CHEMICAL LABORATORY TECHNIQUES

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Summary

Basic laboratory techniques in chemistry have an essential place in the training of a chemist. They provide a good background for experimental skills and for subsequent

independent research. In this chapter, first of all, we shall consider some of the standard pieces of glassware and equipment that you will use in the laboratory.

By assembling suitable apparatus, you can carry out reactions from starting materials to pure products. Sometimes these operations require particular laboratory reactors that work in well-defined operation conditions. At the end of the reaction different techniques are needed in order to separate the products from the reaction mixture, and then the final compound will need to be purified by means of specific operations. The separation of reaction products is obtained by techniques such as extraction with solvents, crystallization, and distillation. These methods, which are almost standardized, are often able to purify organic compounds by separating the impurities. Chromatographic methods are very effective in separating more complex mixtures.

Standard safety practices are an essential part of all laboratory operations. The chemicals employed in the laboratory can be flammable or irritating, and many possess known or as yet undetermined toxic characteristics. Accidents in the chemistry laboratory can be avoided if you enter the laboratory properly prepared for the experiment, if you use common sense in reacting to unexpected situations, and if you rigidly follow basic safety rules that are enforced to ensure your personal safety.

1. Common Laboratory Apparatus

The first section of this article provides an introduction to various pieces of equipment—glassware and nonglassware—that are used in the laboratory.

1.1. Glassware

Glass equipment can be divided into those with ground-glass joints and those without. New techniques of glassworking and the employment of glasses with a very low expansion have allowed the mass production of really interchangeable, standard-taper, ground-glass joints. Therefore standard-taper, ground-glass joint equipment is strongly recommended. In fact, apparatus for a range of experiments can be assembled quickly and easily from relatively few basic items and there is no need to utilize corks or rubber bungs. The advantages of this are:
- corrosive liquids and solids are easily manipulated;
- no impurity is introduced from reaction with corks or rubber bungs (this prevents contaminating the reaction products with impurities);
- all joints are interchangeable and a good "fit" is assured (this is very advantageous in assembling apparatus that have to work under reducing pressure); and
- apparatus may be rapidly assembled.

In the use of ground-glass joints some precautions need to be observed, such as cleaning the joints and the use of a little grease or a thin Teflon sleeve. The dimension of standard taper joints are designated by a number that refer to the external diameter of the smaller end of the cone (in millimeters) and the length of the ground zone (also in millimeters).

Exact details of the various types and size of ground-glass joint apparatus available commercially will be found in the catalogues of the various manufacturers. Some units employed for single ground-glass joint assemblies are shown in Figure 1.
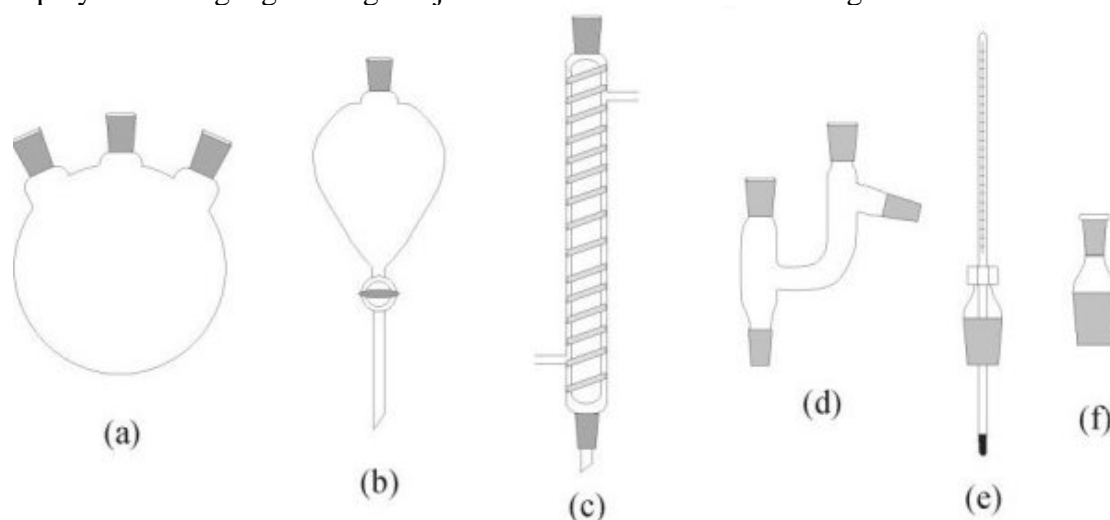

Figure 1. Glass equipment with standard-taper, ground-glass joints
Notes: (a) Three-neck, round-bottom flask; (b) separation funnel; (c) water condenser; (d) Claisen adapter; (e) standard glass screw thread for a thermometer or tube; (f) expansion adapter.

Common glassware without ground-glass joints can generally be used in the chemical laboratory. The main glassware without glass joints are illustrated in Figure 2.
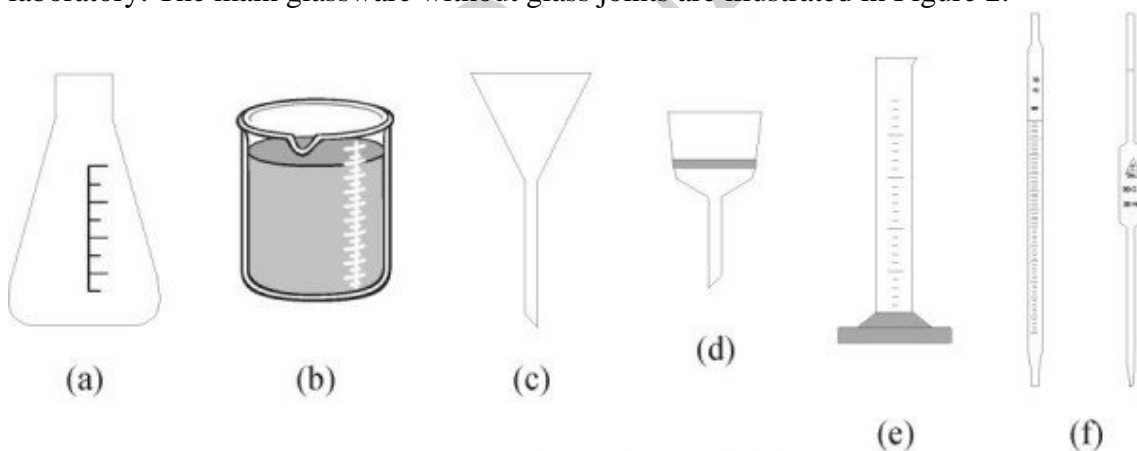

Figure 2. Common glassware without ground-glass joints
Notes: (a) Erlenmeyer flask and (b) beaker, used for temporary storage or transfer of materials; (c) funnel, for transfer of liquids and solids, and for filtration; (d) Buchner funnel, which is made of porcelain and has a perforated porcelain plate to support a filter paper, and is used in conjunction with a suction flask; (e) graduated cylinder, for measuring liquids by volume; (f) graduated pipet, for accurate measurement.

At last vacuum essicators are available to remove water or solvents of relatively low boiling point adhering to solids, under reduced pressure at room temperature, or to keep anhydrous any compounds. The reagent in the bottom of the essicators naturally depends on the exact nature of the substance to be adsorbed—water or organic solvent—or the acid or basic vapor generated during the drying process.

1.2. Heating of the Reaction Mixture

Several methods of heating are commonly encountered in the laboratory, but the ready flammability of a wide range of reagents, coupled with their volatility, always requires vigilance when heating. For these reasons open flames represent an obvious hazard, but hot metal surfaces can also give rise to dangerous situations.

1.2.1. Burners

These can normally only be used for heating aqueous solution in open vessels. When heating beakers, flasks, or flat-bottom vessels with a burner, wire gauze should be placed between the vessel and the flame. This serves both as a support and as a mean of dispersing heat. Burners can also be used for distillation and reflux procedures involving high-boiling-point materials, but in these cases care must be taken to ensure that no flammable vapors come into contact with the flame.

1.2.2. Heating Baths

For temperatures up to 100 °C a water bath or steam bath is generally employed, although water condensing can be a problem if it is necessary to ensure anhydrous conditions within the reaction. Water is placed in the vessel, which is heated by means of the flame. For this reason it may be used for noninflammable liquids or for refluxing of low-boiling-point products, but in this case the presence of a naked flame introduces considerable risks of fire. These baths are normally equipped with a series of overlapping concentric rings, which can be removed to give the right size of support for the particular vessel being heated.

If the laboratory is equipped with a steam service, it is convenient to have a series of steam baths. Alternatively, if the laboratory has no external steam service, to avoid naked flames, an electrically heated bath may be utilized and fitted with a constant level device. A resistance connected to a temperature regulator heats water in the bath.

For temperatures above 100 °C, oil baths are generally used. The bath can be heated with a heating element or on a hotplate. Medical paraffin, glycerol, silicon oil, and cottonseed oil may be employed; it depends on the work temperature. Silicon oils carry a low risk of inflammation, do not give off unpleasant odors, and have a long service life. Synthetic thermal liquids are mainly produced on a hydrocarbon basis and exhibit a low viscosity within the recommended working temperature range. Mineral oil is mainly used for the high temperature range. Unpleasant odors are kept to a minimum.

The silicon fluids are probably the best liquids for oil baths, but they are very expensive for general use. On the other hand, these fluids can be heated to up to 250 °C without loss or discoloration.

In modern equipment, an immersion heating circulator is mounted onto the rear panel of the bath vessel (see Figure 3a). This combines a heater, a temperature control, and a circulating pump for temperature uniformity throughout the bath, which is of great advantage in temperature control. It can be moved from one vessel to another and can

be used with any tank. The heater and control sensors are sometimes located underneath the bath, thus guaranteeing easy cleaning.

Heated baths feature external parts to circulate the fluid to an external system. Heating circulators (Figure 3b) are mainly used for temperature controlling external systems, such as densitymeters, reaction vessels, autoclaves, and viscosimeters. Powerful pumps provide good heat exchange and optimum temperature accuracy.
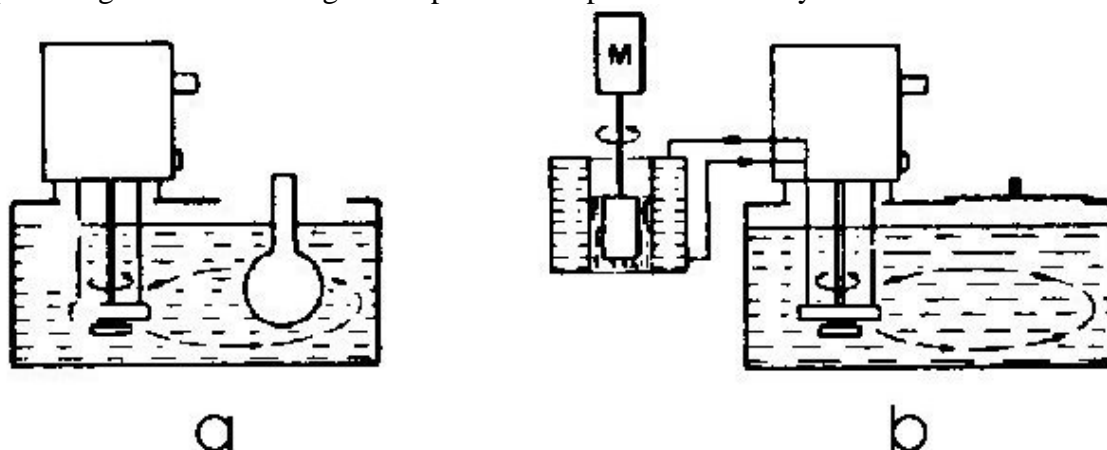

Figure 3. Schematic (a) heated bath and (b) heated circulator bath

Higher temperatures may be obtained with the support of a bath of fusible metal alloys (e.g., Rose's metal: 2 parts of Bi, 1 part of Pb, and 1 part of Sn, melting point 94 °C; woods metal: 5 parts of Bi, 2 parts of Pb, 1 part of Sn, and 1 part of Cu, melting point 71 °C; and a mixture of Pb (37%) and Sn (63%), melting point 183 °C). These metal baths should not be used at temperatures in excess of 350 °C owing to the rapid oxidation of the alloy. Metal baths are solid at ordinary temperatures, and for this reason the flask and the thermometer should be removed from the bath before the latter solidifies.

1.2.3. Electric Hot Plates and Electric Heating Mantles

These items of equipment may also be employed for heating, although their use for several tasks (e.g., distillation) is to be discouraged.

The hot plate/magnetic stirrer is a single device that can heat liquids and stir them with a magnetic stirring bar. One knob controls the rate of stirring and another controls heating. A stirrer hot plate keeps the solution at a constant temperature while stirring. The built-in magnetic stirrer permits efficient agitation of nonviscous solutions by adding an appropriately sized magnetic stirrer bar to the liquid in the container. It is designed for heating flat-bottomed vessels, such as flasks and beakers, in a temperature range from 40 °C to 200 °C. Round-bottomed flasks may be heated using a stirrer hotplate by immersing the flask in a flat-bottomed oil bath. The flat, exposed surface of the hotplate, designed for transferring heat rapidly, makes it extremely dangerous when hot.

The electric heating mantel is a very convenient method of heating, especially for temperature above 100 °C. It consist of an electric resistance embedded within a hemispherical knitted mantle, so that the heat supply is as close to the flask to be heated

as possible. Electric mantels are designed only for heating round-bottomed flasks and can accept a flask of a particular size.

All heating mantels are particularly vulnerable to spillage of liquids, and with constant use this can lay bare the wires within the heating element.

1.3. Cooling of the Reaction Mixtures

Temperatures below that of the laboratory are sometimes required. If it is necessary to obtain temperatures of 0 °C to –5 °C we can use a slush of crushed ice with sufficient water to provide contact with the vessel to be cooled. For temperatures of –5 °C to –15 °C it is usually best to use a mixture of one part of common salt (NaCl) and three parts of crushed ice. Greater cooling may be obtained with five parts of calcium chloride hydrate and four parts of crushed ice (i.e., temperatures of –40 °C to –50 °C).

To reach very low temperatures, solid carbon dioxide is employed. Temperatures of –50 °C can be obtained if it is suspended in solvent—equal volumes of chloroform and carbon tetrachloride—and lower temperatures with different solvents (e.g., with ethanol –72 °C; with diethylether –77 °C; or with chloroform or acetone –77 °C). Even lower temperatures may be obtained using liquid nitrogen (i.e., for temperatures below –100 °C).

Sometimes a heated circulator is combined with a refrigerator unit. Powerful, quiet-running cooling compressors cool them. Refrigerated circulators and cryostats are mainly used when below-ambient temperatures must be reached or maintained or when it is wished to cycle between two temperatures at a control rate. Refrigerated circulators and cryostats feature a wide temperature range (i.e., –90 °C to 150 °C). They are especially suited for controlling open and closed circuits due to their extremely powerful pressure/suction pumps.

These coolers are suitable for individual cooling applications:
- for cooler smaller volumes down to –90 °C,
- for removing reaction heat, and
- for replacing tap-water cooling.

The lowest reliable temperature depends on the quantity of liquid, the type of liquid and its viscosity, and the bath insulation.

1.4. Stirring

Most of the chemical reactions need stirring to mix the reagents or to aid heat transfer. There are three main ways to agitate a mixture: by hand, with a magnetic stirrer, and with a mechanical stirrer; but when a constant stirring is needed for a sustained period, a stirrer motor should be used. Magnetic stirring has many applications, but the most important is probably stirring in closed systems. Magnetic stirrers are easy to use, and have the advantage that they are often combined with a hotplate. Hot-plate stirrers allow you to keep solutions at a constant temperature while stirring.

A rotating field of magnetic force is employed to induce a variable-speed stirring action. The principle of magnetic stirring is shown in Figure 4a. The stirring is accomplished with the aid of a small magnetic bar—coated with Teflon or Pyrex—that is available in various sizes and forms depending on the volume and the viscosity of the liquid. Although bar magnets can be obtained with several sorts of coating, only Teflon-coated stirrers are universally useful (see Figure 4b).
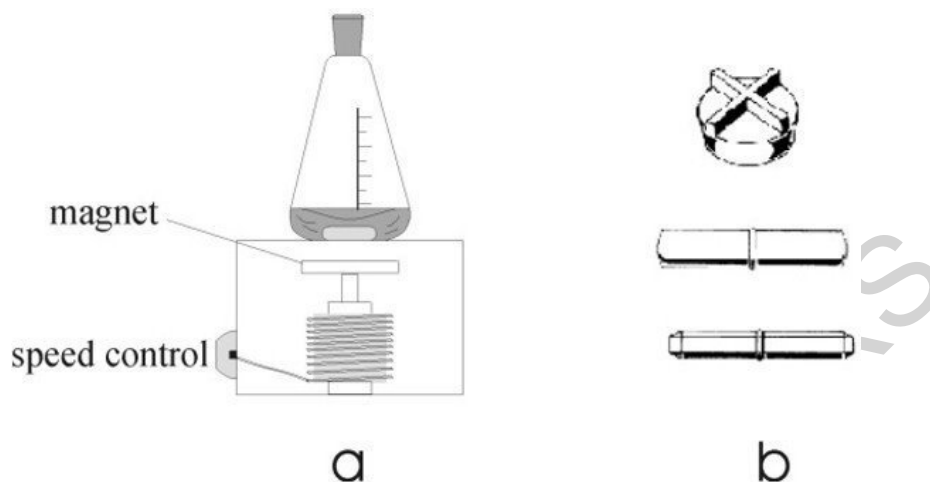

Figure 4. Schematic (a) magnetic stirrer and (b) magnetic bars

There are two types of magnetic stirrers: mechanical and electronic. Most manufacturers of magnetic stirrers use a mechanical approach. They use steel and aluminum for the structural material, and outdated methods of controlling the speed. Adjusting the stirring speed with a mechanical stirrer is fairly inaccurate.

Electronic controls allow the stirrer to control the speed with greater accuracy, and if the motor is running at the maximum operating speed with a load, and the load is suddenly removed, the circuitry will not allow the motor to increase in speed, which would damage the unit. It is advantageous to stir in two directions to obtain homogeneous results. This cannot be done with a mechanical stirrer.

Liquid behavior is important to consider when you begin a mixing project. If the reaction mixture is very viscous or heterogeneous—with a large amount of suspended solid—the magnetic stirrer motor, with its relative low torque, will not be suitable for the purpose, and in these cases a mechanical stirrer should be used.

The stirrer is simply attached to a motor by a flexible connection made out of a sort of pressure tubing. A typical collection of mechanical stirrers is illustrated in Figure 5. The stirrer—either rod or paddle—may be made of glass, metal, or Teflon, depending on the characteristic of the liquid to be stirred.

Figure 5. Several useful types of mechanical stirrer

Proper selection of a suitable motor and type of stirrer requires that you know certain application variables:

- container volume;
- liquid viscosity (the viscosity of most liquids varies inversely with temperature);
- torque requirements (the rotation force required of the mixer measured in lbs*ft or N*m);
- horsepower (hp) requirements (the efficiency required of the mixer motor with regard to torque and to rotation speed); and
- rotational speed (measured in rpm).

The effect of the liquid's viscosity can present problems when the liquid is subjected to a force. Different types of liquid display characteristics when force is applied. The most common types of viscous liquids are:

- *Newtonian liquids*: viscosity remains constant regardless of changes in shear rate or agitation. Liquids displaying Newtonian behavior include water, hydrocarbons, mineral oil, and syrup.
- *Pseudoplastic liquids*: viscosity decreases as shear rate increases, but initial viscosity may be sufficiently great to prevent mixing. Typical pseudoplastic liquids are gels and latex paints.
- *Dilatant liquids*: viscosity increases as shear rate increases. Mixers can bog down and stall after initially mixing such liquids. Dilatant liquids include slurries, clay, and candy compounds.
- *Thixotropic liquids*: viscosity decreases as shear rate or agitation increases, but when agitation is stopped or reduced, hysteresis occurs and viscosity increases. Often the viscosity will not return to the initial value. Thixotropic liquids include soaps, tars, vegetable oils, inks, glue, and peanut butter.

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Biographical Sketches

Davino Gelosa was born in Monza (Milano), Italy, on December 12 1948, and graduated in Agrarian and Natural Science at the University of Milano in 1978. He was technical official at the Politecnico of Milano from 1968 to 1986, then technical coordinator at the Department of Chimica Fisica Applicata

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Andrea Sliepcevich was born in Milano, Italy, on November 22 1968, and graduated in Industrial Chemistry at the University of Milano in 1995. He obtained his Ph.D. in Electrochemical Engineering at the Politecnico of Milano in 1999. From 1999 he has been a junior researcher at the Department of Chimica Fisica Applicata (Politecnico of Milano) where his main fields of research are the kinetic study of homogeneous and heterogeneous catalysis and emulsion polymerization. He has contributed four papers to international journals and two patents.