Lesson 31

Title of the Experiment: Isolation of Clove Oil by Steam Distillation
(Activity number of the GCE Advanced Level practical Guide - 68)

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Introduction:

Essential oils are a group of natural products which can be extracted from plants. Usually they are hydrophobic and contain a volatile aroma. Cloves are the flower buds of *Syzygium aromaticum* (clove tree). Cloves contain approximately 16 % essential oil by mass. Major constituents of clove oil are Euginol (nearly 85 %) and Eugenol acetate (10 %). Their structures are given below (Figure 1).

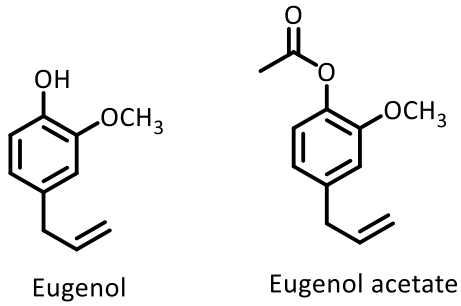

Figure 1: Structures of major constituents of clove oil

Essential oils are normally extracted via steam distillation. There, steam forced through the plant material vaporizes the essential oil from the plant material. Vaporized essential oil can be collected with water into a receiver via condensation. Eugenols (Euginol and Eugenol acetate) contain carbon-carbon double bonds and these can be used to characterize clove oil. A solution of bromine (Br_2) in methylene chloride reacts with the double bonds of euginols and forms a colorless compound as shown in the Figure 2.

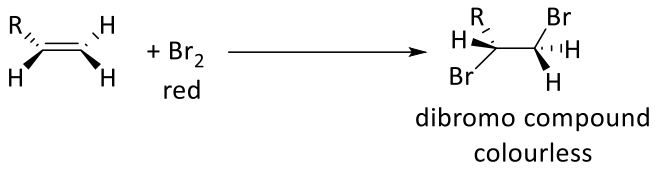

Figure 2: Reaction of euginols with bromine

Theory:

Distillation of heterogeneous mixture (a mixture which contains two immiscible components) is called as co-distillation. If one of the immiscible components is water, the process is called steam distillation. During this heterogeneous mixture distillation, the total vapor pressure over the mixture is approximately equal to the sum of the vapor pressures that would be created by the separate pure liquids at the same temperature. For example, consider a mixture which contains two immiscible components, A and B. The total vapor pressure at a particular temperature (P_{total}) is

equal to the summation of the vapor pressures of pure A (P^o_A) and Pure B (P^o_B) at the same temperature.

$$P_{total} = P^o_A + P^o_B \text{ (Dalton's law)}$$

When the P_{total} is equal to the atmospheric pressure, both components begin to distill. As a result, the boiling point of the heterogeneous mixture will be lower than the boiling point of the most volatile component of the mixture. Therefore, in steam distillation volatile compounds which are unstable at high temperatures or have very high boiling points can be distilled at relatively low temperatures. For example, boiling point of pure Euginol is 254 °C, but during steam distillation both water and Euginol can be extracted at a temperature below 100 ^{o}C, the boiling point of water. Basically, steam distillation can be conducted by two methods – external and internal steam distillation. Here, the distillation will be carried out via internal distillation.

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Learning outcomes:

At the end of the experiment, students will be able to

- understand the principal of steam distillation
- carrying out a steam distillation to isolate clove oil from clove
- characterization of clove oil by using a simple chemical reaction

Laboratory Safety Precaution

- Wear safety goggles all the times while in the laboratory.
- Drain any of the water left in the distillation flask (after the distillation) to the liquid waste container carefully and dump the used cloves in the solid waste container.
- Clean up all glassware with soap and water.
- Methanol is flammable. Keep away from flames or heat sources.
- Bromine solution is corrosive and toxic. Handle carefully.
- Wash your hands thoroughly with soap before leaving the laboratory.

Materials/Equipment:

Freshly ground cloves

100 ml round bottom flask

Distillation head

Heating mantle and Variac

Thermometer

Condenser

Adapter

Rubber tubings

50 ml measuring cylinder

50 ml separatory funnel

Metal stands and clamps

Keck clips

Bromine solution in methylene chloride

Test tubes -2

Methanol

Distilled water

A container for clove oil storage

Procedure:

Weigh 5.00 g of freshly ground cloves to the 100 ml round bottom flask (distillation flask). Add 50 ml of distilled water and couple of boiling chips to the above distillation flask. Set up the distillation apparatus according to Figure 3 using 50 ml measuring cylinder as the receiving flask. Start the flow of water through the condenser according to the indicated direction in Figure 3. Turn on the heating and continue the distillation until little liquid (less than 5 ml) remains in the distillation flask. *If foaming occurs reduce the heat.* Continue heating until 35 ml distillate has been collected. After the distillation, cool the distillate up to room temperature. Transfer the distillate quantitatively to the separatory funnel. Shake the contents and allow the organic and aqueous layers to separate. Carefully transfer the organic layer in to a pre weighed container. Measure the weight of the container with clove oil.

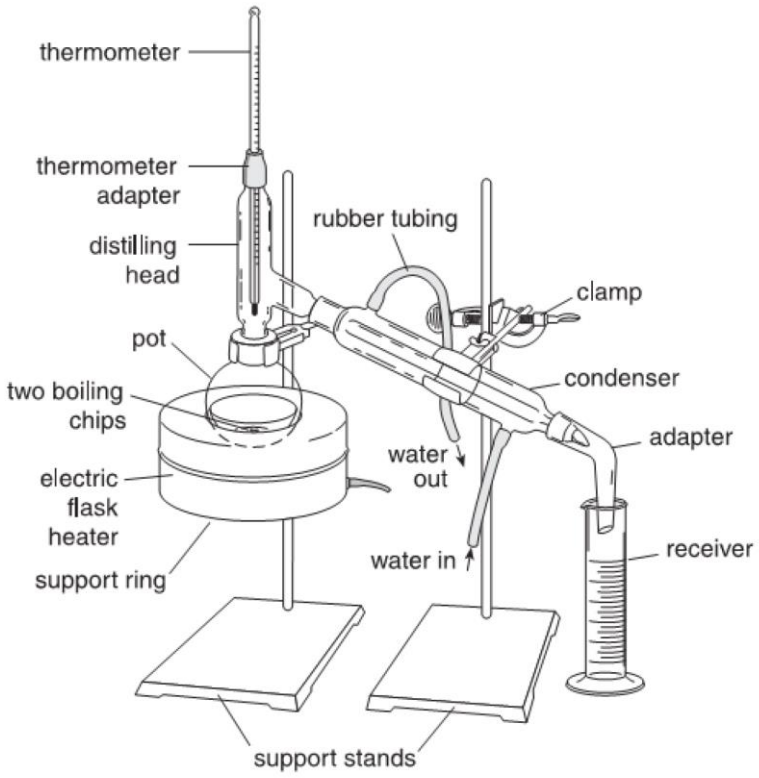

Figure 3: Distillation set up for clove oil extraction

Calculation:

Mass of cloves used = A

Mass of the empty container = B

Mass of the container with isolated oil = C

Mass of product isolated = C-B

$$\% \text{ of oil by mass } = \frac{(C-B)}{A} X\ 100\ \% :$$

Characterization of the Isolated Clove Oil

Obtain two test tubes and label them as “blank” and “test”. Add 1 ml of methanol to both tubes. Add little amount of clove oil to the tube labeled “test”. Add 5 drops of bromine solution to each tube. Gently swirl and record the observations.

Observations:

Blank:

Test:

References:

J. W. Lehman, (1998). “Operational organic chemistry”, 3rd ed. Prentice Hall,.