Pharmaceutical Chemistry Laboratory Course

EXPERIMENT: Distillation

Aim: To determine the boiling point of a pure liquid and an Azeotrope

Introduction

Distillation of a liquid is a convenient method of purification since pure liquids will boil at a well defined temperature and can be recovered simply by condensation. At the boiling point, the gaseous substance exerts the same pressure on its environment as the atmosphere, so that the external pressure dictates the observed boiling point. These principles lead to applications like high pressure cookers and autoclaves where water stays as a liquid at temperatures well above its normal boiling point due to the high pressure inside the vessel. Rotary evaporators present an example at the other extreme, in that liquids are evaporated at temperatures that are much lower than their nominal boiling point by employing a vacuum pump to reduce the pressure above the liquid.

The boiling point of a liquid is therefore defined with reference to standard atmospheric pressure. However, there are several units of pressure in common use with which you should be familiar:

	pascal	**bar**	**atmosphere**	**torr**	**pounds per square inch**
	(Pa)	**(bar)**	**(atm)**	**(Torr)**	**(psi)**
1 Pa	≡ 1 N/m^2	10^{-5}	9.8692×10^{-6}	7.5006×10^{-3}	145.04×10^{-6}
1 bar	100,000	≡ 106 dyn/cm^2	0.98692	750.06	14.5037744
1 atm	101,325	1.01325	≡ 1 atm	760	14.696
1 torr	133.322	1.3332×10^{-3}	1.3158×10^{-3}	≈ 1 mmHg	19.337×10^{-3}
1 psi	6.894×10^{3}	68.948×10^{-3}	68.046×10^{-3}	51.715	≡ 1 lbf/in^2

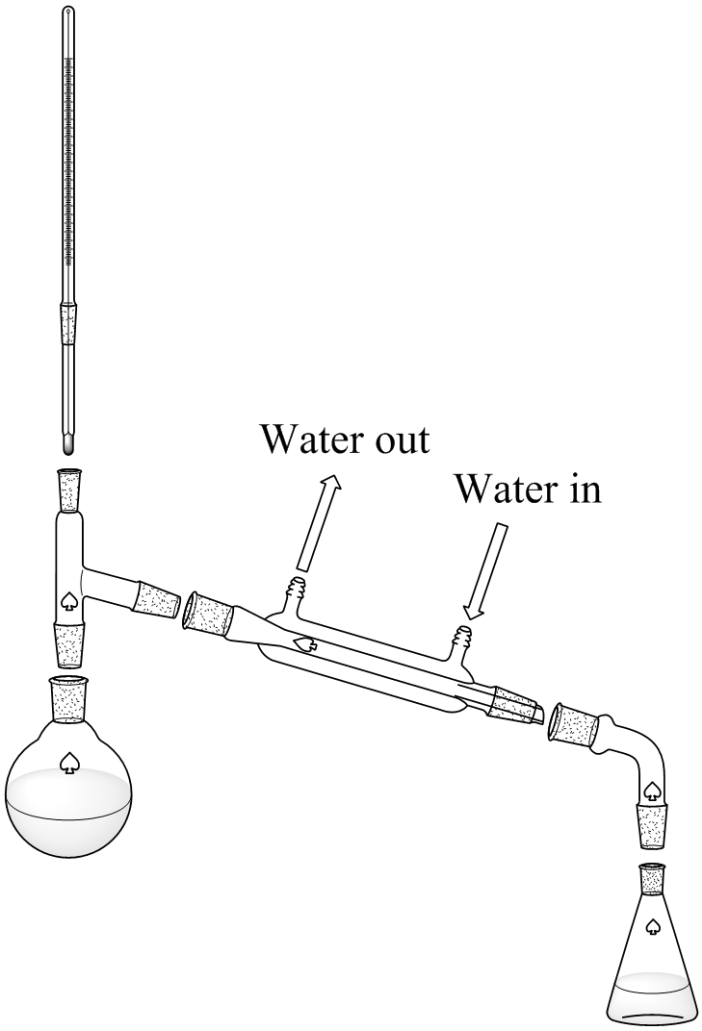
An ***azeotrope*** is a mixture of liquids that cannot be separated by simple distillation. The chemical interaction of different molecules can be sufficiently strong to keep them associated even in the high energy vapour phase. For example, a mixture of ethanol:water (95.63% : 4.37%) boils at 78.2 °C although each component has a higher boiling point (a positive allotrope). Repeated distillation of any mixture of ethanol and water will give compositions that approach this azeotropic ratio. For *negative* azeotropes, the *residue* approaches the azeotropic ratio instead of the distillate. In this experiment, a pure liquid and an azeotrope will be characterized.

Part A: Distillation of a pure compound

Procedure

In the dry 50-mL boiling flask introduce 25 mL of pure, dry methanol, a few boiling chips and connect the flask to the distillation assembly as shown in the diagram. Make certain that all connections are tight. Arrange a graduated cylinder to serve as receiver.

Heat the flask gently until the liquid begins to boil. Adjust the heating rate(height in bath) until the ring of vapor condensation moves up the wall of the flask and past the thermometer into the condenser. Record the temperature when the first drops of distillate collect in the condenser. Continue to distill the liquid slowly (not over 2mL/min) and record the distilling temperature at regular intervals, for example when the volume of the distillate increases by about 5 ml. Discontinue heating when all but 2-3 mL of the liquid remains. Record the temperature range from the beginning to the end of the distillation as the observed boiling point. Transfer the used methanol to a bottle provided for this purpose.



Part B: Distillation and characterization of an azeotrope

Procedure

Repeat the procedure of part A using the ethanol solution provided and a container of known mass as a receiving vessel. Note the appearance of the distillate over the course of the experiment. Add 2 g of anhydrous sodium sulfate to the distillate, seal the top with parafilm and swirl the contents for a few minutes. After standing for a further 15 minute, gravity filter the liquid and record the new weight.

Questions

1. From your data, draw a distillation graph for pure methanol, plotting distilling temperatures on the vertical axis against total volume of distillate on the horizontal axis.

2. Calculate the percent water in the ethanol solution provided.

3. How can you account for the fact that methanol does not form an azeotrope with water?