**Distillation** Separating a Mixture of Liquids

The lab is performed by small groups. Group # _______ Section # _______

This procedure is used to separate volatile substances from non-volatile (or less volatile) substances – or to separate different liquids from each other according to their boiling points. The efficiency of separation depends on the difference in the boiling points and the distance the vapors have to travel in the neck of the boiling flask. A longer neck flask increases the efficiency of the separation. A simple set-up such as this will separate the liquids to a certain extent, but the **distillate** will still be a mixture, somewhat enriched in the lower-boiling component.

**Procedure**

Ethanol and water are the two major volatile components in wine. Pour 250 mL of red wine into the distillation flask, hook up the glassware making sure the connections are tight, turn on the cooling water carefully so the water flows slowly. The thermometer should be adjusted to measure the temperature of the vapors at the point shown in the diagram. Weigh a corked graduated cylinder. Uncork the cylinder and place it to collect the distillate (instead of the Erlenmeyer collection flask shown). Use a heating mantle connected to a power source, instead of a burner. Set the control to HIGH initially, and reduce the power by about 1/3 when the mixture is boiling. Measure the temperature of the vapors when the first drops of distillate come out. Measure the temperature after 5 mL, 10 mL and 15 mL etc every 5 mL until finally 50.0 mL has been collected. Cork the grad cylinder, record the volume carefully to 1 decimal place -- at the bottom of the **meniscus** in the cylinder. Don’t exceed 50 mL since there are no marks above 50 on the cylinder. Carefully sniff the distillate. What do you think? Good stuff? Weigh the corked cylinder with the distillate.

**Data**

<table>
<thead>
<tr>
<th>Volume of Wine Used</th>
<th>Mass of Empty Grad Cylinder &amp; Cork</th>
</tr>
</thead>
</table>

T of vapors at first sign of distillate: _____  T at 20.0 mL _____  T at 40.0 mL _____

T of vapors at 5.0 mL of distillate: _____  T at 25.0 mL _____  T at 45.0 mL _____

T at 10.0 mL _____  T at 30.0 mL _____  T at 50.0 mL _____

T at 15.0 mL _____  T at 35.0 mL _____

Mass of cylinder with distillate: _______

Calculate the mass of the distillate in the cylinder: _______

Calculate the density (g/mL) of the liquid in the cylinder: _______

\[ d = \frac{\text{mass of distillate}}{50.0 \text{ mL}} \]
**Data Analysis.**

Make a graph, plotting the temperature and volume data (T on the y axis, volume 0 – 50 mL on the x axis)

![Graph](image)

**Questions**

1. Look at the graph of temperature vs volume. Assuming the T of pure boiling water is 100°C, Do you think all of the ethanol had boiled out, when 50 mL was collected? If so - at what point do you think all the ethanol had boiled out?

2. The boiling point of pure ethanol is 78°C. At the first sign of condensing vapors (when maybe just one drop of distillate has been collected) - do you think you have pure ethanol? ________ Why or why not?
3. **Graphing** Use a fine graph paper (10 sq/inch or 10/cm) to plot the densities of aqueous ethanol solutions at 20º C (y axis) vs the % composition (x axis).

<table>
<thead>
<tr>
<th>% ethanol</th>
<th>d</th>
<th>% ethanol</th>
<th>d</th>
<th>% ethanol</th>
<th>d</th>
<th>% ethanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
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<td>.902</td>
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<td>50</td>
<td>.913</td>
<td>75</td>
<td>.855</td>
<td>100</td>
</tr>
<tr>
<td>25</td>
<td>.963</td>
<td></td>
<td></td>
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</tbody>
</table>

4. Is the graph linear? _______ Why can’t you use a computer program to find a slope and equation of the line?

5. You have calculated the density of the distillate. Plot the density of the distillate on the graph, and read the % of ethanol/water in the distillate on the x axis ________ % ethanol

6. Assuming all the ethanol did boil out by the time 50 mL had been collected, now you can calculate the % of ethanol in the original wine sample. Since only 50 of the 250 mL of original liquid was distilled, the percent of the ethanol in the wine = % in the distillate x 50/250

\[ \text{=} \quad \text{________%} \]

7. Compare the % of ethanol you calculated above, with the actual % of ethanol in the wine as per the bottle label (or given by the instructor).

Wine brand. read the label % ethanol by volume in wine: ____________ %

Find the % error in the result.

\[ \text{exp. value - true value} \times 100 = \text{% error} \]

\[ \text{________} \text{% error} \]