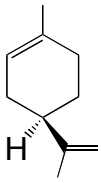
EXPERIMENT 7
ISOLATION OF LIMONENE FROM ORANGE PEELS
Friday, November 4 – Thursday, November 11, 2011

Steam distillation is used to isolate organic compounds that are immiscible liquids with water and have reasonably high vapor pressure at the boiling water temperature. We will use this technique to isolate limonene from orange peels. Limonene is a chiral compound, so we will use polarimetry to determine the specific rotation of our limonene sample and use that data to determine the enantiomeric excess (optical purity) of our limonene sample.

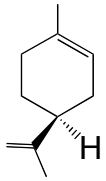
READING ASSIGNMENT:

Technique 13.6: Steam Distillation pp 164-166 in Techniques in Organic Chemistry 3rd Ed.
Technique 16.5-16.7: Optical Activity and Enantiomeric Analysis pp 203-211 in Techniques in Organic Chemistry 3rd Ed.
Experiment 6: Isolation of Essential Oils from Plants pgs 37-39 & 44-46 in Modern Projects 2nd Ed.
Review Extraction Techniques and Ch 5 in your Lecture text book.

Structures of (R)-(+) and (S)-(-)-Limonene



(R)-(+)-Limonene
bp 176°C
density 0.840 g/mL
$[\alpha]_D$ +125.6°



(S)-(-)-Limonene
bp 176°C
density 0.840 g/mL
$[\alpha]_D$ -122.1°

IMPORTANT SAFETY INFORMATION

(R)-(+)-Limonene is an irritant and readily absorbed through the skin**.** Limonene evaporates easily at room temperature and keep the container covered all the time.

EXPERIMENTAL NOTES:

A. Steam Distillation and Isolation of Limonene from Orange Peels

What You Need to Do Before Lab:

- **Bring an orange (unpeeled) to lab**.
- Rewrite the STEAM DISTILLATION procedure ***in your own words***! Note: Peel the orange in lab (or right before). Bring a sealable bag for your orange pieces if you would like to eat them. DO NOT EAT IN LAB!!!
- Rewrite the procedure below ***in your own words*** for the isolation of limonene from the distillate.

Remove approximately 10 mL of the top part containing the organic layer from the distillate into a 30-mL separatory funnel. All of the top layer should be transferred. Add 3 g of sodium chloride and shake the mixture to dissolve the salt completely. Drain off the bottom layer (salt water) until only a small amount of aqueous layer remains at the bottom of the separatory funnel. Transfer the remaining organic and aqueous layers into a centrifuge tube and separate the organic layer following the techniques: **11.5b Microscale Extractions with an Organic Phase Less Dense than Water**, page 127 in Techniques in Organic Chemistry 3rd Ed. Dry the organic layer by adding anhydrous calcium chloride ($CaCl_2$). The organic phase is completely dried when the $CaCl_2$ freely moves and no longer clumps. Let it stand for at least 10 minutes and collect the dried organic layer into a tared clean vial. **NOTE: Give your limonene sample to your TA. Everyone's samples will be combined to make a class sample for the next part of the experiment!**

Data to Record:

- Your observations!
- Mass and appearance of isolated limonene sample to the nearest milligram.

B. Polarimetry of Isolated Limonene

What to Do Before Lab:

- Complete the reading assignment! Pay special attention to sections 16.6 and 16.7
- Write a general procedure for use of polarimeter tubes.

Data to Record:

- Your observations!
- Measure the optical activity of the two limonene samples (R and S limonene in 100% ethanol) provided by the stockroom
- Measure the optical activity of the CLASS limonene. (Note: Your TA will collect and combine all of your products and let you measure the optical activity without dilution.)
- Calculate the specific rotation of all three samples. For the class limonene sample, use the density of limonene as the concentration term.
- Compare the specific rotation of the class sample to the two pure samples.
- Calculate the enantiomeric excess (%ee) for the class sample **using the literature optical rotations** for the pure R and S enantiomers.

NOTES FOR WRITING YOUR DISCUSSION:

Your Theory section should include:

- An overview of steam distillation – include an explanation of why it works!
- A brief discussion of optical activity of compounds

Your Results section should include:

- Appearance and mass of isolated limonene
- Observed specific rotation and calculation of the optical rotation and % ee of the class sample

Your Discussion section should include:

- An evaluation of your results: which enantiomer of limonene is present in excess in orange peels and how you determined that

Your Sources of Error/Future Experiments section should include:

- Comment on the reliability of your results and propose different experiments/ techniques that would address the identification and the determination of the purity of the compound you isolated. How does this affect the conclusions you made in your discussion?