

Lab 6 - Steam Distillation of *d*-limonene

Joshua Fitzpatrick

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1 Introduction

Steam distillation is a technique used to separate a volatile liquid from less volatile impurities by vaporization. The extract compound will vaporize at a lower boiling point than the impurities contained in the mixture, so the impurities will be left behind and only purified compound and water will re-condense and be collected the receiving flask. Further separation of the purified compound is achieved by use of a separatory funnel and rotary evaporator.

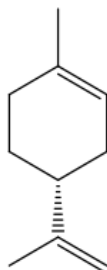


Figure 1: *d*-limonene

2 Materials

3.00g anhydrous sodium sulfate (Na_2SO_4)	1 heating mantle
30mL dichloromethane	stir plate
8.000g orange peels	1 variac
3 boiling chips	2 (50mL) Round bottom flasks
2 keck clips	1 (250mL) Round bottom flask
2 rubber tubes	50mL Erlenmeyer flask
west condenser	cold water source
thermometer adapter	Separatory funnel
vacuum adapter	rotary vaporizer
2 ring stands & clamps	IR spectrophotometer

3 Procedure

A distillation apparatus was assembled according to the graphic shown in *figure2*. using the materials which have been listed. All connections were lubricated and sealed air tight. All glassware was examined for for cracks and the assembly checked for defects before beginning the seam distillation.

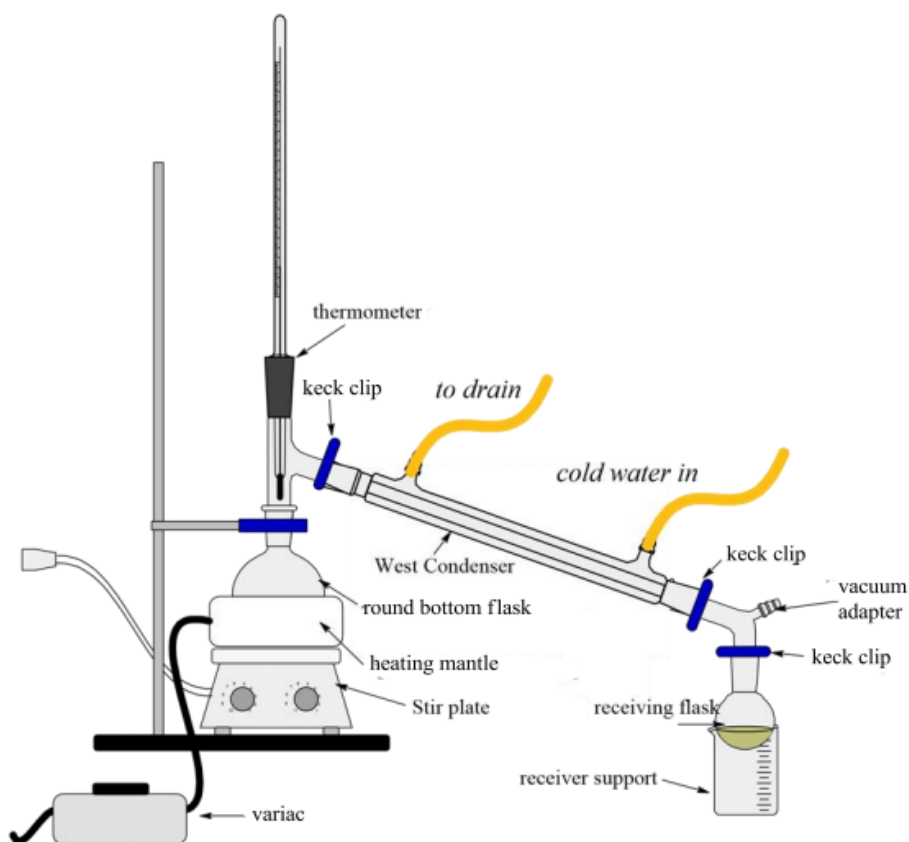


Figure 2: Distillation Apparatus example

8.0000g of orange peels, 150mL water, and 3 boiling chips were added to a 250mL round bottom flask. The flask was then attached to the distillation apparatus. The round bottom flask and thermometer adapter were then wrapped with an insulation of glass wool which was surrounded and held in place by aluminum foil. The water was turned on and began to flow through the west condenser. At this point the heating mantle was plugged in and the variac was set to 120v and the total percent voltage knob was turned to 70v. The as temperature rose the solution began to boil vaporizing the fluid mixture in the flask. The resulting gas condensed as it passed through the west condenser about 4 – 50mL distillate was collected in a 50mL round bottom flask. The distillate was then poured into a separatory funnel and extracted 3 times using 10mL dichloromethane each time. Emulsions were eliminated by adding salt water. The combined organic layers were collected in a 50mL erlenmeyer flask and dried with 3.00g of Na_2SO_4 . The flask was corked and left in a locker until the next lab period.

The following lab period the Na_2SO_4 was removed from the liquid by gravity filtration into pre-weighed 50mL round bottom flask. The remaining solvent was then removed by rotary vaporization. The final weight of the 50mL round bottom flask containing the purified *d*-limonene was weighed.

4 Measurements and Calculations

Experimental Data

Mass orange peels:

8.0000g

Mass 50ML round bottom flask:

Initial (empty) weight = 51.3190g

Final weight (with d-limonene) = 51.3313g

5 Results

d -limonene recovered = $M_f - M_i = 0.0123g = 12.3mg$

6 Conclusion

The objective of this lab was to isolate d limonene, which is contained within the husks of oranges, from any impurities. This was accomplished through a process of steam distillation where the lower boiling point of d limonene was taken advantage of by vaporizing only d limonene leaving the impurities, which had higher boiling points, behind in solution. The vaporized d limonene was recondensed as it passed through the wet condenser and collected in a receiving flask. After drying and removing the remaining solvent by rotary vaporization the receiving flask was weighed with its contents. The total yield of d limonene recovered by steam distillation was 12.3 mg.

Conclusion Responses

- Terpene is a hydrocarbon that makes up a class of fragrant organic compounds produced by a variety of plants and some insects.
- d limonene can be extracted from the rinds both oranges and lemons as well as other citrus fruits.
- Other compounds such as the L enantiomer of limonene can be obtained by steam distillation of pine. Other aromatics and many other temperature sensitive materials can be isolated by steam distillation.