Steam Distillation of Cloves

READ:Landgrebe 4th Ed .p160-161 (Section 7.6) and p369-371, 5th Ed. P 140-141 (Section 7.6) and p 321-322.

I. Introduction

In an earlier experiment we have seen that distillation of a mixture of two miscible liquids depends on the vapor pressure and mole fraction of each of the components. There is a different situation in the distillation of two liquids that are not mutually soluble. In this case the vapor pressure above the mixture is the sum of the partial pressures of the components:

$$P_T = P_A + P_B$$

Each liquid exerts its own vapor pressure independent of the other. Since the vapor pressures are additive, the boiling point of the mixture is lower than the boiling point of either of the components.

As long as separate components are present in the liquid, the mixture will have a constant boiling point. In addition, the distillate will have a constant composition; which is determined by the ratio of the vapor pressures (eq 1). Since the number of moles of a compound (n) is equal to the weight divided by its molecular weight (mw), equation 2 can be derived from the weight ratio of the compounds in the distillate.

$$n_A/n_B = P_A/P_B \qquad (1)$$

$$wt_A/wt_B = P_A/P_B \times mw_A/mw_B \qquad (2)$$

Thus if the molecular weights and partial pressures of the two components are known, the weight ratio of the components in the distillate can be calculated. Conversely, if the vapor pressure and molecular weight of one component and the total pressure during distillation are known, the molecular weight of the second component can be calculated by the weight ratio.

For example, assume that one component is water, the other is an unknown organic compound Y, and the distillation occurs at 99.4 °C at a barometric pressure of 750 mm HG to give a mixture of water and Y in a ratio of 9 g to 1 g. The partial pressure of Y can be calculated from the total pressure and the partial pressure of water at 99.4 °C, which from a handbook of tables is 744 mmHg. Thus:

$$P_Y = P_T - P_{H_2O}$$
$$= 750 - 744 = 6 \text{ mmHg}$$

Then:

$$mw_Y/mw_{H_2O} = wt_Y/wt_{H_2O} \times P_{H_2O}/P_Y$$

Solve for mw Y

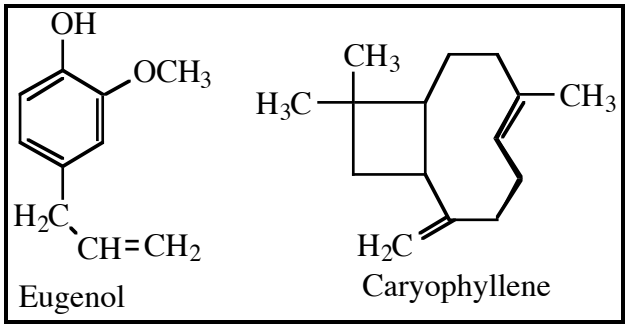
$$mw_Y = 250$$

When one of the two components is water, this process is termed **STEAM DISTILLATION**, and it provides a means of distilling a relatively non-volatile substance at a temperature far below its atmospheric boiling point. Steam distillation is a useful alternative to extraction for the isolation of essential oils from plant materials.

The characteristic aromas of plants are due to organic substances known as essential oils (the term essential does not mean "necessary", but derives from the word essence). Essential oils have been used since antiquity in flavorings and fragrances. These oils occur in all living parts of the plant, they are often concentrated in flowers and seeds. Essential oils are often complec

mixtures of hydrocarbons, alcohols and carbonyl compounds. A multi-billion dollar industry has developed around the extractionof essential oils.

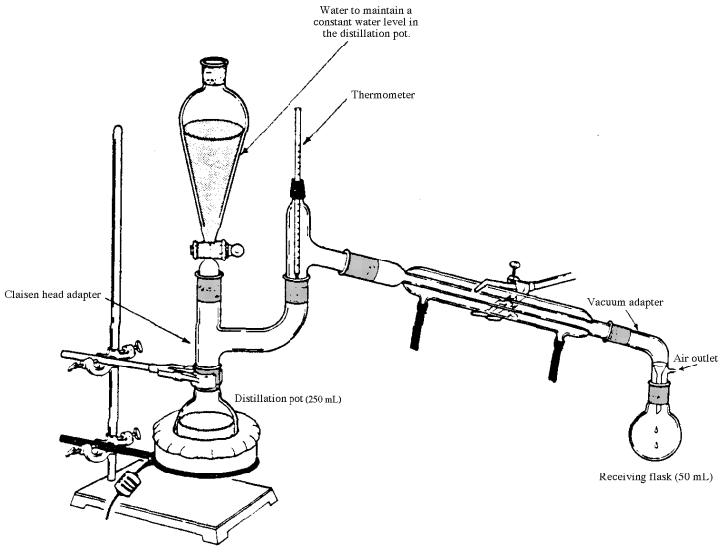
In today's experiment we will isolate oil of cloves from cloves (Eugenia caryophyllata). Cloves have been used as a flavoring agent for food for thousands of years; I for one love cloves in my Easter Ham. In addition, oil of cloves was one of the first know dental pain killers...a few drops of oil of cloves will numb the pain of a tooth ache. One formulation sold on the market for tooth pain relief still contains oil of cloves. Oil of cloves is rich in eugenol (4-allyl-2-methoxyphenol) and contains a trace amount of caryophphyllene.



II. Procedure

A. Follow the procedure described on pages 369-370 part A of Landgrebe. We will use 2x the amount of cloves called for in the experiment (2.0 g), 125 mL of water and a 250 mL round bottom flask.

B. Use the Set-up shown below. Make sure the distillation pot is completely free of any



detergent that may have been used to clean it. Failure to do so will result in foaming and make the distillation difficult. To get rid of the detergent, rinse three times with tap water and, finally, once with deionized water.

C. While you need to keep the water level somewhat constant during the distillation, you should not let the water level get much over half full in the distillation pot. Record the temperature of the distillate.

E. Record the final weight of oil of cloves recovered, calculate the % recovery based on the amount of cloves used. You will analyze your product from this experiment using gas chromatography .

F. Calculate your % recovery of eugenol based on the amount of oil of cloves you used and the GC data..

III. Report(use standard format)

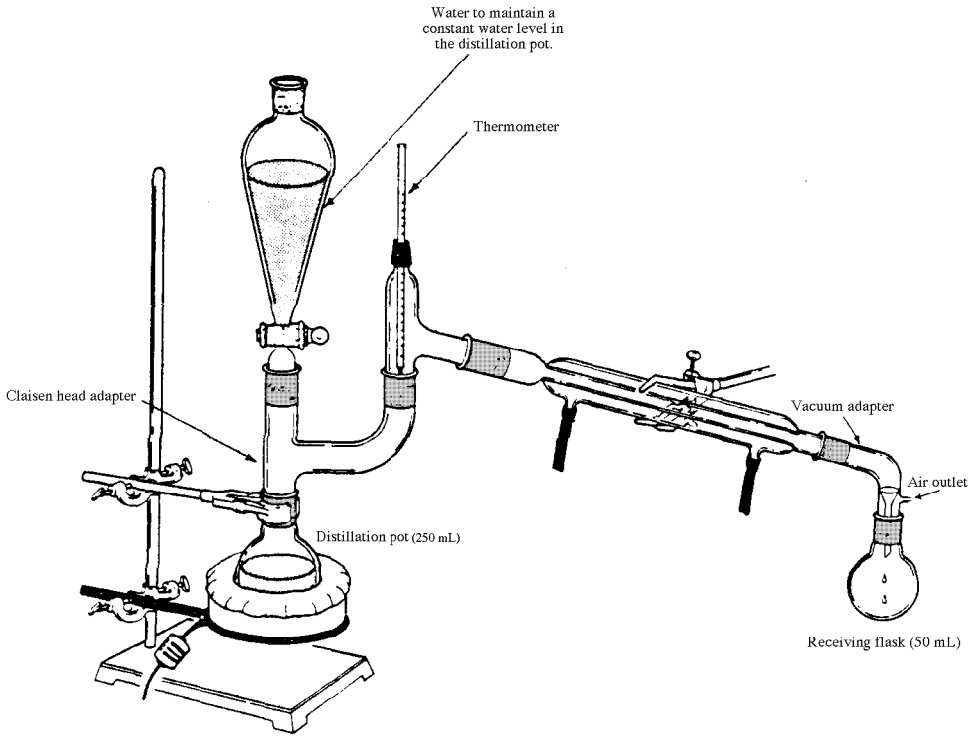
A. Introduction and Experimental details

B. Observations, data, and gas chromatographs and nmr spectrum

C. Show the following on the gas chromatograph.
   a. identity of each peak
   b. calculations for the area under each peak (except solvent peak)

   c. Draw the structure for eugenol and for the impurity caryophyllene. Calculate the percent of each in oil of cloves.

F. Answer problem 16, page162 (5^{th} Ed. Do problem 20 on page 142) .



STEAM DISTILLATION APPARATUS