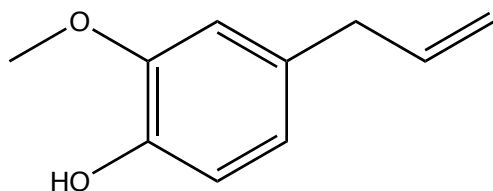


Co-Distillation: The isolation of eugenol from cloves

Purpose

To perform a steam distillation using a microscale distillation apparatus and isolate a natural product from cloves.

Background



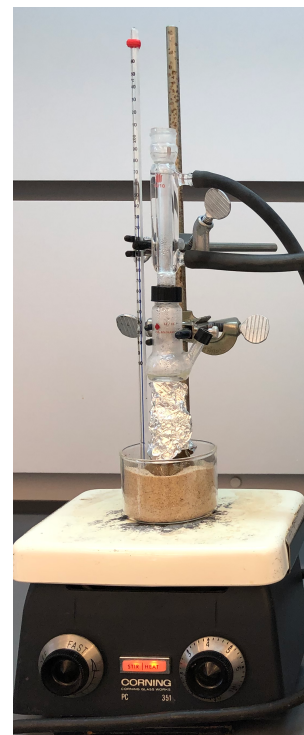
Eugenol

The boiling point of eugenol, an oil found in cloves, is 248 °C, but it can be isolated at a lower temperature by performing a co-distillation with water. Since the distillate will contain both water, eugenol and other oils, the clove oil must be extracted from the water using an organic solvent. After extraction into an organic solvent, the organic layer is separated from the aqueous layer and dried. The clove oil is finally isolated by evaporation of the organic solvent.

Procedure¹

Co-distillation

1. Combine 15 mL of water and 1 g of crushed, ground cloves in a 25-mL round-bottom flask.
2. Add a spin bar to the 25-mL round-bottom flask and assemble the microscale distillation apparatus (the flask, a Hickman still head, and a water condenser).
3. Make certain that the ground cloves are well wetted and soak the ground cloves for 15 minutes.
4. Turn on the cooling water for the condenser and heat the clove-water suspension using an aluminum block and a heating mantle. The temperature of the aluminum block should be maintained at approximately 130 °C and the bottom of the still should be wrapped with aluminum foil. Be aware, heating the suspension too vigorously may resulting in foaming, which will contaminate the distillate with ground cloves.
5. Periodically transfer the distillate from the Hickman head to a 15-



¹ Adapted from *Introduction to Organic Laboratory Techniques: A Microscale Approach*. Pavia, Lampman, Kriz, and Engel. (1999) Saunders College Publishing.

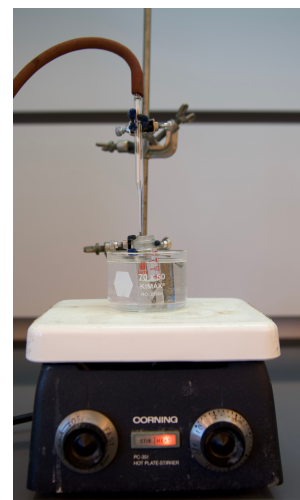
mL screw cap centrifuge tube and continue the steam distillation until 5–8 mL of distillate have been collected.

Extraction

6. To the water–clove oil emulsion add 3 mL of diethyl ether.
7. Cap the tube and shake (remember to vent the tube frequently).
8. Allow the layers to separate and transfer the diethyl ether–clove oil solution to a clean, dry 15-mL centrifuge tube. Make certain that no water is transferred during this step.
9. Add 2 mL of diethyl ether to the water–clove oil emulsion.
10. Cap the tube and shake (remember to vent the tube frequently).
11. Allow the layers to separate and transfer the diethyl ether–clove oil solution to the 15-mL centrifuge tube used in step 8. Make certain that no water is transferred during this step.
12. Add 2 mL of diethyl ether to the water–clove oil emulsion.
13. Cap the tube and shake (remember to vent the tube frequently).
14. Allow the layers to separate and transfer the diethyl ether–clove oil solution to the 15-mL centrifuge tube used in step 8. Make certain that no water is transferred during this step.
15. Dry the diethyl ether–clove oil solution with 2-3 microspatulas of anhydrous sodium sulfate.

Evaporation

16. If there is more than 5 mL of diethyl ether–clove oil solution transfer half of the dried diethyl ether–clove oil solution to a clean, dry, tared 5-mL conical vial otherwise transfer all of the diethyl ether–clove oil solution to the vial.
17. In a fume hood, evaporate the diethyl ether using a hot water bath (approximately 40 °C max. 50 °C).
18. Once the volume of diethyl ether is reduced sufficiently add the remaining diethyl ether to the tared 5-mL vial from step 16. Rinse the drying agent with a few drops of diethyl ether and transfer the diethyl ether rinse to the 5-mL conical vial from step 16.



IR Spectrum

Using the attenuated total reflectance (ATR) crystal collect an infrared spectrum of the eugenol.

Gas Chromatography

During the next lab period the clove oil will be analyzed by gas chromatography.

Report

The report for this experiment is described in the “An Introduction to the Gas Chromatograph: Collecting a chromatogram of clove oil” activity.