

weighings in air with stainless steel weights, and these values can be used to calculate relevant volume directly from the observed weight of water. Plot a graph to find the volume of 1 g of water at the calibration temperature. Fuller tables are given in BS 6696 (1986).

In all calibration operations the apparatus to be calibrated must be carefully cleaned and allowed to stand adjacent to the balance which is to be employed, together with a supply of distilled or deionised water, so they assume the temperature of the room. Flasks also need to be dried, and this can be accomplished by rinsing twice with a little acetone then blowing a current of air through the flask to remove the acetone.

#### Graduated flask

After allowing it to stand in the balance room for an hour, the clean dry flask is stoppered and weighed. A small filter funnel, the stem of which has been drawn out so it reaches below the graduation mark of the flask, is then inserted into the neck and deionised (distilled) water, which has also been standing in the balance room for an hour, is added slowly until the mark is reached. The funnel is then carefully removed, taking care not to wet the neck of the flask above the mark, and then, using a dropping tube, water is added dropwise until the meniscus stands on the graduation mark. The stopper is replaced, the flask reweighed, and the temperature of the water noted. The true volume of the water filling the flask to the graduation mark can be calculated with the aid of Table 3.1.

#### Pipette

The pipette is filled with the distilled water which has been standing in the balance room for at least an hour, to a short distance above the mark. Water is run out until the meniscus is exactly on the mark, and the outflow is then stopped. The drop adhering to the jet is removed by bringing the surface of some water contained in a beaker in contact with the jet, and then removing it without jerking. The pipette is then allowed to discharge into a clean, weighed stoppered flask (or a large weighing bottle) and held so the jet of the pipette is in contact with the side of the vessel (it will be necessary to incline slightly either the pipette or the vessel). The pipette is allowed to drain for 15 s after the outflow has ceased, the jet still being in contact with the side of the vessel. At the end of the draining time the receiving vessel is removed from contact with the tip of the pipette, thus removing a drop adhering to the outside of the pipette and ensuring the drop remaining in the end is always of the same size. To determine the instant at which the outflow ceases, the motion of the water surface down the delivery tube of the pipette is observed, and the delivery time is considered to be complete when the meniscus comes to rest slightly above the end of the delivery tube. The draining time of 15 s is counted from this moment. The receiving vessel is weighed, and the temperature of the water noted. The capacity of the pipette is then calculated with the aid of Table 3.1. At least two determinations should be made.

#### Burette

If it is necessary to calibrate a burette, it is essential to establish that it is satisfactory with regard to leakage and delivery time, before undertaking the actual calibration process. As a test for leakage, the plug is removed from the barrel of the stopcock and both parts of the stopcock are carefully cleaned of all grease; after wetting well with deionised water, the stopcock is reassembled. The burette is placed in the holder, filled with deionised water, adjusted to the zero mark, and any drop of water adhering to the jet removed with a pipette.



## Common Apparatus and Basic Techniques

of filter paper. The burette is then allowed to stand for 20 min, and if the meniscus has not fallen by more than one scale division, the burette may be regarded as satisfactory as far as leakage is concerned.

To test the delivery time, again separate the components of the stopcock, dry, grease and reassemble, then fill the burette to the zero mark with deionised water, and place in the holder. Adjust the position of the burette so the jet comes inside the neck of a conical flask standing on the base of the burette stand, but does not touch the side of the flask. Open the stopcock fully, and note the time taken for the meniscus to reach the lowest graduation mark of the burette: this should agree closely with the time marked on the burette, and it must fall within the limits laid down by BS 846 (1985).

If the burette passes these two tests, the calibration may begin. Fill the burette with the deionised water which has been allowed to stand in the balance room to acquire room temperature: ideally, this should be as near to 20 °C as possible. Weigh a clean, dry stoppered flask of about 100 mL capacity, then, after adjusting the burette to the zero mark and removing any drop adhering to the jet, place the flask in position under the jet, open the stopcock fully and allow water to flow into the flask. As the meniscus approaches the desired calibration point on the burette, reduce the rate of flow until eventually it is discharging dropwise, and adjust the meniscus exactly to the required mark. Do not wait for drainage, but remove any drop adhering to the jet by touching the neck of the flask against the jet, then restopper and reweigh the flask. Repeat this procedure for each graduation to be tested; for a 50 mL burette, this will usually be every 5 mL. Note the temperature of the water, and then, using Table 3.1, the volume delivered at each point is calculated from the weight of water collected. The results are most conveniently used by plotting a calibration curve for the burette.

## Water for laboratory use

### 3.14 Purified water

From the earliest days of quantitative chemical measurements it has been recognised that some form of purification is required for water used in analytical operations, and with increasingly lower limits of detection being attained in instrumental methods of analysis, correspondingly higher standards of purity are imposed upon the water used for preparing solutions. Standards have now been laid down for water to be used in laboratories;<sup>[4]</sup> they prescribe limits for non-volatile residue, for residue remaining after ignition, for pH and for conductance. ISO 3696-1987 recognises three different grades of water, as summarised in Table 3.2:

**Grade 3** is suitable for ordinary analytical purposes and may be prepared by single distillation of tap water, by deionisation, or by reverse osmosis (see below).

**Grade 2** is suitable for more sensitive analytical procedures, such as atomic absorption spectroscopy and the determination of substances present in trace quantities. Water of this quality can be prepared by redistillation of grade 3 distilled water, by distillation of deionised water, or by distillation of reverse osmosis water.

**Grade 1** is suitable for the most stringent requirements, including high-performance liquid chromatography and the determination of substances present in ultratrace amounts. It is obtained by subjecting grade 2 water to reverse osmosis or deionisation, followed by filtration through a membrane filter of pore size 0.2 µm to remove particulate matter. Alternatively, grade 2 water may be redistilled in an apparatus constructed from fused silica.



Table 3.2 Standards for water to be used in analytical operations

Parameter	Grade of water		
	1	2	3
pH at 25 °C	<sup>a</sup>	<sup>a</sup>	5.0-7.5
Electrical conductance at 25 °C (mS m <sup>-1</sup> )	0.01	0.1	0.5
Oxidisable matter = amount of oxygen (mg L <sup>-1</sup> )	<sup>b</sup>	0.08	0.4
Absorbance at 254 nm, 1 cm cell	0.001	0.01	<sup>c</sup>
Residue after evaporation (mg kg <sup>-1</sup> )	<sup>c</sup>	1	2
SiO <sub>2</sub> content (mg L <sup>-1</sup> )	0.01	0.02	<sup>c</sup>

<sup>a</sup> pH measurements in highly purified water are difficult; results are of doubtful significance.

<sup>b</sup> Not applicable.

<sup>c</sup> Not specified.

For many years the sole method of water purification available was by distillation, and distilled water of varying quality was universally employed for laboratory purposes. A modern water-still is usually made of glass and is heated electrically. The current may be interrupted if there is a failure in the cooling water supply or the boiler-feed supply; the current is also cut off when the receiver is full. The equipment should be regularly checked and cleaned to ensure a constant quality of water.

#### Deionised water

Deionised water, often of greater purity than laboratory distilled water, is produced by allowing tap water to pass through a mixture of ion exchange resins. A strong acid resin is used to replace any cations from the water by hydrogen ions, and a strong base resin (OH<sup>-</sup> form) is used to remove any anions. In commercial deionisers the quality of the water produced is monitored by a conductance meter. The resins are usually supplied in an interchangeable cartridge, so that maintenance is reduced to a minimum. A mixed-bed ion exchange column fed with distilled water is capable of producing water with the very low conductance of about  $2.0 \times 10^{-6} \Omega^{-1} \text{cm}^{-1}$  ( $2.0 \mu\text{S cm}^{-1}$ ), but in spite of this very low conductance, the water may contain traces of organic impurities which can be detected by means of a spectrofluorimeter. For most purposes, however, the traces of organic material present in deionised water can be ignored, and it may be used in most situations where distilled water is acceptable.

An alternative method of purifying water is by **reverse osmosis**. Under normal conditions, if an aqueous solution is separated by a semipermeable membrane from pure water, osmosis will lead to water entering the solution to dilute it. But if sufficient pressure is applied to the solution, i.e. a pressure in excess of its osmotic pressure, then water will flow through the membrane from the solution; the process of reverse osmosis is taking place. This principle has been adapted as a method of purifying tap water. The tap water at a pressure of 3-5 atm (300-500 kPa) is passed through a tube containing the semipermeable membrane. The permeate usually still contains traces of inorganic material and is therefore not suitable for operations requiring very pure water, but it will serve for many laboratory purposes, and is very suitable for further purification by ion exchange treatment.



## Common Apparatus and Basic Techniques

The water produced by reverse osmosis is passed first through a bed of activated charcoal which removes organic contaminants, and is then passed through a mixed-bed ion exchange column and the resultant effluent is finally filtered through a submicron filter membrane to remove any last traces of colloidal organic particles.

The **high-purity water** thus produced typically has a conductance of about  $0.5 \times 10^{-6} \Omega^{-1} \text{cm}^{-1}$  ( $0.5 \mu\text{S cm}^{-1}$ ) and is suitable for use under the most stringent requirements. It will meet the purity required for trace element determinations and for operations such as ion chromatography. But it can readily become contaminated from the vessels in which it is stored, and also by exposure to the atmosphere. For organic determinations the water should be stored in containers made of resistant glass (e.g. Pyrex), or ideally of fused silica, whereas for inorganic determinations the water is best stored in containers made from polythene or from polypropylene.

### 3.15 Wash bottles

Traditional glass wash bottles are 500–750 mL flat-bottomed flasks fitted with tubes and jets (Figure 3.3a); they are very rarely used except where nothing else is available. However, a very wide range of **polythene** wash bottles purpose-made for the job are readily available and inexpensive. They usually have ~250 mL capacity, a hard plastic cap and a plastic jet; the jet is inserted through the cap (Figure 3.3b). The bottles are easy to hold in the hand and provide a controllable jet of liquid when squeezed with a gentle pressure. They are virtually unbreakable, but should be kept away from Bunsen flames and hot surfaces; they can be used with a wide variety of solutions and solvents, but seldom with hot liquids.

Polythene wash bottles used with liquids other than water should normally be designated for use only with a specific solvent or solution. This is because the components of some wash solutions may be adsorbed into the polythene and then contaminate any other liquid if the wash bottle is used for another purpose. Repeated fillings and rinsings may be required to clean a bottle before it can be used for a different liquid.

**Note** Wash bottles should always be clearly labelled and colour-coded if they are used for different liquids, but should not be used for concentrated acids or highly corrosive substances.

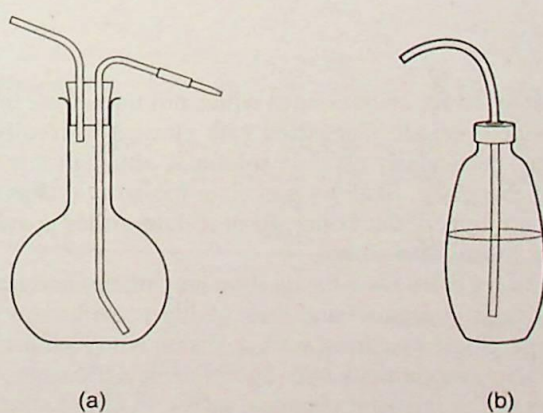


Figure 3.3 Wash bottles: (a) glass, (b) polythene



## General apparatus

### 3.16 Glassware, ceramics, plastic ware

#### Glassware

To avoid introducing impurities during analysis, use apparatus made from resistance glass. A borosilicate glass is preferred for most purposes. Resistance glass is very slightly affected by all solutions, but attack by acid solutions is generally less than by pure water or alkaline solutions; hence alkaline solutions should be acidified, whenever possible, if they must be kept in glass for any length of time. Attention should also be given to watch, clock, and cover glasses; they should also be made from resistance glass. As a rule, glassware should not be heated with a naked flame; a wire gauze should be interposed between the flame and the glass vessel. For special purposes, Corning Vycor glass (96% silica) may be used. It has great resistance to heat and equally great resistance to thermal shock, and is unusually stable to acids (except hydrofluoric acid), water and various solutions.

**Beakers** The most satisfactory beakers for general use are beakers with a spout. The advantages of this form are convenience of pouring; the spout forms a convenient place at which a stirring rod may protrude from a covered beaker, and it forms an outlet for steam or escaping gas when the beaker is covered with an ordinary clockglass. The size of a beaker must be selected with due regard to the volume of the liquid it is to contain. The most useful sizes are from 250 to 600 mL.

**Conical flasks** Conical flasks of 150–500 mL capacity are used extensively in titrations, for mixing liquids and for heating solutions.

**Funnels** Funnels should enclose an angle of  $60^\circ$ . The most useful sizes for quantitative analysis have diameters of 5.5, 7 and 9 cm. The stem should have an internal diameter of about 4 mm and should not be more than 15 cm long. For filling burettes and transferring solids to graduated flasks, a short-stem, wide-necked funnel is useful.

#### Porcelain apparatus

Porcelain is generally employed for operations in which hot liquids are to remain in contact with the vessel for prolonged periods. Compared with glass, it is usually considered to be more resistant to solutions, particularly alkaline solutions, although this will depend primarily upon the quality of the glaze. Shallow porcelain basins with lips are employed for evaporations. Casseroles are lipped, flat-bottomed porcelain dishes provided with handles; they are more convenient to use than dishes.

Porcelain crucibles are very often used for igniting precipitates and heating small quantities of solids because of their cheapness and their ability to withstand high temperatures without appreciable change. Some reactions, such as fusion with sodium carbonate or other alkaline substances, and also evaporations with hydrofluoric acid, cannot be carried out in porcelain crucibles owing to the resultant chemical attack. A slight attack of the porcelain also takes place with pyrosulphate fusions.



## Common Apparatus and Basic Techniques

### Fused-silica apparatus

Two varieties of silica apparatus are available commercially, translucent and transparent. Translucent apparatus is much cheaper and can usually be employed instead of the transparent variety. Silica ware has several advantages: it has a great resistance to heat shock because of its very small coefficient of expansion; it is not attacked by acids at a high temperature, except by hydrofluoric acid and phosphoric acid; and it is more resistant than porcelain to pyrosulphate fusions. And the chief disadvantages: it is attacked by alkaline solutions, particularly fused alkalis and carbonates; it is more brittle than ordinary glass; and it requires a much longer time for heating and cooling than platinum apparatus. Corning Vycor apparatus (96% silica glass) possesses most of the merits of fused silica and is transparent.

### Plastic apparatus

Plastic materials are widely used for a variety of items of common laboratory equipment such as aspirators, beakers, bottles, Buchner funnels and flasks, centrifuge tubes, conical flasks, filter crucibles, filter funnels, measuring cylinders, scoops, spatulas, stoppers, tubing and weighing bottles; plastic items are often cheaper than their glass counterparts, and are frequently less fragile. Although inert towards many chemicals, there are some limitations on the use of plastic apparatus, including the generally rather low maximum temperature to which it may be exposed. Salient properties of the commonly used plastic materials are summarised in Table 3.3.

Teflon is extremely inert; it is so lacking in reactivity that it is used as the liner in pressure digestion vessels in which substances are decomposed by heating with hydrofluoric acid, or with concentrated nitric acid (Section 3.28).

**Table 3.3** *Plastics used for laboratory apparatus*

Material	Appearance <sup>a</sup>	Highest temperature (°C)	Acids <sup>b</sup>		Alkalis <sup>b</sup>		Attacking organic solvents <sup>c</sup>
			Weak	Strong	Weak	Strong	
Polythene (LD) <sup>d</sup>	TL	80–90	R	R*	V	R	1, 2
Polythene (HD) <sup>d</sup>	TL–O	100–110	V	R*	V	V	2
Polypropylene	T–TL	120–130	V	R*	V	V	2
TPX (polymethylpentene)	T	170–180	V	R*	V	V	1, 2
Polystyrene	T	85	V	R*	V	V	most
PTFE (Teflon)	O	250–300	V	V	V	V	V
Polycarbonate	T	120–130	R	A	F	A	most
PVC (polyvinyl chloride)	T–O	50–70	R	R*	R	R	2, 3, 4
Nylon	TL–O	120	R	A	R	F	V

<sup>a</sup> O = opaque; T = transparent; TL = translucent.

<sup>b</sup> A = attacked; F = fairly resistant; R = resistant; R\* = generally resistant but attacked by oxidising mixtures; V = very resistant.

<sup>c</sup> 1 = hydrocarbons; 2 = chlorohydrocarbons; 3 = ketones; 4 = cyclic ethers; V = very resistant.

<sup>d</sup> LD = low density; HD = high density.