FOR DEMONSTRATION OF DISTILLATION

It is best to select two liquids that have distinct and widely separate boiling points for the demonstration. As there is provision for determining the temperature of the liquid in the boiling flask, it is a good idea to choose water as the liquid with the higher boiling point. In this way the temperature of the liquids may be roughly determined by the activity of the water. If a large amount of water is used along with a small amount of another liquid, there will be little observable bubbling as long as the temperature is kept below 100^{o} C. Depending on the availability of materials, and safety considerations, some possible liquids to be used with the water are ammonium hydroxide or ethanol. **NOTE, THESE COMPOUNDS ARE FLAMMABLE. WHEN WORKING WITH THEM IT IS ADVISED THAT AN ELECTRIC HEATING BLOCK BE USED, RATHER THAN AN OPEN FLAME.** The use of a heating block also allows for better control of the temperature, thus ensuring more efficient distillation.

Another possibility for a demonstration is the use of a dilute acid, such as acetic, which has a higher boiling point than water. When using such a compound the water will be distilled out, leaving a more concentrated acid. As the boiling point of water is lower than that of the acid, a correct temperature may be maintained by observing the boiling of the liquid in the flask, and decreasing the heat slightly whenever the liquid begins to boil vigorously.

Following are step-by-step instructions for two distillation activities, one using ethanol and one using acetic acid.

DISTILLATION OF ETHANOL

1. Set up the apparatus as described above, but rather than using the wire gauze on the tripod, place a heating mantle on the tripod.

Adjust the apparatus so that all connections are tight.

2. Remove the boiling flask, and place about 200ml of ethanol and 200ml of distilled water in the flask.

3. Replace the boiling flask in the heating mantle, and set the heating mantle at about 80° C. (Ethanol boils at about 78° C).

4. Turn on the water to the condensing column, and adjust the flow so that there are no bubbles visible.

5. Watch the liquid closely, and when bubbles first start to form turn the temperature up to about 85° C.

6. As the alcohol begins to boil, the vapors will slowly make their way into the distilling bulb. At this point some of the vapor will condense and run back into the boiling flask.

7. As the temperature of the vapor increases, the vapor will begin to enter the condensing column, and drops of distillate will begin to collect in the Erlenmeyer flask.

8. When the volume in the boiling flask is about half of the original volume turn off the heating mantle and allow it to cool.

9. When the apparatus has cooled, remove the distillate flask and demonstrate that the flask contains pure alcohol.

DISTILLATION OF ACETIC ACID

1. Set up the apparatus as described above. In this case there is no danger from fire, so the use of a heating mantle is not required, although it is possible to use one if available.

2. Remove the boiling flask and place in it about 200ml of water and 200ml of glacial acetic acid. **(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 paper.

4. Reposition the boiling flask on the wire gauze (or in the heating mantle) and check the connections for tightness.

5. Begin heating the dilute acid, keeping the solution just boiling. What you want is for the solution to be slightly above a simmer, but not at a full boil. (If using a heating block set the temperature at about 100° C or slightly higher).

6. When the volume in the boiling flask reaches about half of 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, while the solution in the boiling flask should now be a more concentrated acid, with a consequently lower pH.

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 liquid.

By condensing the resulting vapor, it is possible to end up 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 having the lower boiling point in the initial mixture the mixture will not become any hotter than that temperature. However, as 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 two ways around this problem. One is to provide a means of adding more of the mixture to the boiling flask, and so keeping enough of the lower boiling point liquid in the solution. Another is to 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. A third way is to accept that there will be some contamination of the distillate, and use some other means of separation for a 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 when 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 the 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 components are close. A more recent method uses a similar column, but performs the actual separation by means of catalysts.

NOTES

The two activities described above 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. **AS WITH ANY APPARATUS USED IN THE CHEMISTRY CLASSROOM, CARE MUST BE TAKEN TO ENSURE SAFE OPERATION. IT IS THE RESPONSIBILITY OF THE INSTRUCTOR TO TAKE ALL NECESSARY PRECAUTIONS. PROPER PROTECTION SHOULD BE WORN TO PROTECT THE EYES AND SKIN.**

It is advised that any demonstration be carried out at least once by the instructor prior to using it in the classroom, both to ensure that it works as expected and to discover any special problems or equipment. It is a good idea to time the activity carefully, and make certain that there is sufficient class time to complete the procedure.