS	The results have been presented on page 2 of this certifictae including uncertainty of measurement.
UNCERTAINTY OF MEASUREMENT	Uncertainty of measurement has been evaluated in compliance with EA-4/02 M:2013 The expanded uncertainty assigned corresponds to a coverage probability of 95 % and the coverage factor k = 2.

Akredytowane Laboratorium Wzorcujące AP 069

Kierownik ds. technicznych
Laboratorium Pomiarowego
mgr inż. Tomasz Kasprzak

This certificate may be presented or copied as whole document only

Fig. 47. Calibration certificate

CALIBRATION CERTIFICATE issued by ACCREDITED LABORATORY No AP 069

Date of issue: 17 June 2021 | Certificate No: 6076/2252/21 | Page: 2 / 2

CALIBRATION RESULTS The results presented below refer only to the calibration object described on the first page of this certificate

Adjustment device: internal
Adjustment weight: internal

MEASUREMENT ERROR AND REPEATABILITY

Load L g	Load TARE L_T g	Mass reference m_{ref} g	Indication of instrument I g	Measurement error $E(I)$ g	Standard deviation s g	Uncertainty of measurement $U(E)$ g
0,001		0,0010011	0,001001	0,000000	0,0000009	0,000001
0,5		0,5000014	0,500002	0,000000	0,0000013	0,000005
1		0,999997	0,999998	0,000001	0,0000008	0,000005
2		2,000003	2,000003	0,000000	0,0000012	0,000007
5		4,999988	4,999988	0,000000	0,0000013	0,000009

Authorized by: Tomasz Kasprzak

Fig. 48. Calibration certificate - results

11. Features

Modern weighing devices shall be characterized by proper metrologically important parameters and by particular ergonomic and software solutions. Market demands influence the final outcome, i.e. development scope and direction. Users' requests and comments contribute to both, progress and advancement of high resolution balances.

11.1. Speed

Weighing speed as a parameter of electronic balances usually refers to the measurement time, i.e. the time in which the sample weight can be determined. When analyzing the catalog data of scales, marketing content should be clearly distinguished from the actual technical capabilities of the device. In the case of ultra-microbalances and microbalances, the measurement time consists of two cycles. The first is access to the weighing chamber, the second is weighing. The weighing chamber opens and closes in about 1.2 seconds. The operations of placing a load on the pan are difficult to estimate due to the individual characteristics and capabilities of the operators. The actual weighing time of a sample is not significantly dependent on its weight and amounts to about 6 seconds. This is the time needed to obtain a uniquely stable measurement. Of course, by modifying the balance parameters, such as the weighing profile, you can obtain shorter weighing times, but you should remember that usually increasing the measurement speed means reducing the weighing precision. Table 7 shows exemplary weighing times for scales with different scale intervals.

Table 7. Examples of mass measurement times depending on the selected weighing profile

Name of balance	XA 6.4Y PLUS	XA 210.4Y PLUS	AS 220.X2	PS 4500.X2
Sample mass / d balance	0.5g / 0.001 mg	20 g / 0.01 mg	10 g / 0.1 mg	500 g /10 mg
Profile Fast	6 sec.	3 sec.	3 sec.	2 sec.
Profile Fast dosing	9 sec.	7 sec.	3 sec.	2 sec.
Profile User	8 sec.	7 sec.	3 sec.	3 sec.
Profile Precision	15 sec.	14 sec.	6 sec.	4 sec.

The data presented in table 7 shows that the larger the scale division, the smaller the differences in mass measurement times, regardless of the selected profile. The shortest weighing time can be obtained with the Fast profile and the longest with the Precision profile. The Fast Dosing profile is a method of fast weighing, but with very high excitability of the balance (detection of very small changes in weight). Therefore, the measurement time in this profile is usually longer than in the Fast profile.

NOTE
Measurement or measuring off a particular amount of a sample, wherein the resolution is at least 20 million reading unit, shall be approached form accuracy perspective, e.g. 3/20 million., not speed of measurement. Most experienced users find this issue clear.

11.2. Touch-free operation

Manual abilities of an operator may be limited by characteristics of a workplace or by a required testing methodology (suit, gloves etc.). Owing to proximity sensors, RADAWAG microbalances and ultra-microbalances may be operated regardless of the said limitations. These sensors allow the operator to perform various operations: printing, tarring, opening or closing the draft shield, selecting the name of a sample intended for weighing and many more. Sensors operation power may be adjusted which is useful when there is a need for their activity area optimization.

Fig. 49. Proximity sensors

11.3. Wireless operation

Most balances with module design use cables connecting particular components. Such solution is the most common one but not always satisfactory. There are situations when it should be possible to access the draft shield from more than one side. This requires terminal to be located somewhere in a distance. Limited by cable length space between balance and terminal is a serious drawback therefore RADWAG balances use Bluetooth connection allowing to locate the terminal anywhere in a distance (up to 10 meters). This is a convenient solution when placing a balance inside fume cupboard or Glove Box type of chamber.

11.4. Safety

It is frequent that balance parameters optimization takes place in course of validation process, this is performed in order to provide the best possible settings allowing respective weighing accuracy. These parameters should be used throughout balance lifetime. They are protected against unauthorized modification, few access levels accessible. Some particular options and balance functions are enabled for each operator individually, e.g. weighing, printing, saving data etc. The operator logs in using his own password, after completing particular set of operations he or she logs out. It shall be remembered that all measurements are recorded into so called ALIBI memory, wherein data such a date, time, operator name etc. are specified. The protection is based on a system comprising 4-level access structure, owing to that it ensures:

a. ergonomics (a particular operator takes only necessary actions, which is time-saving advantage)
b. stability of balance parameters that are decisive for accuracy (accuracy guaranteed regardless of the operator's manual skills)

11.5. Customization

Customization is simply a process of fitting balance operation to one's needs. The scope of possible modifications depends on a particular balance program. Microbalances and other balances manufactured by Radwag offer vast range of options and functions that may be customized. For a particular user the following can be personalized:

a. Password
b. Access level
c. Language version for an interface
d. RIF card number
e. Possibility of fitting working environment so that is suited individual preferences. One's own profile, referring to used application, shall be created. It is possible to preset the following:
    - parameters concerning result stability – menu for readout
    - information displayed in INFO area
    - quick access buttons
    - default profile, run at the start after logging procedure completion
    - required printout type

11.6. Multi-functional working environment

Weighing module of each balance shall provide measurement accuracy. This is guaranteed by internal adjustment systems like in case of Radwag manufactured balances. the measurement may be monitored by means of so called weighing applications. These can be divided into two groups. The first group comprises applications relating to weighing:

a. dosing
b. checkweighing
c. parts counting
d. % control of sample weight
e. formulas
f. density determination

The second group includes Statistics, SQC, Databases which are designed to process and collect information. Simultaneous operation of numerous applications forms multi-functional environment enabling performance of complicated projects.

11.7. Service support – media module

User-friendly interface features touch panel and it seems to be uncomplicated when it comes to operation. Long-term work proves that software mechanisms may turn out to be ambiguous. Any doubts and uncertainty may be resolved by means of user manuals, however one may apply more convenient method, namely MEDIA module which is supported by Radwag balances. The module supplies balance users with instructional videos on various areas of use.

SUMMARY

Weighing applications are a group of application programs that develops quite dynamically, mainly as a result of comments and needs of users. Therefore, it is difficult to talk about the potential stability of these applications, rather they should be perceived as something that is changing in an innovative way, following the trends and needs of the market.

12. List of tables

13. List of drawings

Advanced Weighing Technologies for Laboratory

www.radwag.com