Experiment 7: Isolation of a natural product by steam distillation

Steam Distillation

$P_T = P_{A\,obs} + P_{B\,obs} + \ldots$

For a binary solution: $P_{T\,obs} = \chi_A P^o_A + \chi_B P^o_B$

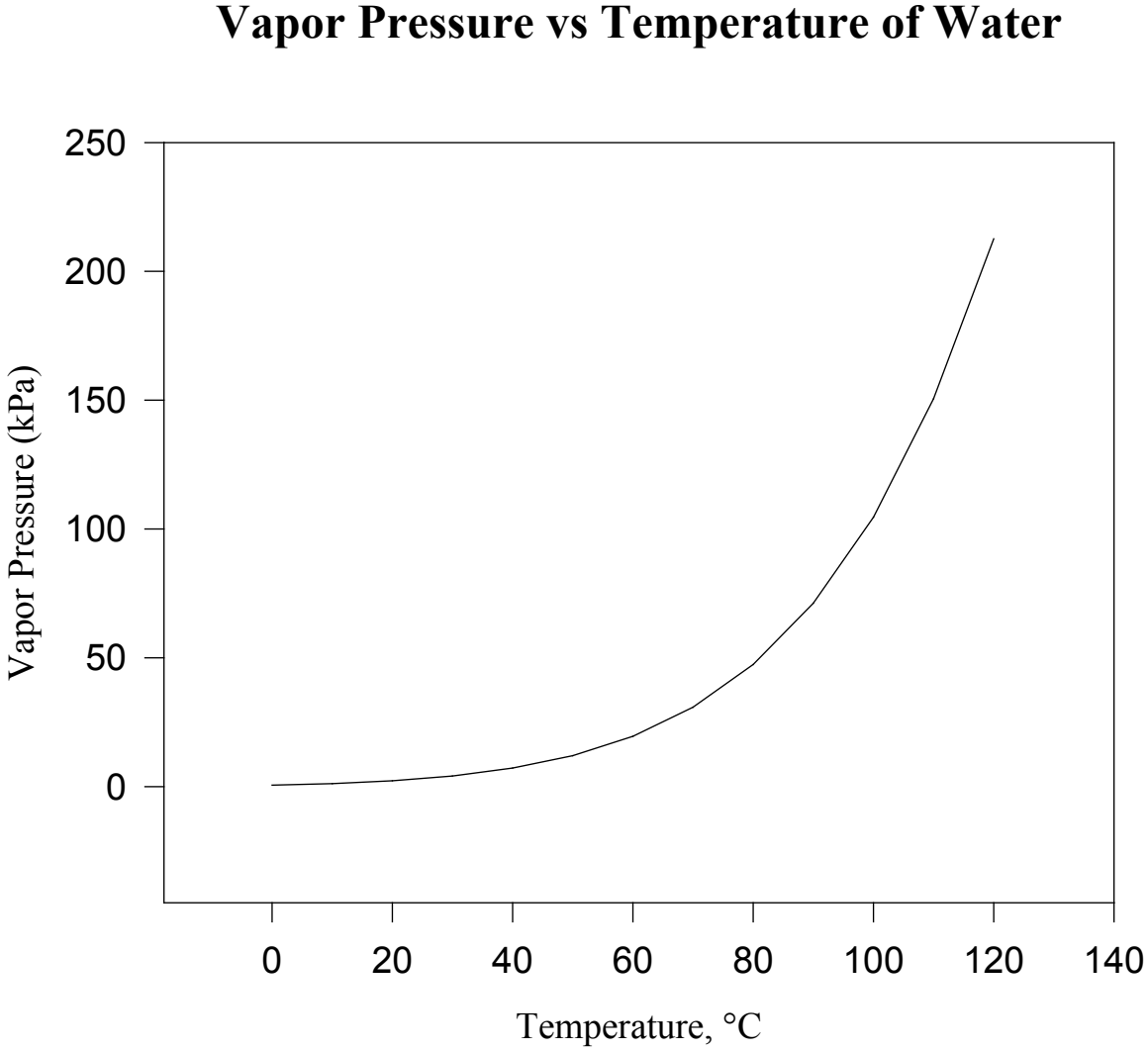
What happens when two immiscible liquids are present?

Are all vapors miscible?

Yes

What is the mole fraction of immiscible liquids?

Approximately 1



What is the maximum temperature two liquids, one being water, will boil?

100 ^{o}C

$$P_{T\,obs} = \chi_A P^{o}_A + \chi_B P^{o}_B \approx P^{o}_A + P^{o}_B$$

Suppose we have an immiscible organic compound with a molecular weight of approximately 150. If we co-distill it with water, how much will co-distill with water if the compound has a vapor pressure of 1 kPa at 100 ° C?

$$P_{T\,obs} \approx P^{o}_{water} + P^{o}_{organic}$$

$$PV = nRT$$

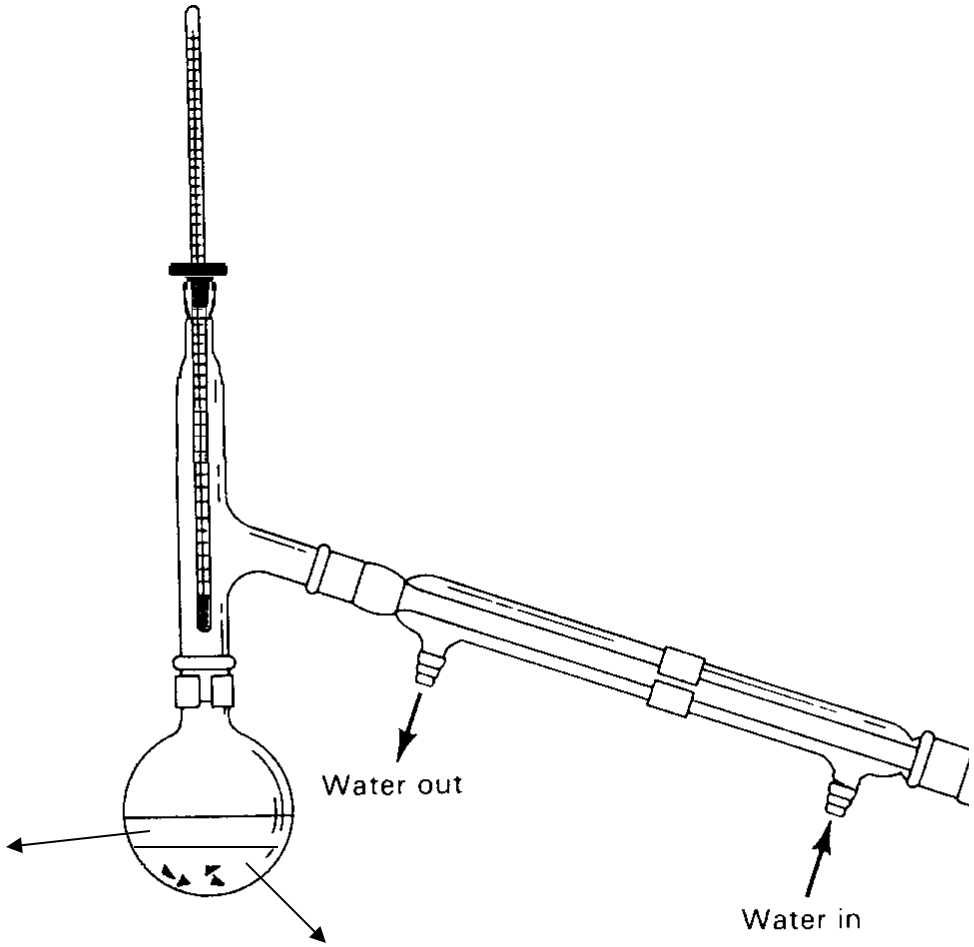
$$P^{o}_{water}V = n_{water}RT$$

$$P^{o}_{organic}V = n_{organic}RT$$

$$P^{o}_{water}/P^{o}_{organic} = n_{water}/\ n_{organic}$$

$$100\ kPa/1\ kPa = [wt_{water}/18]/wt_{organic}/150$$

$$wt_{water}/wt_{organic} = 18*100/150 = 12/1$$



organic

water

Advantage: It is possible to separate volatile compounds from non-volatile compounds at temperatures well below the natural boiling point of the organic component.

Requirements: The compound must be stable in water up to 100 °C

The compound must have a volatility of a few kPa (at the boiling point of water

The IR spectrum will be obtained by allowing the methylene chloride solution you use to extract the spice from water to evaporate. The IR will be run on the residue.

The GC should be run on the methylene chloride solution. This will insure you don't inject too much of your natural product on the column.

You should try to identify the compound by either the GC retention time or its IR or both.

You should use the retention time of methylene chloride as your reference point. Use the difference in retention time between you spice and methylene chloride to determine the structure of your spice, using the reference spectra that will be provided. Be sure to record which GC you used.

Natural products such as spices often have have a several components of volatile oils. You will identify the major component in your spice by comparing the retention of the major component in your gas chromatograph to an authentic sample run under identical conditions. The gas chromatographs of the authentic samples in methylene chloride will be provided to you.

They include the following spices: allspice, caraway, cumin, and cloves.

Steam distillation is one of the major ways natural products are isolated from botanicals.

This week you will isolate a natural product and characterize it by IR and identify it by comparing its retention time to the retention time of an sample from a different source by gas chromatography

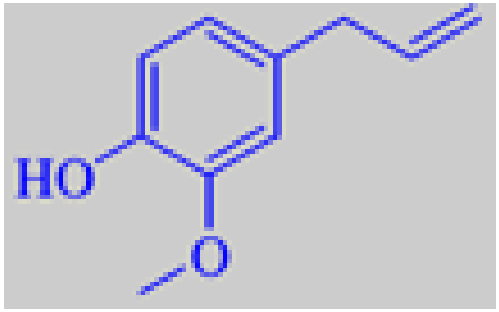
Caraway

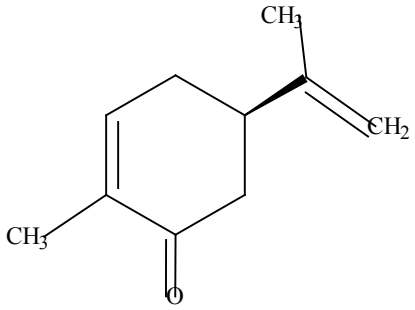
Fennel

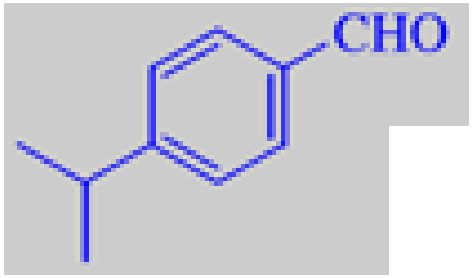
Cloves

Cumin

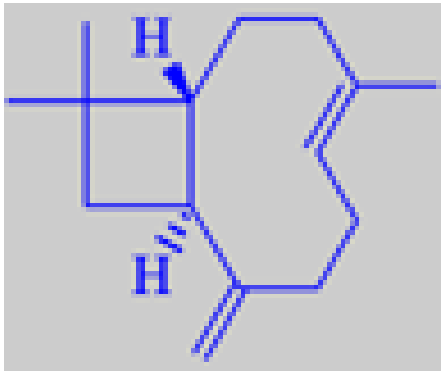
Major Components



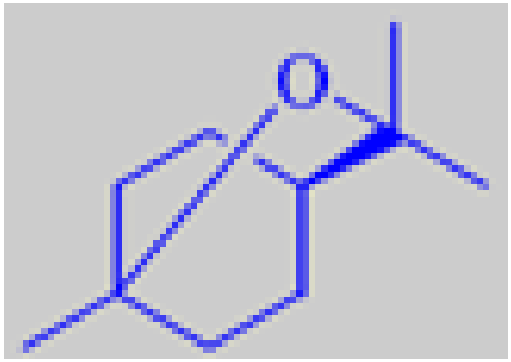




Minor Components



caryophylene



1,8-cineole