

Extraction of Limonene from Citrus Fruit Peels

An Inquiry-Based Experiment

Summary

Reference:

<http://academic.bowdoin.edu/courses/s04/chem226/chem226a/laboratory/images/limoneneisolation.pdf>

R-(+)-limonene gives the characteristic smell of orange peels. It belongs to an important class of organic compounds known as terpenes, and constitutes the major component (~ 90%) of the essential oil isolated from the orange peel zest. Mixtures of volatile and odoriferous compounds, very rich in terpenes, can be obtained by steam distillation of plant tissues. These mixtures are called essential oils.

There are several methods that can effectively isolate limonene from orange peels. Two of the most commonly used methods are by solid-liquid extraction of limonene into an appropriate solvent, and by steam distillation. In the first method a suitable solvent has to be chosen in order to maximize the amount of extracted limonene.

In this project you will exercise collaborative efforts to investigate the following issues related to the isolation of limonene. Attached is a typical experimental procedure for isolating limonene from orange peel zest. You may change part of the experimental details to investigate how the changes affect the efficiency of the extraction process.

1. The efficiencies of the two different methods.
2. The best choice of solvent and temperature for maximizing the amount of extracted limonene.
3. The choice of organic solvent for extracting limonene from the aqueous distillate collected from steam distillation.

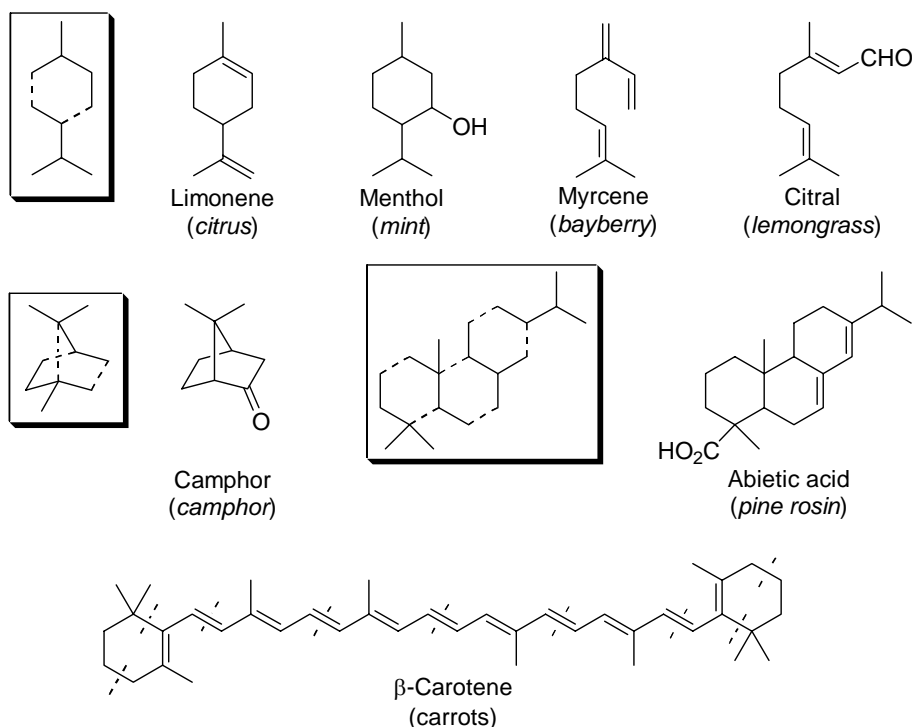
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Lab Documentation

Introduction

Many plants and trees are known to have distinctively pleasant odors due to the presence of essential oils. They are highly volatile substances isolated from odoriferous plants. Although steam distillation is the most common method for isolating essential oils, other processes (such as solvent extraction and mechanical pressing) are sometimes used for certain types of products.

Many compounds present in the essential oils that are responsible for the pleasant odors contain exactly ten carbon atoms. These ten-carbon compounds are known as terpenes if they are hydrocarbons and as terpenoids if they contain oxygen and are alcohols, ketones, or aldehydes. The terpenes are usually grouped according to the number of isoprene units (C_5H_8)



Some examples of terpenes and terpenoids.

Limonene is a naturally occurring hydrocarbon found in the essential oils of spearmint, caraway, lemon, and orange oils. R-(+)-Limonene is the main component

(~90%) of the essential oil of oranges. Orange peel zest (the orange part) contains about 0.5 – 1% of limonene by weight. Two of the most commonly used methods of isolating limonene are solid-liquid extraction of limonene into an appropriate solvent, and steam distillation.

Shown below are two typical experimental procedures for isolating limonene from orange peel. You can compare the effectiveness of these two isolation methods. You may also apply these procedures to other kinds of fruit peels such as lemon and grapefruit, and compare the essential oil content of these different types of fruit peels. Furthermore, instead of strictly following the given procedure, you may also change some of the experimental conditions such as extraction temperature or the organic solvent used to investigate how varying these factors will affect the efficiency of the extraction process.

Experimental Procedure

Method 1: Isolation of Limonene by Extraction into a Solvent

Materials and Apparatus:

Solvent: *n*-hexane, dichloromethane, ethyl acetate, ethanol, diethyl ether

Fruit peels: orange, lemon, grapefruit

Anhydrous magnesium sulfate

5% ethanolic phosphomolybdic acid

TLC plates

Stirring hotplate

Rotary evaporator

General lab glassware kit

50-mL pear-shaped flask

Grater

Use a grater to obtain about 5-8 g of orange peel zest and transfer the peel zest into a 50 mL conical flask. Choose an organic solvent and add 20 mL of that solvent to the grated peel zest. Stir the mixture (with or without heating) for 15 minutes.

Remove the solid residue by simple filtration and dry the filtrate with anhydrous magnesium sulfate (MgSO_4). Add the drying agent in portions with swirling until it no longer clumps together. Remove the drying agent by simple filtration. Wash the drying agent with several small portions of fresh solvent and combine the washings to

the filtrate.

Transfer the dried solution to a pre-weighed 50 mL pear-shaped flask. Evaporate the solvent using a rotary evaporator to obtain the crude limonene.

(Optional) Analyze the crude product by TLC (thin-layer chromatography) using *n*-hexane as the eluent. You may obtain a standard sample of limonene from the laboratory assistant for comparison.

Since limonene is a colorless compound, you need to use phosphomolybdic acid to visualize the spots on TLC plate. Dip the TLC plate into a 5% ethanolic phosphomolybdic acid solution very briefly and then heat the plate with a hot-air gun. The sample spots will appear as deep blue or deep green spots.

Method 2: Isolation of Limonene by Steam Distillation

Materials and Apparatus:

Solvent: *n*-hexane, dichloromethane, ethyl acetate, diethyl ether
Fruit peels of orange, lemon, and grapefruit
Saturated sodium chloride solution
Anhydrous magnesium sulfate
5% ethanolic phosphomolybdic acid
TLC plates
Stirring hotplate
Rotary evaporator
General lab glassware kit
Apparatus set for simple distillation
50-mL pear-shaped flask
Grater

Use a grater to obtain about 5-8 g of orange peel zest and mix the peel zest with 50 mL of water in a 100 mL round-bottomed flask. Attach the flask to a simple distillation apparatus.

Heat the mixture with an oil bath to provide a steady rate of distillation. Allow the distillation to proceed until about 20 mL of distillate has been collected, or wait until no more organic substance distills out.

Transfer the distillate to a small separatory funnel and add 5 mL of dichloromethane to extract the organic substances from the distillate. Collect the organic solution and repeat the extraction two more times.

Transfer the combined organic solutions back to the separatory funnel and extract the organic solution with 20 mL of saturated sodium chloride solution to remove the residual water. Collect the organic solution in a dry 100 mL conical flask and dry the solution with anhydrous magnesium sulfate (MgSO_4). Add the drying agent in portions with swirling until it no longer clumps together.

Remove the drying agent by simple filtration. Wash the drying agent with several small portions of dichloromethane and combine the washings to the filtrate. Transfer the solution to a pre-weighed 50 mL pear-shaped flask. Evaporate the solvent using a rotary evaporator to obtain the crude sample of limonene.

(Optional) Analyze the crude product by TLC using *n*-hexane as the eluent. You may obtain a standard sample of limonene from the laboratory assistant for comparison.

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Data Sheet

Name of Participants: _____

Type of Fruit: _____

Amount of Sample Used: _____

Method Used for Extraction: _____

Solvent Used for the Extraction: _____

Amount of Limonene Obtained: _____ (%)

Remarks / Other Comments: