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Handbook of Weighing Applications
Balances and Scales Used as Measuring and Test Equipment in a Quality System

turning science into solutions

Preliminary Considerations

In many areas of applications, the balance, scale or weight value is only a means to an end. The quantity that is actually of interest is derived from the weight value or mass. For this reason, each booklet of the Handbook of Weighing Applications thoroughly treats a specific topic. For every subject, the individual booklets include an explanation of the general and theoretical principles of the application concerned – this is not always possible without discussing equations according to the laws of physics or mathematical formulas.
Part 3, which is now available, discusses the subject of "Balances and Scales Used as Test Equipment in a Quality System."

An important part of all quality systems is the area covering inspection, measuring and test equipment and its monitoring for accuracy. The quality element "control of inspection, measuring and test equipment" requires that the supplier of a product or service develop and maintain Standard Operating Procedures (SOPs) for inspecting, calibrating and servicing test and measuring equipment. The purpose of these SOPs is to ensure that the supplier´s products conform to defined quality standards. When referring to the control of inspection, measuring and test equipment, we mean an orderly sequence that ensures that the equipment is inspected in a timely fashion and, if necessary, appropriate measures are taken so that the equipment corresponds to the given requirements.

Using the laboratory balance as an example, this chapter explains how one can establish an acceptable level of confidence in the test and measuring equipment being used. Suitability of the equipment is the initial requirement for obtaining reliable results.

Marketing, Mechatronics Division
July 2008

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Motivation

In the meantime, extreme ranges of resolution have been attained in the field of analytical weighing technology. Reaching these new limits, however, has opened up discussion about the competence of individual laboratories. For this reason, most laboratories keep certificates, accreditation documents and written attestations on file. These credentials provide objective evidence of the laboratory´s performance and assure those using the laboratory's services that analytical questions will be answered by an expert.

In addition, the flood of analytical data has confronted laboratory employees with a problem. Namely, they must test and validate many measured values for plausibility and accuracy. Here again, quality assurance measures are essential for correct, comparable and verifiable results, and are fundamental to long-term success.

Regulations and standards of the most prominent quality systems that relate to the control of inspection, measuring and test equipment are:

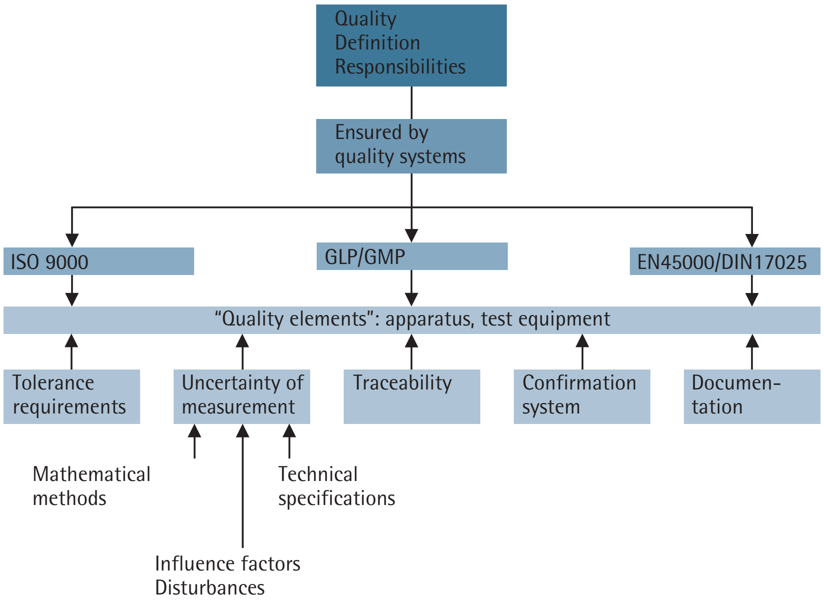
- GLP (Good Laboratory Practice)
- GMP (Good Manufacturing Practice)
- ISO 9000 series
- EN 45000 series

They have been generalized to cover a large number of devices and procedures and, therefore, must be interpreted accordingly.

ISO 10012 provides a more extensive and concrete explanation of the requirements for test and measuring equipment. Accordingly, a series of measures for using the test and measuring equipment can be summarized as a few general, basic requirements.

The objective of each quality system is to provide a product or service with the appropriate quality. But what is quality? The term "**quality**" is defined in the EN ISO 8402 standard as follows:

"Totality of characteristics of an entity that bear on its ability to satisfy stated and implied needs"



Overview of Quality Systems

The following descriptions of the quality management systems intend to highlight the key features and application areas. One thing that all quality systems have in common is in their requirements placed on test equipment. These common requirements describe how equipment qualification is carried out for all test equipment before initial operation and how this equipment that is used daily is tested and calibrated on a regular basis.

Quality management systems are subdivided into various categories. We distinguish between universal and industry-specific quality systems.

Universal Quality Systems

ISO 9000 Series

The ISO 9000 series is a set of widely used international standards applicable to production and the service industry. Considering its general applicability, ISO 9000 does not contain requirements specifically related to laboratories, but is a suitable approach for assuring quality in laboratories. The ISO 9000 series covers voluntary requirements for all areas of production and service. A management representative for quality oversees the integrated quality management and assurance entities. Internal audits are carried out continually, and recertification is done every three years.

The focal points of this quality system lie on the following:

- Internal and external interfaces
- Purchaser-supplier relations
- Corrective action

EN 45000 Series

This quality system involves European-wide recognition of testing laboratories. A testing laboratory accredited for compliance with European Standards obtains the status of an “institution qualified for specific tasks.” This laboratory is accredited for a defined scope of validity and is re-accredited every five years. A typical example of a laboratory accredited for compliance with the EN 45000 standards is a testing laboratory commissioned to perform analyses relating to environmental protection. The particular focal points of a quality system based on European Standards are the following:

- Employee qualification
- Qualification of the processes used
- Accuracy of the results
- Device testing
- Calibration and validation of the method used

Legally Regulated Quality Systems

GLP (Good Laboratory Practice)

GLP is a system of standards applied worldwide and is legally regulated for data used to assess products for safety approval in order to protect people and the environment from hazards. The requirements imposed by GLP refer to the organization and to personnel. An audit for compliance with GLP requirements is performed every four years. A typical example of a GLP-compliant unit is a toxicological or analytical laboratory in a chemicals company that conducts research, or a testing laboratory that is commissioned to perform tests. The focal points of GLP are the following:

- Organizational rules and formal requirements
- Documentation
- Independence of the quality assurance unit

GMP (Good Manufacturing Practice)

This system is prescribed for the pharmaceutical industry and medical device manufacturers. The scope of application for GMP lies in the manufacture and analysis of pharmaceuticals. The focal points of GMP are the following:

- Defined and validated manufacturing processes
- Release of each product lot
- Self-audits

The most important prerequisites for implementing GLP and GMP are listed as follows:

- Organizational structure of the testing facility
- Qualification of personnel
- Quality assurance program
- Testing facilities
- Equipment, materials and reagents
- Test and reference materials
- Standard operating procedures (SOPs)
- Study plans, raw data and test reports
- Filing and preservation of records and materials

If we compare all quality systems with one another, we discover that many areas overlap. These systems differ from one another in their focal points because each system has different objectives. For instance, GLP is a system of documentation that contributes towards improving quality. By contrast, accreditation according to the EN 45000 series entails less work for documentation. For the latter quality system, the focus is on the competence of personnel and the quality of results.

Selection of Suitable Test and Measuring Equipment

Equipment Qualification
The use of inspection, measuring and test equipment in a quality management system requires a detailed description and documentation of the results of measurements and of confirmation. Processes and standard procedures must be traceably documented and these documents filed.

Many leading quality systems, such as GLP/GMP and the ISO9000 and the EN 45000 series, explain how to comply with the standards.

Equipment qualification provides documented evidence that an instrument is appropriate for its intended use to ensure that it will operate on demand, under specified service conditions, to meet system performance and accuracy requirements.

Equipment Qualification is subdivided into 4 sections:

1. Design Qualification	(DQ)
2. Installation Qualification	(IQ)
3. Operational Qualification	(OQ)
4. Performance Qualification	(PQ)

Design Qualification (DQ)
In design qualification, the user defines his or her requirements on the test or measuring equipment. Parameters, such as accuracy, method of measurement, and requirements on the supplier that relate to design validation or services, must be defined and documented before purchasing (procurement). The purpose of design qualification is to ensure that the measuring equipment – in this case, the balance, scale or weighing system – is suitable for the particular application.

The data generated using the test equipment are merely observed values of a quality characteristic, for example, the weight values generated in a laboratory. Systematic and random errors that occur during the weighing process and result from the weighing equipment itself affect the accuracy of these values. Therefore, the result determined by the weighing instrument has a degree of uncertainty, which is called "uncertainty of measurement" and must be indicated as a matter of principle for each weighing process. The factors that play a role in this uncertainty of measurement are explained in the following.

The selection of a suitable measuring instrument must be based on answering the question of how great the uncertainty of measurement may be to allow reliable compliance with the required tolerances. A good approach to answering this question is to apply the "golden rule of metrology" that says that the measurement uncertainty of a measuring device may only be 1/10 of the tolerance of the measured values.

For example, let's suppose that a 10-mg sample is to be weighed to an accuracy of 1 percent, which corresponds to 0.1 mg. According to the "golden rule," the total uncertainty of the balance may not exceed 0.01 mg.

Especially if a cost-intensive process is used, it is important that this criterion be met under economically feasible conditions. Under certain circumstances, a ratio of 1/3 is acceptable if these tolerances are met through suitable measures, such as the frequency of testing to ensure that the test equipment is appropriate.

The basis for the selection of a measuring instrument or test equipment is provided by the manufacturer's technical specifications, such as repeatability, linearity or temperature coefficient. Besides these instrument parameters, additional factors that may affect the results of a measurement must be considered. These include the ambient conditions at the place of measurement, qualification of the operator, test object and test procedure.

Installation Qualification (IQ)
Installation qualification describes startup and the detailed sequence of setting up the measuring equipment. Special attention must be paid to the completeness and correct installation of the equipment supplied. To operate high-resolution analytical and microbalances, you should essentially consider using specially designed anti-vibration balance tables. In addition, the climate conditions (particularly the temperature) should be kept as constant as possible.

Test Methods

Operational Qualification (OQ)
Operational qualification describes the metrological testing of a weighing instrument at the place of installation. Adequately trained personnel must test weighing instruments using the corresponding auxiliary equipment and weights that have the appropriate accuracy. In addition, the test results must be documented in a calibration certificate or test report of the weighing instrument. This testing must be performed at established intervals (known as " intervals of confirmation").

Performance Qualification (PQ)
All manufacturers´ specifications refer to nearly ideal measurement conditions as recommended in the installation and operating instructions. In practice, however, operators frequently operate weighing instruments under conditions that differ from these. Therefore, performance qualification requires verification that the measuring equipment functions as intended in its normal operating environment (e.g., weighing a sample under a laboratory fume hood).

Device Qualification | Final Report
Once all qualification procedures described above have been successfully performed and the adequate performance of the measuring equipment has been verified, equipment qualification along with a final report is completed.

All manufacturer specifications are based on "idealized" weighing conditions. Otherwise, comparisons could not be made between different instruments. But the methods actually used in the field often differ from those used by the manufacturer. Variations in the methods used should be documented appropriately in the SOP, and allowances should be made for deviations in the weighing accuracy that may result. For example, if a hanger for below-balance weighing is used to weigh a magnetic sample, the manufacturer specifications, which were determined under the best weighing conditions, cannot be maintained. In this case, preliminary tests must be run using reference samples to verify the attainable degree of accuracy.

Determination of the Uncertainty of Measurement

Manufacturer specifications, which are the basis for selecting test and measuring equipment, are explained and interpreted in the following sections.

The limits of a weight measurement, i.e., the range within which the defined certainty of measurement is maintained, is called "**weighing range.**"

Repeatability describes the ability to display corresponding results under constant testing conditions when the same load is repeatedly placed on the weighing pan in the same manner. Repeatability is essentially independent of the load on the balance or scale. It can be designated as the most important metrological feature because its influence on the uncertainty of measurement – especially with lighter loads – becomes the dominant factor.

Either the **standard deviation** or the difference between the highest and the lowest result for a defined number of measurements is used to specify this quantity.

Example of a Weighing Series:

Weight No.	Weighing Series
1	9.997g
2	10.002g
3	9.998g
4	10.002g
5	10.001g
6	10.002g
7	10.001g
8	10.000g
9	9.998g
10	10.002g
11	9.997g

The mean value is calculated from the sum of the individual values W1 to Wn, divided by the number "n" of individual values; hence

$$x = \frac{1}{n} \cdot \sum_{i=1}^{n} x_i$$

Using our example, this means:

$$\bar{x} = \frac{9.997 + 10.002 + 9.998 + 10.002 + 10.001 + 10.002 + 10.001 + 10.000 + 9.998 + 10.002 + 9.997}{11}$$

x = **10.000 g**

The difference between the highest and lowest result in the weighing series is calculated as follows:

10.002 g – 9.997 g = 0.005 g

The standard deviation is computed using the following equation:

$$s = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^{n} (x_i - \underline{x})^2}$$

x_i = individual value measured in the weighing series

n = total number of weight measurements

– mean value of the individual results measured

The standard deviation of our example is:

$$s = \sqrt{1 \quad \cdot \left[(9{,}997 - 10{,}000)^2 + (9{,}997 - 10{,}000)^2\right] 0{,}0020976\text{ g}} \approx \textbf{2 mg}$$

For evaluating the quality of a weighing instrument on the basis of its technical specifications, both values (lowest and highest result) are approximately comparable with each other if the minimum | maximum specification is compared with three times the standard deviation. Within the standard deviation times three, you will find 99.7% of all representative values of a weighing series.

The standard deviation corresponds to the spread of the bell curve on either side from its point of inflection. Sixty-eight point three percent (68.3%) of the individual values will be located within this area or, to put it differently, the individual values will fall within the range of $\bar{x} \pm s$ with a confidence interval of 68.3%. In practice, the use of the standard deviation times two has become the norm. This interval has a probability of 95.5%.

This means that 95.5% or 99.7% of all values will be distributed with respect to the mean value within the range defined by the standard deviation times two or three.

The following Figures show a weighing series listed in a chart and plotted as a graph; the eleven individual weights are marked as points.

Frequently, the **relative standard deviation** is also given in percent

$$\left[\frac{s}{x} \cdot 100\ \%\right].$$

In our example, the standard deviation is:

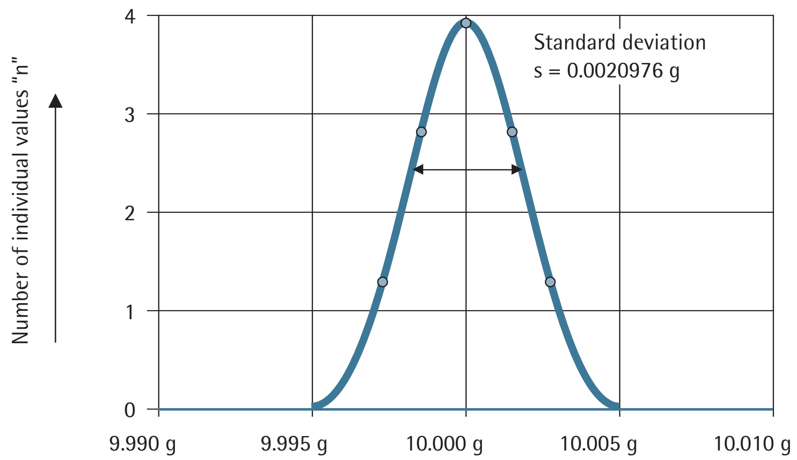
$$= \frac{0.002\ \text{g}}{10.000\ \text{g}} \cdot 100\ \% = 0.02\%$$

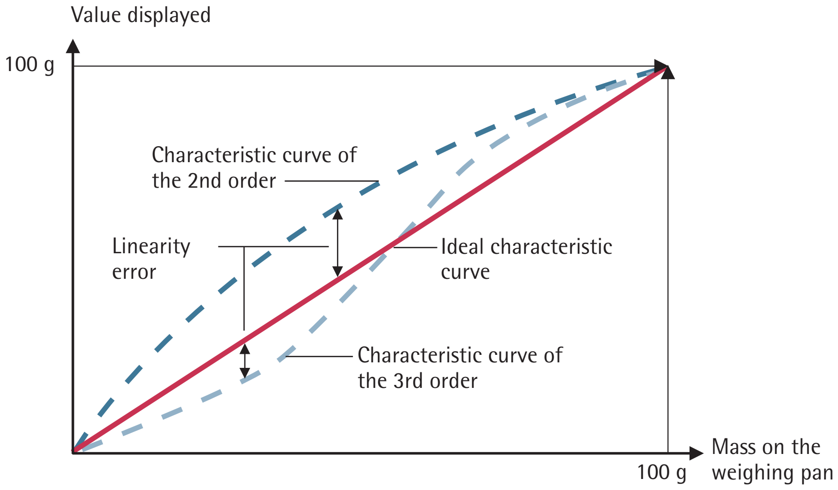
The values of our weighing series given as an example yield the following results:

	Weighing Series
Number of individual values measured "n"	11
Sum of the individual values measured	110.000 g
Mean value	10.000 g
Standard deviation	0.002 g
Approximate (relative) standard deviation	0.00167 g
Repeatability acc. to OIML R76	0.005 g

The **linearity error** (usually referred to as **linearity**) indicates how much a balance or a scale deviates from the theoretically linear slope of the characteristic calibration curve. In the case of an ideal characteristic curve, the mass on the weighing pan will always equal the weight displayed on the balance or scale. If the zero point is correct and the weighing instrument has been correctly calibrated and adjusted at maximum capacity, the linearity can be determined by the positive or negative deviation of the value displayed from the actual load on the pan. Linearity is caused by the specific inherent properties of a weighing instrument and is therefore unavoidable. Two of the most frequent curves are slopes of the 2nd order (convex or concave curve) and of the 3rd order (S-shaped curve).

The maximum deviation between the actual characteristic curve and the linear slope of the two interdependent values – the zero point and the maximum capacity – is defined as linearity. The maximum linearity is given in the data sheets of balances and scales. In some cases (such as an analytical balance), a limited range is specified, for instance, 200 g = ± 50 µg within 2 g = ± 10 µg.





Influence Quantities

The measured result, or weight, can be affected by so-called influence quantities, such as temperature, barometric pressure and humidity. In general, a distinction is made between the temperature coefficient of the zero point and of the sensitivity. Each of these parameters shows a more or less considerable impact on the measured result, affecting both the electronic components and weighing system to an equal extent.

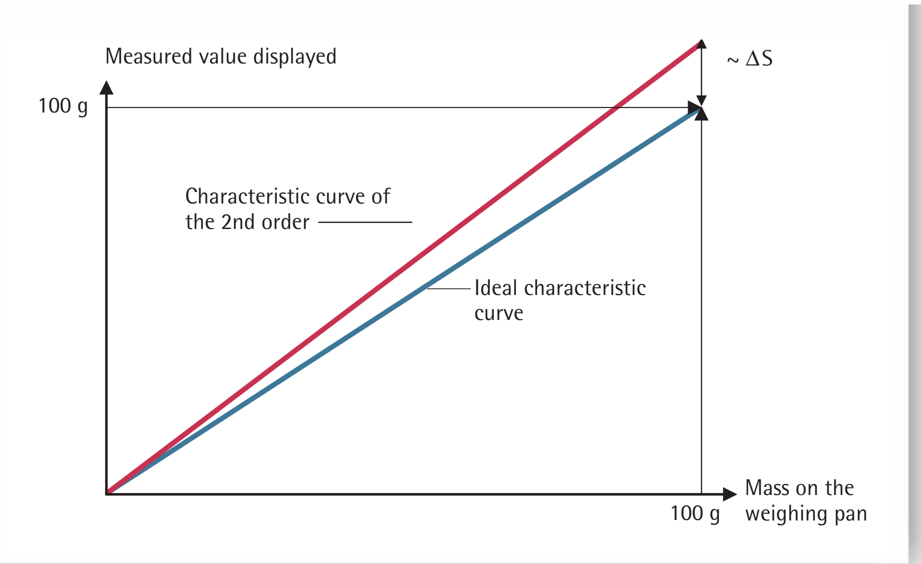
The **sensitivity** is the change in a displayed value divided by the change in the load signal generated by the mass on the pan. If a balance or scale with a digital display has been correctly adjusted, the sensitivity must always be exactly 1.

The equation for the sensitivity is as follows:

$$S = \frac{\Delta D}{\Delta m}$$

where ΔD is the number of scale intervals that correspond to the change in load Δm.

A **sensitivity error** ΔS is caused by using inappropriate calibration weights to adjust a balance or scale. The sensitivity error is always indicated as a relative number, e.g., 20 ppm per K (1ppm = one part per million = 10^{-6}).



If the value of the zero point or of the sensitivity changes because the temperature fluctuates, the **temperature coefficient** is used to characterize this change. If a weight is divided by the change in temperature, this will yield the value of the temperature coefficient.

Example:

Temperature coefficient: $2 \cdot 10^{-6}\ K^{-1}$
Initial sample weight : 10 g
Change in temperature : 5 K

Systematic error due to the temperature coefficient:

$2 \cdot 10^{-6}\ K^{-1} \cdot 10\ g \cdot 5\ K = 0.1\ mg$

The value of the temperature coefficient is the major criterion for judging whether or not the weight readout has stabilized when a balance or scale is exposed to fluctuations in the ambient temperature.

If a light load is left on the balance or scale, over time you will see a **drift in the zero point** Δ ZP on the display. Zero point drift is only important for long-term measurements involving a constant load, as in thermogravimetric and sorption measurements.

The **off-center load error,** also called "**corner load error**," means the change in readout when the same load is placed in various positions on the weighing pan or load plate.

The off-center load error is officially called "eccentric loading error." To verify the error, a weight is placed exactly in the middle of the weighing pan and the balance or scale is tared. Then the weight is placed in 3 to 4 different locations on the edges of the weighing pan; if the pan is rectangular, the weight is placed in the corners. The off-center load error can then be directly read off the display. This value can be negative or positive and usually ranges from 1 to 10 digits or scale intervals. Therefore, especially when you use balances with high resolution, the sample to be weighed should always be placed exactly in the middle of the weighing pan. In addition, other factors that can substantially influence the weighing results must be taken into account: operator, weighing location, sample and weighing procedure. For this reason, it is recommended that the effects of these factors be minimized whenever possible.

In the following, these factors will be dealt with in more detail.

Operator

Today, leading manufacturers offer balances with a readability of up to 0.1 µg and a resolution of up to 21 million digits. It almost goes without saying that the operator must receive proper training in order to capitalize on the accuracy and precision of these instruments.

For instance, the operator must be aware of and strictly comply with basic rules, such as:

- Placing the sample in the middle of the weighing pan (to avoid off-center load errors);
- Attempting to work as consistently as possible (to maintain the specified repeatability);
- Making sure that the balance is set up on a level surface (to prevent a systematic sensitivity error)

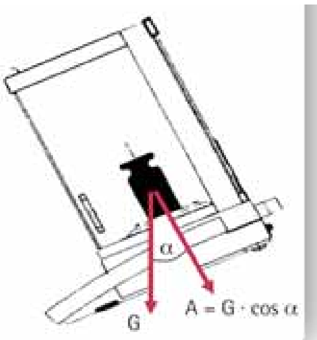
Weighing Location

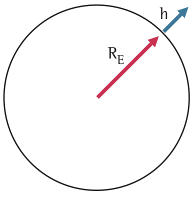
A scale or balance is adjusted in a manufacturing process so that the force transmitted to the weigh or load cell when the scale is loaded is parallel to the direction of the gravitational acceleration and perpendicular to the cell. A level indicator (small spirit level) attached to the scale enables the operator to pinpoint this position exactly, allowing it to be reproduced at all times. This step is called **leveling.**

The importance of leveling a balance or scale will be explained using the following example. Suppose a laboratory bench with an edge length of 1,000 mm is raised at one end by 5 mm. Then the following applies to the angle of inclination:
a= arctan 5/1,000 = 0.2865°

Moreover, the following applies to the force generated by a load in the direction of the weighing axis:
$A = W \cdot \cos\alpha = W \cdot 0.9999875$,
i.e., the weight measured by the tilted balance is 2.5 mg too low for a sample with a mass of 200 g.

Because of the earth´s rotation and geographical features, the **gravitational acceleration** varies depending on where the balance or scale is set up. We therefore recommend that the weighing instrument be adjusted each time it is set up in a new location and before initial startup. During this procedure, a known mass is loaded on the weighing instrument and the adjustment factor is determined from the weight value displayed. Another effect that often goes unnoticed is a change in altitude and how it can influence the gravitational acceleration when, for example, the balance is moved to a higher location. Moving the balance will affect the accuracy of the weight displayed!





g = Gravitational accleration
R_E = Earth's radius
h = Difference in height (altitude)

$$g \cdot (R_E + h) \approx g_K \cdot \left(\frac{R_E}{R_E + h}\right) \approx g_K \cdot 1 - 2 \cdot \left(\frac{h}{R_E}\right)$$

$$g \cdot (R_E + 4\ \mathrm{m}) \approx g \cdot \left(1 - 2 \cdot \frac{4\ \mathrm{m}}{6370000\ \mathrm{m}}\right) = g \cdot 0.9999987$$

The following is obtained in the relation shown below for a difference in altitude of only 4m:

This means that a semi-microbalance, which measures a mass accurately to 200.00000 g, will only measure 199.99974 g for the same mass when set up 4m higher. This underscores the necessity of calibrating and adjusting a balance or scale each time it is moved to a different location.

As a result of the moment of inertia, **mechanical disturbances** register on the balance or scale as periodic or stochastic "weight changes" depending on their attributes. A digital filtering feature on the weighing instrument, which can be activated by selecting a suitable integration time, can reduce these disturbances.

Low-frequency interference, however, is less likely to be filtered out because the filter can no longer differentiate between mechanical interference and a slowly changing weight readout (for example, during filling). We generally recommend that specially designed weighing tables be used for balances that have extremely high resolution. If vibrations in the building cause the disturbances, we recommend that the balance be set up on a lower floor. If this is not possible, the balance should be used with a specially designed wall console.

Mechanical disturbances can be caused by pumps, laboratory shakers, turbulence under laboratory fume hoods, and so forth.

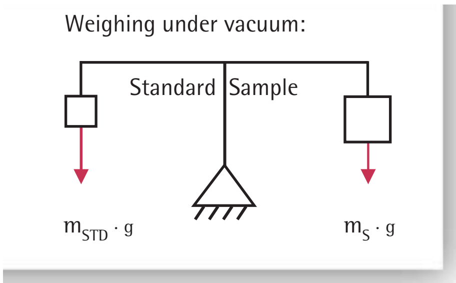
Under normal circumstances, **humidity** as an ambient quantity affecting the weighing procedure can be neglected. However, for balances of older designs and scales with a strain-gauge load cell, the change in humidity must be kept as low as possible as damage caused by corrosion of the connections can occur at high humidity. The humidity also affects the long-term stability of such load cells.

For standard weighing procedures, **barometric pressure** is a negligible source of error.

However, for precision weight measurements ($u_{rel} =< 5 \cdot 10^{-4}$), the **air buoyancy** must be taken into account as it is of considerable importance for assessing the accuracy of the value measured by the balance or scale.

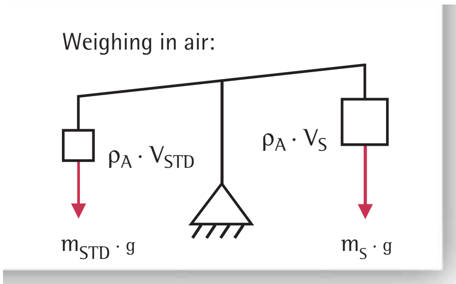
If an object is in a medium, this lifting force opposes the weight of this object. Buoyancy reduces the weight of the mass to be measured by the amount that equals the weight of the displaced medium.

If you consider two materials of the same weight but of a different volume, such as an aluminum cylinder with a density of 2.7 g/cm^3 and a weight standard with a density of 8.000 g/cm^3, both of these are in equilibrium when weighed under vacuum.



m_{STD} = Mass of the weight standard
m_S = Mass of the sample
g = Gravitational acceleration

If you consider the same setup weighed in air, both samples are no longer in equilibrium.



ρ_A = Density of the air
V_{STD} = Volume of the weight standard
V_S = Volume of the sample

This is caused by the different buoyancies resulting from the different material densities and volumes.

Because air has mass (density under standard atmospheric conditions: $\rho_A = 1.2\ \mathrm{mg/cm^3}$), the weight W of a sample depends on the density of its materials and thus on the volume it takes up.

Let Δm be the difference measured between two masses m_{STD} and m_S. You will obtain the actual difference $\Delta' m$ using the following general equation:

$$\Delta' m = m_S - m_{STD} = \Delta m + \rho_A \cdot (V_S - V_{STD}) \quad \text{or}$$

$$m_{STD} - \rho_A \cdot V_{STD} = m_S - \rho_A \cdot V_S$$

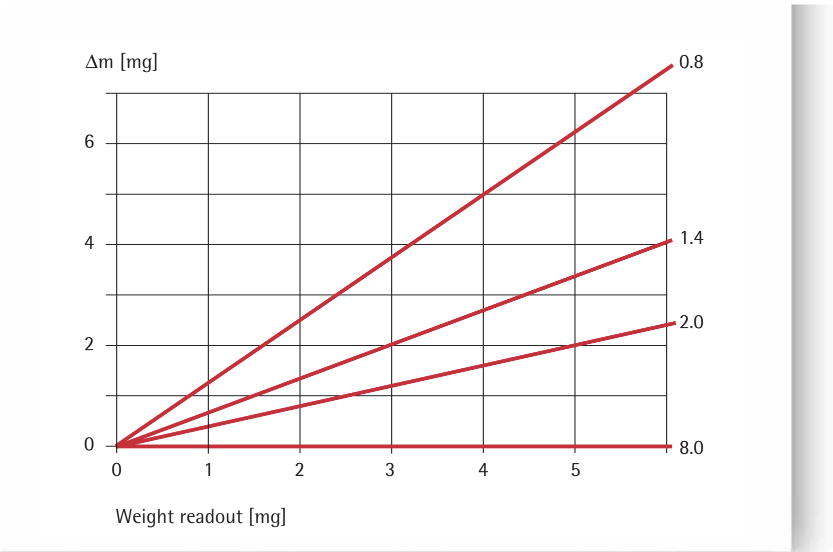
where V_{STD} and V_S are the volumes of the objects of the masses m_{STD} and m_S, and ρ_A is the density of the air according to the conditions prevailing during the weighing procedure.

The mass of the sample is determined according to the density values available,

$$m_s = \frac{1 - \frac{\rho_A}{\rho_{STD}}}{1 - \frac{\rho_A}{\rho_s}}$$

where ρ_{STD} is the density of the standard and ρ_S the density of the sample.

The graph shows how the weight readout of a mass is corrected for air buoyancy as a function of the material density for a few selected density values given in $\mathrm{g/cm^3}$.



Electromagnetic disturbances consist mainly of electromagnetic radiation in the range of a few kHz up to several GHz, which is frequently used for wireless communication:

- Radio communications
- Mobile or closed-circuit radio communications
- Transmission of weights
- Telecommunications through remote control
- Radar transmissions or measurement of noise in electric circuits

Every measuring instrument, in other words a balance or scale, must be able to function properly when exposed to the effects of these electromagnetic disturbances, generally referred to as radio frequency interference. Every balance or scale that is supplied with a Declaration of Conformity (CE mark) has passed the test prescribed by the EC Council Directive 89/336/EEC "Electromagnetic Compatibility" (EMC). This means that the balance or scale has a defined immunity to emissions in residential, commercial and industrial areas including light industrial environments. Based on the results of the EMC test, electromagnetic disturbances have no effect on the weighing results.

The Sample

In the majority of cases, the properties of the sample itself are the cause of inadmissible results. The most important factors that influence weighing accuracy are:

- Electrostatic charges
- Magnetic or magnetizable materials
- Hygroscopic materials
- Sample temperatures that deviate too much from the ambient conditions in the laboratory

Static electricity – or electrostatic charges, which are particularly noticeable when the humidity is low – is characterized by a weight readout that drifts considerably and by poor readability. This phenomenon primarily affects substances that have a low electrical conductivity and can therefore pass on charges (caused by friction with air, internal friction or direct transfer) to their environment only slowly. Examples of these substances are plastics, glass and filter materials as well as powders and liquids.

Depending on the polarity of the charged particles involved, this force either attracts or repels, so a weighing result may deviate in either direction. This effect is based on the interaction of electrical charges that have built up on the sample weighed and on the fixed parts of the balance that are not connected to the weighing pan.

This problem can be eliminated by:

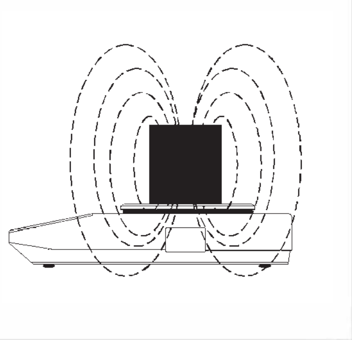
- Shielding the sample (using a metal container)
- Increasing the surface conductivity of the sample by raising the level of humidity inside the draft shield of an analytical balance
- Directly neutralizing the surface charges using so-called static eliminators

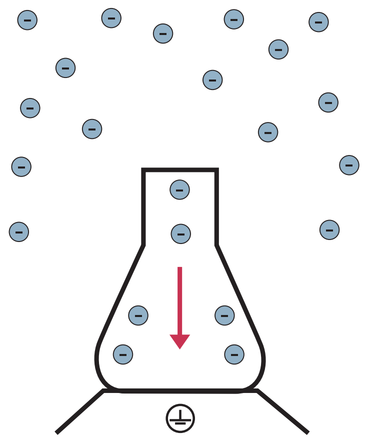
If a sample is **magnetic or magnetizable**, i.e., contains a percentage of iron, nickel or cobalt, forces of a different origin are generated, which also have a significant influence on the weighing result. If the sample is magnetized, as is the stirring bar of a magnetic stirrer, the forces of attraction that this magnet exerts on the magnetizable parts of the balance will override the weight of the sample. Vice versa, the influence that the residual magnetic field of the electromagnetic-force compensating weighing system has on a sample cannot be ruled out. Magnetic forces manifest themselves as a loss of repeatability of the weighing result because they depend on the orientation of the sample within the field of interference. Unlike electrostatic interference, magnetic interference is stable over time.

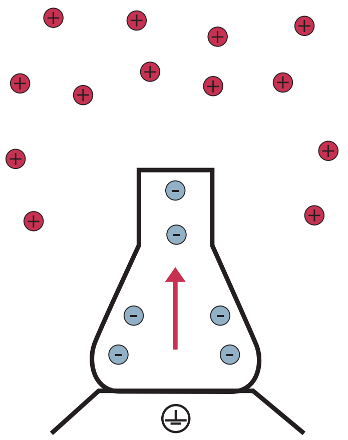

Static electricity eliminator with integrated high-voltage source


Microbalance for weighing filters; with a metallic pan cover


Semi-microbalance with a static electricity eliminator integrated as a standard feature


Example of the pattern of field forces generated by a magnetic or magnetizable sample


Interaction of electric charges that repel one another; the sample appears to be heavier


Interaction of electrostatic charges that attract one another; the sample appears to be lighter

To eliminate problems with magnetic forces, one of the following approaches can be taken:

- Increase the distance between the sample and the weighing pan
- Use a hanger for below-balance weighing (under-scale weigh kit)
- Use a shield made of a soft magnetic material
- Use a special anti-magnetic weighing pan

Hygroscopic samples cannot be precisely analyzed because they absorb moisture, which causes a constant increase in weight. If appropriate steps cannot be taken to keep the humidity to a minimum at the weighing location, we recommend that the sample be weighed in an enclosed container that is suitable for its size.

The **sample temperature** is an influence quantity that is often underestimated. Especially during very precise weighing procedures, it is imperative that the sample be adapted to the ambient temperature. Otherwise, convection currents on the surface of the sample can lead to major errors in measurement. Research has shown that when beakers with a large surface area are used during weighing, temperature differences of a few degrees [°C] can cause the readout to differ in the gram [g] range.

Special anti-magnetic weighing pan

Hanger for below-balance weighing

Traceability of a Measurement

Calibration and Adjustment

The previous sections covered a series of influence quantities that can adversely affect the accuracy of test and measuring equipment in a variety of ways. Therefore, it is hardly surprising that the test and measuring equipment standards used in all quality systems require that errors in measurement be quantified. In addition, measures for eliminating such errors must be specified. This is done through calibration and adjustment.

Calibration checks the deviation between the weight readout on the balance and a reference weight (in the field of weighing technology, this is a weight whose value is indicated on an accompanying certificate). Calibration is the most important source of information for checking a balance's or scale's uncertainty of measurement under actual installation and operating conditions. Therefore, it plays a central role in controlling the accuracy of inspection, measuring and test equipment.

Adjustment always entails corrective intervention in the balance or scale to eliminate the existing error as far as possible. During adjustment, the weight readout is compared to the "correct" value of the calibration weight, and the resulting correction factor is stored in the balance's or scale's processor until the next adjustment. Weighing procedures performed after adjustment are corrected accordingly.

How frequently a balance or scale needs to be adjusted depends significantly on the following parameters:

- The frequency of weighing procedures
- The ambient conditions
- The effects of an incorrect result

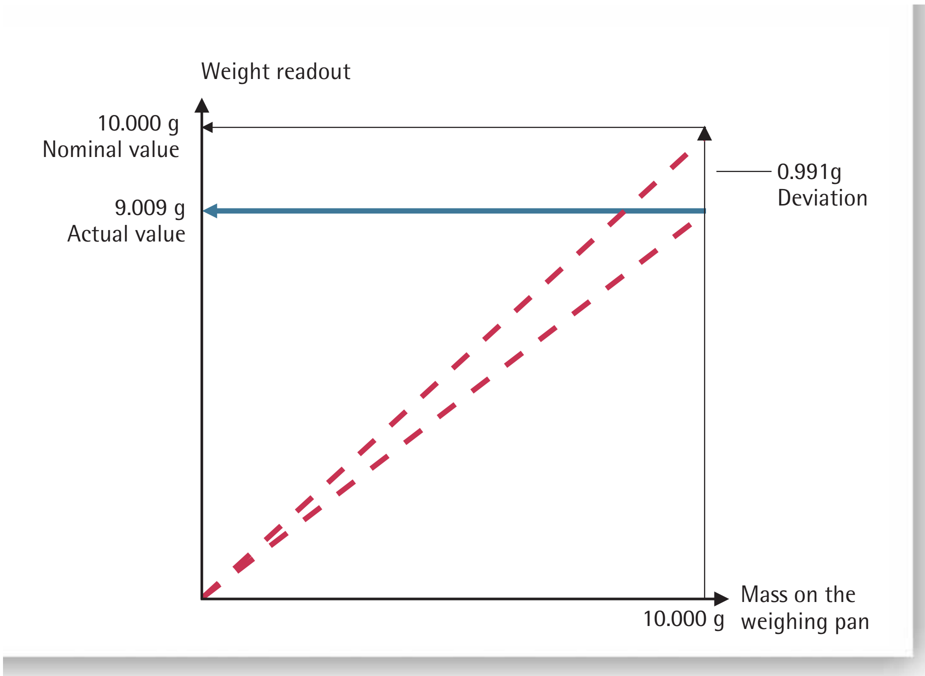
A variety of instruments and methods exist for performing both of these procedures. In general, a distinction is made between internal and external calibration and adjustment.

The **external calibration and adjustment** procedure is used mainly on older-model balances and scales or those with high capacities. Comparison and correction are accomplished using one or more weights whose value and uncertainty must be known and documented.

National testing laboratories, calibration laboratories and qualified manufacturers provide appropriate certificates for this purpose.

For **internal calibration and adjustment**, a reference weight that is built into the balance or scale is used. The exact value of this weight was previously determined during manufacture and stored as a fixed value in the electronically programmable read-only memory (EPROM) of the weighing instrument's processor. On the simplest models, the user places a weight on the balance's or scale's weighing system with the help of a mechanical device. The motorized calibration weight feature, which is operated at the touch of a button, has recently become the standard. The most advanced balances and scales are equipped with a fully automatic calibration and adjustment device that initiates calibration after a preprogrammed or user-defined amount of time has elapsed.

External calibration and adjustment of a precision balance



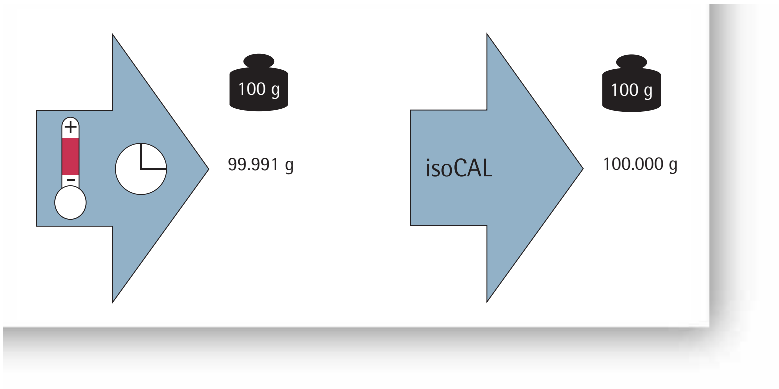
In addition, an internal sensor continuously monitors the balance or scale temperature (as a parameter for determining accuracy) and triggers automatic calibration once a certain temperature difference has been exceeded. This ensures the continuous accuracy of the balance or scale without requiring the user to intervene.

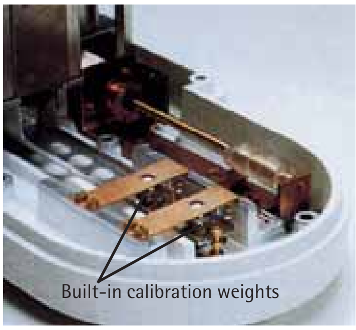
The figure below shows the sequence of functions that take place during fully automatic calibration.

Besides the advantages offered by this convenience feature, internal calibration is generally considered preferable over external calibration.

The internal weights are better protected from dirt and damage and are always at the same temperature as the balance or scale, per se. Moreover, the motorized calibration feature ensures that the weight is placed on the balance or scale in the most reproducible manner possible.
The fully automatic mode ultimately ensures that one of the most important requirements of the test and measuring equipment is fulfilled.

The question is often asked about how the traceability of a balance's or scale's built-in calibration weight can be ensured. This can be accomplished by tracing the internal calibration weight to an extremely precise reference weight from the manufacturer. With regard to its materials and surface properties, the internal weight must possess all of the features of a classified weight. As is the case with all external weights, internal weights must also be tested at certain intervals to ensure that they are within tolerance limits. This is usually done when the balance or scale is serviced.

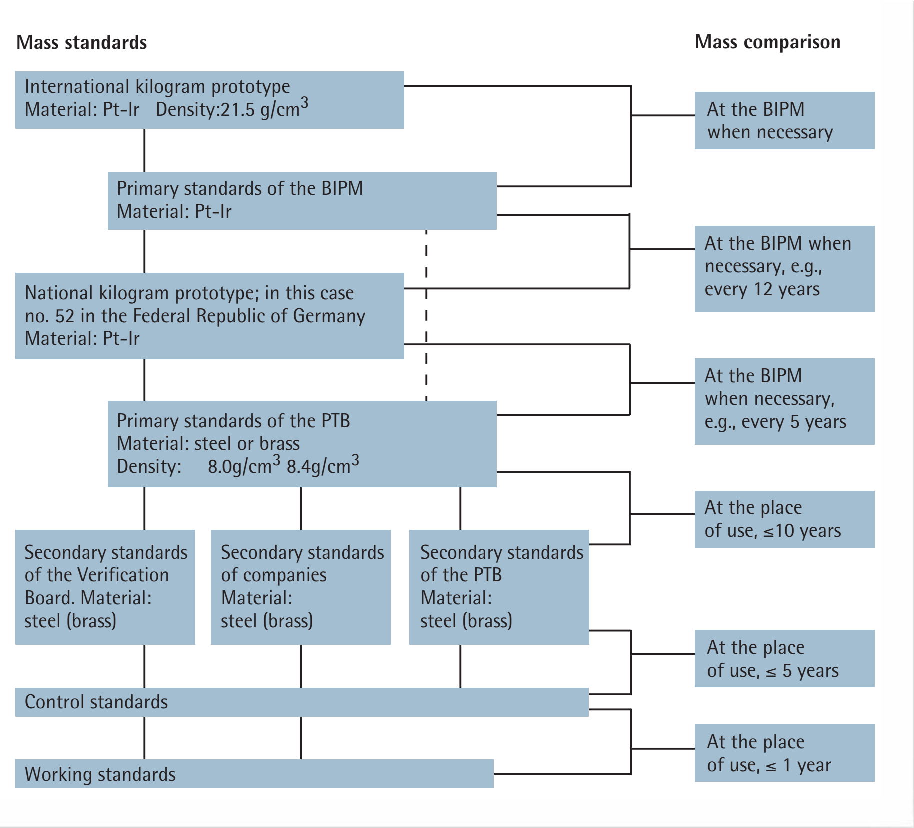




Motorized calibration weights of a micro-balance that are spherically shaped to improve the area-to- volume ratio

Mass and Weights

To enable comparison of the results obtained with various balances and scales, we must be able to trace these results to a defined standard. A balance's weighing results are traced and monitored by comparing them to a standard that represents the value of the measurand (quantity subject to measurement) that is required to be correct. This standard is also traced to the international prototype through an uninterrupted chain of such standards for comparison.



Relation to the Base Unit

Nano-gram	ng	1ng = 0.000,000,000,001 kg
Mikro-gram	µg	1µg = 0.000,000,001 kg
Milli-gram	mg	1mg = 0.000,001 kg
Gram	g	1g = 0.001 kg
Kilo-gram	kg	1 kg base unit
Ton	t	1 t = 1000 kg

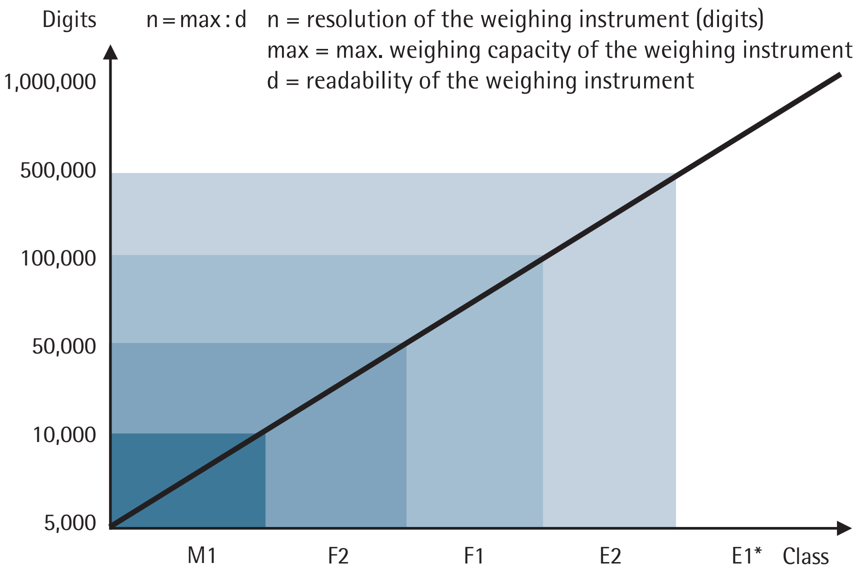
The necessity of tracing other units to the kilogram by mass comparison has given rise to the hierarchical structure of mass standards. In this hierarchy, the uncertainty of

Germany's national kilogram prototype

measurement at a certain level depends on the number of previous mass comparisons.

+ / – in mg

Nominal value	E1	E2	F1	F2	M1	M2	M3
1 mg	0.002	0.006	0.020	0.06	0.20		
2 mg	0.002	0.006	0.020	0.06	0.20		
5 mg	0.002	0.006	0.020	0.06	0.20		
10 mg	0.002	0.008	0.025	0.08	0.25		
20 mg	0.002	0.010	0.03	0.10	0,3		
50 mg	0.004	0.012	0.04	0.12	0.4		
100 mg	0.005	0.015	0.05	0.15	0.5	1.5	
200 mg	0.006	0.020	0.06	0.20	0.6	2.0	
500 mg	0.008	0.025	0.08	0.25	0.8	2.5	
1 g	0.010	0.030	0.10	0.3	1.0	3	10
2 g	0.012	0.040	0.12	0.4	1.2	4	12
5 g	0.015	0.050	0.15	0.5	1.5	5	15
10 g	0.020	0.060	0.20	0.6	2	6	20
20 g	0.025	0.080	0.25	0.8	2.5	8	25
50 g	0.030	0.10	0.30	1.0	3.0	10	30
100 g	0.05	0.15	0.5	1.5	5	15	50
200 g	0.10	0.3	1.0	3	10	30	100
500 g	0.25	0.75	2.5	7.5	25	75	250
1 kg	0.5	1.5	5	15	50	150	500
2 kg	1.0	3,0	10	30	100	300	1000
5 kg	2.5	7.5	25	75	250	750	2500
10 kg	5	15	50	150	500	1500	5000
20 kg	10	30	100	300	1000	3000	10000
50 kg	25	75	250	750	2500	7500	25000



* or E2, DKD calibrated

Documentation

A characteristic element of all quantity systems is the requirement of documentation. Requirements as to the extent and depth of the documentation vary significantly depending on the system being used. In any case, it is helpful to use the rule of five W's as a guide when developing a set of instructions that must be followed. This rule states that procedures must be documented in such a way as to answer the question:

"Who Did What, with What, When and Why?"
In the area of management of test and measuring equipment, experience has shown that this requirement is best met by introducing and maintaining an SOP and a logbook for the weighing instrument. While all aspects of operation are laid out in the SOP, the logbook contains entries about the maintenance, service and repair procedures for the particular balance or scale.

Practical examples of an SOP and a logbook are given in the Appendix of this Handbook.

In particular, the following must be recorded:

- Description and identification of the test and measuring equipment
- Calibration equipment and results
- Defined maximum permissible errors
- Ambient conditions and corresponding adjustments
- Maintenance procedures
- Modification of the weighing instrument(s)
- Identification of the personnel responsible
- Restrictions on the suitability of test and measuring equipment

These items are discussed in more detail in the following sections.

Description and Identification of the Test and Measuring Equipment: This includes general information about the type of weighing instrument (e.g., analytical balance with a motorized draft shield); the most important manufacturer specifications; and the model, serial number or inventory number at the weighing location.

Calibration Equipment and Results: These two factors are decisive for maintaining the desired degree of weighing accuracy. Depending on the resolution of the balance or scale and its construction features (motorized placement of the weight on the weighing pan, fully automatic calibration function), determinations must be made about the nominal value, the maximum permissible errors and how the weights are to be used. The weights or sets of weights employed are also considered test and measuring equipment and must be labeled and identified accordingly. Intervals for recalibration of the weights must also be defined. Especially when there are large deviations in the calibration results, control limits must be defined, and a procedure must be developed for reporting such deviations.

For **defined maximum permissible errors**, the **overall uncertainty** of measurement, which was determined using the test and measuring equipment described above, must be traceable. On the basis of this value, the user can determine whether a balance or scale is suitable for the tolerance indicated in the SOP (e.g., the analysis.)

Ambient Conditions and Corresponding Adjustments
The specifications that characterize the balance or scale are determined by the manufacturer under well-defined standard conditions. In reality, however, certain usually unfavorable conditions often cannot be avoided. For example, if the balance or scale is located under a fume hood in the laboratory or in a place where there are great fluctuations in temperature, the analysis can be adversely affected. Modern balances and scales can be adapted to the ambient conditions at the weighing location by varying the set of parameters in the operating system so that the balance or scale may be used in that location. However, this usually results in the accuracy being reduced.

Example:
For example, if the "stability range" parameter is increased, the balance or scale can deliver accurate results even when it is subjected to a field of interference of a great amplitude. The attainable repeatability however, is sacrificed in the process. In this case, the change in the parameters of the balance or scale operating system and the influence on the uncertainty of measurement must be documented.

Maintenance Procedures

Determinations must be made about

- when the balance or scale should be cleaned,
- who should service it and at what intervals,
- and how to proceed if a repair is necessary.

The results of regularly performed maintenance procedures can also be useful for analyzing the trend of certain deviations. This facilities appropriate definition of the interval of confirmation.

Modification of the Weighing Instrument(s)

A variety of technical applications require that a standard-equipped balance or scale be modified. For example, a hanger for below-balance weighing might be used if either the size of the sample or special ambient conditions (such as magnetic fields, temperature, humidity and so forth) dictate the manner in which the analysis should be conducted. Weighing pans of modified shapes and sizes and analytical balances with specially designed draft shields are also often used. Today, leading manufacturers are in a position to offer their customers application-specific solutions with respect to digital filters or other weighing parameters. Dynamic weighing procedures constitute one of the main application areas for which this type of modification is necessary.

Appointment and Identification of Personnel Responsible for Monitoring Test Equipment

The laboratory manager appoints a person to oversee the test and measuring equipment. This person is responsible for the appropriate use of the balances and scales.

Restrictions on the Suitability of Test and Measuring Equipment

If a confirmation or calibration procedure determines that the test and measuring equipment can no longer operate within the defined maximum permissible errors, even if corrective intervention is taken, the balance or scale should no longer be used for the intended purpose. Of course, it is possible to use the balance or scale for analyses that do not require such a high level of accuracy. In this case, the limited application range must be clearly denoted on the instrument and indicated in the SOP.

Defining the Interval of Confirmation

We use the term confirmation to summarize all activities that ensure that the predefined properties of the test and measuring equipment are maintained. Therefore, the interval of confirmation corresponds to the time interval or number of analyses performed with the test and measuring equipment between two successive inspections. From an economic standpoint, testing should be optimized so that it is performed before a balance or scale exceeds the maximum permissible errors. This is also closely connected to the previously mentioned rule, which states that the uncertainty of measurement of the test and measuring equipment should be much lower than that required by a particular weight measurement application. The following should be taken into account when first defining the interval of confirmation:

- The extent of possible adverse effects on the analysis due to non-conforming test and measuring equipment
- Manufacturer´s recommendation
- Tendency toward component wear and drift
- Environmental influences
- Demands of customers, standards or laws
- Experiences with similar test and measuring equipment

The following questions should be taken into account **if non-conforming test and measuring equipment causes consequential damage:**

1. When should data obtained with a nonconforming instrument be rejected?
2. What additional expenses can result from overfilling expensive substances?
3. Can the customer assert product liability claims in such case?

Manufacturer´s Recommendation

Laboratory balance manufacturers – if they provide service and maintenance for their products – have an extensive amount of data at their disposal with respect to all important features of the balance. This is especially true given various areas of use and ranges of application of lab balances.

Tendency Toward Component Wear and Drift:

On advanced laboratory balances and scales, this tendency can be neglected because these weighing instruments are designed and constructed to keep component wear to a minimum when they are operated according to the manufacturer´s instructions. The readout might drift in individual cases and after prolonged use of the balance or scale due to the electronic components.

Environmental Influences:

The range of uses for balances and scales are specified according to temperature and humidity classes. If a weighing instrument is mainly or constantly subjected to temperatures or levels of humidity that border on the allowable limits of these classes, the specifications will likely be affected and must be taken into account accordingly.

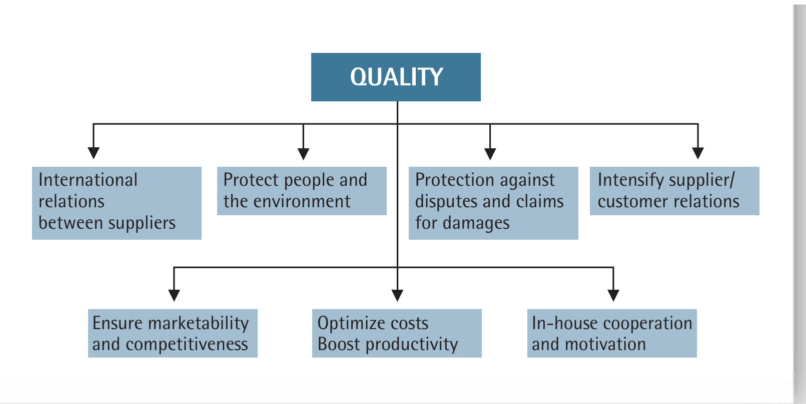
Demands of Customers, Standards or Laws

If the equipment is to be used in sensitive areas with very high security standards (e.g., in the aerospace industry, for medical technology, for pharmaceutical production and so forth), the customer will place high demands on the supplier´s quality system. These demands can go far beyond the standard requirements and, therefore, can have an influence on the control of inspection, test and measuring equipment.

Experience with Similar Test and Measuring Equipment

Because of the multitude of factors that must be considered when defining the interval of confirmation, a general recommendation on how to do so cannot be made. It makes more sense to follow your technical “intuition“ and consider the relevant factors to determine a suitable interval. Statistical data from the current inspection can be used to check calibration and optimize the interval that is initially selected. For example, the interval of confirmation can be gradually adjusted by cutting the test interval in half, if the maximum permissible errors are exceeded, or doubling it if the requirements are met satisfactorily. From an economical standpoint and to ensure the traceability of test results, it may be useful to combine extensive inspections at longer intervals with additional short-term tests or calibration procedures using suitable working standards.

Summary



The control of inspection, measuring and test equipment is an element of functional quality management. It is a prerequisite for objectively demonstrating the performance of a laboratory as well as for introducing and maintaining processes that can be controlled.

This starts with the selection of a suitable test or measuring device based on the tolerances to be tested, which, for instance, are indicated in the laboratory’s SOPs. Measuring equipment suitable for this purpose has an overall uncertainty of measurement that is much lower than the sample with respect to the specifications of the equipment and all factors that have an influence on the measurement.

Suitable SOPs should be indicated in writing to ensure that the test requirements are always met, and all related data should be documented.

Error Calculation

A deviation in the displayed value from the true value is commonly known as an "error" or "deviation"; the standardized term is "error of measurement." In the following, we will use the simpler form "error." We distinguish between two types of error: systematic and random errors.

Systematic Errors
The cause of the error is known, perhaps even the value of this error, or at least an upper limit of error.

Examples:

1. A scale of lengths is not exactly accurate in length; all measurements are made with the same scale of lengths.
2. The same holds true for a weighing instrument; e.g., a balance with an incorrectly adjusted sensitivity.
3. A measuring instrument is adjusted to 20°C, but the measurement is carried out at 25°C (this is important, e.g., in the case of a volumeter.)

Random Errors
The cause of a deviation is either unknown, or this deviation is caused by varying influence factors.

Examples:

1. Friction in a measuring instrument that has mobile components
2. Random fluctuations in the zero point of a weighing instrument
3. Statistical influence of the operator (e.g., parallax errors when the operator reads off the measuring instrument display that has a pointer; or a change on the mass of the object being weighed when the operator touches it with his or her hands)

Note:
There is no hard-set difference between systematic and random errors. By means of additional measurements or information, many random errors can be transformed into correctable systematic errors.

There are mathematical rules for random errors:

Rule 1:
If a measurement is repeated a sufficient number of times and the frequency distribution of the individual values measured are plotted, you will obtain a characteristic curve, the so-called Gaussian curve.

The standard deviation "s" is given as the quantity for repeatability:

$$s = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^{n} (\chi_i - \bar{\chi})^2}$$

n = total number of measurements
χ_i = individual results measured
$\bar{\chi}$ = mean value of the individual results measured $\quad \bar{\chi} = \frac{1}{n} \cdot \sum_{i=1}^{n} \chi_i$

Gaussian distribution ("normal distribution")
$\bar{\chi}$ = average weight; s = standard deviation
68.3% of the weighing results lie within the range of $\chi \pm s$
95.5% of the weighing results lie within the range of $\chi \pm 2s$
99.7% of the weighing results lie within the range of $\chi \pm 3$

Rule 2:
Law of error propagation for sums and differences: In sums or differences, the squares of the absolute individual errors (E) are added and the square root of this sum is taken:

$$E_{Sum} = \sqrt{(E_1)^2 + (E_2)^2}$$

F1, F2 = individual errors

Example:

The gross weight m_G of	210.213 g
And the tare weight m_T of	205.171 g
Yield the mass m_{Net} of	5.042 g

The individual errors of m_G and m_T, respectively, are each 1 mg
Hence, the absolute error of m_{Net} is :

$$E_{Net} = \sqrt{(1 \text{ mg})^2 + (1 \text{ mg})^2} = 1.4 \text{ mg}$$

and the relative error is:

$$\frac{E_{Net}}{m_{Net}} = \frac{1.4 \text{ mg}}{5.042 \text{ g}} = 0.028\% = 2.8 \cdot 10^{-4}$$

Rule 3:
Law of error propagation for products and quotients:
In products or quotients, the square of the relative individual errors are added and the square root of this sum is taken:

$$\frac{E_{Result}}{Result} = \sqrt{\left(\frac{E_1}{Value_1}\right)^2 + \left(\frac{E_2}{Value_2}\right)^2}$$

Example: Density determination in accordance with the equation: $\rho = \frac{m}{V}$

m = mass = 150.27 g ± 0.01 g
V = volume = 173.4 cm^3 ± 0.1 cm^3
ρ = density

$$\rho = \frac{150.27 \text{ g}}{173.4 \text{ cm}^3} = 0.866609 \frac{\text{g}}{\text{cm}^3}$$

$$\frac{E_\rho}{\rho} = \sqrt{\left(\frac{F_m}{m}\right)^2 + \left(\frac{F_v}{v}\right)^2}$$

$$\frac{E_\rho}{\rho} = \sqrt{\left(\frac{0.01 \text{ g}}{150.27}\right)^2 + \left(\frac{0.1 \text{ cm}^3}{173.4 \text{ cm}^3}\right)^2} = 5.80 \cdot 10^{-4}$$

$$E_\rho = \rho \cdot 5.8 \cdot 10^{-4} = 0.8666 \frac{\text{g}}{\text{cm}^3} \cdot 5.8 \cdot 10^{-4} = 0.5 \cdot 10 - 3 \frac{\text{g}}{\text{cm}^3}$$

Final result: $\rho = (0.8666 \pm 0.0005) \frac{\text{g}}{\text{cm}^3}$

Deriving the Uncertainty of Measurement from the Standard Deviation

Only approximately 68% of the measured results lie within the range of ± s from the mean value. Therefore, for practical purposes, the (maximum) uncertainty of measurement "u" is frequently defined as ± 2 s (95% of the measured results lie within the range of ± 2 s from the mean value).

We will use: $u = 2\,s$

Example for Calculating the Uncertainty of Measurement for Samples of Approx. 10 g:

Small amounts (approx. 10g) are to be weighed on a GENIUS ME2545 semi-microbalance with a resolution of 0.1 mg. Ambient conditions are good (no tilting; temperature difference of 5°C max.; none of the containers or objects is electrostatically charged, nor is there any electromagnetic interference.)

- The containers are small and must be correctly centered, as directed in the standard operating instructions. Therefore, the off-center load error can be neglected for 10 g.
- The repeatability/standard deviation is: $\leq \pm 0.07$ mg
- The temperature coefficient for the sensitivity is 1ppm/K => $\leq \pm 1 \cdot 10^{-6}$ /°C, as stated in the technical specifications. Hence, the error for 10 g and $\Delta T = 5°C$ is $\leq \pm$ 10 g $\cdot 1 \cdot 10^{-6}$ /°C $\cdot$ 5°C = $\leq \pm 0.05$ mg
- The max. linearity error is as stated in the technical specifications: $\leq \pm$ 0.15 mg
- The balance has been calibrated and adjusted with a standard E_2 class weight of 200 g (maximum error of 0.3 mg). In relation to a 10-g load, the error is: $\leq \pm 0.015$ mg

The sample´s density is 2.0g/cm^3, with an uncertainty of ±20%; the difference between air buoyancy of the samples and that of the standard weights used to adjust the balance is thus 2.25 mg with an uncertainty of ±20%
± 0.45 g.

The uncertainty of this air buoyancy correction value due to fluctuations in the air density of ±10% is considerably less than that of density fluctuations.

Deriving the Uncertainty of Measurement from the Standard Deviation

With the exception of the repeatability| standard deviation, all values are maximum errors. If the equation of u=2s is used to express the maximum uncertainty of the repeatability and if the air buoyancy has been corrected, the uncertainty of measurement will be as follows:

$$u = \sqrt{(2 \cdot 0.07\ \text{mg})^2 + (0.05\ \text{mg})^2 + (0.15\ \text{mg})^2 + (0.015\ \text{mg})^2 + (0.45\ \text{mg})^2}$$

u= 0.50 g

However, if no correction is made for air buoyancy, a systematic error of 2.25 mg is added to the uncertainty of measurement "u" so that the total deviation can be as much as 2.75 mg.

The uncertainty of measurement of a weighing instrument can be exactly determined over its entire weighing range by calibration in a DKD*-accredited laboratory, which Sartorius has.

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*DKD = German Calibration Service officially recognized throughout Europe

Index

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(German titles have been translated into English in parentheses for convenience.)

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Specifications subject to change without notice.
Printed in Germany on paper that has been
bleached without any use of chlorine.
Publication No.: W--0123-e08071
Order No.: 98649-008-45