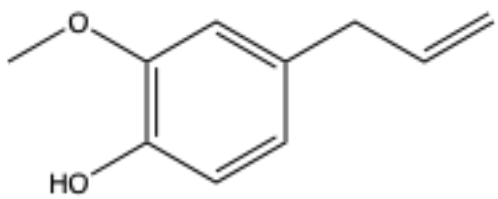
Steam Distillation: The isolation of eugenol from cloves

Purpose

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

Background

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, this process is also known as a steam distillation.



Eugenol

Since eugenol is only slightly soluble in water (2.46g/L at 25°C[1]), the concentration of the eugenol in the vapor over the boiling eugenol–water suspension does not depend on concentration of the eugenol. The relative amounts of eugenol and water in the vapor simply depend on the vapor pressures of the pure materials. The vapor pressure of water at 100 °C is 760 torr, and the vapor pressure of eugenol at 100 °C is approximately 4 torr; therefore, the vapor is roughly 0.5 % eugenol. (Note, the suspension boils when it's vapor pressure is equal to the external pressure. Since both the eugenol and the water are contributing to the vapor pressure of the suspension, the suspension will boil before either pure substance would normally boil.)

Since the distillate will contain both water and eugenol, the eugenol must be extracted from the water using an organic solvent. Once the eugenol is extracted into an organic solvent, the organic layer is separated from the aqueous layer and dried. The eugenol is finally isolated by evaporation of the organic solvent.

Procedure[2]

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.

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[1] https://pubchem.ncbi.nlm.nih.gov/compound/eugenol#section=Solubility accessed 2019/1/24.

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

4. Turn on the cooling water for the condenser and heat the clove–water suspension using a sand bath and a heating mantle.

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

Extraction

6. To the water–eugenol emulsion add 2 mL of CH_2Cl_2.
7. Cap the tube and shake (remember to vent the tube frequently).
8. Allow the layers to separate and transfer the CH_2Cl_2–eugenol solution to a clean, dry 5-mL conical vial. Make certain that no water is transferred during this step.
9. Add 1 mL of CH_2Cl_2 to the water–eugenol emulsion.
10. Cap the tube and shake (remember to vent the tube frequently).
11. Allow the layers to separate and transfer the CH_2Cl_2–eugenol solution to the 5-mL vial used in step 8. Make certain that no water is transferred during this step.
12. Add 1 mL of CH_2Cl_2 to the water–eugenol emulsion.
13. Cap the tube and shake (remember to vent the tube frequently).
14. Allow the layers to separate and transfer the CH_2Cl_2–eugenol solution to the 5-mL vial used in step 8. Make certain that no water is transferred during this step.
15. Dry the CH_2Cl_2–eugenol solution with 2-3 microspatulas of anhydrous sodium sulfate.

Evaporation

16. Transfer the dried CH_2Cl_2–eugenol solution to a clean, dry, tared, 5-mL conical vial.
17. Rinse the drying agent with a few drops of CH_2Cl_2 and transfer CH_2Cl_2 rinse to the 5-mL conical vial.
18. In a fume hood, evaporate the CH_2Cl_2 using a hot water bath (approximately 40 °C max. 55 °C) and a still.

IR Spectrum

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

Report

For this experiment, you will write an experimental, a formal description of the procedure you followed to isolate the eugenol. Remember to report the percent recovered for the eugenol at the end of your experimental and attach an IR spectrum of your product to the report.