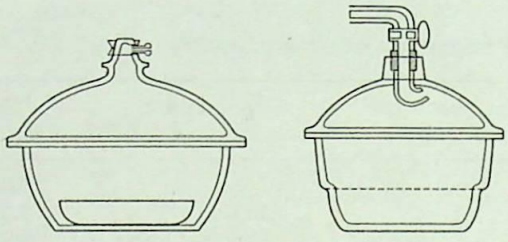


Figure 3.4 Vacuum desiccators

Convenient types of **vacuum desiccator** are illustrated in Figure 3.4. Large surfaces of the solid can be exposed; the desiccator may be evacuated, so drying is much more rap than in the ordinary Scheibler type. These desiccators are made of heavy glass, plastics even metal, and they are designed to withstand reduced pressure; nevertheless, no desi cator should be evacuated unless it is surrounded by an **adequate guard** in the form of stout wire cage.

For most purposes the vacuum produced by an efficient water pump (20–30 mm me cury) will suffice; a guard tube containing desiccant should be inserted between the pum and the desiccator. The sample to be dried should be covered with a watch or clock glas so that no solid is lost during removal or admission of air. Air must be admitted slow into an exhausted desiccator; if the substance is very hygroscopic, a drying train should b attached to the stopcock. In order to maintain a satisfactory vacuum within the desiccato the flanges on both the lid and the base must be well lubricated with vaseline or oth suitable grease. In some desiccators an elastomer ring is incorporated in a groove in th flange of the lower component of the desiccator; when the pressure is reduced, the ring compressed by the lid of the desiccator, and an airtight seal is produced without the nee for any grease. The same desiccants are used as with an ordinary desiccator.

Dry boxes

Dry boxes (glove boxes) are used for manipulating materials that are very sensitive atmospheric moisture (or to oxygen); they consist of a plastic or metal box with glass clear plastic windows on the upper side and on the sidewalls. Rubber or plastic glov are fitted using airtight seals through the front side of the box, and by placing the han and forearms into the gloves, manipulations may be carried out inside the box. One end the box is fitted with an airlock so that apparatus and materials can be introduced into th box without disturbing the atmosphere inside. A tray of desiccant placed inside the bo maintains a dry atmosphere, but to counter the unavoidable leakages it is usual to supp a slow current of dry air to the box; inlet and outlet taps are provided to control th operation. If the box is flushed out before use with an inert gas (e.g. nitrogen), and a slo stream of the gas is maintained while the box is in use, materials which are sensitive oxygen can be safely handled. A detailed discussion of their construction and use is give in the literature.[6]

3.20 Stirring apparatus

Stirring rods

Stirring rods are made from glass rod 3–5 mm in diameter, cut into suitable lengths. Both ends should be rounded by heating in the Bunsen or blowpipe flame. The length of the stirring rod should be suitable for the size and shape of the vessel, e.g. a spouted beaker requires a stirring rod that projects 3–5 cm beyond the lip when in a resting position. Glass stirring rods should not be used for stirring viscous liquids as they can cause serious hand injuries if they break. A short piece of Teflon or rubber tubing (or a rubber cap) fitted tightly over one end of a stirring rod of convenient size provides the so-called **policeman**, used for detaching particles of a precipitate adhering to the side of a vessel which cannot be removed by a stream of water from a wash bottle; it should not, as a rule, be employed for stirring, nor should it be allowed to remain in a solution.

Boiling rods

Boiling liquids and liquids in which a gas, such as hydrogen sulphide or sulphur dioxide, has to be removed by boiling can be prevented from superheating and 'bumping' by the use of a boiling rod (Figure 3.5). This consists of a piece of glass tubing closed at one end and sealed approximately 1 cm from the open end; the open end is immersed in the liquid. When the rod is removed, the liquid in the open end must be shaken out and the rod rinsed with a jet of water from a wash bottle. This device should not be used in solutions which contain a precipitate.

Stirring may be conveniently effected with the so-called **magnetic stirrer**. A rotating magnet induces a variable-speed stirring action within closed or open vessels. The actual stirrer is a small cylinder of iron sealed in Pyrex glass, polythene or Teflon, which is caused to rotate by the rotating magnet.

The usual glass paddle stirrer is also widely used. It works in conjunction with an electric motor controlled by a transformer or solid-state speed device. The stirrer may be connected directly to the motor shaft or to a spindle actuated by a gearbox which forms an integral part of the motor housing; it is possible to obtain a wide variation in stirrer speed.

Under some circumstances, e.g. the dissolution of a sparingly soluble solid, it may be better to use a **mechanical shaker**. They range from wrist action shakers which accommodate small or medium-sized flasks, to powerful shakers which can take large bottles and give their contents a vigorous agitation.

3.21 Filtration apparatus

The simplest filtration apparatus is a filter funnel fitted with a filter paper. To promote rapid filtration, the funnel angle should be as close as possible to 60° and the funnel stem should be 15 cm long. Filter papers are made in varying grades of porosity, so choose one appropriate to the filtration (Section 3.32).

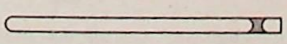


Figure 3.5 Boiling rod

In the majority of quantitative determinations involving the collection and weighing a precipitate, it is convenient to collect the precipitate in a crucible for direct weighing and various forms of **filter crucible** have been devised for this purpose. Sintered-glass crucibles are made of resistance glass and have a porous disc of sintered ground glass fused into the body of the crucible. The filter disc is made in varying porosities, 0 is the coarsest and 5 the finest; the range of pore diameter for the various grades is as follows.

Porosity	0	1	2	3	4	5
Pore diameter (μm)	200–250	100–120	40–50	20–30	5–10	1–

Porosity 3 is suitable for precipitates of moderate particle size, and porosity 4 for fine precipitates such as barium sulphate. These crucibles should not be heated above about 200 °C. Silica crucibles of similar pattern are also available; although expensive, they have certain advantages in thermal stability.

Filter crucibles with a porous filter base are available in porcelain (porosity 4), in silica (porosities 1, 2, 3, 4) and in alumina (coarse, medium and fine porosities); they can be heated to much higher temperatures than sintered crucibles. Nevertheless, the heating must be gradual otherwise the crucible may crack at the join between porous base and glazed side.

Large quantities of material are usually filtered through a **Buchner funnel**, or one of the modified funnels in Figure 3.6. In the ordinary porcelain funnel and the 'slit sieve' glass funnel (Figure 3.6(a) and (b)) two good quality filter papers are placed on the plate; the transparent glass funnel is preferable since it is easy to see whether the funnel is perfectly clean. In the Pyrex funnel with a sintered glass plate (Figure 3.6(c)) no filter paper is required, so strongly acidic and weakly alkaline solutions can be readily filtered with this funnel. In all cases the funnel of appropriate size is fitted into a filter flask (Figure 3.6(d)) and the filtration conducted under the diminished pressure provided by a filter pump or vacuum line. One of the disadvantages of the porcelain Buchner funnel is that, being of one piece construction, the filter plate cannot be removed for thorough cleaning and it is difficult to see whether the whole of the plate is clean on both sides. In a modern polythene version the funnel is made in two sections which can be unscrewed for inspecting both sides of the plate.

Also available are special disposable filter funnels with removable filter plates and capacities of up to 30 mL; they are particularly useful in filtering small quantities of

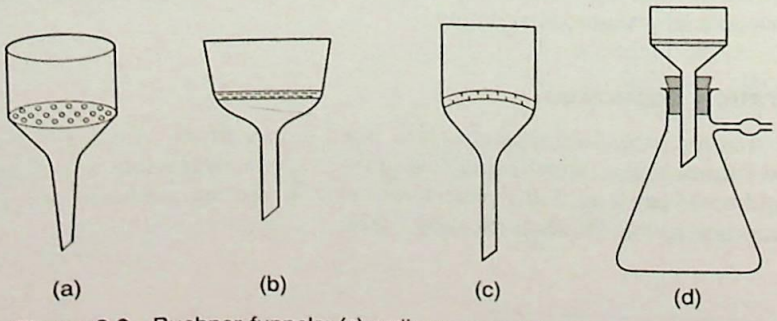
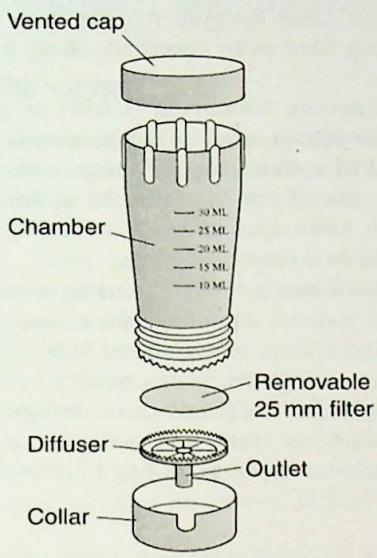
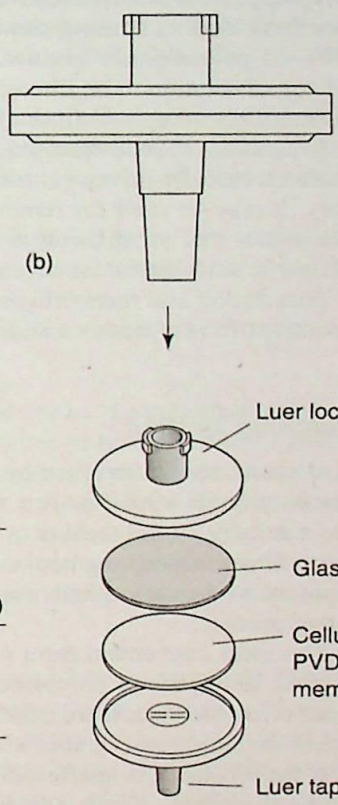


Figure 3.6 Buchner funnels: (a) ordinary porcelain, (b) 'slit sieve' glass, (c) Pyrex, (d) Buchner funnel fitted to a filter flask





Syringe filter diameter (mm)	Capacity (mL)	Typical hold-up volume (µL)
4	0–2	10
13	2–5	50
25	>4	<100

Figure 3.7 (a) Disposable filter funnel: construction (courtesy Whatman International Ltd, Maidstone, Kent). (b) Construction of a syringe type filter.

radioactive materials and proteins (Figure 3.7(a)). The choice of filter should achieve a compromise between separation time and separation quality. For many filtrations, single-use cartridges made of plastic are more convenient than traditional filter funnels and folded papers. But these throwaway items come in such a huge range of sizes, shapes and compositions that choosing the appropriate cartridge may seem quite daunting to start with. However, if each parameter in turn is compared against the proposed experiment, the choice is normally fairly straightforward. The most widely used form is a sealed flat filter in a plastic housing fitted with male and female Luer lock (syringe) fittings on either side of the filter (Figure 3.7(b)).

The pore size of the filter is normally considered first; it can range from 1.0 µm down to less than 0.1 µm (since these membranes are made of plastic materials, the pore size is remarkably constant across the membrane). For many chemical separations, a pore size of 0.45 µm is often chosen as a good compromise between effective filtration and speed. However, where very finely divided materials are anticipated or where biological activity

must be minimised, then 0.2 µm filters are used. Once the pore size has been determine the actual material for the filter and its housing need to be specified, along with the si of the filter.

For many aqueous solutions, a cellulose acetate membrane (CAM) or glass fibr polypropylene membrane can be used, but for non-aqueous or aggressive solutions, t membrane and its housing can be constructed of a wide range of other materials such PTFE or polysulphone plastics. Equally the size of the filter can be optimised for t volume of solution to be filtered, ranging from 3 mm discs which will filter up to 2 mL, 25 mm discs which will filter 100 mL of liquid in a reasonable time.

Separation of solid from liquid is sometimes better achieved by using a **centrifuge**. small, electrically driven centrifuge is a useful piece of equipment for an analytical labo atory; it may be used for removing mother liquor from recrystallised salts, for collectin precipitates that are difficult to filter, and for washing certain precipitates by decantatio It is particularly useful when small quantities of solids are involved; centrifuging, followe by decantation and recentrifuging, avoids transference losses and yields the solid phase i a compact form. Another valuable application is the separation of two immiscible phase

3.22 Weighing bottles

Most chemicals are weighed **by difference** through placing the material inside a stoppere weighing bottle which is then weighed. The requisite amount of substance is shaken ou into a suitable vessel (beaker or flask), and the weight of substance taken is determined b reweighing the weighing bottle. In this way, the substance dispensed receives the minimu exposure to the atmosphere during the actual weighing process, important if the materi is hygroscopic.

The most convenient form of weighing bottle is fitted with an external cap and mad of glass, polythene or polycarbonate. A weighing bottle with an **internally** fitting stopp is not recommended; there is always the danger that small particles may lodge at the upp end of the bottle and be lost when the stopper is pressed into place.

If the substance is unaffected by exposure to the air, it may be weighed on a watchglas or in a disposable plastic container. The weighing funnel (Figure 3.8) is very useful, pa ticularly when the solid is to be transferred to a flask. Having weighed the solid into the scoo shaped end, flattened so it will stand on the balance pan, the narrow end is inserted into th neck of the flask and the solid washed into the flask with a stream of water from a wash bottl

Woodward and Redman[2] have described a specially designed weighing bottle whic will accommodate a small platinum crucible; when a substance has been ignited in th crucible, the crucible is transferred to the weighing bottle and subsequently weighed insid This obviates the need for a desiccator. If the substance to be weighed is a liquid, it placed in a weighing bottle fitted with a cap carrying a dropping tube.

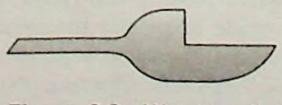


Figure 3.8 Weighing funnel

Reagents and standard solutions

3.23 Reagents

The purest reagents available should be used for quantitative analysis; wherever possible, use reagents of analytical quality. Certain manufacturers market chemicals of high purity, and each package of these analysed chemicals has a label giving the manufacturer's limits of certain impurities.

With the increasingly lower limits of detection being achieved in various types of instrumental analysis, there is an ever growing demand for reagents of correspondingly improved specification, and some manufacturers are now offering a range of specially purified reagents.

In some instances, where a reagent of the requisite purity is not available, it may be advisable to weigh out a suitable portion of the appropriate **pure** metal and to dissolve this in the appropriate acid.

The label on a bottle is not an infallible guarantee of the contents. Purity may be compromised for several reasons:

(a) Some impurities may not have been tested for by the manufacturer.
(b) The reagent may have been contaminated after its receipt from the manufacturers; the stopper may have been left open for some time, exposing the contents to the laboratory atmosphere, or there may have been accidental return of an unused portion of reagent to the bottle.
(c) A solid reagent may not be sufficiently dry, perhaps due to insufficient drying by the manufacturers, perhaps due to leakage through the stoppers, or perhaps a combination of the two.

However, if the analytical reagents are purchased from a reputable manufacturer, if no bottle is open for longer than absolutely necessary, and if no reagent is returned to the bottle, then the likelihood of these errors is considerably reduced. Liquid reagents should be poured from the bottle; a pipette should never be inserted into the reagent bottle. Particular care should be taken to avoid contamination of the stopper of the reagent bottle. When a liquid is poured from a bottle, the stopper should never be placed on the shelf or on the working bench; it may be placed upon a clean watchglass, and many chemists cultivate the habit of holding the stopper between the thumb and fingers of one hand. The stopper should be returned to the bottle immediately after the reagent has been removed, and all reagent bottles should be kept scrupulously clean, particularly round the neck or mouth of the bottle.

If there is any doubt as to the purity of the reagents used, they should be tested by standard methods for the impurities that might cause errors in the determinations. Where a chemical required for quantitative analysis is not available in the form of analytical reagents, the purest commercially available products should be purified by known methods (see below). The exact mode of drying, if required, will vary with the reagent; details are given for specific reagents in the text.

3.24 Purification of substances

If a reagent of adequate purity for a particular determination is not available, then the purest available product must be purified; for inorganic compounds this is most commonly