DISTILLATION : PURIFICATION OF A MIXTURE OF MISCIBLE LIQUIDS

EXPERIMENTAL TECHNIQUES REQUIRED

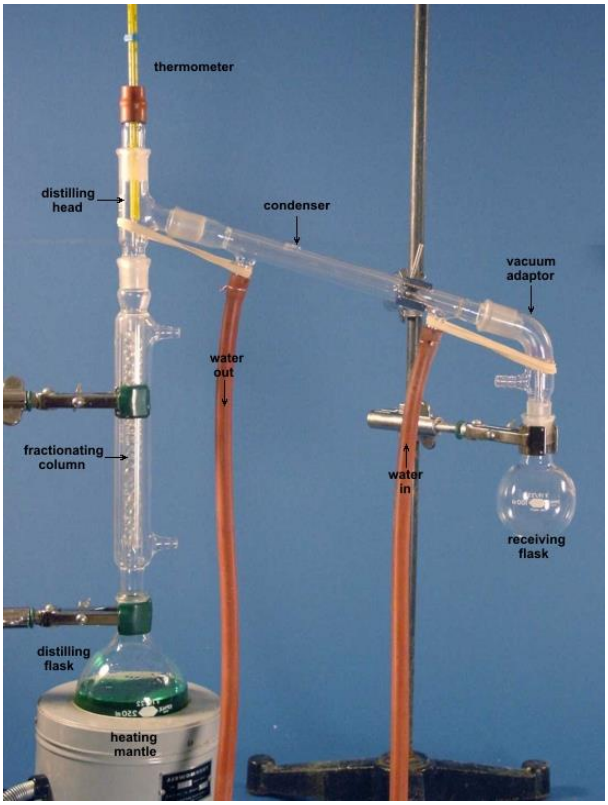
Fractional distillation (T 10), greasing glass joints (T 11), gas chromatography (T 13)

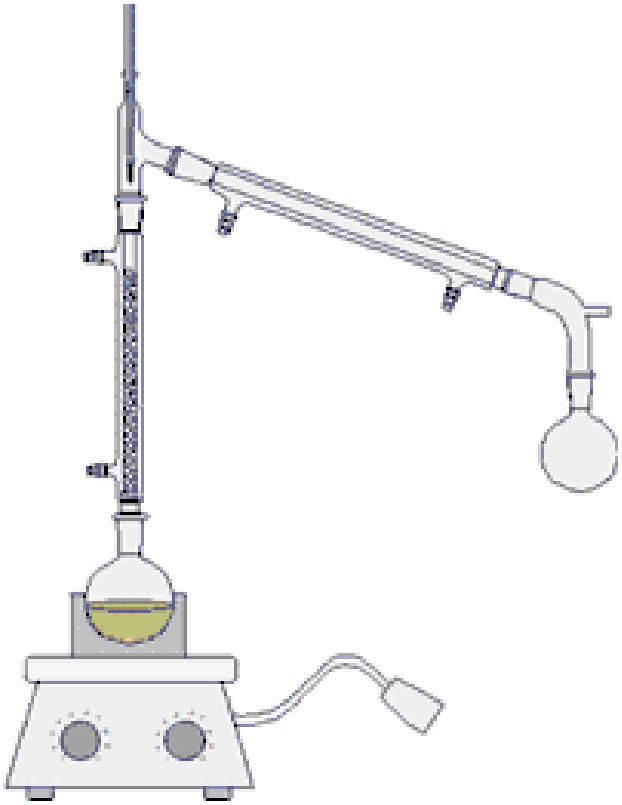
EXPERIMENTAL PROCEDURE

Work individually and write an individual report

- **Make sure you don't distill the distillation flask to dryness.**
- **Work in a fumehood whenever possible.**
- **Pay attention to the position of power cords : keep them away from the hot surfaces of hot plates.**
- **Hot stirrers / metal blocks and hot glassware ! Avoid burns !**

NOTE: In the equipment set up shown in the distillation technique document, a heating mantle with a heating controller to heat the round bottom flask. In your equipment set up (and like the reflux apparatus used in other experiments), the heating mantle and heating controller will be replaced by an engineered (shaped) aluminium block sitting on the top of a stirrer hot plate. This is a more modern version of a heating mantle with some distinct benefits.





Record your unknown sample number. Measure 30-35mL of the sample (*i.e*. the initial sample volume) and transfer it into the "distillation flask", a 50mL round bottom flask (it should be between about 1/2 to 2/3 full).

Assemble the fractional distillation apparatus as described in the distillation techniques document.

Make sure your TA checks the set up before you start to heat the distillation flask.

Start heating the distillation flask with the hotplate / aluminium block, monitor the progress and increasing the heat gradually as required until your mixture starts to boil. When boiling starts, turn down the power slightly but keep an eye on it and readjust if required to keep it boiling gently (note that heating too rapidly will typically reduce the efficiency of the fractionation process and lead to poorer separation = poorer results). You might be able to observe a ring of condensate in the fractionating column (it can also be detected by the carefully touching the outside of the column starting at the top and working down, at the condensate level it will be "hot"). The condensate ring should gradually rise up the column as heating continues. If the condensate stops rising, increase the heat a little. Once the condensate ring reaches the top of the column and the distillation head, the first fraction will start to collect. Continue to fractionate the unknown mixture, recording the temperature at the distillation head after each ml of condensate is collected. When the temperature starts to increase rapidly, record the volume collected, transfer the condensate to a flask, and quickly replace the measuring cylinder to collect the second fraction. Once fractionation is complete, make a preliminary identification of the components based on the boiling points of the fractions. From a graph of temperature vs the *total volume* of distillate collected for the distillation of the mixture, estimate the composition of the mixture in terms of the identity of components and their relative proportions.

Test the water solubility of each of your fractions. Place two drops of the liquid in a small test tube and add about 0.5 mL of water - carefully shake the test tube to ensure mixing. If the compound dissolves completely then you should see only one layer. If the sample only dissolves partially you may see a swirling appearance in the water, due to change in the refractive index, this is an indication of *some* solubility.

Measure the density (by determining the mass of a known volume *e.g.* 5ml) of each of the fractions to aid in your identification of the components.

Based on your results and the information provided in the data table in the background document, identify the components of your mixture.

CLEAN UP

- Discard your distilled fractions and distillation flask residue into the organic waste drum in the fume-hood.
- Remember that you heated the aluminium block and it might be hot!

REPORT

Before writing any Chem 353 laboratory report, we strongly recommend that you review section 9 in the introductory section of the Chem 353 student laboratory manual that discusses how to write reports and/or from "writing reports" on the course website. Students often don't get the grades they would like because they make errors that are addressed in that section of the manual. These are avoidable errors.

The report for this experiment is to be completed in the template provided. Remember that more it not necessarily better. It is important to be accurate and concise rather than verbose and vague. Proper English should be used and it should be written in your own words.