Most graduated glassware is manufactured in high-quality heat-resistant glass and last for many years if carefully treated. Plastic graduated equipment is also available, this is not usually suitable for accurate work as surfaces deteriorate rapidly and are o difficult to clean adequately.

Cleaning of glass apparatus

All such glassware must be perfectly cleaned with detergents and free from grease, oth wise the results will be unreliable. One test for cleanliness of glass apparatus is that being filled with distilled water and the water withdrawn, only an unbroken film of wa remains. If the water collects in drops, the vessel is dirty and must be cleaned. Vario methods are available for cleaning glassware. Many commercially available detergents suitable for this purpose, and some manufacturers market special formulations for cleani laboratory glassware; some are claimed to be especially effective in removing contamin tion due to radioactive materials.

Teepol is a relatively mild and inexpensive detergent which may be used for cleani glassware. The laboratory stock solution may consist of a 10% solution in distilled wat For cleaning a burette, 2 mL of the stock solution diluted with 50 mL of distilled water a poured into the burette and allowed to stand for 30–60 s; the detergent is then run off a the burette is rinsed three times with tap water followed by several times with distill water. A 25 mL pipette may be similarly cleaned using 1 mL of the stock solution deionis with 25–30 mL of deionised water.

A method which is frequently used consists in filling the apparatus **carefully** wi chromic acid cleaning mixture – a nearly saturated solution of powdered sodium dichr mate or potassium dichromate in concentrated sulphuric acid – and allowing it to stand f several hours, preferably overnight. The acid is poured off then the apparatus is thorough rinsed with deionised water and allowed to drain until dry. Potassium dichromate is n very soluble in concentrated sulphuric acid (about 5 g L^{-1}), whereas sodium dichroma ($Na_2Cr_2O_7 \cdot 2H_2O$) is much more soluble (about 70 g L^{-1}); that is why sodium dichromate usually preferred for the cleaning mixture, besides the fact it is much cheaper. From tim to time it is advisable to filter the sodium dichromate–sulphuric acid mixture through gla wool placed in the apex of a glass funnel; this removes small particles or sludge that a often present and which may block the tips of burettes.

A very effective degreasing agent, claimed to be much quicker-acting than the cleani mixture, is obtained by dissolving 100 g of potassium hydroxide in 50 mL of water, and aft cooling, making up to 1 L with industrial methylated spirit.[2] **Handle this with great car**

The capacity of a glass vessel varies with the temperature, so a temperature is defin at which the vessel's capacity is intended to be correct. A temperature of 27 °C is accept for use in tropical climates where the ambient temperature is consistently above 20 °C.

3.7 Graduated flasks

A graduated flask, also known as a volumetric flask, is a flat-bottomed, pear-shaped vess with a long narrow neck. A thin line etched around the neck indicates the volume that holds at a certain definite temperature, usually 27 °C (both the capacity and temperatu are clearly marked on the flask); the flask is then said to be graduated **to contain**. Flas with one mark are always taken **to contain** the volume specified. A flask may also b marked **to deliver** a specified volume of liquid under certain definite conditions, but the flasks are not suitable for exact work and are not widely used.

The mark extends completely around the neck, in order to avoid errors due to parallax when making the final adjustment; the lower edge of the meniscus of the liquid should be tangential to the graduation mark, and both the front and the back of the mark should be seen as a single line. The neck is made narrow so that a small change in volume will have a large effect upon the height of the meniscus; the error in adjustment of the meniscus is accordingly small.

The flasks should be fabricated in accordance with specifications* and the opening should be ground to standard (interchangeable) specifications and fitted with an interchangeable glass or plastic (commonly polypropylene) stopper. They should conform to either class A or class B specification.

Graduated flasks are available in the following capacities: 1, 2, 5, 10, 20, 50, 100, 200, 250, 500, 1000, 2000 and 5000 mL. They are employed in making up standard solutions to a given volume; they can also be used for obtaining, with the aid of pipettes, aliquot portions of a solution of the substance to be analysed.

3.8 Pipettes

There are three kinds of pipette:

Transfer pipettes have one mark and deliver a constant volume of liquid under certain specified conditions.

Graduated or measuring pipettes have graduated stems which are employed to deliver various small volumes as required.

Syringe pipettes have fixed or variable volume and are usually employed for dispensing large numbers of identical volumes very quickly.

Transfer pipettes

Transfer pipettes consist of a long glass tube with large central cylindrical bulb; a calibration mark is etched around the upper (suction) tube, and the lower (delivery) tube is drawn out to a fine tip. The graduated or measuring pipette is usually intended for the delivery of predetermined variable volumes of liquid; it does not find wide use in accurate work, for which a burette is generally preferred. Transfer pipettes are constructed with capacities of 1, 2, 5, 10, 20, 25, 50 and 100 mL; those of 10, 25 and 50 mL capacity are most frequently employed in macro work. They should conform to ISO 684-1984 and should carry a colour code ring at the suction end to identify the capacity. As a safety measure, an additional bulb is often incorporated above the graduation mark. They may be fabricated from soda-lime or Pyrex glass, and some high-grade pipettes are manufactured in glass which has been subjected to an ion exchange process that strengthens the glass and also leads to a greater surface hardness, thus giving a product which is resistant to scratching and chipping. Pipettes are available to class A and class B specifications.

The filling of pipettes should never be carried out by mouth suction, and the pipette should never be placed to the lips, irrespective of which liquids are being measured.

To use transfer pipettes, a suitable **pipette filler** is first attached to the upper or suction tube. These devices are obtainable in various forms; a simple version consists of a rubber or plastic bulb fitted with glass ball valves which can be operated between finger and thumb. The valves control the entry and expulsion of air from the bulb and thus the flow of liquid into and out of the pipette.

* The specifications laid down by the International Standardisation Organisation based in Geneva; in the above example the relevant reference is to ISO 384-1978.

Before measuring out the volume of liquid required, rinse the pipette with a sm amount of the liquid then discard it. The pipette should then be filled with the liquid to ab 1–2 cm above the graduation mark. Any adhering liquid is removed from the outside of t lower stem by wiping with a piece of filter paper, and then by careful manipulation of t filler, the liquid is allowed to run out slowly until the bottom of the meniscus just reach the graduation mark; the pipette must be held vertically and with the graduation mark at e level. Any drops adhering to the tip are removed by stroking against a glass surface. T liquid is then allowed to run into the receiving vessel, the tip of the pipette touching t wall of the vessel. When the continuous discharge has ceased, the jet is held in contact w the side of the vessel for a **draining time** of 15 s. At the end of the draining time, the t of the pipette is removed from contact with the wall of the receptacle; the liquid remaini in the jet of the pipette must not be removed either by blowing or by other means.

A pipette will not deliver constant volumes of liquid if discharged too rapidly. T orifice size must produce an outflow time of about 20 s for a 10 mL pipette, 30 s for 25 mL pipette and 35 s for a 50 mL pipette.

Graduated pipettes

Graduated pipettes consist of straight, fairly narrow tubes with no central bulb, and are al constructed to a standard specification, and are colour-coded in accordance with ISO 17 Three different types are available:

Type 1 delivers a measured volume from a top zero to a selected graduation mark.
Type 2 delivers a measured volume from a selected graduation mark to the jet, i.e. t zero is at the jet.
Type 3 is calibrated to **contain** a given capacity from the jet to a selected graduation ma and thus to **remove** a selected volume of solution.

For type 2 pipettes the final drop of liquid remaining in the tip must be expelled, which contrary to the usual procedure. These pipettes are therefore distinguished by a white sandblasted ring near the top of the pipette.

Syringe pipettes or micropipettes

Syringe pipettes or micropipettes are now very common in laboratories and are used pa cularly for dispensing toxic solutions and large numbers of repeat volumes for multi analyses. They may be of fixed or variable volumes. They have a push-button design which the syringe is operated by pressing a button on the top of the pipette; the plun travels between two fixed stops and a reliable constant volume of liquid is delivered. Syri pipettes are fitted with disposable plastic tips (usually of polythene or poly-propyle which are not wetted by aqueous solutions, thus helping to ensure constancy of the volu of liquid delivered. The liquid is contained entirely within the plastic tip and so, by replac the tip, the same pipette can be employed for different solutions. They are available delivering volumes of 1 µL to 10 mL, and the delivery is reproducible to within about 1

The dispensing of volumes smaller than 1 µL is usually carried out using special nee syringes of the type employed for gas chromatography (Chapter 9). Micrometer pipettes also available for the dropwise dispensing of solutions. They are fitted with a microme control that operates the plunger of the syringe, which has a stainless steel needle tip. T small volume of liquid delivered at any time is measured accurately by the micrometer sc

3.9 Burettes

Burettes are long graduated cylindrical tubes of uniform bore terminating at the lower end in a glass or polytetrafluoroethylene (PTFE) stopcock and a jet. The PTFE taps have the great advantage that they do not require lubrication.

It is sometimes advantageous to employ a burette with an extended jet which is bent twice at right angles so that the tip of the jet is displaced by some 7.5–10 cm from the body of the burette. Insertion of the tip of the burette into complicated assemblies of apparatus is thus facilitated; and if heated solutions have to be titrated, the body of the burette is kept away from the source of heat. Burettes fitted with two-way stopcocks are useful for attachment to reservoirs of stock solutions.

As with other graduated glassware, burettes are produced to both class A and class B specifications in accordance with ISO 385 (1984), and class A burettes may be purchased with certificates. All class A and some class B burettes have graduation marks which completely encircle the burette; this is a very important for avoiding parallax errors when taking readings.

In addition to the volume requirements, limits are also imposed on the length of the graduated part of the burette and on the drainage time.

When in use, a burette must be firmly supported on a stand, and various types of burette holder are available for this purpose. The use of an ordinary laboratory clamp is not recommended; the ideal type of holder permits the burette to be read without removing it from the stand.

Lubricants for glass stopcocks

The object of lubricating the stopcock of a burette is to prevent sticking or 'freezing' and to ensure smoothness in action. The simplest lubricant is pure vaseline, but this is rather soft, and unless used sparingly, portions of the grease may readily become trapped at the point where the jet is joined to the barrel of the stopcock, eventually blocking the jet. Commercial lubricants for stopcocks are available from laboratory suppliers. Silicone-based lubricants should not be used as they tend to creep and cause contamination of the inside of the burette.

To lubricate the stopcock, the plug is removed from the barrel and two thin streaks of lubricant are applied to the length of the plug on lines roughly midway between the ends of the bore of the plug. Upon replacing in the barrel and turning the tap a few times, a uniform thin film of grease is distributed round the ground joint. A spring or some other form of retainer may then be attached to the key to lessen the chance of it becoming dislodged when in use.

Using a burette

The burette is thoroughly cleaned using one of the cleaning agents described in Section 3.6 and is then well rinsed with distilled water. The plug of the stopcock is removed from the barrel, and after wiping the plug and the inside of the barrel dry, the stopcock is lubricated as described in the preceding paragraph. Using a small funnel, about 10 mL of the solution to be used are introduced into the burette, and then after removing the funnel, the burette is tilted and rotated so that the solution flows over the whole of the internal surface; the liquid is then discharged through the stopcock. After repeating the rinsing process, the burette is clamped **vertically** in the burette holder and then filled with the solution to a little above the zero mark. The funnel is removed, and the liquid discharged through the stopcock

until the lowest point of the liquid meniscus is just below the zero mark; the jet is inspe to ensure that all air bubbles have been removed and that it is completely full of liqui

To read the position of the meniscus, the eye must be at the same level as the menis in order to avoid errors due to parallax. In the best type of burette, the graduations carried completely round the tube for each millilitre (mL) and halfway round for the o graduation marks; parallax is thus easily avoided. To assist in reading the position of meniscus, use a piece of white paper or cardboard, its lower half blackened by paint with dull black paint or by pasting on a piece of dull black paper. When this is placed that the sharp dividing line is 1–2 mm below the meniscus, the bottom of the menis appears to be darkened and is sharply outlined against the white background; the level the liquid can then be accurately read. A variety of burette readers are available fr laboratory supply houses, and a home-made device which is claimed to be particula effective has been described by Woodward and Redman.[2] For ordinary purposes, readi are made to 0.05 mL; for precision work, readings should be made to 0.01–0.02 mL us a lens to assist in estimating the subdivisions.

To deliver liquid from a burette into a conical flask or other similar receptacle, pl the fingers of the left hand behind the burette and the thumb in front, and hold the tap the right-hand side between the thumb and the fore and middle fingers. In this way th is no tendency to pull the plug out of the barrel of the stopcock, and the operation is un complete control. Any drop adhering to the jet after the liquid has been discharged removed by bringing the side of the receiving vessel into contact with the jet. During t delivery of the liquid, the flask may be gently rotated with the right hand to ensure t added liquid is well mixed with any existing contents of the flask.

3.10 Weight burettes

Weight burettes are used for work demanding the highest possible accuracy in transferri various quantities of liquids. As the name implies, they are weighed before and after transfer of liquid. A very useful form is shown in Figure 3.2(a). There are two groun glass caps; the lower cap is closed and the upper cap has a capillary opening; this mea

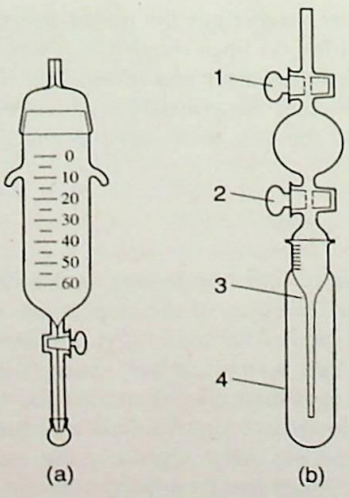

Figure 3.2 Weight burettes: (a) a very useful form, (b) the Lunge–Rey pipette

there is negligible loss by evaporation. For hygroscopic liquids, a small ground-glass cap is fitted to the top of the capillary tube. The burette is roughly graduated in 5 mL intervals. The titre thus obtained is in terms of weight loss of the burette, and for this reason the titrants are prepared on a weight/weight basis rather than a weight/volume basis. The errors associated with a volumetric burette, such as drainage, reading and change in temperature, are avoided, and weight burettes are especially useful when dealing with non-aqueous solutions or with viscous liquids.

The **Lunge–Rey pipette** is shown in Figure 3.2(b). There is a small central bulb (5–10 mL capacity) closed by two stopcocks (1, 2); the pipette (3) below the stopcock has a capacity of about 2 mL, and is fitted with a ground-on test-tube (4). This pipette is especially useful for weighing out corrosive and fuming liquids.

3.11 Piston burettes

In piston burettes the delivery of the liquid is controlled by movement of a tightly fitting plunger within a graduated tube of uniform bore. They are particularly useful when the piston is coupled to a motor drive, forming the basis of automatic titrators. Piston burettes can provide automatic plotting of titration curves; they also allow a variable rate of delivery as the end point is approached, so there is no danger of overshooting.

3.12 Graduated (measuring) cylinders

Cylinders are graduated in capacities from 2 to 2000 mL. Since the surface area of the liquid is much greater than in a graduated flask, the accuracy is not very high. This means graduated cylinders cannot be used for work demanding even a moderate degree of accuracy, but they are okay for rough measurements.

3.13 Calibration of graduated apparatus

For most analytical purposes, graduated apparatus manufactured to class A standard will prove satisfactory, but for work of the highest accuracy it is advisable to calibrate all apparatus for which a **recent** test certificate is unavailable. The calibration procedure involves determining the weight of water contained in or delivered by the particular piece of apparatus. The temperature of the water is observed, and from the known density of water at that temperature, the volume of water can be calculated. Tables giving density values are usually based on weights in vacuo (Section 3.4), but the data given in Table 3.1 is based on

Table 3.1 *Volume of 1 g of water at various temperatures*

Temp. (°C)	Volume (mL)	Temp. (°C)	Volume (mL)
10.00	1.0013	22.00	1.0033
12.00	1.0015	24.00	1.0037
14.00	1.0017	26.00	1.0044
16.00	1.0021	28.00	1.0047
18.00	1.0023	30.00	1.0053
20.00	1.0027		