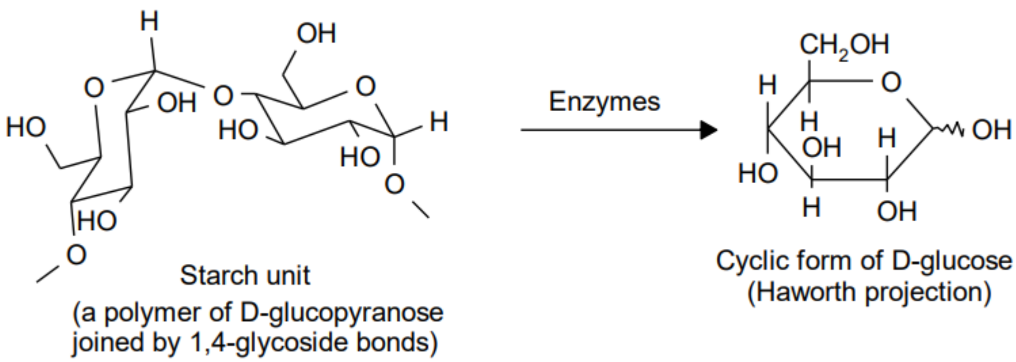
DISTILLATION OF WINE

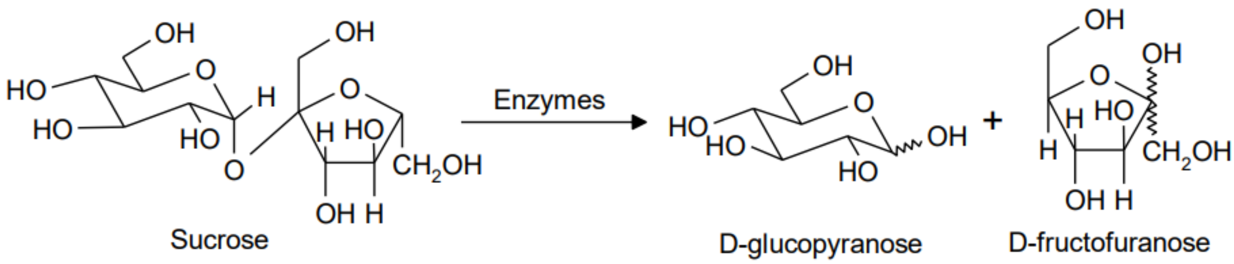
Important concepts:

Simple distillation, fractional distillation, condensate, distillate, azeotrope, boiling point, miscibility

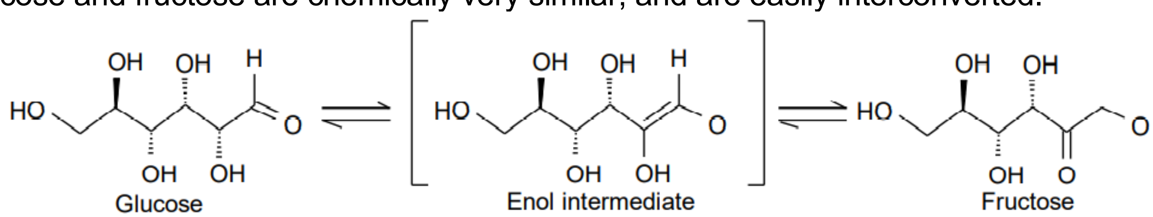
Introduction:

Alcoholic beverages are made by using yeast to convert starch or sugar to ethanol, CH_3CH_2OH. Starch is a polymer, made of many molecules of the sugar glucose linked together, and sucrose is a disaccharide composed of glucose and fructose linked together. Enzymes (proteins that act as catalysts) in the yeast cleave the sugar-sugar linkages and convert starch to glucose, or sucrose to glucose and fructose:

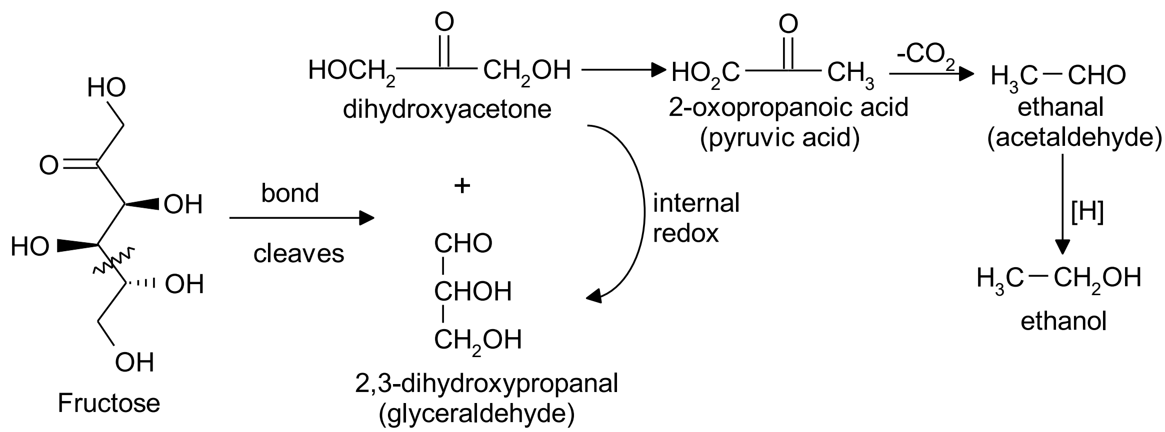




Glucose and fructose are chemically very similar, and are easily interconverted.



Fermentation is the conversion of sugar to ethanol: this process is outlined in a very simplified form below. Each of the 5 reactions shown actually involves several steps, and each is promoted by enzymes.



In this experiment you will isolate *almost* pure ethanol from red wine by distillation (a process similar to that used to prepare stronger beverages like whisky or vodka, which contain about 40% ethanol). Since ethanol (bp 78 °C) is more volatile than water (bp 100 °C), ethanol can be distilled off preferentially. However, the boiling point difference is not large enough for a good separation to be achieved by a simple distillation (***refer to lab manual – simple distillation***). Thus, a fractionating column (***refer to lab manual – fractional distillation***) is employed to increase the proportion of ethanol obtained. In simple terms, this is achieved by the increased surface area in a fractionating column which helps facilitate the vapour (water/ethanol mixture) to condense back to a liquid. As the water will preferentially condense instead of ethanol, the ethanol purity of the vapour will increase moving up the column. This results in higher purity ethanol vapour which condenses after the fractionating column.

Pure ethanol cannot be obtained by fractional distillation because ethanol and water have hydrogen-bonding interactions that cause them to form an azeotrope: a constant boiling mixture with a fixed composition that behaves as a pure compound. The vapour in equilibrium with an azeotropic liquid has the same composition as the azeotrope. For ethanol/water, a mixture of 95.5% (by weight) ethanol and 4.5% water boils at 78.15 °C, below the boiling point of pure ethanol (78.3 °C), meaning that 95% ethanol is an azeotrope. Thus, no matter how efficient the distilling apparatus, only 95% pure ethanol can be obtained by distillation. To prepare 100% ethanol, the water can be removed chemically by reacting with calcium oxide, or by removal of the water as an azeotrope with yet another liquid.

Hints for success:

- Do not throw any of your distillate away until clean up at the end of the lab.
- Be particularly careful about how you position the thermometer bulb with respect to the condenser. The thermometer bulb should sit right at the opening of the condenser so it accurately measures the temperature at which the distillate is coming off. Record the temperatures on the data chart given.
- Use a single clamp to hold the apparatus: Attach the clamp firmly to the round-bottom flask at the ground-glass joint. Please do not put a clamp on the condenser - the glass is thin and easy to break. Use rubber bands to hold the condenser to the stillhead adapter, and the receiver adapter to the condenser. Check that all of the joints are tight to avoid losses of vapour/condensate.
- When pipetting, do not jam the bulb on to the end of the pipette: hold the bulb firmly against the end of the pipette as demonstrated.

- Use the analytical balance to obtain masses for this experiment.

Your grade for this experiment will include marks for the % ethanol obtained from each distillation, so it is vital to take your time to do the distillations slowly and carefully!

CAUTION

You will be heating with a micro burner. Make sure that no flammable solvents are out on the benches. Ethanol is flammable, but on this scale there is little danger of ignition. If the ethanol vapour does ignite, don't panic, simply *snuff out the flames by placing a piece of glassware or a book over the mouth of the vessel.*

Never leave a lighted micro burner unattended!!

Note: You must present your prelab to the TA before the lab

Part 1: SIMPLE DISTILLATION

Equipment Checklist for Parts 1 and 2

2 Corks
Condenser*
2x Erlenmeyer flask, 25mL
10 mL Graduated cylinder
Fractionating column*
Receiver adapter*
Reduction adapter*
Round bottom flask, 250 mL*
Still head*
Thermometer
Thermometer adapter*
2x test tube, 18 x 150 mm

* indicates pieces with ground glass joints.

Procedure

- Use a graduated cylinder to measure out 100 mL of red wine and pour it into the round-bottom flask.
- Place a few boiling stones into the round-bottom flask before you begin, these will prevent localized superheating and "bumping" of the wine.
- Assemble the apparatus as indicated in Figure 1. Have the demonstrator check that everything is set up correctly before you begin heating.
- Start with a fairly large micro burner flame, but *turn it down when boiling starts* (in about 5 minutes). It is helpful to use a chimney to protect the flame from air currents in the laboratory. Boil gently and distil at a rate of about 1-2 drops/sec (about 1 mL every 1-2 min). Heating too quickly will cause the wine to foam up into the condenser, and sometimes even into the distillate!

- Collect the distillate in a clean, dry 10 mL graduated cylinder, and *note the temperature of the vapour as each mL of distillate is collected*, tabulating the results on the form provided. Once you have collected about 10 mL of distillate you may have to turn up the flame to maintain the rate of distillation. Continue distilling and noting the temperature.
- When the graduated cylinder is full, quickly empty the contents into a clean, dry test-tube and replace the cylinder. Stopper the test-tube and set it aside; you will later find the percentage of ethanol in this first 10 mL of distillate. Stop the collection when you have collected about 15 mL of distillate in total. Do not waste too much time getting to this point. Remember, the last few readings we need are for plotting the graph. You will notice that the temperature stops rising i.e. reads the boiling point of water. The whole distillation should take only about 30-40 minutes. Turn off the micro burner and allow the apparatus to cool while you analyze the distillate.

To determine the percent ethanol in the sample collected, you will measure the mass of a known volume of the sample to determine its density.

- Weigh a clean, dry 25 mL Erlenmeyer flask to 3 decimal places, and note the number on the sheet provided. This is the tare weight of the flask.
- Pipette 5.00 mL of the distillate into the **tared** 25 mL Erlenmeyer flask then weigh it again immediately, to 3 decimal places.
- Calculate the density of the distillate in mg/mL.
- The composition can be determined from the density by reading it from the % ethanol versus density graph provided. (Note: This graph was obtained by actual experiment.)

⇨Do not discard any of the distillate. ⇦

Plot a graph of temperature versus volume of distillate (mL), as outlined in the questions.

Part 2: FRACTIONAL DISTILLATION

Procedure

- **Pour all of the distillate collected during Part 1 back into the round-bottom flask, so that you once again have 100 mL of wine (ignoring small losses due to evaporation and manipulation)**, then add some more boiling chips to the flask.
- Set up the apparatus for fractional distillation by attaching the fractionating column between the round-bottom flask and the stillhead adapter, as shown in Figure 2.
- Start with a fairly large flame, but turn it down when boiling starts. Do not hurry the process: good separation depends on allowing equilibration of the vapours. Distil at a rate of about 1 drop per second (about 1 mL every 1-2 min), and note the temperature as each mL is collected as before. After you have collected about 5 mL of distillate you may have to turn up the flame to maintain the rate of distillation.
- When the temperature of the vapour starts to rise abruptly (it should have stayed close to 78 °C so far), quickly transfer the distillate from the graduated cylinder to a clean, dry test-tube and stopper it. Replace the graduated cylinder and continue to note the temperature and the volume, stopping the distillation when the temperature plateaus at a constant reading or when you have ~ 13 mL collected.

Measure the density of the lower-boiling fraction by weighing 5.00 mL of it as before to find its composition. Note the total volume that was collected for the lower-boiling fraction.

Plot the temperature versus volume of distillate on the **same** sheet of graph paper as the simple distillation graph.

WASTE DISPOSAL: Pour the left over wine down the sink. Dispose of the distilled ethanol in the flammable container provided.

Hand in your prelab, data sheets and answers to the questions.

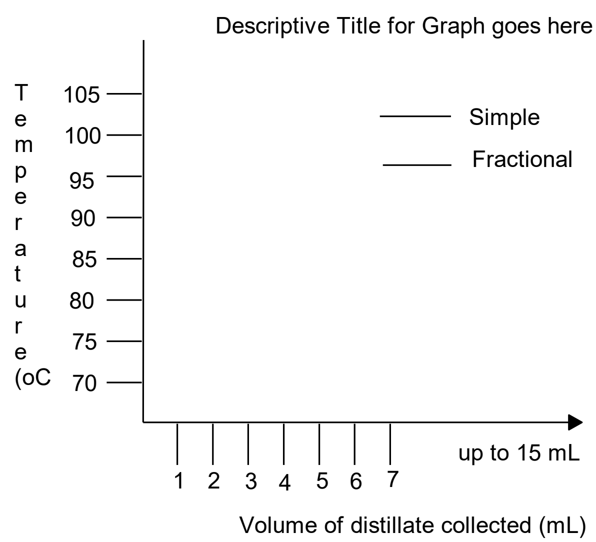
Questions:

1. Plot a graph of temperature versus volume with data for both the simple and fractional distillations plotted on the same axes, on one sheet of graph paper. See the example below. Why does the temperature rise abruptly in the fractional distillation and gradually in the simple distillation? (**8 marks**)

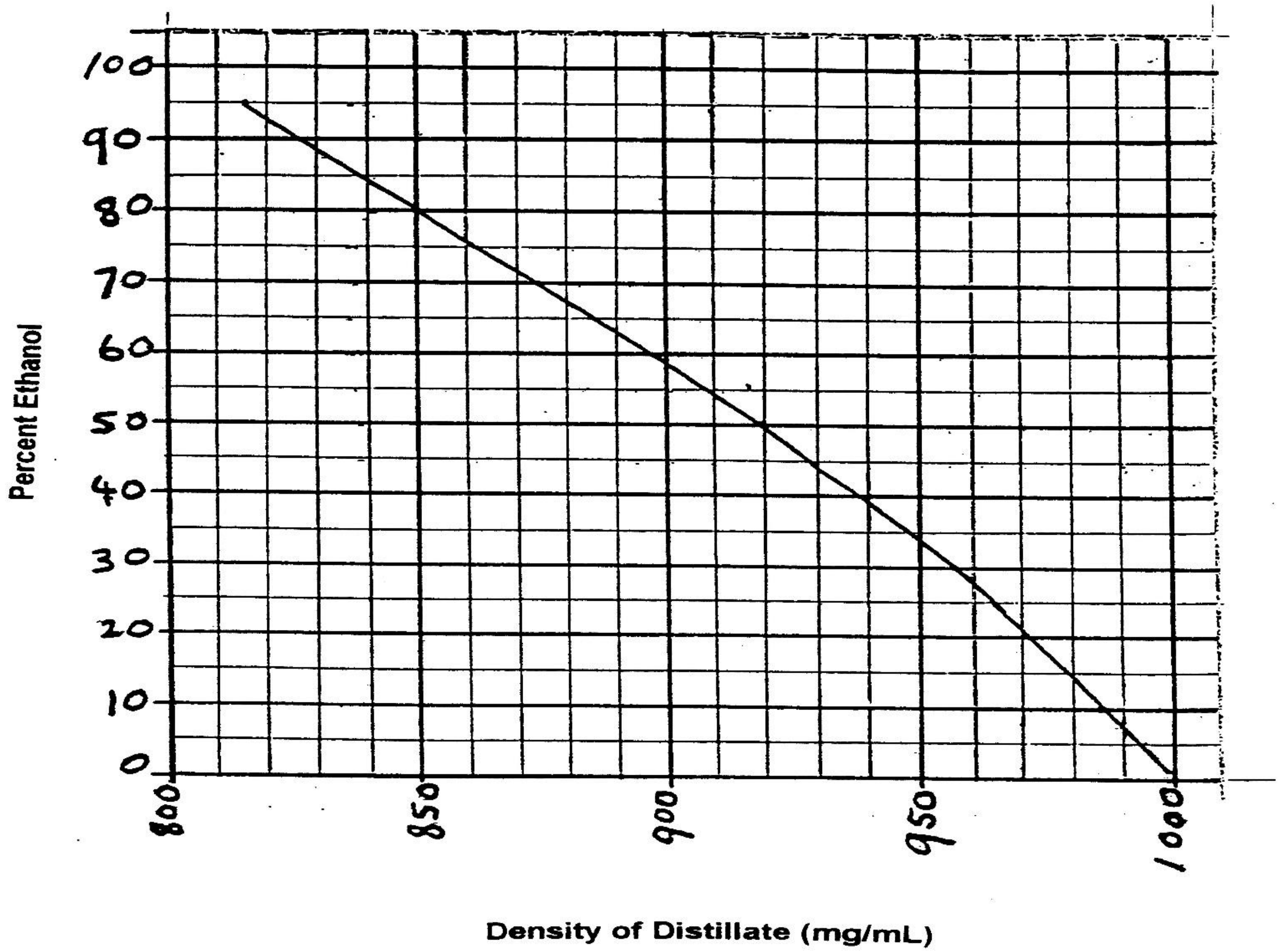
2. Can we get 100% ethanol from either one of these distillations and why/why not? (**4 marks**)

3. Compare the percentage ethanol recovered from the simple distillation and the fractional distillation. (**4 marks**)

Properly reference any materials used for the writing of this report in the references section in ACS format.



Percent Ethanol in Water versus Density of Solution



Name:____________________________________ Lab Section: ____________

Part 1: Data Recorded during the Simple Distillation

Volume of distillate collected (mL)	Temperature (°C)	Volume of distillate collected (mL)	Temperature (°C)

Weight of 5.00 mL of distillate + 25 mL Erlenmeyer flask ______________________g

Weight of empty 25 mL Erlenmeyer flask (tare weight) ______________________g

Weight of 5.00 mL of distillate ______________________g

Concentration of ethanol (from graph) ______________________%

Calculation of density: (Pay attention to units and significant figures.)

Name:_______________________________________ Lab Section: ______________

Part 2: Data Recorded during the Fractional Distillation

Volume of distillate collected (mL)	Temperature (°C)	Volume of distillate collected (mL)	Temperature (°C)

Volume of lower boiling fraction collected _____________________mL

Weight of 5.00 mL of distillate + 25 mL Erlenmeyer flask ______________________g

Weight of empty 25 mL Erlenmeyer flask (tare weight) ______________________g

Weight of 5.00 mL of distillate ______________________g

Concentration of ethanol (from graph) ______________________%

Calculation of density: (Pay attention to units and significant figures.)