Steam Distillation

Source: AEM Handout, adapted from D. L. Pavia, G. M. Lampman, G. S. Kriz, R. G. Engel **Introduction to Organic Laboratory Techniques (A Microscale Approach)** Saunders: 1990, p 87-95.

Procedure:

Assemble a microscale steam distillation apparatus using a 10 mL round bottom flask, a Hickman head (with a septum cap over its collection port!), and a water-cooled reflux condenser, as shown in your text (shown there for simple distillation…). Heat a sand bath to about 130°C in preparation for the distillation. During the distillation, you should adjust the sand bath temperature to distill efficiently, but not allow frothy bubbles to boil up into the Hickman head.

To steam distill oil of cloves and to isolate eugenol, place about 0.4 g (record actual weight to 3 decimal places) of ground cloves into the round bottom flask of your apparatus; add about 4 mL of water and a boiling stone. Begin heating the mixture to provide a steady rate of distillation. Raise or lower the round bottom flask in the sand to control the temperature of the mixture. If a higher temperature is needed, you may also push the sand towards the flask with a spatula, or increase the setting of the power-mite. Some care should be exercised so that the mixture does not froth too much! Do NOT let the frothy mixture in the round bottom flask boil up into the Hickman head!

During the distillation, you may need to replace the water lost through distillation by adding water to the boiling mixture using a Pasteur pipette (which is passed through the condenser and Hickman head into the round bottom flask). As the distillation proceeds, collect the distillate with a different Pasteur pipette and use it to rinse the walls of the Hickman head at appropriate intervals. Notice that your Hickman head has a collection port with a septum cap through which you can collect your product; keep this port capped unless you are removing distillate! When a good bit of distillate has collected in the Hickman head, transfer it to a 5 mL <u>conical</u> vial (vial #1). Continue collecting the distillate in vial #1 until you have obtained at least 2.5 mL of distillate (leaving space for adding about one mL of methylene chloride in the liquid-liquid extraction below).

The essential oil must now be extracted from this aqueous solution into methylene chloride. To do this, add 1.0 mL of methylene chloride to vial #1 containing the distillate. Cap the vial securely and shake it vigorously with frequent venting. Allow the layers to separate. Using a disposable pipette, transfer the lower methylene chloride layer to a second, dry, vial (vial #2, screw-capped or conical). Repeat this extraction procedure with two more 1.0 mL portions of methylene chloride, combining all three methylene chloride extracts in vial #2. Be very careful not to transfer any of the water layer to the vial that contains the methylene chloride extracts; if there are visible drops of wa-

ter in that vial, it will be necessary to transfer the methylene chloride solution with a dry pipette to another dry conical vial! Dry the methylene chloride solution in vial #2 by adding three to four microspatulafuls of granular anhydrous sodium sulfate (Na_2SO_4) to it. Let it sit for 5 or 10 minutes with occasional swirling.

While the organic solution is being dried, clean and dry another 5 mL conical vial (vial #3) and weigh it accurately. Make a microscale gravity filtration device using a disposable pipette plugged tightly with a small piece of filter paper. With a clean, dry pipette transfer the dried organic layer through this gravity filtration device into the dry, tared vial #3. Use small amounts of clean methylene chloride to rinse the first flask and the filtered solid Na_2SO_4 to be sure of complete transfer of the solution into the vial #3. Evaporate the methylene chloride from the solution in vial #3 by heating it very gently on a sand bath in the hood until nothing but a drop of oily residue remains. You may want to tap the vial with your finger to provide agitation in order to facilitate removal of the remaining solvent.

When the solvent has been removed and the flask is back at room temperature, weigh vial #3 with its eugenol contents. By difference, compute the mass of eugenol that you have isolated and calculate the percentage recovery of this oil from the original amount of spice used.

Obtain the infrared spectrum of the oil as a pure liquid. Include the spectrum in your laboratory notebook, along with an interpretation of the principal peaks.

Clean-up and Disposal: The material remaining in the still pot can be thrown in the trash can since it is non-hazardous. The filtered sodium sulfate can also go in the trash can. After weighing your eugenol sample (oil of cloves) and taking its IR spectrum, you may wash it down the sink with water. (You could also take it home to a special family member! ;-)).