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PROFESSOR: While working in the laboratory, you will often be required to separate liquid mixtures or to purify liquid from non-volatile substances. This separation and purification can be done by utilizing the liquid's boiling point through a technique called distillation. Distillation works through the processes of vaporization and condensation. Vaporization is the transformation of a liquid to a gas, and condensation is the conversion of a gaseous species into a liquid.

To perform a distillation, a mixture of two miscible liquids is heated until the liquid with the higher vapor pressure reaches its boiling point. This vapor travels up the inside of the flask, through a distilling head, and into a condenser. The cold water jacket on the condenser transforms the vapor back into liquid, which then travels into the receiving flask. In this manner, distillation effectively separates lower boiling point compounds from higher boiling point compounds.

Three types of distillations are commonly used in the laboratory. Depending on your liquid solution, you will have to decide which type of distillation will be the most effective. The first type of distillation is a simple distillation. This technique is used when separating miscible liquids that boil below 150 degrees Celsius at 1 atmosphere from either non-volatile impurities or from a second liquid that boils at least 25 degrees Celsius higher than the first.

The second type of distillation is the fractional distillation. This technique should be used to separate liquid mixtures that have less than a 25 degree Celsius difference in boiling point. The third type of distillation is a vacuum distillation. This technique should be used to separate miscible liquids that boil above 150 degrees Celsius at 1 atmosphere.

In this video, "Distillation Part 1," we will discuss simple and fractional distillations. In "Distillation Part 2," we will discuss vacuum distillations. To carry out a simple distillation, you will need a cork ring, a round bottom distilling flask, two ring stands, a stir bar and magnetic stirrer, a distilling head, a condenser, a vacuum adapter, grease, 3 yellow keck clamps, a thermometer and thermometer adapter, 3 round bottom receiving flasks, 2 extension clamps and fasteners, a heating mantle, an iron ring, a variable voltage transformer, or Variac; 3 hose clips, 2 pieces of rubber tubing, and a container for an ice water bath.

The first step in performing a distillation is choosing the proper sized distillation flask. The distilling flask should not be more than 1/2 full or less than 1/3 full when the distillation begins. If the flask is overfilled, the solution will likely overheat or bump into the condenser. Bumping will cause impurities to be collected in the receiving flask, rendering the distillation worthless. If the flask is less than 1/3 full, then you'll have a greater chance of losing some product because a substantial amount of the material will be needed just to fill the flask and distilling head with vapor.

While we're on the topic of size, we'd like to speak briefly about heating mantles. If you do not have an appropriately sized heating mantle, then use one that is too big. Never use a heating mantle that is too small because heat is not easily transferred between the mantle and flask due to poor contact, which causes the mantle to burn out. If the mantle is too big for the flask, then add sand to fill in the empty space. After you have chosen a distilling flask, transfer your sample into it and add an appropriately sized stir bar. If you will not have access to a magnetic stirrer, then add a few boiling stones to the distilling flask instead.

The next step is the assembly of the distillation apparatus, which is likely one of the most difficult glassware assemblies you'll ever encounter. The neck of the distilling flask is clamped onto one of the ring stands with an extension clamp and fastener. A heating mantle, which is plugged into the Variac, rests on an iron ring below the distilling flask. If you're using a stir bar, then place a magnetic stirrer underneath the heating mantle. Leave enough room below the iron ring so that the heating mantle can be dropped away from the distilling flask if necessary.

The distilling head is attached to the top of the distilling flask, and a condenser is attached to the side arm of the distilling head. The vacuum adapter is attached to the other end of the condenser, and this joint is attached to the second ring stand with an extension clamp and fastener. A small receiving flask is clamped to the vacuum adapter with the yellow keck clamp, and an ice water bath is placed underneath.

The thermometer and thermometer adapter are attached to the top of the distilling head and clamped in place with the green keck clamp. The top of the thermometer bulb should be aligned with the bottom of the side arm on the distillation head. Yellow keck clamps are used to secure both ends of the condenser. Rubber tubing is attached to the water inlet and outlet of the condenser and secured with hose clips. Finally, place 2 clean, dry receiving flasks near the distillation apparatus. Before continuing, confirm that all of the joints have been lightly greased and that no keck clamps are used on any joint that will become hot.

After assembling the distillation apparatus, attach the tubing at the bottom of the condenser to the water source, place the end of the tubing at the top of the condenser into a sink. Remember, water goes in at the bottom and out at the top to discourage the formation of air bubbles. Double check that all of the joints are secured with hose clips and start the water flow.

The water should flow at the slowest rate that is necessary to keep the condenser cold. If the flow rate is too fast, the chance of the tubing popping off the condenser and spring all over your workspace greatly increases. To perform this simple distillation, turn on the Variac and heat the sample to a gentle boil. Make sure that the vacuum adapter remains to the air. Never heat a closed system, or it will explode.

A ring of condensate will move up the flask and into the distilling head. When the vapor reaches the height of the thermometer bulb, the temperature reading will jump dramatically, indicating the formation of a droplet of liquid on the thermometer bulb. If the condensate ring completely stops rising at any point, slightly increase the setting on the Variac.

After the vapor reaches the condenser, it transforms back into liquid, flows down the condenser and into a small receiving flask. When approximately 10 drops have been collected in the small receiving flask, quickly replace it with the larger one. The first couple of drops of a distillation are always discarded because they may contain some lower boiling impurities.

At this point, the distillate should be dropping into the receiving flask at a rate of 10 drops per minute. If the rate is faster, then the heat is too high, and higher boiling point impurities may have escaped into the receiving flask. If the rate is too slow, then the distillation will occur over too long of a time period.

After the lower boiling liquid has been completely distilled, no drop of liquid will be on the thermometer bulb, and the temperature will plunge dramatically. At this point, immediately replace the large receiving flask with a new, small receiving flask to collect the last few drops of liquid. These drops may contain some higher boiling impurities that should not be collected in the main receiving flask. Record the temperature range of when the mixture first started to boil until right before the dramatic drop in temperature. This is the boiling point of the distillate. If the range is within 2 degrees Celsius, your distillate is fairly pure.

After you have collected the final few drops of liquid, turn off the heat source. A simple distillation is performed more effectively if certain guidelines are followed. First, make sure all the joints on the apparatus are tight and that the water lines are secured. Second, fill the distilling flask between 1/3 and 1/2 full prior to distillation. Third, allow the distillation to proceed at a rate of 10 drops per minute. Fourth, use separate receiving flasks for the beginning, middle, and end of the distillation to disallow low and high boiling impurities into the distillate. Fifth, never let the distillation flask run dry. A flask that is allowed to run dry may overheat and break.

The second type of distillation that we will cover is the fractional distillation. This technique should be used if you need to separate two liquids that have a small difference in boiling point-- less than 25 degrees Celsius. Fractional distillation is more effective at separating liquids with similar boiling points because it utilizes a fractionating column.

A fractionating column contains column packing with a large surface area and works by promoting a lot of tiny distillations on the surface of this packing. Specifically, when the liquid mixture is heated to boiling, the resulting vapor is richer in the lower boiling component. The vapor moves out of the flask and condenses on the bottom few centimeters of the fractionating column.

Now the condensed droplet is richer in lower boiling component, and the liquid in the distilling flask is richer in the higher boiling component. If that liquid droplet is reheated, it will vaporize and condense a little further up the fractionating column. This distillate will have an even higher percentage of the lower boiling component. This process is repeated until the vapor reaches the top of the fractionating column, where theoretically, it should be pure in lower boiling component.

To assemble the apparatus for our fractional distillation, place a fractionating column between the distillation flask and the distillation head. Alternatively, your lab may have all-in-one distillation glassware that contains a fractionating column. Perform the fractional distillation in the same manner as the simple distillation.

To ensure an effective fractional distillation, heat the distilling flask very slowly. Too much heat causes the distillation to occur too rapidly, disallowing the liquid vapor equilibria on the surface of the fractionating column. If too little heat is used, then the column may lose heat faster than it can be warmed up by the vapor, preventing the vapor from reaching the top of the column.

If the column seems to be losing heat faster than it can be warmed up by the vapor, you may insulate the column by wrapping it with glass, wool, or cotton and aluminum foil. As a general rule, a slow steady distillation, where one drop is collected every 2 to 3 seconds, is reasonable. Distillation is an effective technique for separating impure fine liquids. When used correctly, pure contents and good yields are readily attained.

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