**Isolation of (+)-Limonene from Orange Zest**

**Introduction**

Limonene is an example of terpene, a class of natural products biosynthesized by the assembly of isoprene units into various structures. In this lab’s case, Limonene is responsible for the odor of an orange. Organic chemists use terpenes and other natural products as chiral starting materials for complex chemical syntheses or as inspirations for pharmaceuticals.  
The main purpose of this lab is to isolate limonene from the peelings of citrus fruit; through steam distillation and we will analyze isolated limonene with IR spectroscopy and polarimetry.

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**Physical Data and Hazards:**

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| --- | --- | --- | --- | --- | --- |
| **Chemical Formula & Name** | **Molecular Weight (g/mol)** | **Melting Point (C)** | **Boiling Point (C)** | **Density (g/cm3)** | **Hazards** |
| *Menthol* | 156.27 | 36-38 | 212 | 0.890 |  |
| *Limonene* | 136.24 | -74.35 | 176 | 841.10 (kg/m^3) | Irritant |
| *Ethanol* | 46.07 | -114 | 78.37 | .7893 | Toxic and flammable |
| *Sodium Sulfate* | 142.04 | 884 | 1429 | 2.66 | Irritant |

*Sources: Handbook for Organic Chemistry,* ***CRC Handbook of Chemistry and Physics*** *(especially Section C: "Physical Constants of Organic Compounds" ), available at the information desk in the Science Library (in Norlin) and in the Organic Chemistry Stockroom.*

**Safety Precautions**

*Pure limonene is an irritant and flammable.*

*Ethanol is flammable and toxic*

*Do not cut yourself with the knife or peeler you use to remove the rind from the oranges.*

**Wastes**

Place all waste from this lab, including peeled oranges, into the bucket in the fume hood. Do not pour pulp into the sink

**Procedure**

1. Peel orange with a sharp knife. Remove the skin/exterior (zest).
   1. The white portion underneath the zest doesn’t contain much limonene
2. Weigh the mass of the zests.
3. Place them in the blender
4. Add 50ml of water
5. Blend the mixture.
   1. Make sure to place the lid on the blender before blending it.
   2. Until it’s a smooth puree
6. Place 50 ml of the puree into the round bottom flask.
7. Add a couple boiling chips
8. Attach the flask to the steam distillation system.
9. Use a 10 ml graduated cylinder for receiving flask.
10. Place the heating mantle under the distillation flask.
    1. Make sure it is connected to the Variac; heat it to 6-7.
    2. Do not heat it too quickly.
11. Periodically add 5 ml to 10ml portions of water to the distillation flask to replace the water that has already been distilled.
    1. Maintain the liquid level to half full
    2. Add water by removing the thermometer adapter and pour it through the y adaptor.
12. An upper layer in the receiving flask would be forming of limonene. When it is close to full, remove the layer by using a Pasteur pipet and put it into a clean, tared vial.
    1. Tare the vial beforehand.
13. Pour the remaining water into a separate beaker and then place the graduated cylinder back as a receiving flask.
14. Continue this process for about 45 minutes.
15. If you see some water in the tared vial, add sodium sulfate and then pipet it into a clean tared vial.
16. Weigh the mass of the limonene
17. Obtain an IR spectrum
18. Measure the optical rotation of the sample in ethanol using a polarimeter.