

Micro Scale Measurement

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Sławomir Janas

MICRO SCALE MEASUREMENT

Installation at the workstation • Usage precautions • The best measuring capacity Ambient conditions effect • Optimization as a balanced process SOP – monitoring of metrologically important parameters • Diagnostics measures Weighing applications (filter weight measurement, light loads measurement, measuring liquids) Conformity with regulations (legal metrology and practice, GMP, pharmacy-USP)

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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.

Research Laboratory Manager RADWAG Balances and Scales

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TABLE OF CONTENTS

1. Introduction	•••••
	<u>6</u>
2. Installation At The Workstation	6
2.1. Workstation	7
2.2. Acclimatization	8
2 Operating Conditions	
<u>5. Operating conditions</u>	10
4. Measuring Capacity – how to obtain precise measurement	<u> 11</u>
	14
5. Ambient conditions effect	<u> 15</u>
5.1. Workstation and its location	15
5.2. Personnel	15
5.3. Workplace temperature	16
5.3.1. Self-heating	17
5.3.2. Systems for temperature control	19
5.3.3. Accuracy and variable temperatures influence	19
5.4. Moisture content and the weighing process	20
5.4.1. Disturbed humidity and the measurement	21
5.4.2. Humidity influence on sample weight measurement	22
5.5. Air flow	23
5.6. Unbalanced electrostatic charges	24
5.7. Ground vibrations	25
6 Ontimization as a halanced process	27
6.1. Optimization – effect on metrological performance	27
6.2. Optimization of speed	32
7. SOP – monitoring of metrologically important parameters	<u> 33</u>
7.1. Repeatability of indications	<u> 33</u>
7.2. Eccentricity	<u> 34</u>
7.3. Linearity	35
8. Diagnostics measures	37
8.1. Adjustment	37
8.2. Autotest GLP	38
8.3. Autotest FILTER	40
8.4. Ambient conditions module	42
0. Weighing applications	
9. Weigning applications	<u></u> 44
9.1. Filters weight measurement	44
9.2. Light loads net measurement	45
9.3. Liquids checkweighing and control	46
9.4. Comparative weighing (comparison)	47
9.5. Measurement of heavy weight samples	48

10. Conformity with regulations
10.1. Legal metrology and practice50
<u>10.2. GMP – metrology for industry51</u>
<u>10.3. Pharmacy</u>
10.4. Environmental protection52
11. Features
53
<u>11.1. Speed</u> <u>53</u>
11.2. Touch-free operation54
11.3. Wireless operation54
<u>11.4. Safety</u>
11.5. Customization
11.6. Multi-functional working environment56
11.7. Service support – media module56

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.



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.



- 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 mc significant issues is the amount of time needed for balance acclimatization. The said time is 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 t' temperature stabilization period. Actually the period about few hours long, therefore PRECI. 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 therm 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 sh be aware of the fact that for such a case, metrologically important parameters may significant 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.



Fig. 5. Microbalance design, weighing module external (b) and internal housing (a)

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 necessar for most measuring devices. This influences the adopted approach.



Providing working conditions that allow the best possible measuring performance is a sta operation. It is always difficult to estimate how much does variation dynamics of ambient conditions influence the measurement. The most basic test concerns balance indic: b repeatability, the said test means determination of standard deviation being an outcome of serier of measurements. Knowing the standard deviation value, one may consider whether it is possit to obtain even lower rate. It is worth to mention that obtained standard deviation is a ra 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 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 influent of ambient conditions on measurement indication.

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

4. Measuring Capacity – how to obtain precise measurement

This seemingly simple issue turns out to be a bit sophisticated problem in reality. It is due to the fact that measuring capacity, understood as "ACCURATE" and "PRECISE" measurement, is a sum of many factors.



Fig. 7. Accuracy and precision in metrology

In order to determine sample weight, one has to assume that the obtained result is both, real and accurate. In order to provide confirmation for the above mentioned, it is necessary to check balance indications by means of mass standards. The procedure is quite simple. First, balance adjustment must be carried out, next a respective mass standard shall be put on the weighing pan, i.e. such mass standard, weight of which is contained within weighing range for a real sample weight. If balance indicates correct mass standard weight (including its error) then it is certain that the sample weight will be estimated correctly as well. Even serious deviation from an expected value, indicated in course of mass standard measurement, may be used for determination of real sample weight. This deviation shall be treated as a systematic error.

The main problem while checking accuracy, is the necessity of having a mass standard characterized by an appropriate accuracy class and weight value (equal to sample weight value). This problem is one of economic origin since mass standard purchase and maintenance costs are high. It is assumed that as a result of service adjustment processes the balance is characterized by a linear relation, therefore it is enough to test it by means of one mass standard. Its weight may be freely selected out of ½Max ÷ Max range. Such approach does not take into account the effect of linearity – which is assumed to be of minor importance.

The above described solution is used quite often for daily, simple tests. Despite theoretically linear relation between an indication and the loading, it is recommended to check the readings by means of mass standards of weight similar to weighed samples. This may prove that balance accuracy is indeed correct as far as its weighing range is concerned.

Those users who operate the full weighing range, may find documents on Quality Control highly useful. A good example of such documents may be those provided by Radwag, like Q.C. Test Report.

	RADWAG	
Q.C. TE	ST REPORT	2
		1.
	Certificate number 441020	5
acka 28, 26-600 Radom, Polska	Data of issue 40.45.0014	
vw.radwag.pl	Date of issue 10.15.2014	C
roduct specification		
Model PS 1000 P2	Max capacity [Max] 1000 ~	0
Serial number 441036	Readability (d) 1 mg	4
Software version 2.0.0		-
Soliware version 5.0.0		
Product code		C
	-	-
est ambient conditions		
Temperature 23 °C	Date of test 10.15.2014	
Humidity 51 %	Time 11:21	
Pressure 1001 hPa		<

Estimation of measurement precision is much easier since it is characterized by repeatability of indications. It is possible to speak about a precise measurement when exactly the same or insignificantly different results are obtained. This test is performed by means of mass standards, although it may be carried out using any object of constant over time weight. This is an ideal solution for those who want to determine repeatability of indication for a packaging, glass flask, initial sample weight (differential weighing), clean filter (differential weighing) etc. usually 6-10 measurements are performed and standard deviation is calculated out of the measurement series according to the following formula:

 $sd = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n-1}}$

where: s - standard deviation

x_i - successive measurement

- \overline{x} arithmetic mean of a series of measurements
- n number of measurements within measurement series

It may happen that an exceptional situation occurs during the test -i.e. all the measurements are of equal values. For such an instance standard deviation is calculated according to the following equation:

Sd=0,41 d

d - reading unit.

The measurement precision depends on repeatability of indications, and on the following:

- ambient conditions (dynamicity of changes)
- manual and intellectual skills of the user (knowledge, abilities, shock-free measurement)
- test duration,
- used equipment (the measurement is sometimes performed by means of an under-pan weighing system, not necessarily the weighing pan)

The best measuring capacity therefore may be perceived as

- repeatability od indications, when "precise sample" weight is of no interest to us. This occurs during differential weighing where difference of sample weights is crucial (weight before and after the process end)
- accurate and precise measurement, which takes place when one is to measure determined weight value of a particular sample

It is advisable to select such balances for respective procedures that provide compliance with weighing accuracy requirements, simultaneously assuring that some reserve is left. This is to be done by assessment of technical specification comprised within product folders. Even for this kind of approach, measuring capacity shall be determined in a workplace characterized by ambient conditions existing there typically.

(1)

4.1. Analysis of measuring capacity

There are two decisive parameters when it comes to measuring capacity: repeatability indications and linearity. Problem of eccentricity may be omitted owing to adopted testin, methodology – i.e. the sample has to be placed in the very centre of a weighing pan each time. It is assumed that the eccentricity error for such methodology is relatively small, few reading unit Deviation being a result of indefinite drifts of zero indication may be minimized and this is carried out through zeroing procedure prior each measurement. Effect of weighing result variation resulting from sensitivity drifts may be considered to be insignificant when measurement takes little time, about 6 - 15 seconds, and when the balance adjustment is performed periodically.

0.00000

XA 52.3Y Semi Micro-Analytical Balance

- Max capacity [Max] 52 g
- Readability [d]
 0,01 mg
- Repeatability
 0,01 mg
- Linearity ±0,03 mg

For this balance it is possible to measure sample weight with readability of \pm 0,03 mg, it is due to the linearity error it is characterized with. This means that for a sample weighing 12,65446, the weighing result shall be included within the following range: 12,65443 g \div 12,65449 g.

5 g

MYA 5.3Y Microbalance

- Max load [Max]
- Readability [d] 1 μg
- Repeatability 1 µg
- Linearity ± 5 μg

nce, the sample weight may be measured with tolerar

For this balance, the sample weight may be measured with tolerance of 10 μ g, i.e. for weight of 12,654465 the weighing result shall be included within the following range: 12,654460 g ÷ 12,654470 g.

Balance linearity is a decisive parameter when it comes to accuracy of sample weight determination. This is fundamental information for those whose task is to determine precisample weight. For processes wherein weight variation is crucial, i.e. processes where the samsample is measured several times in a row, repeatability of indications is more important parameter. Measurement deviations should not be taken for uncertainty of determination of the 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.

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.

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 ($d=1\mu g$ or $0,1\mu g$). 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 cour of 24-hour-long cycle. The second in turn concerns balance construction, more specifically i internal temperature. Temperature stability depends on changes that take place within the whole laboratory space.



Fig. 8. Diagram of temperature changes occurring within 24-hour-long cycle, easily noticeable temperature growth starting at 6 a.m. is a result of personnel presence

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 I understood here rather as insignificant temperature oscillations around the specified value. Quit 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 or 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 Laboratory of the order of $1 \div 2^{\circ}C$ / hour, do not affect weight measurement.

5.3.1. Self-heating

Microbalance temperature stability is an effect of both external temperature and balance selfheating. 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. 9. Microbalance



Fig. 10. Operator testing the balance

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.

In order to observe whether the balance has reached stable temperature or not one may use ambient conditions module, such one that is comprised within 3Y series microbalances. Upr pressing ambient conditions icon, the module displays values of current temperature, moistu content, atmospheric pressure and air density.



Fig. 11. Ambient conditions module

Different colours of icons inform whether:

- max value has not been exceeded,
- temperature or moisture content variation dynamics is not greater than the limit value

H < 85 %	H > 85 %	H < 85 %	H > 85 %
T < 35°C	T < 35° C	T > 35°C	T > 35°C
∆H < 10%/h	∆H > 10%/h	∆H < 10%/h	∆H > 10%/h
∆T < 5°C/h	∆T < 5°C/h	∆T > 5°C/h	∆T > 5°C/h

Fig. 12. Ambient conditions module icons – colour designation

Time period for thermal stabilization depends on difference between balance temperature and temperature of room intended for weighing procedure performance. The greater the difference, the longer the time needed for stabilization.

2. Systems for temperature control

Some standards specify temperature range appropriate for the laboratory needs, e.g. 20 °C \pm 1 °C according to EN 12341 standard: "Air quality. Determination of the PM10 fraction of suspended particulate matter". Means of meeting this requirement has not been specified therefore it may be freely adopted, nevertheless one has to be aware so as not to introduce too much air flow to the laboratory space while trying to provide stable temperature. This turns out to be quite complicated issue. Various limitations such as cost and available room, cause that air conditioning is one of the most frequently used solutions. Unfortunately air conditioning is almost always a source of heavy air flow acting against correct measurement indication. For such an instance it is advisable to apply supplementary draft shield which limits air flows within balance neighbourhood. Draft shields have been commonly used for devices other than microbalances. Sometimes it happens that relocation of the workstation is helpful, providing that the new place is free from strong air flows.

1. Temperature stability may be reached by means of natural air circulation. Attempts to reach stable ambient temperature within a specified temperature range, i.e. 20 °C ÷ 35 °C, shall be made.

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. 13. Correction of accuracy of indications by means of automatic adjustment

- Temperature variation apart from being a cause of weighing module size changes may influence characteristics modification of both, electromagnetic transducer and electronics. As a result of the above mentioned, the balance accuracy may worsen, the sample may be weighed with a more serious error. This calls for need of automatic adjustment eliminating the error. Automatic adjustment is a standard solution of all Radwag laboratory balances.
- 2. Using the balance in various temperatures requires a bit different approach to the methodology of weight measurements. Adjustment shall be performed more often, prior the measurement most preferably.

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 $\% \div$ 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.

Humidity absorption process relates to samples as well, among the samples there are powders or cellulose filters. While being weighed the samples put on weight quite fast therefore unexperienced operators may get impression that the balance operates incorrectly.



Fig. 14. MYA 5.3Y/F – cellulose filter of 90 mm diameter

1. Disturbed humidity and the measurement

Maintaining stable level of humidity, e.g. $50\% \pm 5\%$, 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 metrologically important parameters of the balance.

For balances with reading unit smaller than 0,01 mg, the metrologically 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.

🖹 ΝΟΤΕ

- 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.

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.

Guidelines:

- 1. prior weighing the samples should be acclimatized to the ambient temperature, desiccator shall be applied for acclimatization purposes
- 2. for gross measurement, the packaging size shall be selected respectively to sample size
- prior weighing the sample shall be mixed in order to provide homogeneity, it may happen that surface layers of the sample contain more moisture

Example processes where there are problems with sample weight instability:

a. differential weighing of cellulose filters, measurement of dustiness GUIDELINES:

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)

GUIDELINES:

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

GUIDELINES:

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. 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 par 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. 15. 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.

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

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.



Fig. 16. Unbalanced electrostatic charges

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.

7. Ground vibrations

First balances were probably even-armed levers and their operation was based on comparison of weight of loads placed on both sides of the lever. Design of most contemporary balances with electromagnetic type of conversion is similar. It comprises a weighing pan upon which the load is deposited, on one side, and a coil suspended in a magnetic field, on the other side. The coil compensates gravity force applied to the load. As a consequence of such design the measuring system is susceptible to shocks and vibrations. It cannot be stated with 100 percent certainty that all ground vibrations disturb the measurement. Ground vibration disturbance may be a result of balance construction, amount of vibrations, their variation and size of a sample.

The main evidence for occurrence of significant vibrations is instability of indication. The sample weight determination is impossible when the observed result is stable for a short time only. What kind of corrective actions may be taken in order to prevent the above?

- 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 antivibration table is an example of such workstation



Fig. 17. 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 metrologically important parameters in real working conditions, the method is called Service Autotest.

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 setpoints. 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 metrologically 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.

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.

What shall be understood by scope of balance adjustment?

There are two systems of setup available. The first one is a service menu, the second, uses menu. Service menu parameters shall not be described here since they are important for technicians and therefore can be found in a service menu only. As for the user menu it comprises three components, each of them described below. Modification of any of these components influences the weighing process.

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.

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

Instruction relating to typical situations.

a. Sample is being weighed, the indication oscillates around stable result

Result oscillation, i.e. plus/minus change of a specified value, may be a result of inappropriately selected balance settings. If insignificant deviations occur (few reading units), one may eliminate them by modifying balance parameters settings. In case of more serious oscillations it is necessary to eliminate disturbance source first, and adjust balance parameters next.

OPERATION

Swap the filter value one level up (e.g. medium to slow), Value Release shall be Fast + Reliable.

b. Sample is being weighed, stable result increases few units up changing from one state to another

Obtaining indirect stable results is an effect of incorrectly selected Value Release parameters. Absorption of humidity by sample gives similar response. Here, one may observe constant sample weight growth – corrective actions may turn out to be inefficient.



Fig. 18. Stability of a result over time (A – unstable results, wide disperse of indications, B-C – stable result, narrow disperse of indications)

OPERATION

Swap the filter value one level up:

- Fast to Fast + Reliable,
- Fast + Reliable to Reliable.

If result variation is an effect of humidity absorption it is necessary to acclimatize the sample to existing ambient conditions. Another solution is to analyse the first initial stable measurement since any successive measurements reflect quickness of humidity absorption. Rarely used operation is to measure a sample in a tight packaging. The sample weight shall include packaging weight then.

c. Sample is being weighed, the result increases/decreases continuously

NOTE

When continuous change of result is observed, wherein the change is either plus change or minus change, it shall be assumed that one of two possible processes has occurred. The first one is sample absorption of humidity. This especially refers to hygroscopic samples that may acquire moisture from the environment (weight increase), or loose moisture content from the surface (weight decrease).



Fig. 19. Hygroscopic samples

Examples of such samples are absorbent papers, cellulose filters, powders, plastic, cardboard, fibrous materials or materials containing cellulose. As for the secon process, it is occurrence of unbalanced electrostatic charges. Depending on their type, intensity or distance the indication drift increases or decreases.

OPERATION

Changing balance operation parameters brings no effect. In case of hygroscopic sample two solutions are possible: sample stabilization in the working environment, reducisample surface or providing packaging for it when carrying out weighment. Trying to reduce humidity level at a workplace might turn out to be helpful, but as it is well known moisture content reduction may not be easy to perform, especially when it comes maintenance of stability.

One may limit electrostatic charges occurrence by means of volume ionization of a sa Such attempt improves measurement stability. Number of ions needed for ionization and measurement methodology are selected experientially. A highly useful devic f ionization purposes is DJ-02 or DJ-03 ionizer.



Fig. 20. Electrostatic charges ionizer

- a charges type selection (plus, minus, mix)
- b number of generated ions (low, medium, high)
- c charges generating fields

d. Wide dispersion of indications for the same sample

Prior taking any action, one shall realize that repeatability of indication entirely depends on ambient conditions. Being aware of the above, it is necessary to diagnose both, working conditions and the balance, with regard to temperature stability. Dispersion may also be caused by processes occurring within a sample (evaporation, absorption, electrostatics).

OPERATION

Actions respective to an observation outcome shall be taken. If the result keeps changing by few reading units regardless of displayed stability marker, swap Value Release filter one value up:

- Fast to Fast + Reliable,
- Fast + Reliable to Reliable.

Check value of Ambient Conditions parameter – UNSTABLE value is the recommended one When in doubt perform measurement using mass standard.

e. Indication does not return to zero value upon unloading

Upon unloading the weighing pan, ambient conditions stabilization takes place inside the draft shield. The stabilization may be interrupted by air flows (air conditioning), ground vibrations, lack of balance acclimatization. The same criteria is used for weight measurement with unloaded weighing pan and for sample weight measurement.

OPERATION

Carefully take the samples off the weighing pan avoiding shocks, while sliding the draft shield door use the most gentle movements, check Value Release option – when the parameter is set to FAST value it may cause temporary display of unstable results.

2. Optimization of speed

Balance parameters may be adjusted in order to provide shorter time needed for measurement performance. It is true that shorter measurement means worsening of metrologically important parameters such as repeatability of indications.

a. method 1 – parameters adjustment

It is possible to shorten measurement time by setting two parameters. Lower filter shall be used, e.g. VERY FAST, Value Release shall be set to Fast option. This generates about 25 – 30 % shorter measurement when compared to standard settings. Even for such fast measurement stable result and good repeatability are possible providing that ambient conditions are also stable.

b. method 2 – limitations of resolution

Resolution is calculated as a quotient of the maximum capacity and a reading unit. For an exemplary microbalance the resolution is:

R = Max/d = 5 g/0,000001 g = 5 000 000

Any balance settings are optimized for such resolution, also those that apply to speed of measurement. Each balance menu offers an option enabling electronic resolution limitation, it is performed by means of LAST DIGIT OUT.

Available options, last digit:

- always
- when stable
- never

Setting value 'never' for Last Digit parameter, limits balance resolution to 500 000 intervals. Criteria for measurement speed change automatically.

7. SOP – monitoring of metrologically important parameters

In most cases information on metrologically 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.

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 orfor 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:

 $-3 \cdot sd \le x \le +3 \cdot sd$ with 99,7 % probability $2 \cdot sd \le x \le +2 \cdot sd$ with 95,5 % probability $-1 \cdot sd \le x \le +1 \cdot sd$ with 68 % probability



Fig. 21. Standard deviation - interpretation

The lower SD value, the more reliable measurement (more intense concentration of results around the average value). Deviation occurring during weighment of mass standard determines whether the measurement is precise or not.

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. 22. 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 weighment of mass standard, wherein the mass standard is placed in points 2-5, and a weighing result obtained by weighment 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:

 $\mathsf{E}_{\mathsf{ECC}-2} = \mathsf{I}_2 - \mathsf{I}_1$

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: Max = 5 g 1/3 Max = 1,6 g (required mass standards: 1 g, 500 mg, 100 mg) Test mass standard = 2 g

Max 21 g 1/3 Max = 7 g (required mass standards: 5 g, 2 g) Test mass standard = 10 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 standarc 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. 23. Balance linearity – testing method 1 linearity point only 500 mg mass standard, (ballast weight is at the bottom of a draft shield)



Fig. 24. 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.

8. Diagnostics 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 \div 0,0001), 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, ultramicrobalance. Means of balance adjustment operation at the moment of balance connection to mains has been presented below.



Indication variation presented in the graph above, blue line, is a result of balance self-heating process. Red line shows temperature changes. Variation of indication accuracy is temporarily corrected, first by temperature adjustment, then by time adjustment. The correction is performed automatically and it provides correct indications right after completion of the adjustment procedure.

Providing that the external temperature is stable, successive adjustment processes shall be performed in accordance with a specified time interval. One shall keep in mind that manual adjustment procedure is possible to be carried out at any moment (by means of a respective button).

Methodology for sample weight measurement shall assure that the balance indication is as reliable as possible. Since the balance reliability between successive adjustments is usually unknown it is necessary to perform the adjustment procedure prior measurement, it can be done by means of a particular button. The adjustment may be understood as correct balance sensitivity estimation. See the figure below.



Fig. 26. Correction of sensitivity for microbalance with Max 5 g

The graph clearly shows that potential problem of sensitivity variation is important when sample weight is $\frac{1}{2}$ max \div Max. For less heavy samples repeatability of indications is more critical parameter, this the repeatability here that decides on measurement accuracy. Size of sensitivity variation depends on both, dynamicity of ambient conditions changes and microbalance design. Technical specification defines sensitivity stability with reference to temperature changes, usually 1 ppm/⁰C.

It is possible to track sensitivity variation over time for real working conditions, nevertheless it does require a great number of tests to be performed. Such approach is not always economically justified.

2. Autotest GLP

Undoubtedly it is repeatability of indications that is considered to be the most metrologically 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.

Autotest GLP: Report		
Balance type	MYA 3Y	
Balance ID	0	
Operator	Admin	
Application revision	L1.4.15 K	
Date	2014.09.30	
Time	13:42:13	
Number of measurements	10	
Balance weighing interval	0.000001 g	
Internal weight mass	17.673852 g	
Filter	Slow	
Value release	Fast and reliable	
Temperature: Start	23.99 °C	
Temperature: Stop	23.96 °C	
Humidity: Start	58 %	
Humidity: Stop	58 %	
	/	
Deviation for Max	0.000004 g	
Repeatability	0.0000017 g	

Adjustment weight value is almost always contained within ³/₄ Max ÷ Max range. Judging by that it may be easily noticed that repeatability applies to a high measuring limit. The question is how, if ever, this value may be applied to weighing within low measuring range?

NOTE

Repeatability value is constant for a low range extent. This initial point is defined as load of about 1000 d – applying lighter load does not result in improved repeatability of indications. Heavier loads application causes repeatability value rise, taking MYA 11.3Y as an example

- load up to 0,2 g sd=1,5 μg
- load up to 0,2 g do 5 g sd=2,0 μg
- load up to 5 g do 11 g sd=2,5 μg

As it has been proved, loads of 11 g weight value bring only 1 μ g better repeatability.

With standard deviation determined through Autotest, it may be expected that while weighing samples of lower weight values one can obtain better repeatability. Dispersion of indication is little influenced by testing method (manual one is worse) and characteristics of a given sample.

Adjustment is part of the GLP procedure. After adjustment it is possible to calculate deviation of accuracy for max. load. While analysing this parameter one should be aware of the fact that so called error of accuracy includes deviation which is an effect of repeatability of indications.

Access path in microbalance program structure: SETUP/OTHER/Autotest GLP

3. Autotest FILTER

Trying to optimally select appropriate range for Filters and Value Release might be quite sophisticated process. Some balances require precision, other as short measurement as possible. What shall one set then and how it should be evaluated? Autotest FILTER is helpful for those who are in doubts. Autotest FILTER is a new ergonomic application at a user disposal, each laboratory balance manufactured by Radwag features it.

Operating principle for this application consists in determining standard deviation and length of time for weighing, wherein all settings combinations for Filter/Value Release have to be taken into account. After completed procedure the balance displays results which help the user to decide which setting is the one that provides an optimal result:

the shortest time for measurement

----- Autotest Filter: Report ------

the best repeatability

Balance type	MYA 3Y
Balance ID	876570
Operator	Service
Application revision	L1.4.15 K
Date	2014.09.30
Time	15:04:57
Reading unit	0.000001 g
Internal weight mass	17.673852 g
Temperature: Start	24.27 °C
Temperature: Stop	24.39 °C
Humidity: Start	63 %
Humidity: Stop	64 %

First part of the report contains data on a balance and working conditions parameters. The 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.



Fig. 27. Autotest FILTER - results

----- Autotest Filtr: Raport ------

Filter Value release Repeatability Stabilization time

Filter Value release Repeatability Stabilization time Very fast Fast 0.0000023 g 8.851 s

Very fast Fast and reliable 0.0000027 g 9.309 s

Very fast Reliable 0.0000074 g 10.588 s

Fast Fast 0.0000024 g 10.049 s

Fast Fast and reliable 0.0000046 g 10.795 s

Fast Reliable 0.0000038 g 12.074 s

Average Fast 0.0000056 g 9.922 s

Average Fast and reliable 0.0000028 g 10.663 s Filter Value release Repeatability Stabilization time

Signature

Average Reliable 0.0000019g 13.233 s

Average Reliable 0.0000019g 13.233 s

Slow Fast 0.0000033 g 9.974 s

Slow Fast and reliable 0.0000016 g 11.096 s

Slow Reliable 0.0000052g 13.253 s

Very slow Fast 0.0000034 g 10.067 s

Very slow Fast and reliable 0.0000029 g 10.896 s

Very slow Reliable 0.0000066 g 13.818 s

Access path in microbalance program structure: SETUP/OTHER/Autotest FILTER

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. 28. Ambient conditions module

Microbalance comprises two temperature sensors (T1, T2), and a sensor of humidity and air pressure. Different colours of icons inform:

- which parameter value has been exceeded,
- if dynamics of a given parameter variation is not too high.

Below options are possible.

H < 85 %	H > 85 %	H < 85 %	H > 85 %
T < 35°C	T < 35°C	T > 35°C	T > 35°C
∆H < 10%/h	∆H > 10%/h	∆H < 10%/h	∆H > 10%/h
∆T < 5°C/h	∆T < 5°C/h	∆T > 5°C/h	∆T > 5°C/h

The said parameters may be recorded by means of external sensors. Such sensor is installed using 1,5-meter-long cable – it measures parameters of ambient conditions, not those that exist inside a particular balance.



Fig. 29. Microbalance with an external THB sensor

Correctness of all sensors indications may be confirmed by calibration certificate (Accredited Laboratory).

9. Weighing applications

Balance as a universal weighing device may be used for determination of different objec' weight. In order to obtain precise measurement one has to apply a particular methodology. most cases it is the methodology that decides on the final measurement result. Therefor, knowledge on balance operation, sample structure, disturbance and possibilities of settingr modifications is beneficial when it comes to performance of precise measurement. Prior taki 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 metrologically 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. su 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 + absorption is insignificantly different, around tens of hundreds of milligrams. The measureme, applies to low extend of the weighing range. This is crucial information number two. Regular filt 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 the measurements?

a. Repeatability of indication

Decisive factor when it comes to measurement reliability is repeatable determination of a same 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 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 structu 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 humidi 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 testing this is controlled through so called blank tests (zero tests). This provides certainty 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. Whe

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.



Fig. 30. F series microbalance (for filters weighing) with an ionizer

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.

Access path to this function: SETUP/PROFILE/HOME/READOUT/AUTOZERO or another one, not requiring use of Setup button: GREY AREA/PROFILE/READOUT/AUTOZERO

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.

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



Fig. 32. 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 I 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 evapor 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 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 samp, 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 functio, 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 of decrease. Susceptibility of measuring system to external disturbing factors also increases.

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 weighments 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.











HEATING

CUTTINGPAINTINGAIR DUST (PM)Fig. 33. Technological processes for comparison processes

MASS STANDARDS COMPARISON

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 0.099	9239 0.1000681 -0.00014385	
2 0.1000684 0.0999	239 0.1000696 -0.0001451	
3 0.1000685 0.099	9238 0.1000688 -0.00014485	
4 0.1000690 0.099	9242 0.1000690 -0.0001448	
5 0.1000695 0.099	9243 0.1000693 -0.0001451	
6 0.1000693 0.099	9248 0.1000691 -0.0001444	
Mean average -(0.000144683333 g	
Standard deviation C	.000000482355 g	
Number of cycles 6	B2A	
Method A	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 comparisor from the moment of order placement, through calibration, to calibration certificate issuing.

Operation of comparators combined with software for calibration process control influences increase of capacity, provides reliable indications and complete documentation for calibration and reduces labour costs.

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 ³/₄ 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. <u>Repeatability of indications</u>

Repeatability is a metrologically 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. <u>Balance linearity</u>

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.

49

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 metrologically important parameters.

10.1. Legal metrology and practice

Legal regulations resulting from WELMEC law acts, OIML guidelines and drafts are common known mainly due to the fact that WELMEC is a globally operating organization. WELMECapproved methods and procedures are world-wide spread and used in course of indivia 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 f balances of I accuracy class:

MPE	Load
■ 0,5 e	$0 e \le m \le 50\ 000 e$
■ 1e	$50000 e < m \le 200000$
 1,5e 	200 000 e < m

Stating that balance complies to legal regulations means that errors occurring while measureme 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 narrowerification scale interval is 1 mg and that reading units and MPE values are as follows:

d=0,1 mg	MPE = 5 d
d=0,01 mg	MPE = 50 d
d=0,001 mg	MPE = 500 d (microbalance)
d=0,0001 mg	MPE = 5 000 d (ultra-microbalance

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 deviction accordance with legal regulations decide to perform adjustment procedure. This provider information on errors of the particular balance. It may be concluded that legal metrolos, regulations are not relevant when it comes to high resolution balances. Still, the testimethodology is of importance and use.

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

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

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. Americar 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

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

Chapter 1251 is not compulsory, it supplies the reader with explanation and definitions of terms that are pivotal when it comes to electronic weighing equipment:

- sensitivity
- accuracy
- linearity
- eccentricity

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:

- a. perform series of 10 measurements, using mass standard
- b. use mass standard of much greater weight than expected MSW value (USP 1251)
- c. calculate standard deviation using the obtained results
- d. calculate MSW, to do it standard deviation value must be multiplied by constant value equal 2000

MSW = 2000 sd

e. MSW value depends on repeatability of indication, whereas repeatability depends on balance settings (optimization) and on ambient conditions

🖹 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.

Apart from metrological perspective there are also functional requirements relating to balance use and safety. They refer to the following aspects:

- multi-levelled system of authorization,
- user logging mechanism,
- record of settings modifications, etc.

For more detailed guidelines refer to 21 CFR Part 11.

10.4. Environmental protection

Balances used for performance of environmentally related processes require conformity to legal regulations binding for a particular region and to requirements of respective standards. As it has been presented before, standards specify quite high MPE values, therefore from the metrological point of view, most balances meet the specified requirements, and there is still some margin. As a consequence of the above it is necessary to subject a given balance to temporary verification, performed by a respective body.

Particular standards concerning environmental protection, and regulations they provide, define balance by its reading unit. There are no other requirements. It is the user who has to establish how much metrologically important parameters influence the indication.

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

Speed approached to as a parameter of electronic balances usually refers to measurement duration, i.e. to amount of time within with the sample weight value is determined. As such, the parameter may by understood differently by independent subjects. It may be defined as follows:

Time of measurement is time flow after which sample weighing result falls within a specified tolerance.

This time is relatively short, it may take 2-3 seconds, whereas actual time needed for the result to stabilize takes usually around 6 - 7 seconds. While reading balance data contained within brochures and catalogues, one shall be able to distinguish between marketing content and real balance performance characteristics.

In case of ultra-microbalances and microbalances, time of measurement consists of two cycles. The first one concerns draft shield access procedure, the second one weighing procedure. It takes about 1,2, seconds to open and close the shield. Trying to estimate how long it takes to deposit a load on a weighing pan is quite impossible, it shall not be established here. Time needed for weighing does not depend on sample weight and it normally takes about 6 seconds.



Fig. 34. Speed of measurement for 1 g heavy sample

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.

Wherever highly precise accuracy is demanded, it may happen that time needed for measurement performance is intentionally lengthened.

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. 35. 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.

Internal batteries installed inside the panel provide 8-hour-long, continuous operation.



Fig. 36. Balance-terminal wireless connection

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.





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