MANUAL For Organic Chemistry Glassware

INTRODUCTION

Now that you are the proud possessor of an "SGA" Compact 2-Layer Kit*, not only do you have the finest glassware obtainable anywhere, but you'll be able to appreciate the many exclusive built-in features of the molded polystyrene case — designed for your convenience, namely:

- Compact . . . easy to carry, easy to store.
- 2-Layer design requires 25% less space than other kits of similar type.
- Light in weight . . . but engineered for strength and safe handling.
- Pocket in cover — for filing manual.
- Extra cavities — for storing small articles.
- Slot in tray accommodates stock thermometer (available from us).
- Special neoprene (rubber) connectors for thermometers.
- Drying tube standardly supplied.

Prior to using your "SGA" Compact 2-Layer Kit, we suggest that you take time to examine both the case and the contents. Notice particularly the fine workmanship of the glassware — fashioned by our craftsmen from heat-resistant borosilicate glass.

NOTE — After opening the case, remove the tray (with the adapters and tubes) and *place it into the recess provided inside the cover.* All of the glassware items will thus be easily accessible.

*Patent Pending

⅋ = STANDARD TAPER GROUND JOINTS

The ⅋ symbol on the glassware in this kit indicates that the ground joints comply with Commercial Standard CS-21 of the National Bureau of Standards. Ground joints made to these specifications have a "⅋" (standard taper) of 1 mm per 10 mm length. The symbol ⅋ (standard taper) is followed by the size of the ground joint; thus the 19 represents the large diameter and the 22 is the length of the ground joint which is 22 mm. All ground joints in this kit are ⅋ 19/22 size.

METHODS OF ASSEMBLING GROUND JOINT GLASSWARE

To begin with, before assembling any of the equipment, be sure to remove any dirt or foreign matter from the ground surfaces of the joint members.

There are several methods that can be used to assemble ground joint glassware. For example, you can apply a thin coating of lubricont (SGA S-8090 or similar) around the upper end of the *inner* joint member, working the two parts back and forth to obtain uniform distribution. Too much lubricant should be avoided becouse it can lead to serious contamination of reaction solutions; in fact, it actually increases the chance of frozen joints.

Often it is undesirable to use lubricants of any kind. If this is the case, here is the method we recommend for assembling ground joints: Hold the *outer member* stationary. Insert the *inner member* gently until both are well seated. Now apply force gently and simultaneously exert a slight twist of the inner member *to the right.* If properly done, you'll find it is difficult to pull the joints apart; however, a separation is quickly and easily effected by simply twisting the inner member slightly outward and *to the left.*

Should it be more convenient to twist the inner member to the left in assembling, simply reverse the procedure in disassembling and twist to the right and separate. This is a simple method of

assembly and is adequate for most laboratory set-ups. Some practice may be required to master this method, but it will save time for it eliminates the need of lubricating joints and the subsequent cleaning thereof.

CAUTION: Do not use too great a force when inserting one joint into another, and do not rotate one joint inside another. Slight pressure, coupled with a very slight twist, is all that is required. If either is excessive, the joints will stick and can not be separated easily.

FROZEN JOINTS

"Frozen Joints" is the expression used to describe difficulty being experienced in separating joints at the end of an experiment. A simple way to overcome the problem is to gently heat the *outer joint* with steam or a luminous bunsen flame. This will cause the outer joint to expand, resulting in the separation of the two parts; or you can soak the joint in a strong detergent solution (SGA G-9040). It's a little more time-consuming, but remarkably effective.

To avoid frozen joints in those operations likely to couse this difficulty, we recommend careful use of a lubricant prior to the experiment. Upon completion, the lubricant is easily removed with a towel moistened with an appropriate solvent. For high vacuum work you require a grease with a very low vapor pressure, e.g., silicone.

CONDENSERS

There are two condensers in each kit. The one with the smallest diameter is the West-type. The larger diameter type also doubles as a distilling column and is pravided with indentations to support packing material. Examples of efficient packing materials are stainless steel sponge (SGA S-5190) and helices (SGA JD-5360 glass or SGA JD-5420 stainless steel). You will notice that on

both condensers the water inlet and outlet connections have been placed at the extreme ends of the jackets to assure maximum efficiency.

The West-type condenser has been designed so that when used at an angle, the water inlet and outlet will be at the rear and approximately 15° below horizontal. This prevents the water hoses from collapsing, insures that the condenser jacket will fill, eliminates air pockets, and maximizes the cooling effect on the inner tube. Please note that if the condenser is correctly clamped, the drip will be *downward.* Either condenser can be used without water, in which case the interior of the jacket should be *dry.* When using the condenser in series, the upper one should be water cooled, with the lower one acting as an air condenser.

SPECIAL NEOPRENE CONNECTORS

Your set is provided with several sleeve-type neoprene (rubber) connectors, permitting you to adjust the position of the thermometer or bleed tube. Each has a small hole at one end (6¼ mm) to accommodate a thermometer* or bleed tube (usually 6½-7 mm O.D.). The other end, with a larger opening, serves as a sleeve and fits snugly over the glass adapter. Before inserting, wet thermometer, using water or glycerine to minimize friction. Thermometers or bleed tubes should always be held as close as possible to the opening in the rubber connector so as to prevent breaking these items. In a distillation, the position of the thermometer bulb is important if accurate boiling points are to be recorded. The *top of the mercury bulb* should be just below the center of the opening into the condenser — never above the opening.

*Thermometer tubing is known to vary. If your tubing should fit too loosely, a few turns of a rubber band on the outside of the rubber connector will produce a snug fit.

FLASKS

Before using any flask, acquire the habit of inspecting it for cracks or other defects. Also, when you start to apply heat, do it *gradually.* Remember . . . an intense flame may cause strains in the glass and eventual breakage. Never overheat the flask or permit it to become dry during distillations because damage to the flask is apt to occur.

Organic liquids tend to superheat and boil irregularly. When this happens, you will understand why it is called "bumping". This can be minimized by adding small solid pieces known as boiling stones. These are inert, sharp-edged and porous.

TEFLON PLUG

The 125 ml separatory funnel, supplied with some kits, has an interchangeable Teflon* stopcock plug with 1-to-5 taper. Since Teflon has a natural lubricity, no lubricant is required. As Teflon is also chemically inert, it is unaffected by most chemicals. In addition, the Teflon plug features our exclusive "Tite-Seal" design. Coupled with the mirror-smooth inner surface of the stopcock barrel, you are assured of a perfect fit that will neither leak nor freeze. A fine thread on the plug permits adjusting to just the right tension — maintained by an O-ring. (See "Recommendations for Proper Care of Teflon Plugs" enclosed with these kits).

DRYING TUBE

A bent-type drying tube is supplied with each standard "SGA" Kit to prevent desiccants from dropping into the system. One end of the tube has indentations plus a ₹ 19/22 inner joint; the opposite end has a stopper and vent tube.

*duPont Trademark

Suggested methods of using this tube are shown in Figure 1-A and Figure 2-A. Before using the tube, it must be filled with an appropriate drying agent. The indentations and the stopper with vent tube will retain large grain desiccants, but finer grains may require *glass wool* packing at each end.

CLEANING AND DRYING

The best way to clean most soiled glassware is with a warm aqueous detergent solution and an appropriate brush. Some reactions generate a tar residue that is not easily removed, but is generally soluble when soaked in an organic solvent such as acetone, ethanol, kerosene, etc.

Once cleaned, the glassware can be dried by allowing it to stand or by passing *warm* air through it. To speed up this process, the piece can be rinsed with acetone. However, acetone is a very good solvent for polystyrene, the material from which your storage case is made. So if you use acetone, or other organic solvents, take care that it does not contact the polystyrene case. The glassware should be *clean and dry* before you return it to the case. Incidentally, common acids, alkalies, and alcohols do not affect polystyrene.

SAFETY PRECAUTIONS

1. Use reasonable care to avoid accidents.
2. Wear goggles at all times.
3. Use a safety shield when experiments are conducted at reduced pressures.
4. Be sure adequate ventilation is available when you use vapor-emitting solvents.
5. Cuts and burns should be treated immediately.

While it is necessary to carry out some organic reactions in a sealed system, this is never done with equipment such as you have in this kit. You should always be sure that any gases generated in the system can escape to the atmosphere.

ASSEMBLING APPARATUS

On subsequent pages you will see how your set enables you to carry out a variety of chemical operations. Study them carefully.

There is a tendency among beginners to use too many clamps. As a general rule, try to keep the clamps to a minimum. When inner and outer joints are put together properly (see Methods of Assembling Ground Joint Glassware, page 2), they do not separate from one another. However, to minimize the chance of breakage, you can use rubber bands to keep the parts together. In (Fig. 2, page 10), suggested uses of bands are indicated by arrows labeled "R.B.". When a flask having an outer joint is directly beneath a part with an inner joint, it is necessary to support the flask. This is usually done by an iron ring and a wire gauze (see illustrations). Steadiness is provided by traditional clamps and holders, and recommended points of attachment are indicated by arrows marked "Clamp". When two clamps are required, only one need be tight. If you tighten both clamps, you may inadvertently put heavy strains on the setup and this can lead to breakage.

Always remember, the best time to take an apparatus apart is right after the completion of the experiment. Delay may result in frozen joints.

SIMPLE REFLUX

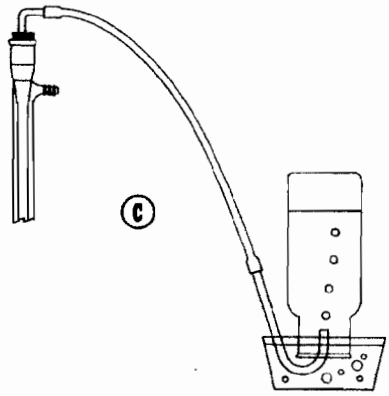
(FIGURE 1)

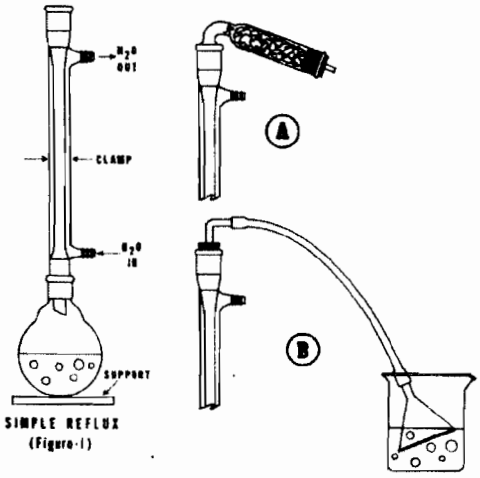
In this setup, a reaction takes place while the solvent is boiling. The vaporized solvent is condensed back to liquid on the water-cooled surface of the condenser and drops back into the boiling solution.

If anhydrous conditions are required, use the drying tube as shown in "A". A freshly filled drying tube is recommended. In this way, you can be sure that you have an open system. After several uses, the drying agent may coalesce and plug up the tube, causing a sealed system. Always check your drying tube for this condition.

Occasionally a gas such as HCl may be liberated as the reaction proceeds. Such water soluble gases are trapped as shown in "B". Note how the position of the funnel prevents sucking the trapping liquid back into the reaction mixture. The rim of the funnel should not be immersed too deeply in the trapping fluid.

If it is desirable to collect a gaseous product, the setup shown in "C" is useful.

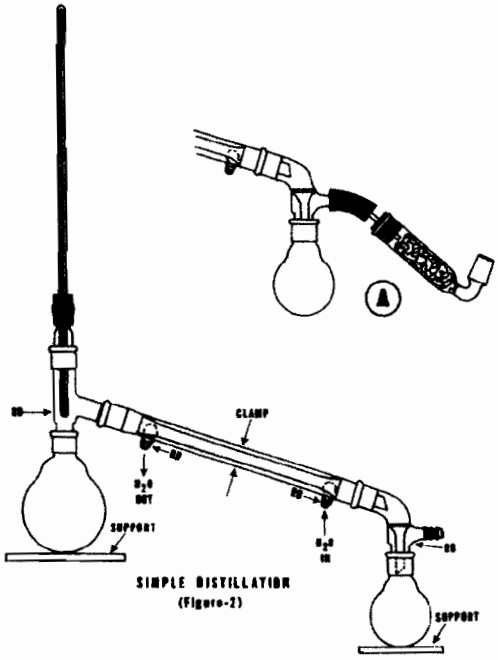




SIMPLE REFLUX
(Figure-1)

SIMPLE DISTILLATION
(FIGURE 2)

In many reactions the products can be separated on the basis of boiling points. The more volatile components are vaporized and condensed first. The condensed vapors, called the distillate, run into the receiver as shown. When the difference in boiling points is large, the simple set-up shown here is adequate. When the product has a boiling point close to other substances in the reaction mixture, it is necessary to use a fractionating column as shown in Figure 3. When a moisture-sensitive product is to be distilled, the atmosphere within the distilling system can be kept anhydrous by attaching the drying tube to the adapter outlet as shown in "A". Be sure the rubber stopper is firmly in place.

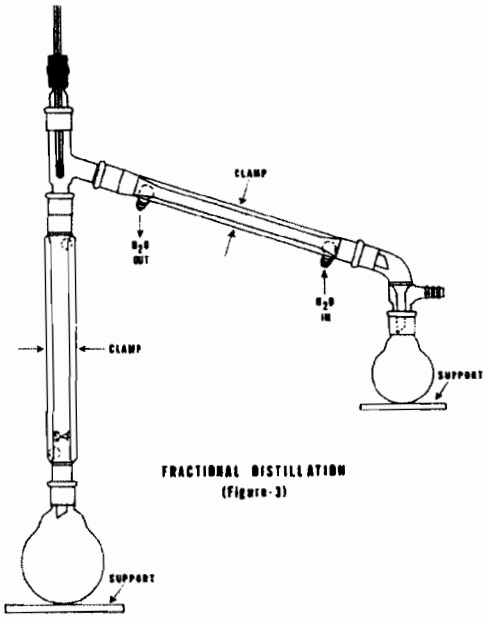


SIMPLE DISTILLATION
(Figure-2)

FRACTIONAL DISTILLATION
(FIGURE 3)

This permits the separation of materials having closely similar boiling points. Better results are obtained if the column is packed with a material which provides a lot of surface. Examples of efficient packing materiol are stainless steel sponge (SGA S-5190) and helices (SGA JD-5360 glass or SGA JD-5420 stainless steel).

Suggested uses of Rubber Bands are noted as "R.B." in Figure 2. Rubber bands can likewise be used in the apparatus shown in Figures 3, 4, 5A and 6.



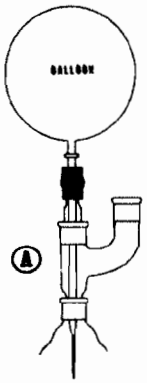
FRACTIONAL DISTILLATION
(Figure-3)

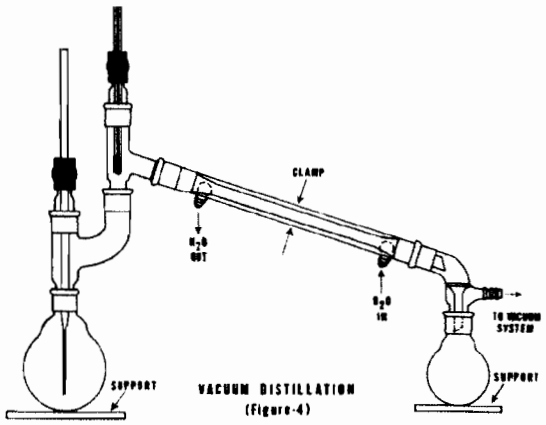
VACUUM DISTILLATION
(FIGURE 4)

The atmospheric boiling point is the temperature at which the vapor pressure of a liquid equals atmospheric pressure. By reducing the gas pressure over a liquid, it is possible to boil the liquid at a lower temperature. Distillations at reduced pressure are called vacuum distillations. The reduced pressure is maintained by a vacuum pump or a water aspirator. In the latter case, a trap should be inserted into the line (between the adapter and aspirator) to prevent water from backing-up into the apparatus.

A bleed tube reaching to the bottom of the distilling liquid is required to minimize superheating and bumping. At reduced pressure, boiling stones soon lose their effectiveness. The bleed tube must be drawn out to an extremely fine capillary. This is then carefully inserted into the rubber adapter and adjusted so that the tip just reaches the bottom of the flask. Your rubber connector is designed to fit 6½ to 7 mm glass tubing.

It is sometimes desirable to distill in an inert atmosphere. In such cases, a balloon filled with dry nitrogen can be put over the upper end of the bleed tube as shown in "A".

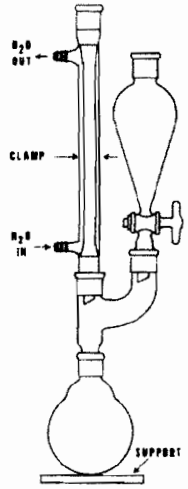




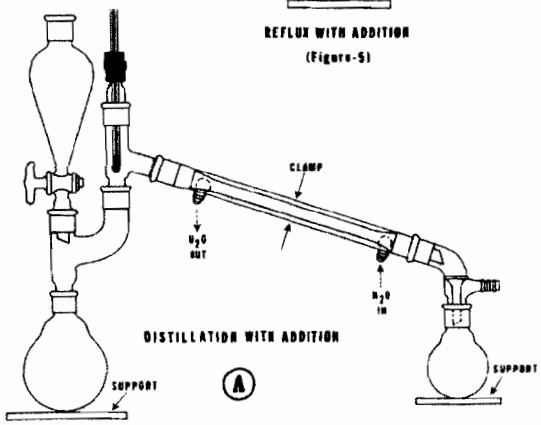
VACUUM DISTILLATION
(Figure-4)

ADDITION
(FIGURE 5)

Often it is advantageous to add one of the reactants slowly while a solution of the other reactant is under reflux. A separatory funnel is used for this, as shown. A similar set-up is possible for addition during a distillation, see "A".



REFLUX WITH ADDITION
(Figure-5)

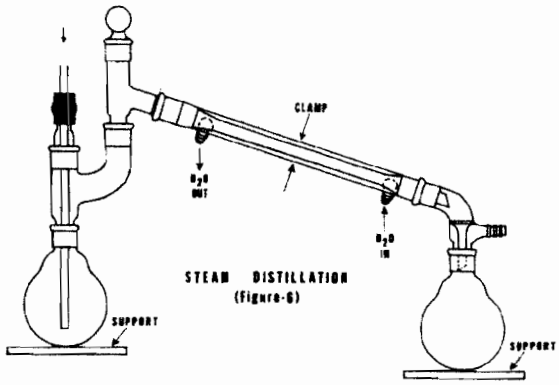


DISTILLATION WITH ADDITION
(A)

STEAM DISTILLATION
(FIGURE 6)

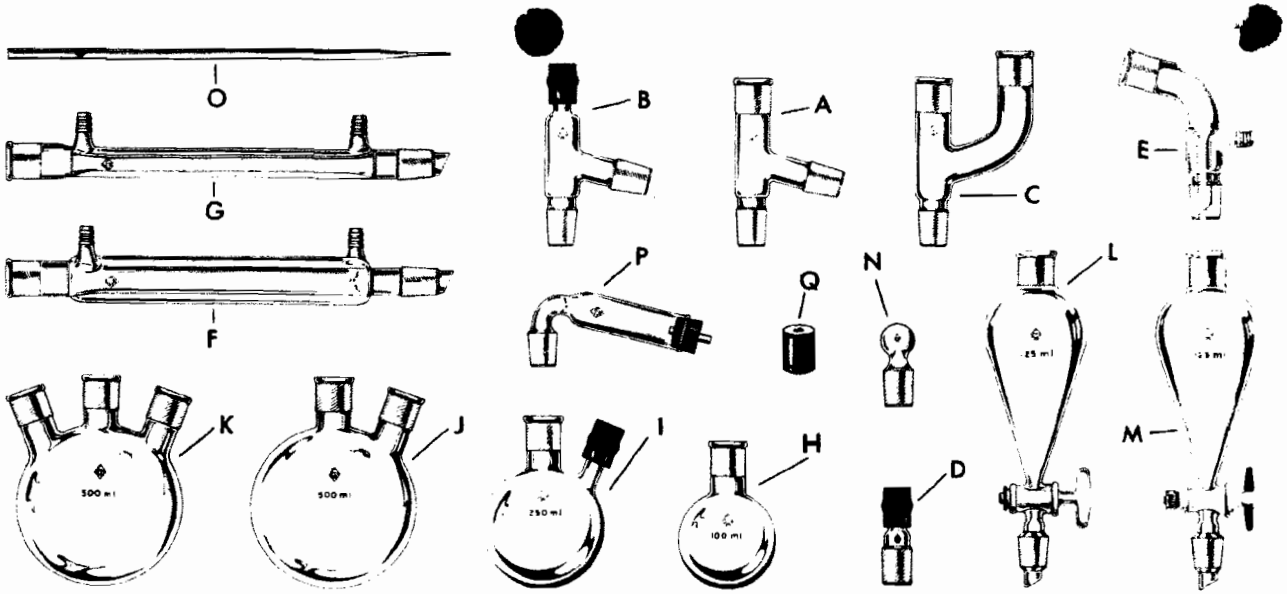
Water soluble compounds can often be distilled by passing steam through them. At the temperature of the steam, the vapor pressure of the compound may be so high that a considerable fraction of the condensed vapor is the desired product. Nitrobenzene is a good example of such a compound.

The apparatus for steam distillation is very similar to the one used for vacuum distillation. The significant difference lies in the size of the bleed-in tube. In this case, the lower end of the tube is not drown out to a fine capillary. It is *not* necessary to use a thermometer in this set-up.



STEAM DISTILLATION
(Figure-6)

The illustrations shown and explained are only examples of popular experiments. Many more set-ups can be made to conduct other experiments with the glassware in this kit.



PARTS LIST - "SGA" COMPACT 2-LAYER KITS WITH ₹ 19/22 JOINTS

ITEM	CAT. NO.	EACH	DESCRIPTION	CATALOG NOS. JM-1380X (Kit No. 1)	JM-1381X (Kit No. 2)	JM-1382X (Kit No. 3)	JM-1383X (Kit No. 4)
A	JM-1882X	$ 5.25	Adapter, Connecting, 75° angle	1	—	1	1
B	JM-1832X	4.25	Adapter, thermometer, con. 75° angle	—	1	—	—
C	JM-1572X	5.80	Adapter, Claisen	1	1	1	1
D	JM-2012X	1.75	Adapter, Outlet, with connector	1	1	1	1
E	JM-2162X	4.90	Adapter, Vacuum	1	1	1	1
F	JM-3420X	7.00	Condenser, Column	1	1	1	1
G	JM-3486X	7.10	Condenser, West	1	1	1	1
H	JM-5435X	2.15	Flask, 25 ml, round bottom	1	1	1	1
H	JM-5435X	2.15	Flask, 50 ml, round bottom	1	1	1	1
H	JM-5435X	2.15	Flask, 100 ml, round bottom	1	1	1	1
H	JM-5435X	2.50	Flask, 250 ml, round bottom	1	1	—	—
H	JM-5435X	2.70	Flask, 500 ml, round bottom	1	1	—	—
I	JM-5532X	3.15	Flask, 250 ml, with side tube & connector	—	—	1	1
J	JM-5544X	4.50	Flask, 500 ml, two neck	—	—	—	1
K	JM-5870X	6.25	Flask, 500 ml, three neck	—	—	1	—
L	JM-6438X	8.60	Funnel, Squibb, 125 ml, glass plug	1	1	—	—
M	JM-6439X	10.70	Funnel, Squibb, 125 ml, Teflon Plug	—	—	1	1
N	JM-7306X	1.30	Stopper, solid	1	1	1	1
O	JM-7745X	.35	Tube, bleed	1	1	1	1
P	JM-1438X	2.25	Tube, drying with stopper & vent tube	1	1	1	1
Q	JM-7014X	.15	Neoprene connector	1	2	2	2