

Name: Sapna Gupta; **Partner:** Perfect Partner

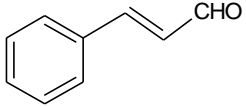
Title: Steam Distillation

Date: 29 September 2013

Purpose: Isolation of cinnamaldehyde from cinnamon using steam distillation.

Abstract and Theory: Cinnamaldehyde can be extracted from cinnamon using hot water (steam) as it is not soluble in water. The boiling point of a substance is lowered when two immiscible substances are boiled together (Raoult's Law). This principle is what is used to extract essential oils from natural products. The essential oil, cinnamaldehyde will be extracted using steam distillation, which is set up as a simple distillation apparatus. After distillation the product will be extracted from water using dichloromethane. The product will then be purified and characterized by IR spectroscopy.

Table of Quantities

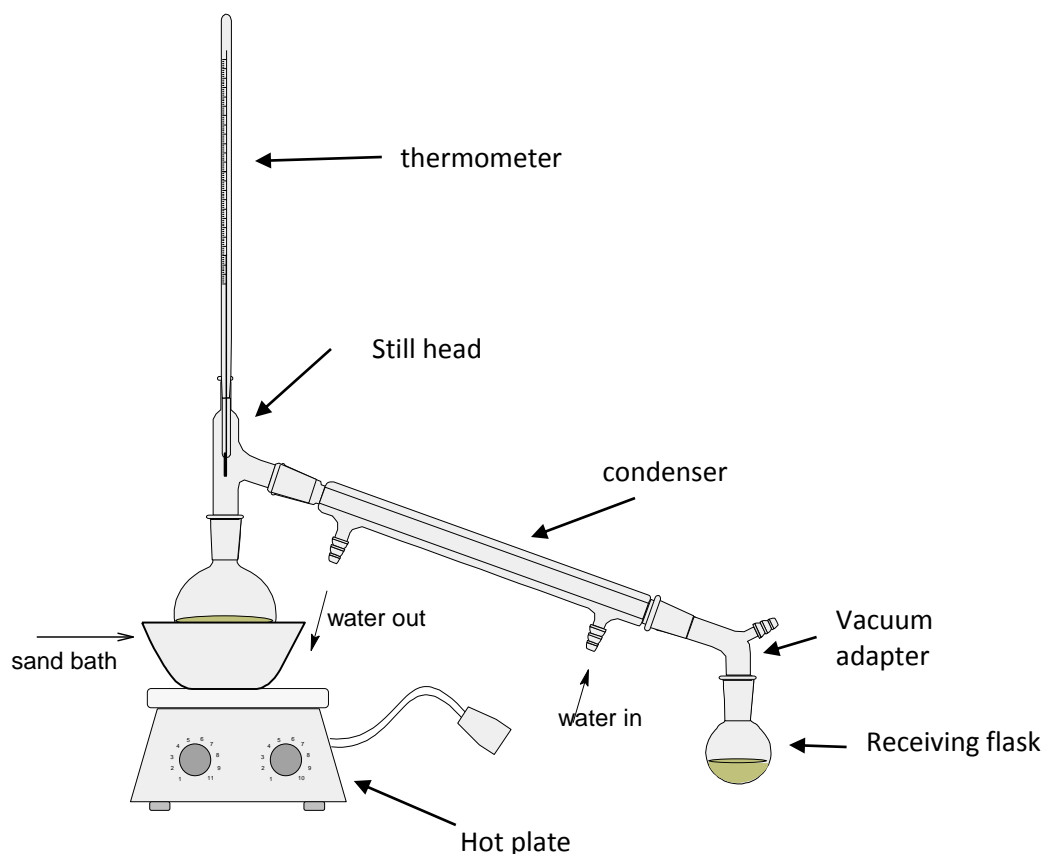
Substance	Formula/ Structure	Amount	Mol wt g/mol	Mpt °C	Bpt °C	Density g/mL	Sol. In H ₂ O
Cinnamon		1 g					No
Dichloromethane	CH ₂ Cl ₂	~ 7.0 mL	84.93		39.8	1.33	No
Sodium sulfate (anhydrous)	Na ₂ SO ₄	Small amount	142.04	880- 888		2.66	No
Cinnamaldehyde			132.16		248		No

Reaction: None

Should be finished up to here and submitted before starting lab.

Procedure

- 1) Placed 1.02 g of cinnamon powder and 10.00 mL of water in a 25 mL RBF (round bottom flask). The water will generate the steam in situ during the extraction process. A boiling chip was added to promote even boiling.
- 2) The RBF was attached to a water condenser and set up for simple distillation.
- 3) A thermometer was placed on the still head to make sure the temperature was maintained at or above 100 °C. (See diagram below).



- 4) The hot plate was turned on and steam distillation was commenced.
- 5) Sand bath on the hot plate was used to provide an even heating of the round bottom flask.
- 6) The distillate was collected until there were a few drops were left in the RBF. The distillate collected had a cloudy appearance indicating there was something mixed in the water.
- 7) A total of 7.5 mL of distillate was collected.
- 8) The distillate was transferred to a 25 mL centrifuge tube.

- 9) About 2 mL of dichloromethane was added to the centrifuge tube and the tube was agitated to extract the cinnamaldehyde into the organic layer. The dichloromethane is the lower layer as it is more dense than water.
- 10) The lower organic layer was pipetted into a clean dry test tube.
- 11) The extraction with 2 mL dichloromethane was done twice more to extract as much as cinnamaldehyde as possible. All the dichloromethane extracts were collected in the same test tube.
- 12) A small amount of sodium sulfate was added to the dichloromethane to dehydrate any water present. A little bit more sodium sulfate was added as droplets of water were seen floating on the surface of the dichloromethane.
- 13) A clean dry test tube was weighed (5.96 g) and the dried dichloromethane was decanted into this test tube.
- 14) This test tube was then placed in a hot water bath (prepared by the instructor) in the hood to evaporate the dichloromethane. Evaporation was continued until no change in volume was observed in the test tube.
- 15) The test tube was dried externally, cooled to room temperature and weighed with the product (6.09 g).
- 16) Analysis: A small sample of the product was placed on the sample holder of the IR machine and the spectrum was taken.

Calculations

$$6.09 \text{ g} - 5.96 \text{ g} = 0.13 \text{ g product}$$

No percent yield was required.

Conclusions

0.13 g of product was obtained. The product was clear and colorless liquid. It smelled like cinnamon. To verify the identity of the product IR was taken. The peak at 1720 cm^{-1} indicated the presence of an aldehyde, which is in the product; and the peak at 3040 cm^{-1} indicates the aromatic stretch of the benzene ring. These two peaks indicate that the product was indeed cinnamon.