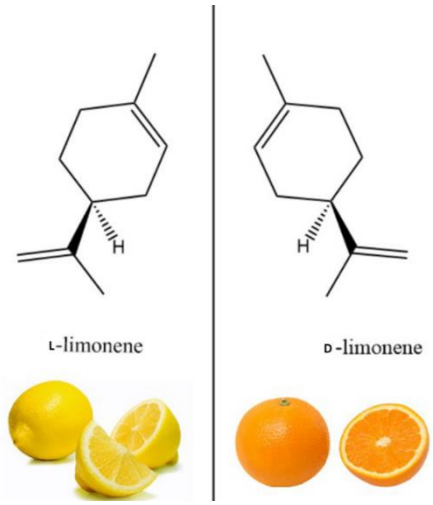
PW 5: Extraction of Limonene contained in oranges

1. Introduction

Limonene is a versatile compound with a wide range of applications in food, cosmetics, and industry, making it an important substance derived from citrus fruits.



Limonene is a colorless, aromatic liquid classified as a monocyclic monoterpene. It is primarily found in the peels of citrus fruits, such as lemons, oranges, and limes, and is responsible for their characteristic citrus scent. Here are some key points about limonene:

- Chemical Formula: $C_{10}H_{18}$
- Boiling Point: Approximately 176°C.
- Molecular Structure: Limonene has a cyclic structure, which contributes to its unique properties and aroma.

Limonene is abundant in the essential oils of citrus fruits, particularly in their peels, it can also be found in other plants and is often used in flavoring and fragrance applications.

Monoterpenes are a class of organic compounds composed of two isoprene units, resulting in a structure that typically contains 10 carbon atoms ($C_{10}H_{18}$). They can have various structures, including linear (acyclic) and cyclic forms.

2. Objectives of the manipulation

- ✓ Extraction to extract limonene from orange peel.
- ✓ Become familiar with the Hydro-distillation process.
- ✓ Become familiar with the decantation process.

3. Equipment and chemicals

Equipment	Chemicals
 **Round bottom flask**	Orange peels

 **Graduated cylinder**	Distilled water
 **Separating funnel**	Diethyl ether
 **Refregirant**	
 **Heating mantle**	
 **250 mL beakers**	
 **Magnetic bar**	
 **100ml Erlenmeyer**	

Hydro-distillation setup

4. Operating mode

Before starting, check if the glassware is clean and dry.

Confirm that the tank-heater is set to a temperature that does not exceed 90°C.

✓ **Preparation of Orange Peels:**

- Grate the outer orange-colored rind of two oranges. Weigh the grated peels (m=15g) to determine the extraction yield later.

✓ **Setting Up Hydro-Distillation:**

- Place the grated orange peels into a 250 cm^3 round-bottomed flask.
- Add 100 cm^3 of distilled water to the flask to facilitate hydro-distillation.

✓ **Heating the Mixture:**

- Connect the flask to a heating mantle and begin heating the mixture.
- As the water heats, it will produce vapor, which carries the aromatic compounds from the orange peels.

✓ **Condensation:**

- The vapor rises into the column head and enters a water-cooled condenser, where cold tap water circulates around it, cooling the vapor back into liquid form.

✓ **Collecting the Distillate:**

- The distillate, which is collected in a graduated cylinder, consists mainly of water but is highly aromatic, indicating the presence of essential oils from the peel.

✓ **Maintaining Water Levels:**

- Use a dropping funnel to add additional distilled water to the boiling flask as needed. This prevents the orange peels from burning during the heating process.

✓ **Stopping Distillation:**

- Continue the hydro-distillation until approximately 70 mL of distillate has been collected. This amount is typically sufficient for effective extraction.

✓ **Separatory Funnel Extraction:**

- Transfer the collected distillate into a separatory funnel. Add a suitable volume of ether, which is a non-miscible liquid with water, to extract the orange essential oil effectively.

✓ **Agitation and Degassing:**

- Gently agitate the separatory funnel to mix the layers. Be cautious, as ether is highly volatile. After agitation, allow the funnel to degas, releasing any trapped gases.

✓ **Separation of Phases:**

- Let the separatory funnel sit undisturbed to allow proper separation between the aqueous phase (water) and the organic phase (ether and orange essential oil).

✓ **Collecting the Essential Oil:**

- Once the layers have separated, carefully drain the aqueous phase from the bottom of the funnel. Retain the organic phase, which contains the ether and orange essential oil.

✓ **Evaporation of Ether:**

- To isolate the orange essential oil, evaporate the ether. This can be done using an evaporating dish or a rotary evaporator, ensuring that the oil is collected in a suitable container.

5. Questions

- What is the purpose of hydro-distillation in the extraction of limonene from orange peels?
- What type of flask is used for the hydro-distillation process, and why is it preferred?
- Why is distilled water added to the orange peels in the round-bottomed flask?
- What is the significance of using a heating mantle in this extraction process?
- What does the aroma of the collected distillate indicate about its composition?
- Why ether is chosen as the solvent for extracting limonene from the distillate?
- Calculate the yield of the reaction.