PRACTICAL LABORATORY SKILLS TRAINING GUIDES

Measurement of Mass

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Co-ordinating Author

RS•C

VAM
VALID ANALYTICAL MEASUREMENT

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Preface

Production of this set of five Training Guides and CD-ROMs was supported under contract with the Department of Trade and Industry as part of the National Measurement System Valid Analytical Measurement (VAM) programme.

The guides were written by staff at LGC in collaboration with members of the SOCSA Analytical Network Group whose assistance is gratefully acknowledged. They include liquid and gas chromatography, the measurement of mass, volume and pH.

Training has formed an essential part of the VAM programme since its inception in 1988. Many training courses on topics aimed at improving the quality of measurements have been developed. However, in working with groups of analytical scientists it has become clear that the basic skills required in an analytical laboratory are not covered on courses or readily available in paper format.

These guides are aimed at filling this gap and are aimed at those working at the bench. For each topic they include a limited amount of theory to explain the essential features but the main emphasis is on what to do to ensure reliable results. They contain references to further reading for those who wish to study the topics in more depth.

To help laboratory managers assess the competence of the trainee there are a limited number of exercises suggested. The chromatography modules also have a trouble shooting section.

The CD-ROMs cover Practical Laboratory Skills and have links to websites where more information may be obtained.

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Measurement of Mass

1 Introduction

Measurement of mass is one of the most frequently carried out operations in an analytical laboratory. Accurate mass measurements are required for such purposes as:

- obtaining a known quantity of a sample for analysis
- preparation of analytical reagents
- preparation of calibration standards

Although modern analytical balances seemingly make weighing objects to 4 or 5 decimal places (0.0001 to 0.00001 gram) a simple and routine procedure, the operation is by no means foolproof. The analyst should approach the weighing of a sample, reagent or any other artefact with the same care and critical appraisal that is (or should be) applied to the more 'exciting' parts of an analytical investigation.

This document provides guidance on various important aspects of mass measurement as it is commonly carried out in analytical laboratories.

2 Mass and Weight

Although in everyday speech the terms 'mass' and 'weight' tend to be used interchangeably, they actually have distinctly different meanings. Mass is the amount of material in an object and does not change with the environment in which the object is located. Weight, in contrast, is a force arising from the interaction of the mass with the earth's gravitational field, which varies with location.

Correctly speaking, we use a balance to determine the *mass* of an object on the basis of its weight, *i.e.* the downward force the object exerts on the balance pan. A measurement of weight is able to provide a mass value since the influence of gravity is eliminated by the calibration of the balance, which is carried out in the same location (gravitational field) in which the object is measured.

Buoyancy is another factor affecting weight (and therefore measured mass). This is discussed in Section 11.

3 Units of Mass Measurement

In the international system (SI) of measurement units the base unit of mass is the kilogram (kg). In routine analytical work it is usually more convenient to work in terms of grams (g), where 1000 g = 1 kg (or 1 g = 10^{-3} kg). Other sub-divisions and multiples of the gram that are likely to be encountered are given in Table 1.

Table 1 *Sub-divisions and multiples of the gram*

Unit	*Symbol*	*Relationship to the gram*
femtogram	fg	1 fg = 10^{-15} g
picogram	pg	1 pg = 10^{-12} g
nanogram	ng	1 ng = 10^{-9} g
microgram	µg	1 µg = 10^{-6} g
milligram	mg	1 mg = 10^{-3} g
gram	g	1 g = 1 g
kilogram	kg	1 kg = 10^{3} g
(metric) tonne	t	1 t = 10^{6} g

4 Terminology and Concepts Used in Mass Measurement

Various terms and concepts are encountered when using balances for weighing operations and the most important of these are described below.

Capacity: the maximum mass that can be measured on a particular balance (*e.g.* 220 g).

Readability (or Resolution): the number of decimal places provided by the read-out of the balance (*e.g.* 0.0001 g).

Stabilization (Response) Time: the time required for the reading to stabilize after the application of an object to the balance pan (*e.g.* 3 seconds).

Tare: A facility that enables the balance reading to be set to zero when an object is on the pan.

Zeroing: the act of setting the balance reading to zero whilst the pan is empty.

Precision: the standard deviation observed for consecutive, replicate weighings of the same object, by the same operator on the same balance, with the balance being re-zeroed before each measurement. A typical precision for a 4-figure balance in good condition operated as described would be 0.0001 g or less. For a 2-figure balance a typical precision would be 0.01 g or less. The term repeatability standard deviation is also used for this type of precision.

Linearity: a balance would be said to exhibit perfect linearity if two masses that differed by a particular factor gave read-outs that differed by exactly the same factor. Perfect linearity is not capable of being achieved in practice or of being demonstrated experimentally. However, manufacturers' specifications indicate that errors in a reading due to non-linearity would not typically exceed 0.0002 g over the entire range of a 4-figure balance with a 200 g capacity.

Check Weight: an object (usually metal) with a known mass, used for checking or calibrating the reading of a balance.

Class of Weight: weights are classified by the International Organisation of Legal Metrology (OIML) according to their material of construction and maximum permissible error (tolerance). Table 2 below shows examples of the classification.

Table 2 *Classification of weights according to OIML*

OIML Classification	*Material of construction*	*Nominal mass value/g*	*Permitted tolerance/g*
E1	Integral* stainless steel without markings or adjustment chamber	50	±0.00003
E2	Integral* stainless steel without markings or adjustment chamber	50	±0.0001
F1	Stainless steel with a screw-knob	50	±0.0003
F2	Plated brass	50	±0.001
M1	Brass or cast iron, with a painted finish	50	±0.003

* One-piece weights, with no detachable parts. Other weights may have an adjustment chamber which allows addition/subtraction of suitable material to provide an actual mass that is identical to the nominal mass.

Calibrated Weight: a weight with a mass value and an associated uncertainty that has been formally established by a calibration laboratory, in a manner that ensures the traceability of the mass value to the national standard of mass; in the UK this is held at the National Physical Laboratory (NPL). Calibrated weights are normally available as sets, covering a range of mass values and are accompanied by a certificate documenting all relevant information. Calibrated weights typically have an uncertainty that is 1/3 to 1/5 of the permitted OIML tolerances.

National Standard of Mass: the UK copy (No. 18) of the international prototype of the kilogram, kept at the NPL.

International Prototype of the Kilogram: an artefact kept at the International Bureau of Weights and Measures (BIPM), Paris, made of platinum–iridium and the mass of which defines the SI unit of the kilogram.

SI (Système International): the International System (SI) of measurement units; there are seven 'base' units of measurement and many more 'derived' units. The kilogram is one of the seven base measurement units.

Traceability: a measurement result is said to exhibit traceability if it can be related to a recognised reference of appropriate quality (usually a national or international standard) through an unbroken chain of comparisons, each comparison having a known uncertainty. Ideally, all results should have such traceability.

Calibration: measurements carried out on weights with a known mass value (such as certified weights), with the purpose of comparing the measured value with the known value. Subsequent adjustment of the balance may be required if the difference between the measured and known values lie outside acceptable limits. Such adjustments are usually made by the balance manufacturer or an experienced service agent.

Uncertainty of Measurement: the uncertainty quoted for a measurement is the parameter that describes the range within which the true value is expected to lie, at a stated level of confidence, usually 95%. It is often reported as a ± quantity. It represents the total uncertainty and, as such, includes *all* sources of uncertainty, not only the uncertainty arising from the precision (repeatability standard deviation) of the measurement. An uncertainty with a confidence level of 95% is often described as an expanded uncertainty with a coverage factor of 2.

Buoyancy: the uplifting force on a weighed object due to the fluid in which it is immersed, usually air. The effect of buoyancy is that the mass actually measured based on weight-in-air is slightly different from the mass that would be obtained if the object was weighed in a vacuum. The difference between mass based on weight-in-air and the mass in vacuum is small, typically ≤ 0.1% and buoyancy effects may therefore be ignored in most routine analytical work. The magnitude of the difference actually depends on the difference between the density of the weighed object and the density of the standard weights used to calibrate the balance, since the standard weights are also subject to buoyancy effects. For a weighed object with the same density as that of the calibrated weights there is no buoyancy effect. For a weighed object with a density of 1 g mL^{-1}, the buoyancy effect will lead to a mass based on weight-in-air that is 0.1% less than the mass based on weight-in-vacuum. For a weighed object with a density greater than that of the standard weights, the buoyancy effect leads to a mass based on weight-in-air that is greater than the mass based on weight-in-vacuum. (See also Section 11).

Furthermore, it is a generally accepted convention (OIML Recommendation 33) that all weighings and all calculations and reporting of results use mass values determined on a weight-in-air basis. According to the convention, the weighings pertain to standard air conditions (density 1.2 kg m^{-3}, temperature 20 °C), and a balance calibrated with standard weights with a density of 8000 kg m^{-3}. A consequence of this is that any errors in weighings due to buoyancy effects primarily arise from any departure from the standard air conditions at the time of weighing. Such errors will not usually exceed about 0.01% of the measured mass value. This convention leads to the concept of the conventional mass value.

Conventional Mass Value: for an object at 20 °C, the conventional mass value is the mass of a hypothetical reference weight of density 8000 kg m^{-3} which balances the object, in air of density 1.2 kg m^{-3}. The concept finds particular application in the mass values of calibrated weights. The formally calibrated weight value is a conventional mass value and enables balances to be calibrated in air, using a calibrated weight that may be regarded as having a density of 8000 kg m^{-3}, even if the actual density of the certified weight is different from this value.

5 Types of Balance Used in Analytical Laboratories

A number of different types of balance are likely to be found in a typical analytical laboratory. They are distinguished by specific features, as discussed below.

5.1 Maximum Capacity and Readability

Balances from individual manufacturers differ somewhat in their specifications, but Table 3 gives typical weighing capacities and associated readabilities for a range of balance types.

Table 3 *Types of balance used in analytical work*

Typical maximum capacity	*Typical readability*	*Type of balance*
10 g	0.001 mg	Micro balance 6-figure balance
150 g 500 g	0.01 mg or 0.1 mg 1 mg	Analytical balance 3, 4 or 5-figure balance
1000 g 5000 g 30 000 g	0.01 g 0.1 g 0.1 g or 1 g	General purpose or 'top-pan' balance

Balances capable of weighing to 0.001 mg (6 decimal places) are commonly referred to as micro balances. They are normally used for weighing quantities of < 0.1 g.

Balances weighing to 0.01 mg, 0.1 mg or 1 mg (5, 4 or 3 decimal places) are commonly referred to as analytical balances. They are normally used for weighing quantities of about 0.1 to 100 g.

The higher capacity balances (1 to 30 kg) are used for such applications as preparing large quantities of reagents and weighing bulk samples. For weighing a nominal mass of, say, 2 kg, a balance with a maximum capacity of 5 kg would normally be selected in preference to a balance with a maximum capacity of 30 kg.

5.2 Balance Features

The single-pan balance is nowadays the most commonly used balance for routine analytical work. It has largely replaced the two-pan balance, being simpler to use and maintain. The object to be weighed is placed on the pan and the mass of the object is displayed by means of a digital read-out, which is usually electronic, but may be a mechanical dial in older balances. In the latter case the final digit may be obtained from a moving vernier scale.

Balances with a capacity of about 500 g or less and a readability of ≤1 mg are normally provided with a moveable shield that encloses the pan from draughts when a reading of mass is being taken. With balances of higher capacity and readability ≥ 10 mg, the pan is usually open to the ambient atmosphere of the laboratory.

Many balances have a tare facility which allows, for example, an empty container to be placed on the pan of the balance and the displayed mass then to be re-set to zero. A sample, reagent or other chemical may then be transferred to the 'tared' container and the mass transferred is immediately given by the newly displayed mass value. The tare facility saves time and avoids the need to include the mass of the empty container in the calculation of the mass of the substance weighed.

Many balances also have a printer attached, so that the displayed weight may be printed out directly to give a permanent record, reducing the scope for operator error in data transcription. Such a record should be signed and dated as soon as possible after the weighing operation.

5.3 Operational Principles

In appearance a typical balance consists of a single pan (usually of stainless steel), which, for analytical balances, is enclosed by a moveable transparent shield to protect the pan from draughts. The balance is provided with a digital scale, which gives a read-out of the mass of the object being weighed. Push-button controls enable the balance to be zeroed and tared as required.

The single-pan balance is usually constructed to one of two basic designs, which although outwardly similar in appearance, differ in their operational principles.

5.3.1 Mechanical Beam Balance

A fixed weight is permanently attached to one end of an asymetrical beam. The pan is attached to the other end of the beam, together with a series of weights which can be mechanically lifted from the pan assembly. When an object to be weighed is placed on the pan, a group of weights of similar mass are removed from the assembly. As a consequence the balance operates under a constant-load, regardless of the mass of an object being weighed. The weights in the balance are not accessed directly by the operator, but *via* control knobs on the outside of the balance. In a micro or analytical balance the pan is suspended below the beam, whilst in a top-pan balance the pan is supported above the beam. The read-out of the mass is obtained by a mechanical or electronic digital scale. Such balances are sometimes referred to as 'single-pan, two-knife edge' balances and 'constant-load' balances. The knife edges are those acting as a fulcrum for the balance beam and the attachment point for the balance pan.

5.3.2 Electromagnetic Force Compensation Balance

In this design a coil is rigidly attached to the balance pan and the pan assembly is placed in the field of a magnet. When an object to be weighed is placed on the pan, the pan is lowered, causing an increase in the current in the coil. A magnetic counter-force is thereby generated which returns the pan to its original position. The increase in current in the coil is measured as a voltage on a digital voltmeter and the mass of the object is directly proportional to the measured voltage. A digital read-out of the mass of the object is therefore readily obtained. Such balances are sometimes referred to as 'electronic' balances, to distinguish them from those described previously and operating on mechanical principles. Electronic balances have the advantage of no moving internal weights and they are less susceptible to disturbance by vibration. They are available as micro, analytical and general purpose 'top-pan' balances.

6 Location of Balance

Attention should be paid to manufacturers' recommendations regarding the environmental requirements for the location of the balance in the laboratory. Important factors to be considered include the following.

Draughts: the balance location should be chosen to minimise draughts from doors, windows, passers-by, other equipment, *etc*. Air conditioning may also cause unwanted draughts.

Vibration: all balances are subject to vibration to a greater or lesser extent, although electronic balances are usually less affected. For measurements to 0.01 g or less, it is important to locate the balance on a vibration-free surface. A solid bench top of stone or slate at least 40 mm thick, free-standing (*i.e.* away from any walls) and isolated from any other work apart from weighing activities is preferred. Also, the bench top should be preferably mounted on brick pillars on a concrete floor. It should not be mounted on wood or a suspended floor. Anti-vibration mounts should not be used, since the balance will tilt slightly in use, as the weights are re-positioned during a weighing operation. In extreme cases of vibration physical damage may be caused and some read-outs may be blurred.

Level Surface: the surface on which the balance is mounted should be level and the balance feet should be adjusted, using the spirit-level device to show when the balance is level.

Cleanliness: the balance should be located in a generally clean area, free from dusts, water and chemical splashes, corrosive substances, aerosols and organic vapours.

Temperature: ideally the ambient temperature should be stable to within ±3 °C. Temperature fluctuations can cause gradients in the balance mechanism and also draughts by air convection.

Humidity: humidity is relatively unimportant provided condensation does not occur.

Magnetic Fields: these should be avoided as they may cause permanent changes in the response of the balance.

Electrical Interference: electronic balances are subject to electrical interference. They should be left on at all times and if mains-borne interference is a serious problem, an appropriate filter plug should be fitted.

7 Balance Calibration

If a balance is to provide accurate mass values it must be properly calibrated so that the readings are traceable to the national standard of mass (the kilogram) held in the UK by NPL (National Physical Laboratory). *Via* this link, the readings are also traceable to the international standard kilogram, held at the International Bureau of Weights and Measures, Paris. In this way, readings obtained on a properly calibrated balance in a laboratory in the UK will be comparable with readings obtained on properly calibrated balances in any other laboratory, either in the UK or overseas.

Although it is possible for balance calibration to be carried out by the user laboratory (following manufacturers' instructions), it is preferable, and often more convenient, for this operation to be carried out by the manufacturer or an experienced service organisation. The body carrying out the calibration should preferably be accredited by the United Kingdom Accreditation Service (UKAS) for such work. The calibration procedure involves examination and testing of various aspects of balance performance, such as:

- repeatability
- linearity
- zero and tare mechanisms
- eccentric or off-centre loading

A series of calibrated weights are applied to the balance pan and the response is observed. UKAS stipulates that 10 different calibrated weights should be used, to provide adequate coverage of the working range. Adjustments are then made, as appropriate, to ensure that the balance readings are within specification.

The calibrated weights have conventional mass values, that is the calibrated mass value is reported on a weight-in-air basis, where the mass is that of a hypothetical weight of density 8000 kg m^{-3}, which balances the calibrated weight, in air of density 1.2 kg m^{-3} at 20 °C. The practical effect of this is that the balance is calibrated with a calibrated weight having a density of 8000 kg m^{-3} (even though the actual density may be different from this) and the balance then gives masses of any weighed object on a 'weight-in-air' basis. This is consistent with normal UKAS practice for analytical laboratories to report mass values on a weight-in-air basis. It also complies with OIML International Recommendation No. 33 on the Conventional Value of the Result of Weighing in Air. Only in exceptional cases, involving the highest accuracy should other conventions be used, such as reporting results on a weight-in-vacuum basis.

A record should be kept of the balance calibration, usually in the form of a certificate of calibration issued by the calibrating body. Re-calibration should be carried out at regular intervals, typically annually.

Some balances have a calibration button that re-calibrates the read-out using internal calibration weights; occasionally this re-calibration is done automatically. However, this facility should not be regarded as a substitute for an annual service and full re-calibration. The calibration button should only be used (if at all) before a series of weighings are carried out. It should not be used part way through a series of weighings.

8 Checking the Accuracy of a Balance

Although all balances should be subject to a full calibration procedure on an annual basis, preferably carried out by an external organisation, intermediate checks by the laboratory should be carried out to confirm that the balance readings are of acceptable accuracy. Such checks do not need to be as extensive as the full calibration procedure. Depending on the laboratory's experience of a particular balance and on the frequency of its use, the in-house checks are carried out at regular specified intervals (*e.g.* daily, weekly, monthly), or immediately before the balance is actually used.

The checks are most effectively done by placing a weight of known value (preferably a calibrated weight) on the pan and comparing the measured mass value to the true value. Provided the difference does not exceed a given critical value, the balance is deemed to be functioning satisfactorily. The particular critical value may be established in various ways, for example:

- by a fitness-for-purpose approach – "What is the accuracy required in a mass measurement for the particular application?"
- by a performance-based approach – "What accuracy should be attainable in day-to-day use of the balance?"

Since a particular balance is likely to be used for many different applications and as modern scientific balances are very reliable even when used by relatively inexperienced operators, the second (performance-based) approach is generally more convenient to use.

As an example, experience at LGC has shown that using a 4-figure balance with a capacity of about 200 g, a measured mass value and the corresponding certified value can be expected to agree to within ±0.001 g, over the entire mass range. In actual fact, the agreement is usually considerably better than this, typically to within ±0.0003 g or less.

Based on such experience, a particular standard operating procedure (SOP) for checking the accuracy of balances requires that 4-figure balances be checked on the day of use, before use, with certified weights of 5 and 50 g. In both cases the SOP stipulates that the measured and certified values should agree to within ±0.001 g.

Such a criterion provides an accuracy that is more than fit-for-purpose, since an error of 0.001 g in 5 g is equivalent to a relative error of only 0.02%. Compared to other likely errors in a chemical analysis, such an error is trivial and may be ignored.

In Table 4 the differences between the measured and the certified mass values that are stipulated as acceptable in the SOP are compared for a range of balance types.

If the acceptable difference is exceeded, the measurements should be repeated, making sure that the balance pan is clean, the door is closed and that other operational requirements (see Section 6) are being met. If the measured mass values are still outside the acceptable limits, the balance must be withdrawn from use and re-calibrated by an experienced service engineer before it is used again.

Table 4 *Acceptable differences between measured and certified mass values*

Type of balance	*Readability/g*	*Nominal mass/g*	*Acceptable difference between measured and certified mass/g*
Micro	0.000001	0.001	±0.00002
		0.1	±0.00002
Analytical	0.0001	5	±0.001
		50	±0.001
Top-pan	0.01	500	±0.1
	0.1	5000	±5

9 Use of the Balance

The manufacturers' instruction manual should be consulted before use of a particular balance. The following points give general guidance on important aspects of the weighing process.

9.1 Choice of Balance

The choice of balance depends on the quantity to be weighed and the nominal accuracy (number of decimal places) required in the weighing. For most routine purposes criteria for balance selection are suggested in Table 5.

Table 5 *Balance selection for a particular weighing operation*

Nominal quantity to be weighed/g	*Recommended balance*	*Nominal accuracy obtained*	
0.01	Micro (6-figure)	0.000001 g	(0.01%)
0.1	Analytical (5-figure)	0.00001 g	(0.01%)
1	Analytical (4-figure)	0.0001 g	(0.01%)
10	Analytical (4-figure)	0.0001 g	(0.001%)
100	Top-pan (2-figure)	0.01 g	(0.01%)
1000	Top-pan (1-figure)	0.1 g	(0.01%)
10 000	Top-pan (0-figure)	1 g	(0.01%)

Such a balance selection will provide mass values with a nominal relative accuracy of 0.01% or better, which is more than adequate for all routine analytical work. In fact, it would nearly always be acceptable to weigh to a nominal accuracy of 0.1%, thus, for example, a weighing of 0.1g could be satisfactorily done on a 4-figure analytical balance. A balance should not normally be used at less than 5% of its maximum capacity. It is not recommended that a balance is used at more than about 95% of its capacity, as the nominal mass to be weighed may exceed the capacity.

Once a particular balance has been identified as suitable for a particular task, checks should be made to ensure that it has been calibrated for the range being used. This is most readily done by locating the certificate of calibration and checking that