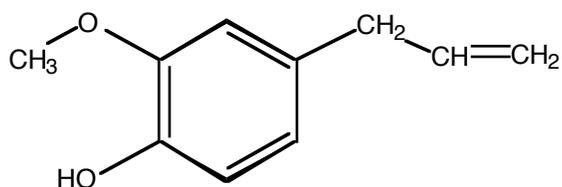


Experiment 7: Steam Distillation

The flora and fauna of the world contain a vast array of organic molecules that can be isolated, studied and put to use in improving the quality of our lives. Many of these *natural products* find applications as flavors, fragrances, dyes and pharmaceuticals. Beneficial compounds that are obtained from plants and animals can also be modified by chemical means, for instance, through the alteration of one or more functional groups, to obtain molecules with even better properties. The essential oils of seeds, leaves and flowers are examples of sources of natural products that have proven to be of use to humans. The flavor of vanilla, the antiseptic agent camphor, the solvent turpentine and the insect repellent citronella are all essential oils that have been extracted from plant material.

The clove tree produces flower buds which, when harvested and dried, become the familiar spice of the same name. It has long been known that cloves contain an ingredient with an anesthetic property that is particularly effective against dental pain. Before modern medicine developed better remedies, people often chewed on cloves to dull the pain of a toothache. The active compound responsible for this effect is found in the essential oil of cloves, which makes up approximately 16-18% of the clove by weight. The major component of this oil is the compound *eugenol*.



Eugenol

Eugenol has a boiling point of 255° C. It would be difficult to isolate this oil by simple distillation since this high temperature could lead to its decomposition as well as the decomposition of other components of the cloves. However, eugenol can be isolated relatively easily from cloves using the technique of *steam distillation*. Read pp 194-196, Technique 12.6, in *LTOC*. See also Technique 12.5 on pp 193-4. Once the aqueous distillate containing the clove oil has been obtained, the oil can be separated from the water by extraction into the organic solvent diethyl ether.

Outline the steps of the following procedure:

You will work with a partner for this experiment.

Weigh out **15 g** of whole cloves and crush them in a mortar and pestle. Place the cloves in a 250 mL round bottom flask and add 30-40 mL of water to completely cover them. Set up the apparatus as shown in the diagram on [Figure Page, Expt. 7](#). Clamp the steam trap in place. Do not attempt to spread apart the bent glass tubing pieces, or they will break. Refer also to the sample apparatus found in the dispensing fume hood. You do not need to monitor the temperature, so the thermometer can be replaced with a ground glass stopper. Use a drop or two of glycerin on the bleed tube, even if it appears to move easily in the thermometer adapter without it. The bleed tube need only be inserted an inch or two into the rubber tubing. *For now*, pull the bleed tube up so that the bottom of it is just above the surface of the liquid. Place a thermowell under the round bottom flask on a ring clamp.

Turn on the process chilled water for the condenser (remember to open “return” before “supply”). Turn on the thermowell and warm the flask for about 5 minutes. Open the screw clamp on the steam trap, then carefully turn on the steam. When the water turns to steam, push the bleed tube down so that it extends almost to the bottom of the round bottom flask. **Be very careful—steam is hot!** Slowly close the screw clamp, and adjust the flow of steam with the handle so that it is low enough that water does not splash over into the condenser due to excessive turbulence, and high enough that water does not back up into the bleed tube. If steam is coming out of the vacuum adapter, it should be turned down. Check the steam trap often to see if it is filling up with water, and open the screw clamp to drain it when necessary.

Use a 125 mL Erlenmeyer flask to collect the distillate. Transfer the distillate periodically to a 100 mL graduated cylinder (you will need a second flask or beaker to collect the drops which fall during this transfer); this way, if any material foams over into the condenser, the entire distillate will not be contaminated. If a portion of the distillate is contaminated in this way, you will have to re-distill that portion. You will collect a total of 100-120 mL of distillate.

When you have collected 50-60 mL of distillate, pour it into a separatory funnel. You will extract the eugenol from the distillate with one 12 mL portion of diethyl ether followed by two 8 mL portions. The upper layer will be the organic layer. Add 12 mL of diethyl ether to the distillate in the separatory funnel. Alternately shake and vent the sep funnel. Drain the lower layer into a clean beaker. Pour the upper layer from the *top* of the separatory funnel into a 250 mL Erlenmeyer flask. Pour the lower layer from the beaker back into the sep funnel. Extract this aqueous solution with an 8 mL portion of ether, and then add the organic layer to the solution in the Erlenmeyer flask. Repeat the extraction of the aqueous layer with another 8 mL portion of ether.

Pour the ether solution from the Erlenmeyer flask back into the separatory funnel, and add 15 mL of saturated sodium chloride solution. Shake the mixture thoroughly, allow the layers to separate and drain the lower, aqueous layer (this layer can be discarded down the drain). Pour the ether layer from the top of the separatory funnel into an Erlenmeyer flask.

You will use two drying agents to remove water from the organic solution. Add just enough anhydrous sodium sulfate to the ether solution so that it no longer clumps together but swirls freely. Swirl the flask for a minute or two to complete the drying process. Decant the ether solution from this drying agent through a cotton plug in a funnel and collect the solution in a *dry* 125 mL Erlenmeyer flask. Add 5 mL of ether to the sodium sulfate left behind, swirl the mixture, and decant this liquid through the funnel into the 125 mL flask. Next, add just enough anhydrous magnesium sulfate to the ether solution so that it no longer clumps together.

Weigh a clean, *dry* 40 mL vial. Decant the ether solution from the magnesium sulfate through a cotton plug in a funnel and collect the solution in the tared vial. Add 5 mL of ether to the drying agent left in the Erlenmeyer flask, swirl the mixture, and decant this liquid through cotton into the vial. Place the vial in a Centrifan to evaporate the ether.

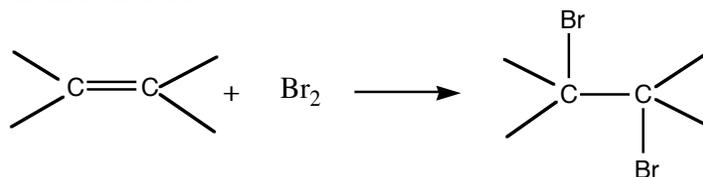
You will need to repeat the entire extraction and drying procedure with the second 50-60 mL portion of distillate that you collect. When you are ready to stop the steam distillation, **first open the screw clamp!** Again, **be careful not to burn yourself!** Once the screw clamp is opened, turn off the steam. Immediately remove both the glass stopper and the thermometer adapter from the apparatus set-up. Turn off the thermowell.

Remove the vial from the Centrifan and decant the second portion of ether extract through cotton into the vial. Evaporate the ether in the Centrifan until a viscous, yellow oil remains. Weigh the crude clove oil.

Use a large Buchner funnel and side-arm flask from the support room to filter the clove and water mixture that remains in the 250 mL round bottom flask. Do not use filter paper. Pour the filtrate down the sink, and throw the cloves in the trash.

Test for Unsaturation

You will test your clove oil, which contains eugenol, for the presence of carbon-carbon double bonds. Bromine dissolved in cyclohexane is a reagent that is frequently used to test for unsaturation. Most alkenes will react rapidly with bromine via an *addition* reaction as shown below:



The bromine reagent is dark red in color, and the visual result of a positive test for unsaturation is the disappearance of this red color. Compounds without carbon-carbon double bonds, on the other hand, are much less reactive toward bromine.

Include the following procedure in your **outline**:

CAUTION: Bromine is a highly corrosive substance that causes severe burns and gives off irritating fumes. Carry out the test in a fume hood and wear gloves when handling the reagent.

Place 3 drops of isooctane in a small test tube, and add 1 mL cyclohexane (about 20 drops). Add dropwise with shaking a solution of bromine in cyclohexane. Record the number of drops necessary to develop a persistent (for 1 minute) reddish color. [**Note:** if the color still does not persist after 15 drops of bromine have been added, you may stop adding bromine.]

Repeat the test in a clean test tube using 3 drops of octene in place of isooctane.

Repeat the test by adding 1 mL of cyclohexane to the clove oil in the vial, and then adding the bromine in cyclohexane. Record the number of drops necessary for the color to persist.

Dispose of all solutions containing cyclohexane and/or bromine into the appropriate **Laboratory Byproducts** jar.

Name _____ Date _____
T. A. _____ Lab period _____

Results and Calculations (to be handed in at the next lab period)

Calculate the percent recovery of oil from cloves.

Number of drops of bromine reagent required for color to persist...

...for isooctane _____

...for octene _____

...for clove oil _____

What conclusions can you draw from the results of the bromine tests?