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2.1.7. BALANCES FOR ANALYTICAL PURPOSES

The scope of this chapter is limited to balances used for analytical purposes. It does not cover balances used for manufacturing or other purposes. Any weighings performed as part of tests prescribed to establish compliance with a monograph of the European Pharmacopoeia must be carried out according to the principles outlined in this chapter.

Information about significant digits and rounding for mass values prescribed in a monograph or any other chapter of the European Pharmacopoeia can be found in the General Notices (under Quantities).

PRINCIPLE

A balance is an instrument used to determine the mass of an object. The SI unit for mass is the kilogram, but its submultiples, e.g. µg, mg or g, are often used.

While weighing instruments make use of different physical principles of mass determination, the majority are based on the weight, i.e. the gravitational force, F_G (expressed in newtons), exerted by Earth on the object being weighed. Weight is defined by the following expression:

$$F_G = m \cdot g \qquad (1)$$

m = mass of the object in kilograms;

g = local acceleration due to gravity in metres per square second (≈ 9.81 m·s^{-2} at sea level).

The two most common gravitational force weighing principles are force compensation (typically used in electronic balances) and mass comparison with a known mass (typically used in mechanical balances).

Depending on the measuring principle of the balance, the mass is either measured directly (e.g. beam balance) or is calculated from the weight using equation (1) (e.g. balance using electromagnetic force compensation).

ELECTRONIC BALANCES

Most balances used are electronic and based on force compensation. In practice, the gravitational force exerted on the object being weighed can be compensated by:

– elastic deformation: the object being weighed presses down onto a spring element that reacts with a compensation force F_c, expressed in newtons, given by the following equation:

$$F_C = c \times \Delta_S \qquad (2)$$

c = spring constant in newtons per metre;

Δs = length change due to elastic deformation in metres; strain gauges are used to measure the strain or elongation and convert it into electrical resistance;

– an electromagnetic force that holds the load cell in equilibrium. In most high-resolution balances, this is the Lorentz force which is generated by a current inside a coil surrounding a permanent magnet.

MECHANICAL BALANCES

Most mechanical balances are equal-arm beam balances. An equal-arm beam balance performs a mass comparison with a two-arm lever and two weighing pans. The mass of the object being weighed is compensated for by counter weights of known mass at the opposite end of the lever. The counter weights are chosen in such a way that the beam of the balance is in equilibrium position.

EQUIPMENT

Balances can be further classified according to their scale interval, *d*, also known as readability. This is the smallest increment of mass that can be indicated on the balance.

Type (*sub-type*) of balance	**Readability, *d* (in grams)**
Precision	10^{-1} to 10^{-3}
Analytical	≤ 10^{-4}
Semi-micro	10^{-5}
Micro	10^{-6}
Ultra-micro	10^{-7}

Most balances in use today show the result of weighings on a digital display. Mechanical balances, e.g. beam balances, without such displays are rarely used.

Balances generally have some means of showing that the indication has stabilised and can be recorded or printed. Balances may be connected to other equipment which documents the result of the weighing procedure, e.g. printers or electronic systems such as laboratory information management systems.

Balances may be built into analytical equipment used for tests such as loss on drying, thermogravimetry and dynamic vapour sorption that determine properties of a sample by measuring changes in its mass, under defined conditions. Balances used for these purposes also fall within the scope of this general chapter, but more specific requirements than given here may be found in the corresponding general chapters.

INSTALLATION AND LOCATION

It is recommended to follow the manufacturer's instructions when installing a balance. It is particularly important to ensure that the installation conditions and location do not adversely affect the performance of the balance.

The environmental parameters that influence the performance of balances include:

– temperature, including changes of temperature caused by direct sunlight;

– ambient humidity and pronounced changes thereof. The optimum relative humidity (RH percentage) during a weighing operation is between 40 per cent and 60 per cent;

– barometric pressure;

– air currents: generated by heaters, air conditioners or devices with ventilators (e.g. computers or large laboratory equipment), or any airflow in doorways or areas of high traffic (corridors) and, in the case of toxic and other special materials, when the balance is placed inside a fume cupboard;

– dust;

– electrostatic forces: electrostatic charging can be significantly reduced by using metal weighing vessels or antistatic devices. Balances must always be grounded (e.g. via the electric plug). Low relative humidity increases the risk of electrostatic charging;

– magnetic forces (e.g. RF generators, magnetic fields from other laboratory equipment);

– vibrations.

Irrespective of the construction materials, the weighing bench should be stable, non-magnetic and vibration-proof. It should also be protected against electrostatic charges (e.g. by grounding).

A balance used for analytical purposes is designed to measure small masses. The weighing pan of these balances is generally located inside an enclosure to reduce dust collection and the influence of air currents on the operation of the balance.

It is important to level balances correctly; most balances have a bubble level which must be brought to the centre by modifying the height of the feet of the instrument, others are equipped with an electronic levelling system. Balances must be adjusted after levelling, using either built-in weights (if available) or external calibrated weights.

Balances must be allowed to warm up after they are connected to the power supply. Typically, this may take from a few minutes for precision balances to up to several days for ultra-micro balances, depending on the model. Electronic balances should be left powered up, as this allows them to stay in thermal equilibrium.

WEIGHING VESSELS

Special care must be taken to ensure that the weighing vessel and the closure are made from an inert material that is compatible with the sample. The size of the weighing vessel must not compromise the repeatability and accuracy of the weighing process. Small weighing vessels tend to give the most accurate results. However, it may sometimes be more practical to use larger weighing vessels, for example, a volumetric flask in the case of samples that are to be diluted after weighing to avoid potential transfer errors.

If considered necessary, the influence of the weighing vessel on the repeatability and accuracy of the measurements may be assessed by including the vessel as a tare in the corresponding equipment performance checks.

Care should also be taken to ensure that weighing vessels composed of materials with a high degree of electrical insulation (e.g. glass and plastic) are not electrostatically charged.

Weighing vessels are made of non-magnetic materials to prevent magnetic interference with the components of electronic balances.

Vessels suitable for weighing solid materials include weighing paper, dishes and funnels, or sealable vessels such as bottles, vials, and flasks, which can also be used for liquids.

Weighing dishes are typically made from a polymer, glass or a metal such as aluminium. Antistatic weighing dishes are available for use with materials that retain static electricity.

Weighing funnels are typically made from glass or from a polymer. The design of this type of weighing vessel combines attributes of a weighing dish and a transfer funnel, which can simplify the transfer of a weighed powder to a narrow-necked vessel such as a volumetric flask.

The weighing vessel and the sample must have the same temperature as their surroundings and the balance.

EQUIPMENT PERFORMANCE

Weighing instruments must be periodically calibrated and checked to ensure compliance with pre-defined requirements. Performance checks must be carried out between calibrations.

Minimum requirements for performance checks are given below. The frequency of the qualification and performance checks is defined in each user's quality management system.

CALIBRATION

Calibration is part of balance qualification and is performed by the user or by a suitable competent body. Its aim is to establish traceability of measurement results to SI units (metrological traceability). The calibration results include measurement uncertainty and are documented in a calibration certificate. To further maintain such traceability, it is recommended to perform calibration before any maintenance operation is carried out on the balance that significantly alters its measurement performance. 'Significant' operations include repairs, transfer of the balance to another location or mechanical adjustment of one or more weighing parameters. The balance must be re-calibrated after significant operations. Re-calibration is not necessary after less significant operations, which include levelling the balance or adjustments using built-in weights.

PERFORMANCE CHECKS

Performance checks are carried out to evaluate the random and systematic error of a balance; they consist of measuring precision and accuracy respectively and comparing the results obtained to pre-defined acceptance criteria. Balances are considered suitable if none of these errors exceeds 0.10 per cent.

In practice, performance checks focus on the two weighing parameters that most significantly affect the performance of the instrument, i.e. repeatability, for precision, and sensitivity as the main component of the accuracy of the balance.

Accuracy is also impacted by two other parameters: eccentricity and linearity. A quadratic addition of the errors of these individual parameters, rather than a more conservative linear addition, provides a more realistic approach to the assessment of the accuracy of the balance because the three individual parameters are known to be largely independent of each other, and it is considered unlikely that they will occur simultaneously and have the same algebraic sign. Therefore, the acceptance criterion for each individual parameter can be set at 0.05 per cent, i.e. half the overall accuracy tolerance of 0.10 per cent. While accuracy is impacted by all three parameters, the impact of eccentricity and linearity is typically less than that of sensitivity. Hence, during a performance check on accuracy, it can be considered sufficient to investigate sensitivity only (at 0.05 per cent), all the more since the three individual parameters impacting accuracy are thoroughly evaluated during calibration.

Repeatability

In most cases, the net mass of the material being weighed is considerably smaller than the maximum capacity of the balance. When weighing such small quantities, one of the major contributors to measurement uncertainty is random error. This is estimated by the standard deviation of the indications that are obtained according to the following procedure.

Use a single weight denomination preferably in the lower end of the measurement range, for example not more than 5 per cent of the maximum capacity of the balance. However, if this yields a test load below 100 mg, 100 mg should be used, as smaller weights are difficult to handle. Zero the instrument, place the chosen test load on the weighing pan and record the indication. Repeat the whole procedure, including zeroing, at least 10 times.

The repeatability is satisfactory, if:

$$\frac{2 \times s}{m_{snw}} \times 100 \leq 0.10 \qquad (3)$$

NOTE: if $s < 0.41 \times d$, where d is the readability (scale interval) of the balance, replace s by $0.41 \times d$;

s = standard deviation of the indicated values (e.g. in grams);

m_{snw} = smallest net weight (e.g. in grams). This value is defined by the user as the smallest net amount of substance that will be weighed on the balance.

The lower limit of "$0.41 \times d$" for the standard deviation originates from the rounding error of the balance. Given that weighing operations comprise two readings (tare and net sample weight), and as the rounding error allocated to a single reading is calculated as "$0.29 \times d$", in this case the propagation of errors by a quadratic sum gives "$0.41 \times d$".

Based on the result of the repeatability test, the minimum weight ($m_{\min}$) of the balance can be determined. The "minimum weight" is the smallest net sample mass that can be weighed on the balance, whilst continuing to comply with the repeatability test criterion. It is given by the following equation:

$$m_{\min} = 2000 \times s \qquad (4)$$

NOTE: if $s < 0.41 \times d$, replace s by $0.41 \times d$

Sensitivity

The sensitivity test assesses the parameter that most significantly influences the accuracy of the balance.

The sensitivity deviation increases approximately linearly with the load, and thus is more significant in the upper part of the weighing range. In addition, as the influence of the random error is dominant at the lower end of the measuring range, using a test load with a mass below 5 per cent of the capacity of the balance to determine the error of sensitivity is not meaningful.

Sensitivity is assessed using a single test load with a mass of between 5 per cent and 100 per cent of the capacity of the balance. Zero the instrument, place the selected test load on the weighing pan and record the indication.

The sensitivity is satisfactory, if:

$$\frac{|I - m|}{m} \times 100 \leq 0.05 \qquad (5)$$

m = nominal weight of the test load, or its conventional mass (see conditions below), e.g. in grams;

I = indication, e.g. in grams.

It is generally sufficient to use the nominal weight as the test load value for the assessment, as long as the relative maximum permissible error of the test load (i.e. the maximum permissible error of the test load divided by the nominal weight) is not more than one third of the sensitivity test specification (0.05 per cent). If this ratio cannot be achieved, the conventional mass value of the test load (stated on the calibration certificate of the weight) must be used for the assessment. In this case, the user must ensure that the weight uncertainty divided by the nominal weight is not greater than one third of 0.05 per cent.

USE OF REFERENCE WEIGHTS

The reference weights used for calibration and the sensitivity test (and any other optional test for assessing the balance accuracy) comply with either OIML R-111 or ASTM E-617 standards (notably concerning metrological traceability). Other weights can be used for the repeatability test, provided their mass does not change during the test.

USE OF BUILT-IN WEIGHTS

In addition to testing weighing instruments with external weights, it is accepted practice to adjust the instruments by means of built-in weights. This makes it possible to reduce the frequency of sensitivity tests with external reference weights. For electronic balances with a built-in weight, daily sensitivity testing with an external reference weight is not considered necessary, but a test with external weights should nevertheless be conducted periodically since it allows detection of any problems with the built-in weights themselves.

WEIGHING PROCEDURE

The balance and weighing vessel must be appropriate for the quantity to be weighed and the expected performance. Both the balance and the weighing vessel must be clean and dry. The gross weight of the weighing vessel plus the material to be weighed must not exceed the maximum capacity of the balance.

It is important that samples and weighing vessels are equilibrated to the ambient laboratory temperature in order to avoid weighing errors. For example, a flask that is warmer than ambient air warms up this air, which then flows upward along the flask and reduces the apparent weight of the contents by viscous friction.

Place the weighing vessel on the balance, taking care to centre it on the weighing pan. Avoid handling the vessel with bare hands as this may affect the temperature and relative humidity. Tweezers can be used instead. After the balance display stabilises, tare the balance, and without spilling, add the desired amount of material to the weighing vessel. Allow the balance display to stabilise and record the mass. If a material transfer is necessary, take care to perform this operation quantitatively or, if it cannot be guaranteed that the entire amount has been transferred, weigh the weighing vessel again and note the difference.

SAMPLES

For volatile, deliquescent or hygroscopic samples, it may be advantageous for analysts to use a vessel with a small opening or a vessel equipped with a gas-tight closure, ensuring the closure is put in place rapidly following weighing. The analyst can also tare both the vessel and closure prior to the operation, then add the material, close the vessel and record the indication.

ELECTROSTATIC SAMPLES

Problems may be experienced when weighing electrostatic samples, since electrostatic forces may render them difficult to handle and can lead to incorrect and non-reproducible results. In such cases, an anti-electrostatic system can be helpful. Plastic and glass containers should be avoided. Metal containers usually reduce or prevent electrostatic charging. The risk of electrostatic effects is higher in working environments with low relative humidity, e.g. below 40 per cent RH.

MAGNETIC SAMPLES

Magnetic forces between a magnetic sample and parts of the balance or the environment add to gravity and may lead to incorrect and non-reproducible results. Special care should therefore be taken when weighing these samples, for example, by shielding them with a suitable material such as mu-metal. It may also be useful to increase the distance between the sample and metallic parts of the balance, as magnetic forces decrease with increasing distance.