3

Common apparatus and basic techniques

3.1 Introduction

The development of advanced instrumentation and modern analytical procedures has tended to lead many people into believing that basic scientific techniques and simple apparatus are of less importance with respect to obtaining accurate, reproducible and reliable results. The importance of being able to handle simple quantitative equipment and of following well-established and set routines for cleanliness and orderly working cannot be emphasised strongly enough for the analyst seeking to maintain high standards of working. The following points should become second nature to the practising analyst:

1. Benches should always be kept clean and tidy and all spillages of both solids and liquids cleared away immediately.
2. All glassware must be scrupulously clean (Section 3.6) and must be rinsed with distilled or deionised water before use. The outsides of vessels may be dried with a lint-free glass cloth which is reserved exclusively for this purpose, and which is frequently laundered, but the cloth should not be used on the insides of the vessels.
3. The working surface of the bench should be free from reagent bottles and apparatus for which there is no immediate use. Reagent bottles must be replaced on the reagent shelves immediately after use. Apparatus for which no further immediate use is envisaged should be returned to the locker.
4. If a solution, precipitate, filtrate, etc. is set aside for subsequent treatment, the container must be labelled with felt tip pen or with adhesive label so that the contents can be readily identified, and the vessel must be suitably covered to prevent contamination of the contents by dust.
5. It should be regarded as normal practice that all determinations are performed in duplicate.
6. A stiff covered notebook of A4 size must be provided for recording experimental observations as they are made.

All experimental observations must be recorded immediately after they are made in the laboratory observation notebook. Each determination should be given a clear title and date and should include a brief outline of the procedure followed along with a full account of any special features associated with the determination. It will be convenient to divide the

experimental page in the observation notebook into three columns, a wider one on the left-hand side for indicating the observations and two smaller columns on the right-hand side to record the data for duplicate determinations.

The record must conclude with the relevant calculations, so the equations for the principal chemical reactions should be shown together with a clear exposition of the procedure for calculating the result. Finally, appropriate comments should be made about the degree of accuracy and the level of precision.

Any chart or printed data of analytical results in the form of spectra or chromatograms, etc. should be permanently attached to the laboratory observation notebook. These results should be checked for their consistency and should not be accepted just because they have come from a computer.

Safety. Safety in the laboratory is essential at all times. You are responsible for the safety of any other person as well as your own. Many chemicals encountered in analysis are poisonous and must be carefully handled. The dangerous properties of concentrated acids and widely recognised poisons, such as potassium cyanide, are well known, but the dangers associated with halogenated solvents, benzene, mercury and many other chemicals are frequently overlooked. That is why it is essential to carry out a safety assessment of the chemicals and processes involved in any analysis before work is started for the first time.

Many operations involving chemical reactions are potentially dangerous, and recommended procedures must be carefully followed and obeyed. All laboratory workers should develop safety consciousness and familiarise themselves with local safety requirements, which may include the compulsory wearing of lab coats and safety spectacles, and the position of first-aid equipment.[1] Safety standards in laboratories have been greatly tightened in very many countries and established safety standards which are monitored by government officials frequently have to be observed.

Balances

3.2 The analytical balance

Most quantitative chemical processes depend at some stage upon the accurate measurement of mass for which an analytical balance is employed. The operation is called weighing, and invariably reference is made to the weight of the object or material which is weighed.

The weight of an object is the force of attraction due to gravity that is exerted upon the object:

$$w = mg$$

where w is the weight of the object, m its mass and g is the acceleration due to gravity. Since the attraction due to gravity varies with altitude and with latitude, the weight of the object is variable, whereas its mass is constant. However, it has become the custom to

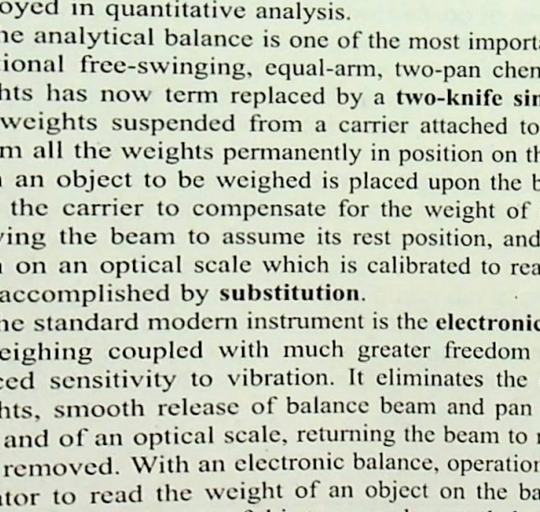

Figure 3.1 Two-knife single-pan balance

employ the term 'weight' synonymously with mass, and it is in this sense that 'weight' i employed in quantitative analysis.

The analytical balance is one of the most important tools of the analytical chemist. Th traditional free-swinging, equal-arm, two-pan chemical balance together with its box o weights has now term replaced by a **two-knife single-pan balance** with a dial-operate ring weights suspended from a carrier attached to the pan support (Figure 3.1). In thi system all the weights permanently in position on the carrier when the beam is at rest, an when an object to be weighed is placed upon the balance pan, weights must be remove from the carrier to compensate for the weight of the object. Weighing is completed b allowing the beam to assume its rest position, and then reading the displacement of th beam on an optical scale which is calibrated to read weights below 100 mg. Weighing i thus accomplished by **substitution**.

The standard modern instrument is the **electronic balance**, which provides convenienc in weighing coupled with much greater freedom from mechanical failure, and greatl reduced sensitivity to vibration. It eliminates the operations of selecting and removin weights, smooth release of balance beam and pan support, noting the reading of weigh dials and of an optical scale, returning the beam to rest, and replacing weights which ha been removed. With an electronic balance, operation of a single on–off control permits th operator to read the weight of an object on the balance pan immediately from a digita display; most balances of this type can be coupled to a printer which gives a printed recor of the weight. The majority of balances incorporate a **tare** facility which permits th weight of a container to be cancelled out, so that when material is added to the containe the weight recorded is simply the weight of material used. Many balances of this typ incorporate a self-testing system which indicates the balance is functioning correctly eac time it is switched on; and they also include a built-in weight calibration system. Operatio of the calibration control leads to display of the weight of the standard incorporated withi the balance, and thus indicates whether any correction is necessary. A more satisfactor calibration procedure is to check the balance readings against a series of calibrated analy tical weights.

Electronic balances operate by applying an electromagnetic restoring force to the sup port for the balance pan; when an object is added to the balance pan, the resultant displace ment of the support is cancelled out. The magnitude of the restoring force is governed b the value of the current flowing in the coils of the electromagnetic compensation system

and this is proportional to the weight placed on the balance pan; a microprocessor converts the value of the current into the digital display in grams.

The balance must be protected from draughts and from dust, and the balance pan is situated within an enclosure provided with glass doors which can be opened to provide access to the pan. The rest of the balance, including the electrical components, is contained in a closed compartment attached to the rear of the pan compartment.

A wide range of electronic balances called top pan balances are available for rough weighing. With these top-pan balances it is not necessary to shield the balance pan from gentle draughts, and weighings can be accomplished very rapidly and with the usual facility of the results being recorded with a printer.

3.3 Care and use of analytical balances

No matter what type of analytical balance is employed, due attention must be paid to the manner in which it is used. The following remarks apply particularly to electronic balances:

1. Never exceed the stated maximum load of the balance.
2. Keep the balance clean. Remove dust from the pan and from the floor of the pan compartment with a camel-hair brush.
3. Objects to be weighed should never be handled with the fingers; always use tongs or a loop of clean paper.
4. Objects to be weighed should be allowed to attain the temperature of the balance before weighing, and if the object has been heated, sufficient time must be allowed for cooling.
5. No chemicals or objects which might injure the balance pan should ever be placed directly on it. Substances must be weighed in suitable containers, such as small beakers, weighing bottles or crucibles, or on watch glasses. Liquids and volatile or hygroscopic solids must be weighed in tightly closed vessels, such as stoppered weighing bottles.
6. Addition of chemicals to the receptacle must be done outside the balance case. It is good practice to weigh the chosen receptacle on the analytical balance, to transfer it to a rough balance, to add approximately the required amount of the necessary chemical, and then to return the receptacle to the analytical balance for reweighing, thus giving the exact weight of substance taken.
7. Nothing must be left on the pan when the weighing has been completed. If any substance is spilled accidentally upon the pan or upon the floor of the balance compartment, it must be removed at once.
8. Avoid exposing the balance to corrosive atmospheres.

The actual weighing process will include the following steps.

1. Brush the balance pan lightly with a camel-hair brush to remove any dust.
2. The object to be weighed must be at or close to room temperature. With the balance at rest, place the object on the pan and close the pan compartment case.
3. Set the on–off control of the balance to the 'on' position, observe the value shown on the digital display and record it in the notebook. If the balance is linked to a printer, confrm that the printed result agrees with the digital display. Return the control to the 'off' position.
4. When all weighings have been completed, remove the object which has been weighed, clear up any accidental spillages, and close the pan compartment.

These remarks apply particularly to analytical balances of the macrobalance range; mic balances and ultramicrobalances must be handled with special care, particularly with re to the temperature of objects to be weighed.

3.4 Errors in weighing

Be aware of the potential sources of error that can occur in weighing, other than th arising from a defective balance. There are three sources of error that need to be conside in detail:

1. Changes relating to the weighing vessel or the substance occurring between succes weighings.
2. Buoyancy of the air and its effect upon the object, the container and any weights.
3. Human errors in recording results.

Changes in the weighing vessel

Changes in the weight of the weighing vessel may arise from absorption or loss of mo ture, electrification of the surface caused by rubbing, a difference in temperature betwe the weighing vessel and the balance case. These errors may be largely eliminated wiping the vessel gently with a linen cloth, and allowing it to stand at least 30 min proximity to the balance before weighing. Hygroscopic, effloresent and volatile s stances must be weighed in completely closed vessels. Substances which have been hea in an air oven or ignited in a crucible are generally allowed to cool in a desiccator c taining a suitable drying agent for alone 30–35 min.

Buoyancy effects

Buoyancy occurs when an object is immersed in a fluid and its true weight is diminish by the weight of the fluid it displaces. If the object and the weights have the same dens hence the same volume, no error will be introduced on this account. If the substance a lower density than the weights, as is usual in analysis, the substance will displac greater volume of air than the weights, and it will therefore weigh less in air than i vacuum. Conversely, if a denser material (e.g. one of the precious metals) is weighed, weight in a vacuum will be less than the apparent weight in air.

The weight of an object in vacuo is equal to the weight in air **plus** the weight of displaced by the object **minus** the weight of air displaced by the weights. It can easily shown that

$$W_v = W_a + d_a\left(\frac{W_v}{d_b} - \frac{W_a}{d_w}\right)$$

where
W_v = weight in vacuo
W_a = apparent weight in air
d_a = density of air
d_w = density of the weights
d_b = density of the body

The density of the air will depend on the humidity, the temperature and the pressure. For an average relative humidity (50%) and average conditions of temperature and pressure in a laboratory, the density of the air will rarely fall outside the limits 0.0011 and 0.0013 g mL^{-1}. It is therefore permissible for analytical purposes to take the weight of 1 mL of air as 0.0012 g.

Since the difference between W_v and W_a does not usually exceed 1–2 parts per thousand, we may write

$$W_v = W_a + d_a\left(\frac{W_a}{d_b} - \frac{W_a}{d_w}\right)$$

$$= W_a + W_a\left\{0.0012\left(\frac{1}{d_b} - \frac{1}{8.0}\right)\right\} = W_a + kW_a/1000$$

where

$$k = 1.20\left(\frac{1}{d_b} - \frac{1}{8.0}\right)$$

If a substance of density d_b weighs W_a grams in air, then $W_a k$ milligrams should be added to the weight in air to obtain the weight in vacuo. The correction is positive if the substance has a density lower than 8.0, and negative if the density of the substance is greater than 8.0.

Human errors

Many weighing errors are due to human mistakes made in checking the weights on the balance pan, or in reading the digital display on electronic balances. The correct reading of weights is best carried out by checking the weights as they are added to and removed from the balance. Any digital display should be read at least twice, paying very careful attention to the position of the decimal point.

Graduated glassware

3.5 Units of volume

For scientific purposes the convenient unit to employ for measuring reasonably large volumes of liquids is the cubic decimetre (dm^3) or litre (L) and for smaller volumes the cubic centimetre (cm^3) or millilitre (mL).

3.6 Graduated apparatus

The most commonly used pieces of apparatus in titrimetric (volumetric) analysis are graduated flasks, burettes and pipettes. Graduated cylinders and weight pipettes are less widely employed. Each of them will be described in turn.

Graduated apparatus for quantitative analysis is generally made to specification limits, particularly with regard to the accuracy of calibration, in two grades class A and class B. The tolerance limits are closer for class A apparatus; this apparatus is intended for work of the highest accuracy. Class B apparatus is employed in routine work.