TECHNIQUE

4 LABORATORY GLASSWARE

You will find an assortment of glassware and equipment in your laboratory desk; some items will be familiar to you from your earlier lab experiences and other items may not. If your laboratory is equipped for miniscale experimentation, you will find specialized glassware called ***standard taper glassware,*** which has carefully constructed ground glass joints designed to fit together tightly and interchangeably. Standard taper glassware is available in a variety of sizes. If you will be carrying out microscale experimentation, you will use scaled-down glassware designed for the milligram and milliliter quantities of reagents used in microscale work. There are two types of microscale glassware commonly used in the undergraduate organic laboratory—microscale standard taper glassware with threaded screw cap connectors and the Kontes/Williamson microscale glassware that fastens together with flexible elastomeric connectors.

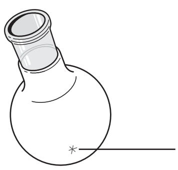

FIGURE 4.1
Round-bottomed flask with a star crack.

SAFETY PRECAUTION

Before you use any glassware in an experiment, check it carefully for cracks or chips. Glassware with spherical surfaces, such as round-bottomed flasks, can develop small, star-shaped cracks (Figure 4.1). Replace damaged glassware. When cracked glassware is heated, it can break and ruin your experiment and possibly cause a serious spill or fire.

4.1 Desk Equipment

A typical student desk contains an assortment of beakers, Erlenmeyer flasks, filter flasks, thermometers, graduated cylinders, test tubes, funnels, and other items. Your desk or drawer will probably have most, if not all, of the equipment items shown in Figure 4.2. Make sure that all glassware is clean and has no chips or cracks. Replace damaged glassware.

4.2 Standard Taper Miniscale Glassware

Standard taper glassware is designated by the symbol $ \mathsf{\$} $. All the joints in standard taper glassware have been carefully ground so that they are exactly the same size, and all the pieces fit together interchangeably. We recommend the use of $ \mathsf{\$} $ 19/22 or $ \mathsf{\$} $ 14/20 glassware for miniscale experiments. The numbers, in millimeters, represent the diameter and the length of the ground glass surfaces (Figure 4.3). A typical set of $ \mathsf{\$} $ 19/22 glassware found in introductory organic laboratories is shown in Figure 4.4.

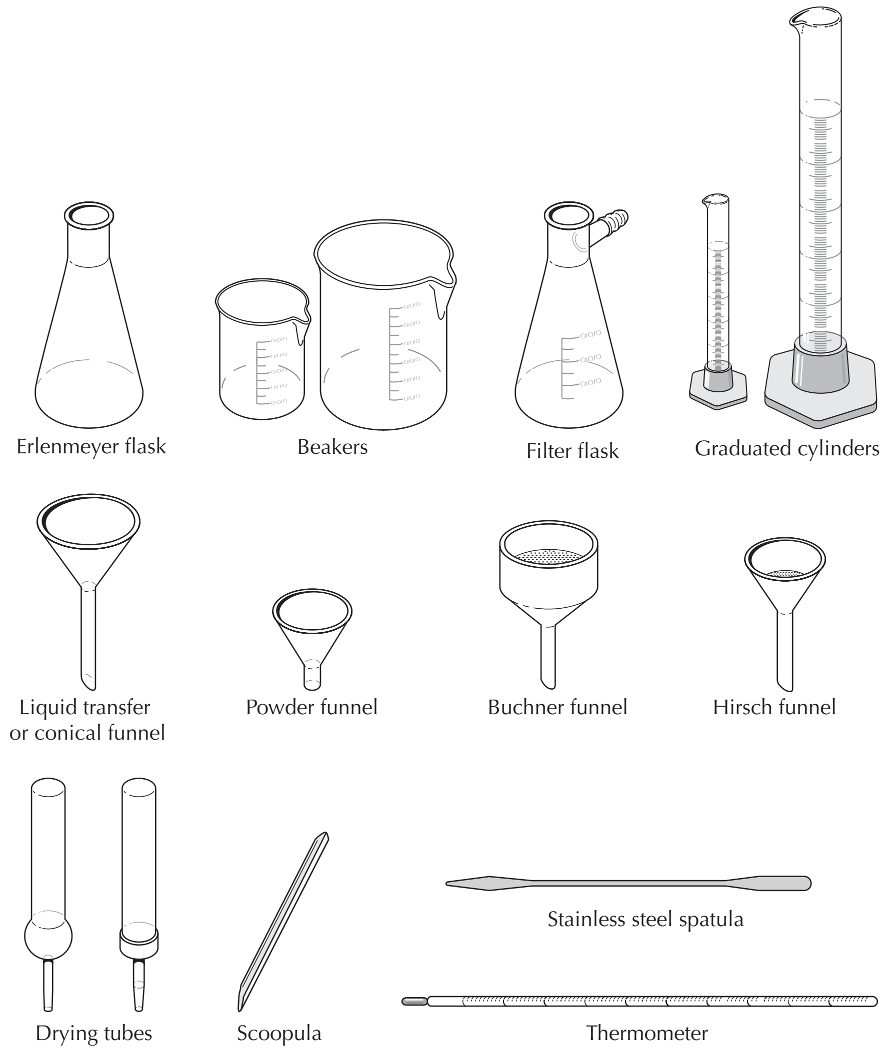

FIGURE 4.2 Typical equipment in a student desk.

Greasing Ground Glass Joints

Because standard taper joints fit together tightly, they are not always put together dry but are often coated with a lubricating grease. The grease prevents interaction of the ground glass joints with the chemicals used in the experiment that can cause the joints to "freeze," or stick together. Taking apart stuck joints, although not impossible, is often not an easy task, and standard taper glassware (which is expensive) frequently is broken in the process. **Note:** Microscale glassware with ground glass joints is never greased unless the reaction involves strong bases such as sodium hydroxide or sodium methoxide.

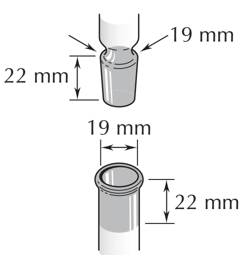

FIGURE 4.3 Dimensions of $ 19/22 ground glass joints.

Types of grease for $ joints. Several greases are commercially available. For general purposes in an undergraduate laboratory, a hydrocarbon grease, such as Lubriseal, is preferred because it can be

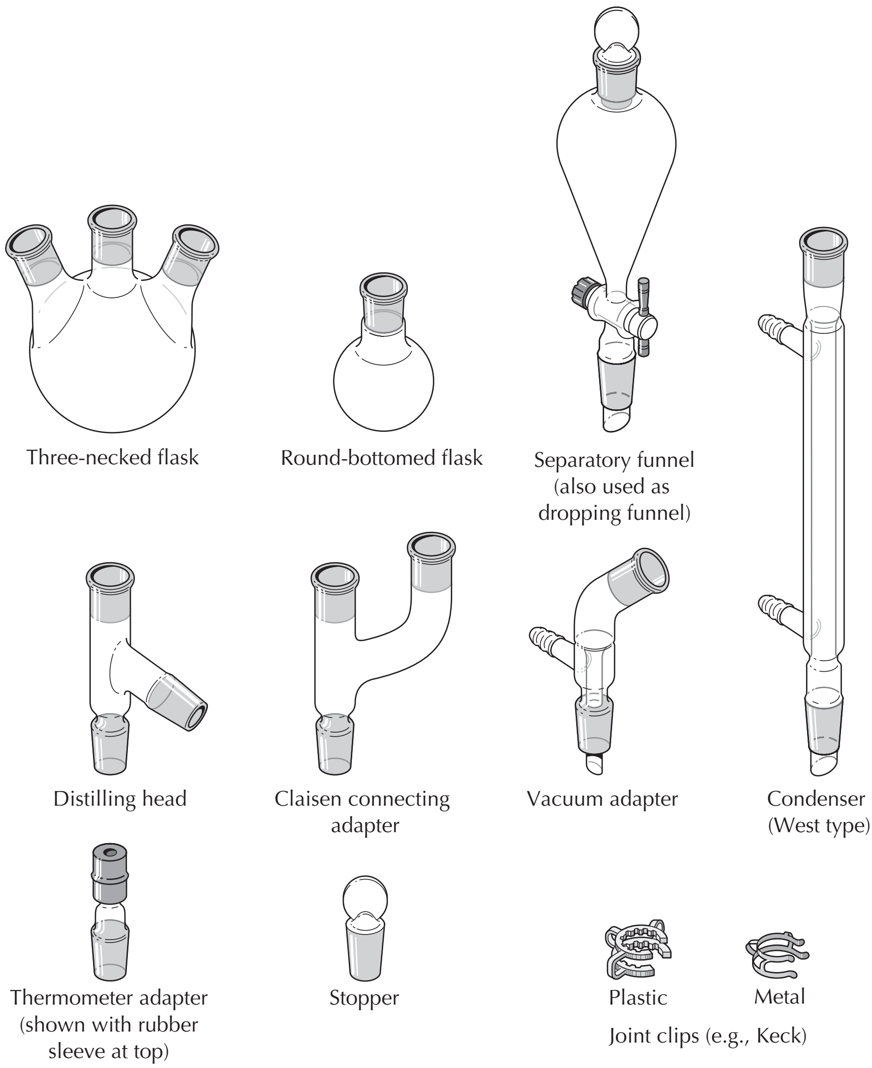

FIGURE 4.4 Standard taper glassware for miniscale experiments.

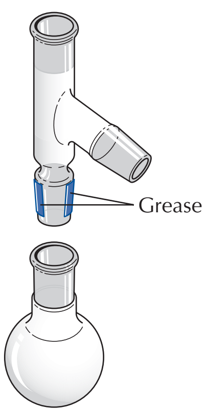

FIGURE 4.5 Apply two *thin* strips of grease almost the entire length of the inner joint about 180° apart.

removed easily. Silicone greases have a very low vapor pressure and are intended for sealing a system that will be under vacuum. Silicone greases are nearly impossible to remove completely because they do not dissolve in detergents or organic solvents.

Sealing a standard taper joint with grease. To seal a standard taper joint, apply two thin strips of grease almost the entire length of the inner joint about 180° apart, as shown in Figure 4.5. Gently insert the inner joint into the outer joint and rotate one of the pieces. The joint should rotate easily and the grease should become uniformly distributed so that the frosted surfaces appear clear.

Using excess grease is bad practice. Not only is it messy, but worse, it may contaminate the reaction or coat the inside of reaction flasks, making them difficult to clean. Just enough grease to coat the entire ground surface thinly is sufficient. If grease oozes above the top or below the bottom of the joint, you have used too much. Take the joint apart, wipe off the excess grease with a towel or tissue, and assemble the pieces again.

Removing grease from standard taper joints. When you have finished an experiment, clean the grease from the joints by using a brush, detergent, and hot water. If this scrubbing does not remove all the grease, dry the joint and clean it with a towel (for example, a Kimwipe) moistened with toluene or hexane.

SAFETY PRECAUTION

Toluene and hexane are irritants and pose a fire hazard. Wear gloves and work in a hood. Place the spent solvent in the appropriate waste container.

4.3 Microscale Glassware

When the amounts of reagents used for experiments are in the 100–300-mg or 0.1–2.0-mL range, microscale glassware is used. Recovering any product from an operation at this scale would be difficult if you were using 19/22 or 14/20 standard taper glassware; much of the material would be lost on the glass surfaces. Two types of microscale glassware are commonly used in undergraduate organic laboratories—standard taper glassware with threaded screw cap connectors or Kontes/Williamson glassware that fastens together with flexible elastomeric connectors. Your instructor will tell you which type of microscale glassware is used in your laboratory.

Standard Taper Microscale Glassware

The pieces of microscale standard taper glassware needed for typical experiments in the introductory organic laboratory are shown in Figure 4.6. The pieces fit together with 14/10 standard taper joints.

Grease is NOT used with microscale glassware, except when the reaction mixture contains a strong base, because its presence could cause significant contamination of the reaction mixture. Instead, a threaded cap and O-ring ensure a tight seal and hold the pieces together, thus eliminating the use of clamps or joint clips. Place the threaded cap over the inner joint; then slip the O-ring over the tapered portion. Fit the inner joint inside the outer joint and screw the threaded cap tightly onto the outer joint (Figure 4.7). A securely screwed connection effectively prevents the escape of vapors and is also vacuum tight.

Kontes/Williamson Microscale Glassware

The various pieces of Kontes/Williamson microscale glassware used in typical experiments in the organic laboratory are shown in

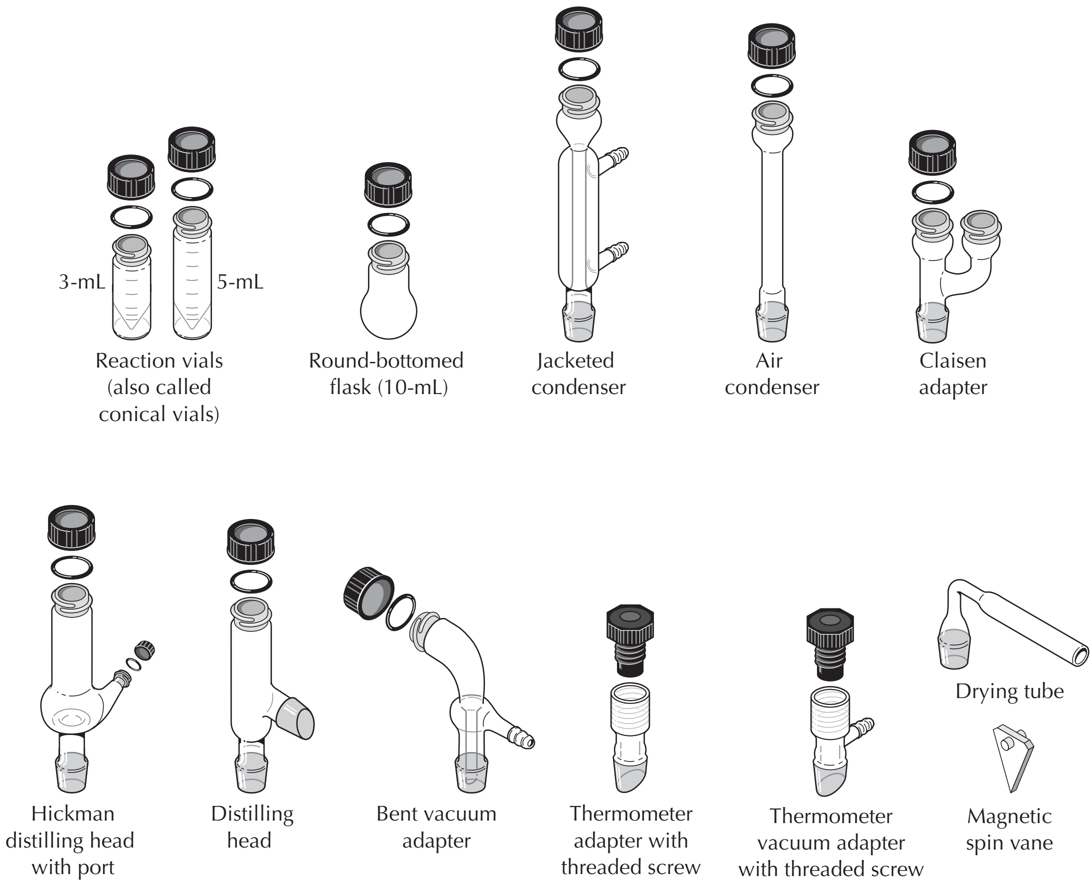

FIGURE 4.6 Standard taper microscale glassware.

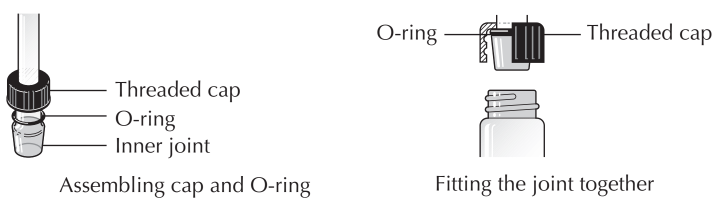

FIGURE 4.7 Assembling a standard taper joint on standard taper microscale glassware.

Figure 4.8. This type of microscale glassware fits together with flexible elastomeric connectors that are heat and solvent resistant.

Grease is NOT used with this type of glassware connector. A flexible connector with an aluminum support rod fastens two pieces of glassware together and provides attachment of the apparatus by way of a two-way clamp to a ring stand or vertical support rod. One piece of glassware is pushed into the flexible connector, and then the second piece is pushed into the other end of the connector, as shown in Figure 4.9. The flexible connector effectively seals the joint and prevents the escape of vapors.

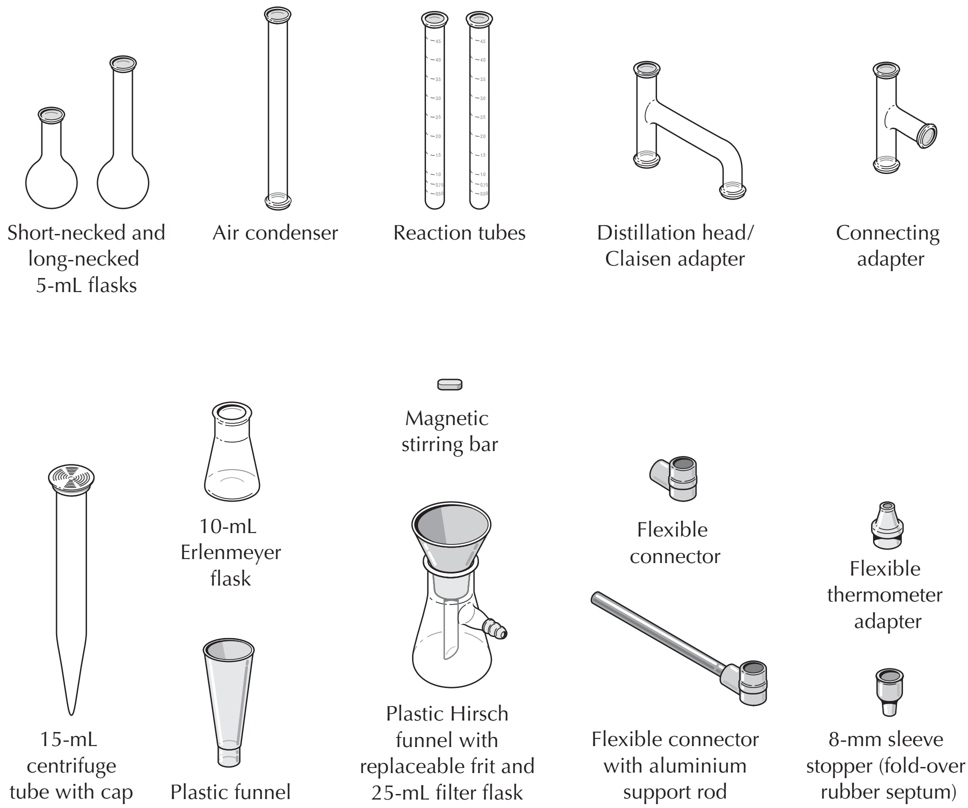

FIGURE 4.8 Williamson microscale glassware and other microscale apparatus. (Manufactured by Kontes Glass Co., Vineland, NJ.)

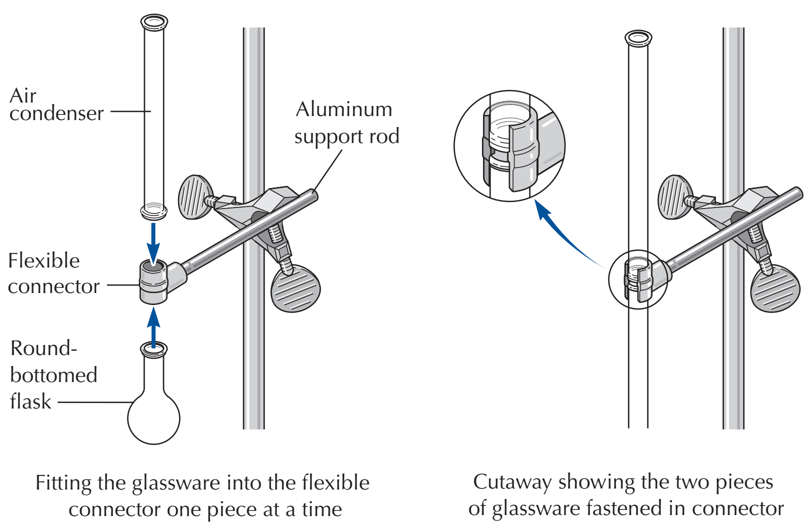

FIGURE 4.9 Assembling Williamson microscale glassware with a flexible connector.

4.4 Cleaning and Drying Laboratory Glassware

Part of effective laboratory technique includes cleaning the glassware before you leave the laboratory, a practice that ultimately saves time and reduces everyone's exposure to chemicals. Clean glassware is essential for maximizing the yield in any organic reaction, and in many instances glassware also must be dry. Try not to have to wash something immediately before using it, because then you will waste time while it dries in the oven.

Cleaning Glassware

Strong detergents and hot water are the ingredients needed to clean most glassware used for organic reactions. Scrubbing with a paste made from water and scouring powder, such as Ajax or Bon Ami, removes many organic residues from glassware. Organic solvents, such as acetone or hexane, help dissolve the polymeric tars that sometimes coat the inside of a flask after a distillation. You may want to wear gloves when cleaning glassware. A final rinse of clean glassware with distilled water prevents water spots.

SAFETY PRECAUTION

Solvents such as acetone and hexane are irritants and flammable. Wear gloves, use the solvents in a hood, and dispose of them in the flammable (nonhalogenated) waste container.

A solution of alcoholic sodium hydroxide* is usually an effective cleanser for removing grease and organic residues from flasks and other glassware.

SAFETY PRECAUTION

Strong bases, such as sodium hydroxide, cause severe burns and eye damage. Skin contact with alkali solutions starts as a slippery feel to the skin followed by irritation. Wash the affected area with copious amounts of water. Wear gloves and eye protection while cleaning glassware with alcoholic NaOH solution.

Drying Glassware

Dry glassware is needed for most organic reactions. The easiest way to ensure dry glassware is to leave all glassware washed and clean at the end of each lab session. It will be dry and ready to use by the next laboratory period.

Oven drying of glassware. Wet glassware can be dried by heating it in an oven at 120°C for 20 min. Remove the dried glassware from the oven with tongs and allow it to cool to room temperature before using it for a reaction.

*Made by dissolving 120 g of NaOH in 120 mL of water and diluting to 1 L with 95% ethanol.

Drying wet glassware with acetone. Glassware that is wet from washing can be dried more quickly by rinsing it in a hood with a few milliliters of acetone. Acetone and water are completely miscible, so the water is removed from the glassware. The acetone is collected as flammable (nonhalogenated) waste; any residual acetone on the glassware is allowed to evaporate into the atmosphere. There is an environmental cost, as well as the initial purchase price and later waste disposal costs, in using acetone for drying glassware.

TECHNIQUE 5 MEASUREMENTS AND TRANSFERRING REAGENTS

Whether you are carrying out miniscale or microscale experiments, you need to accurately measure both solid and liquid reagents as well as the temperature in reaction and purification procedures. Methods for weighing solids and liquids, measuring liquid volumes, transferring solids and liquids without loss, and measuring temperature are described in this chapter.

5.1 Using Electronic Balances

Your laboratory is probably equipped with several types of electronic balances for weighing reagents. How do you decide which one to use to determine the mass of a reagent or product? As a general rule, a top-loading balance that weighs to the nearest centigram (0.01 g) is satisfactory for miniscale reactions using more than 2–3 g of a substance. However, in miniscale reactions where reagent quantities of less than 2 g are used, as well as for the small quantities of reagents used in microscale reactions (100–300 mg), all reagent quantities should be determined on a balance that weighs to the nearest milligram (0.001 g). A top-loading milligram balance has a draft shield to prevent air currents from disturbing the weighing pan while a sample is being weighed (Figure 5.1a).

When a quantity of less than 50 mg is required in a microscale reaction, its mass should be determined on an analytical balance (Figure 5.1b) that weighs to the nearest 0.1 mg (0.0001 g). Close the doors of the balance while weighing the sample.

Care of Electronic Balances

Electronic top-loading and analytical balances are expensive precision instruments that can be rendered inaccurate very easily by corrosion from spilled reagents. If anything spills on the balance or the weighing pan, clean it up immediately. Notify your instructor right away if the spill is extensive or the substance is corrosive.