Steam Distillation
Updated 1/28/2025

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.
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Procedure:

Collect the glassware needed for a microscale steam distillation apparatus: 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 (Padias 4th edition p 166, shown there for simple distillation…). Heat a sand bath to a setting appropriate to boil water in preparation for the distillation. During the distillation, you should adjust the sand bath heat to distill efficiently, but not allow frothy bubbles to boil up into the Hickman head.

To steam distill oil of cloves to isolate eugenol, place about 0.5 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. Attach your Hickman Head and a water cooled condenser. 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! Clamp the apparatus to your monkey bars, with the Hickman head port slightly tilted for easier distillate collection. Perch your sand bath on the lab jack and raise it to the round bottom.

Heat the mixture to provide a steady rate of distillation. Do NOT let the frothy mixture in the round bottom flask boil up into the Hickman head! 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. If the round bottom mixture starts to look too dry during the distillation, you may need to add more water to it. This can be achieved 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, the distillate will pool in the Hickman head trough. When a good bit of distillate has collected, transfer it to a 5 mL <u>conical</u> vial with a fresh pipette (label this vial container #1) . Continue collecting the distillate in container #1 until you have obtained at least 2.5 mL of distillate (leave space to add about 1 mL of ethyl acetate in the liquid-liquid extraction below).

The oil of cloves must now be extracted from this aqueous solution into ethyl acetate. To do this, add 1.0 mL of ethyl acetate to container #1 which holds the distillate. Gently mix the two phases using your pipette. Allow the layers to separate. Transfer the lower aqueous layer to your centrifuge tube (label this container #2).

Transfer the ethyl acetate layer left behind in container #1 to a screw cap vial (label this container #3).

Add another 1.0 mL of ethyl acetate to the aqueous layer that is now in container #2. Gently mix with your pipette. Allow the layers to separate. Transfer the lower aqueous layer back to container #1. Combine the ethyl acetate layer in container #2 with the first extraction of ethyl acetate in container #3.

For the final extraction, add another 1.0 mL of ethyl acetate to container #1 with the aqueous phase extracted above. Gently mix the two phases with your pipette. Allow the layers to separate. Transfer the lower aqueous layer into container #2. Pour the ethyl acetate layer in container #1 into container #3 with the other ethyl acetate extracts.

If any noticeable water remains in the ethyl acetate extracts, remove the blobs of water at the bottom of container #3 by touching the tip of a fresh pipette to the water blob. The water should move up the pipette from capillary action. With no visible signs of water left in the ethyl acetate, add 3-4 micro spatulas of sodium sulfate drying agent. Let the sodium sulfate sit for 10 minutes to soak up any extra dissolved water.

While the organic solution is being dried, clean and dry another 5 mL conical vial (label this container #4) 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 ethyl acetate through this gravity filtration device into the dry, tared container #4. Use small amounts of clean ethyl acetate to rinse container #3 and the filtered solid Na_2SO_4 to be sure of complete transfer of the solution into container #4.

Evaporate the ethyl acetate from the solution in container #4 by heating it very gently on the lowest setting of your hotplate 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 container #4 with its oil of cloves. By difference, compute the mass of the oil of cloves 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! ;-)).