Lab N °06: Extraction of limonene contained in oranges

Objectives

During the session, implement the following experimental techniques:

- Hydrodistillation
- Extraction to extract limonene from orange peel.
- Decantation.

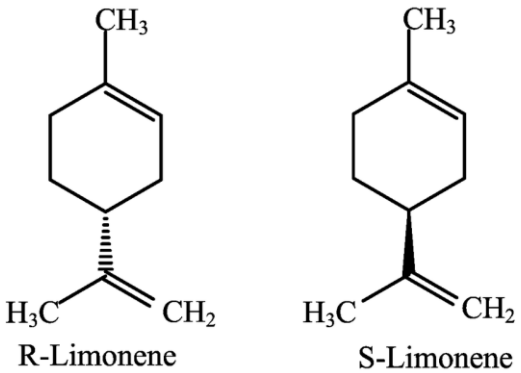
Introduction

Oranges contain not only water and carbohydrates but also significant amounts of acidic compounds like vitamin C (ascorbic acid), citric acid, and malic acid. Additionally, oranges contain aromatic compounds, comprising just around 0.02% of the orange's mass, yet crucial for the sensory experience of the fruit.

The primary aroma in oranges is limonene, a cyclic monoterpene belonging to the terpene family. Limonene exists in two enantiomeric forms, possessing identical physical properties but opposite specific optical rotation powers.

Limonene, a colorless liquid aliphatic hydrocarbon, is the major component of citrus fruit peel volatile oil. It is relatively stable and can be distilled without decomposition, although it may crack to form isoprene at high temperatures. Found abundantly in citrus fruit peels and essential oils, limonene imparts a pleasant lemon-like aroma.

Limonene comes in two forms: D-limonene and L-limonene, both contributing to its characteristic fragrance. Commonly used in fragrances, soaps, shampoos, and cosmetics, limonene also serves as a flavoring agent in various food products.



R-Limonene S-Limonene

Physical Properties

Limonene is colorless liquid oil at room temperature with a characteristic odor. It is insoluble in water but soluble in alcohol and ether. It has boiling point of 74^0C

Materials & Equipment

Materials	Equipment
Oranges peels	Hydro distillation setup
Diethyl ether	100 mL graduated cylinder
Distilled water	Separatory funnel
	Erlenmeyer flask
	Spatula

Procedure

1. Grate the ***outer*** orange colored rind of two oranges and weigh them (m = g) (to determine the extraction yield).
2. Place the peels in the $250\ cm^3$ round bottomed flask and add distilled water $100\ cm^3$ to proceed with hydro-distillation
3. Heat the mixture using a heating mantle. Water vapor forms and carries with it the aromas contained in the orange peels. This gaseous mixture rises into the column head and then enters a water-cooled condenser, through which cold tap water flows.
4. The distillate is collected in a graduated cylinder.
5. The dropping funnel contains water, allowing additional water to be added to the flask to prevent the orange peels from burning under heating.
6. The collected distillate mainly consists of water, but it is highly aromatic, indicating that it indeed contains the aromas present in the orange peel.
7. Stop hydro-distillation when approximately 70 mL of distillate is collected.
8. Transfer the distillate into a separatory funnel for extraction, to which ether is added. Ether is a non-miscible liquid with water, in which orange essential oil is highly soluble.
9. Proper use of the separatory funnel requires regular agitation and degassing. Ether is a highly volatile liquid.
10. Allow the separatory funnel to rest to ensure proper separation between the aqueous phase containing water and the organic phase containing ether and orange essential oil.
11. Once the extraction is complete, evaporate the ether to collect the orange essential oil.

Questions

1. Draw the setup used for extracting orange peels and name all its parts.
2. What is hydro-distillation (steam distillation)
3. Specify the role of the condenser.
4. Calculate the yield of the prepared extract.