



Distillation Apparatus

CG-0800/CG-0801

INSTRUCTION SHEET

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ABOUT THIS MANUAL

The following symbols are used in this guide:



This symbol indicates a CAUTION. Cautions warn against actions that can cause damage to equipment. Please read these carefully.



This symbol indicates a WARNING. Warnings alert you to actions that can cause personal injury or pose a physical threat. Please read these carefully.

NOTES and TIPS contain helpful information.



Fig. 1—The CG-0801 Student Distillation Apparatus contains everything shown here. CG-0800 does not include the stand and clamp.

HOW IT WORKS

The principle of distillation is fairly simple, although the practice may sometimes involve a great deal of work. No two liquids have exactly the same boiling point. Therefore, it is possible to separate any mixture of two liquids by raising the temperature of the mixture to a point where one of the liquids will boil but the other will remain a liquid.

By condensing the resulting vapor, it is possible to end with two containers, each containing a relatively pure liquid. While this is true in theory, in practice, the case is somewhat different. As long as there is a fairly high concentration of the liquid with the lower boiling point in the initial mixture the mixture will not become any hotter than that temperature. However, the more volatile liquid boils away, and the concentration drops. A point will be reached where there is not enough of the volatile liquid to carry away the heat, and the solution will quickly reach the boiling point of the other liquid in the mixture. This forces us to make a choice between halting the distillation process before the two liquids are completely separated, or carrying on to the point where some of the higher boiling point liquid will be evaporated and condensed, thereby contaminating the distillate.

In practice, there are several ways around this problem.

- Provide a means of adding more of the mixture to the boiling flask to keep enough of the lower boiling point liquid in the solution.
- Closely monitor the temperature of the vapor at the top of the distillation column, and turn the heat down whenever the temperature begins to climb.
- Accept that there will be some contamination of the distillate, and use some other means of separation for the final purification.

These methods work well when there is a sufficient difference in the boiling points of the two liquids. However, when the difference is slight, or whenever there are several components present that have similar boiling points, other methods must be used. One of these methods is fractional distillation, which relies not on the boiling point, but upon relative density of the resulting vapors. When several components in a mixture have differing boiling points, the one with the lowest boiling point will have a lighter vapor than the second, which will have a lighter vapor than the third, and so on.

If the vapors are collected in a tall vertical column, therefore, the first component will rise to the top of the column, with each succeeding component below it. It is then possible to draw the various fractions off of the column by means of valves at appropriate levels. This is the method sometimes used in oil refineries.

In laboratory use, such columns are used when extreme purity is required, or when the boiling points of the compounds are close. A newer method used a similar column, but performs the actual separation by means of catalysts.

NOTES

The two activities described herein are not intended to limit the instructor, or to show all the possibilities for the use of this apparatus. The instructor will undoubtedly find other uses for the apparatus, and should feel free to modify the activities described. THIS IS LIKE ANY APPARATUS USED IN THE CHEMISTRY CLASSROOM. CARE MUST BE TAKEN TO INSURE SAFE OPERATION. IT IS THE RESPONSIBILITY OF THE INSTRUCTOR TO TAKE ALL NECESSARY PRECAUTIONS.

It is advised that any demonstrations be carried out at least once by the instructor prior to using it in the classroom, both to insure that it works as expected, and to discover any unique scenarios. Time the activity carefully, and make certain that there is sufficient class time to complete the procedure.

PARTS LIST

This student distilling apparatus contains:

- (1) 250mL round-bottom boiling flask
- (1) Liebig condenser-300mm
- (1) 3-way standard taper adapter
- (1) 250mL collection flask (Erlenmeyer type)
- (1) Support Stand and Clamp (CG-0801 apparatus only)

NOTE: In addition, you will also need a heating mantel or metal tripod base, wire gauze heating square, a thermometer and tubing which are not included in the kit.

SETUP

1. Obtain the appropriate sized heating mantel or use a metal tripod base (not included).
2. If you are not using the provided round bottom boiling flask, place a wire gauze heating square (not included) on the tripod base stand. The wire gauze is not necessary for the round bottom type flasks.
3. Attach the metal rotating clamp to the support stand rod (included in CG-0801).
4. Attach the condenser to the support rod using the clamp.



CAUTION: To avoid breaking the condenser, ensure the condenser is securely fastened and the stand assembly is stable.

5. Adjust the height of the condenser so that it is level with the boiling flask.
6. Place the 3-way adapter into the boiling flask, and then connect it to the condenser tube.

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- Place the collection flask in the position to collect the fraction from the end of the condenser tube.

NOTE: A 250mL Erlenmeyer type flask is included, but any size flask is suitable.

- Attach $\frac{1}{4}$ " ID, $\frac{7}{16}$ " OD silicone water supply tubing to the bottom supply inlet of the condenser and to the drain at the top inlet.
- Suspend a thermometer in the boiling flask so that it will hang just above the level of the solution in the flask. The apparatus is ready for use.

DEMONSTRATION OF DISTILLATION

Choose two liquids which have distinct and widely separate boiling points for the demonstration. In the demonstration, we will distill the liquid with the lower boiling point out of the other liquid. Since the vapor temperature of the liquid in the boiling flask is monitored, one option is to choose water as the liquid with the higher boiling point. This way, the temperature of the liquids may be roughly determined by the activity of the water.

Little observable bubbling is produced when a large amount of water is used with a small amount of another liquid and the temperature is kept below 100°C. Depending on the availability of materials and safety considerations, ammonium hydroxide or ethanol may be used with water.



WARNING: THESE COMPOUNDS ARE FLAMMABLE! WHEN WORKING WITH THEM, WE RECOMMEND USING AN ELECTRIC HEATING MANTEL RATHER THAN OPEN FLAME (FOR EXAMPLE, A BUNSEN BURNER). THE USE OF A HEATING MANTEL/BLOCK ALSO ALLOWS FOR BETTER CONTROL OF THE TEMPERATURE, ENSURING MORE EFFICIENT DISTILLATION.

Another possibility for a demonstration is to use water with a dilute acid, such as acetic. Acetic acid has a higher boiling point than water. When using such a compound the boiling point of the water is lower than that of the acid. You can maintain the temperature by observing the boiling of the liquid in the flask and decreasing the heat slightly whenever the liquid begins to boil vigorously.

Distillation of Ethanol

- Set up the apparatus as described in "Setup" on page 3, but replace the wire gauze and tripod with a heating mantel.
- Adjust the apparatus so that all the connections are tight.
- Remove the boiling flask, and place 100mL of ethanol and 100mL of distilled water in the flask.
- Place the boiling flask on the heating mantel. Set the heating mantel to about 80°C. (Ethanol boils at 78°C.)

CG-0800/0801 (Student Distillation Apparatus)

5. Turn on the water to the condensing column, and adjust the flow so that there are no bubbles visible inside the condenser tube.
6. Closely monitor the heating liquid. When bubbles begin to form, turn the temperature up to 85°C.
7. As the ethanol begins to boil, the vapors slowly make their way into the 3-way adapter. At this point some of the vapor condenses and runs back into the boiling flask.
8. As the temperature of the vapor increases, the vapor begins to enter the condensing column and drops of distillate collect in the Erlenmeyer flask.
9. When the volume in the boiling flask is reduced to half the original volume, turn off the heating mantle and allow the apparatus to cool.
10. When the system is cool, remove the distillate flask and demonstrate that the flask contains pure alcohol.

Distillation of Acetic Acid

1. Set up the apparatus as described in “Setup” on page 3. In this case there is no danger of fire, so the use of a heating mantle is not required, however if one is available, use it.
2. Remove the boiling flask and place in 100mL of water and 100mL of glacial acetic acid.



WARNING: ALWAYS USE CAUTION WHEN HANDLING ACID. ALWAYS ADD ACID TO WATER, NOT WATER TO ACID!

3. Have the students test the diluted acetic acid for pH, using a pH meter or pH test strips.
4. Reposition the boiling flask on the tripod stand.

NOTE: Use the wire gauze when using a flat bottom type boiling flask, and verify that all connections are tight.

5. Begin heating the dilute acid. (If you are using a heating mantle, set the temperature at about 100°C or slightly higher.)
6. When the volume in the boiling flask is reduced to half the original volume, turn off the heat and allow the apparatus to cool.
7. Have the students test both the distillate and the liquid in the boiling flask for pH. The distillate should be mostly distilled water, with a pH of about 7. The solution in the boiling flask should now be a more concentrated acid, with a lower pH.

REPLACEMENT PARTS

- CG-0273** 250mL Round Bottom Boiling Flask with 24/40 joints
- CG-0103** 250mL Erlenmeyer Flask
- 5459** Clamp for ring stand, 10-35mm jaws
- 503084** Small 6" V-Base/Frameworks
- 503075** Stainless Steel Pole, 12mm OD, 60cm long, M8 thread

Optional Parts

- CG-0274** 500mL Round Bottom Boiling Flask with 24/40 joints
- CG-0102** 125mL Erlenmeyer Flask
- CG-0282** 250mL Flat Bottom Boiling Flask with 24/40 joints
- CG-0283** 500mL Flat Bottom Boiling Flask with 24/40 joints
- CG-0112** 250mL Erlenmeyer Flask with 24/40 joints
- 502147** Triangular metal base with 24" pole
- 503085** Large 10" V-base/Frameworks

WARRANTY

WPI (World Precision Instruments, Inc.) warrants to the original purchaser that this equipment, including its components and parts, shall be free from defects in material and workmanship for a period of 30 days* from the date of receipt. WPI's obligation under this warranty shall be limited to repair or replacement, at WPI's option, of the equipment or defective components or parts upon receipt thereof f.o.b. WPI, Sarasota, Florida U.S.A. Return of a repaired instrument shall be f.o.b. Sarasota.

The above warranty is contingent upon normal usage and does not cover products which have been modified without WPI's approval or which have been subjected to unusual physical or electrical stress or on which the original identification marks have been removed or altered. The above warranty will not apply if adjustment, repair or parts replacement is required because of accident, neglect, misuse, failure of electric power, air conditioning, humidity control, or causes other than normal and ordinary usage.

To the extent that any of its equipment is furnished by a manufacturer other than WPI, the foregoing warranty shall be applicable only to the extent of the warranty furnished by such other manufacturer. This warranty will not apply to appearance terms, such as knobs, handles, dials or the like.

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Claims and Returns

Inspect all shipments upon receipt. Missing cartons or obvious damage to cartons should be noted on the delivery receipt before signing. Concealed loss or damage should be reported at once to the carrier and an inspection requested. All claims for shortage or damage must be made within ten (10) days after receipt of shipment. Claims for lost shipments must be made within thirty (30) days of receipt of invoice or other notification of shipment. Please save damaged or pilfered cartons until claim is settled. In some instances, photographic documentation may be required. Some items are time-sensitive; WPI assumes no extended warranty or any liability for use beyond the date specified on the container

Do not return any goods to us without obtaining prior approval and instructions from our Returns Department. Goods returned (unauthorized) by collect freight may be refused. Goods accepted for restocking will be exchanged or credited to your WPI account. Goods returned which were ordered by customers in error are subject to a 25% restocking charge. Equipment which was built as a special order cannot be returned.

Repairs

Contact our Customer Service Department for assistance in the repair of apparatus. Do not return goods until instructions have been received. Returned items must be securely packed to prevent further damage in transit. The Customer is responsible for paying shipping expenses, including adequate insurance on all items returned for repairs. Identification of the item(s) by model number, name, as well as complete description of the difficulties experienced should be written on the repair purchase order and on a tag attached to the item.

** Electrodes, batteries and other consumable parts are warranted for 30 days only from the date on which the customer receives these items.*



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