Lecture Notes
Isolation of Clove Oil Using Steam distillation

Steam Distillation
- Steam distillation is used to isolate, separate or purify compounds that have high boiling point or have low decomposition point.
- The compound to be distilled must be insoluble or only slightly soluble in water.
- The clove oil and the water will co-distill. Remember that both the water and the clove oil exert a vapor pressure. For this experiment water has the higher vapor pressure compared to the clove oil, consequently more water will distill.
- Most compounds, regardless of their boiling point, will distill at temperatures below pure that of boiling water. According to Dalton’s Law $P_{\text{total}} = P_1 + P_2$. Boiling will occur when the total pressure equals 760 torr. The vapor pressure of pure water is 100°C is 760 torr. Since the vapor pressure of the second compound adds to the total pressure, the mixture will distill less than 100°C.

Extraction of clove oil from water.
- The clove oil is separated from the water codistillate by dichloromethane extraction.
- Any aqueous layer that might have been separated along with the extract will be removed with sodium sulfate anhydrous. This process of removing water or any aqueous layer is called "drying."
- The dichloromethane is removed by vacuum distillation using the rotary evaporator. This will be done by the instructor.
- The clove oil is weighed to determine percent yield.

Classification Test of eugenol (clove oil)
- The eugenol isolated will be tested for unsaturation using the potassium permanganate Test and aromaticity with the ferric chloride test.

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![Figure 1: Structures for (a) eugenol and (b) eugenol acetate](image)

- **Br₂ Tests**

![Br₂ Tests](image)
- KMnO₄ test is the oxidation of an alkene to a diol.

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\begin{align*}
3 \text{alkene} + 2 \text{KMnO}_4 + 4 \text{H}_2\text{O} & \rightarrow 3 \text{1,2 diol} + 2\text{MnO}_2 + 2 \text{KOH} \\
& \quad \text{(purple)} \quad \text{(brown)}
\end{align*}
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- Ferric Chloride test - presence of a phenol gives a colored solution.

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\begin{align*}
6 \text{ArOH} + \text{Fe}^{3+} & \rightarrow \text{[Fe(OAr)₃]}^{3⁻} + 6 \text{H}^⁺ \\
& \quad \text{(yellow)} \quad \text{(blue, green, red, or purple)}
\end{align*}
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Procedure Modifications:
Part 1. Distillation of Cloves
- Steam distillation apparatus modification. **Please note the sample setup.** Set up the distillation using a 30 mL separatory funnel.
- Add 1 boiling chip into the boiling flask containing the cloves.
- Keep the flame moving. Maintain boiling but keep the temperature so that no bumping occurs up into the condenser.

Part 2. Extraction of Clove Oil
- Do this part in the hood.
- Remember your glass joints have been greased. When transferring your distillate and extract, you need to avoid pouring from the greased joint. Transfer the liquid using a pipet.

Part 3. Drying of the extract.
- Do this part in the hood.
- The evaporation of the methylene chloride will be done using the rotary evaporator. Parafilm your flask during transport.

Part 4. Classification test
- This part can be done on your table.