# Chirality - isolation of limonene from citrus <br> fruits essay 

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Terpenes are a large and varied class of hydrocarbons produced by a wide variety of plants. Many essential oils belong to the terpene class. They are made up of two or more five carbon units that are called isoprenes. Limonene is a terpene that can be separated from orange peels almost 100\% in the (R)-(+)-limonene form. Other citrus fruits like lemons, grapefruits, limes, and tangerines contain the compound as well. The purity of the oil extracted from the oranges can be determined by physical properties such as optical rotation and refractive index.

In this experiment, limonene will be isolated by steam distillation. Normally, big molecules such as limonene require very high temperatures to boil. In the presence of water, liquids which are immiscible with water tend to boil below its normal boiling point. Steam distillation looks like this: Source: http://mywadud. wordpress. com Normal distillation would require extremely high temperatures and would be more time consuming. Distilling in the presence of water, however, helps to avoid this problem by keeping the temperature low.

Polarimetry measures how much a substance is able to interact with plane polarized light. Anisotropic crystalline solids and samples that have an excess of one enantiomer of a chiral molecule, can rotate the orientation of plane-polarized light. These substances are said to be optically active. In order to be optically active, the substance must have a chiral center. A chiral center consists of a carbon that has four different groups attached to it. A sample that has only one enantiomer of a chiral molecule is said to be optically pure.

The compound can either may either rotate plane polarized light to the left or the right. The enantiomer that rotates light to the right, or clockwise when viewing in the direction of light propagation, is called the dextrorotatory (d) or (+) enantiomer, and the enantiomer that rotates light to the left, or counterclockwise, is called the levorotatory (I) or (-) enantiomer. The magnitude and the direction of rotation are measured to give the observed rotation. The observed rotation will have to be corrected for the length of the cell used and the concentration of the solution.

The corrected observed rotation can be compared to literature values in order to identify an unknown compound. The simplest way of measuring optical purity is using an instrument called a polarimeter. The simplest polarimeter consists of a monochromatic light source, a polarizer, a sample cell, a second polarizer, which is called the analyzer, and a light detector.

The analyzer is oriented 90 oto the polarizer so that no light reaches the detector. Optical purity is measured in terms of enantiomeric excess or \% ee. The formula is $\%$ ee $=[?]$ observed/ [? pure $* 100 \%$. When a ray of light passes from air into a block of glass, the direction of the light changes. The amount of bending that takes place depends on the nature of the glass and the wavelength of the light being used. In all these investigations the yellow light emitted by sodium ions is used. It has a wavelength of 589 nanometers. The refractive index is the ratio of the velocity of light in air to the velocity of light in liquid and is always greater than one. Refractive index, much like melting point or boiling point, can be used to characterize liquids.

Refractive index is measured on a refractometer. Compensation needs to be made for the instrument because white light, instead of sodium vapor light, is used. The temperature can be corrected by adding 0.00045 for every degree above 20oC. Experimental Procedure: The skin was peeled off an orange and the white pulp was carefully scraped off with a metal scoop. The peelings were weighed and taken to the stock room to be blended. Meanwhile the steam distillation was set up according to the picture provided. The orange peel slush was transferred to a $500-\mathrm{mL}$ round bottom flask.

A $50-\mathrm{mL}$ graduated cylinder was attached to the apparatus and the water and heat was turned on, allowing the steam distillation to begin. The mixture was heated to a gentle boil and then the distillation was allowed to begin. The heat was turned off after the temperature of the steam stayed constant. The distillation continued until approximately 40 mL of the liquid was obtained. The 40 mL of distillate was transferred to a 250 mL separatory funnel. A small amount of dichloromethane was used to wash the receiver after the transferal.

The distillate was extracted with 10 mL of dichloromethane three times, each time using a little extra dichlormethane to wash each layer. The organic layer on the bottom was collected for each extraction. All organic layers were collected in a 125 mL Erlenmeyer flask. Anhydrous sodium sulfate was used to dry the solution for fifteen minutes. Meanwhile, a vacuum filtration apparatus was set up accordingly. A 250 mL filter flask was obtained and pre-weighed. The solution containing the limonene was vacuum filtrated while it was kept warm in a steam bath.
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Additional Sodium Sulfate was added to the solution to remove excess water. The solution was then washed 3-4 times with dichloromethane and the vacuum filtration continued. The vacuum pump was turned off when the flask contained only a small amount of oil. The flask was then weighed. After the limonene was isolated in part one, a few drops of it was used to find the refractive index. A Pasteur pipette was used to place a few drops of the oil on the measuring prism. This part was handled by a TA. The result obtained was corrected using the formula for 200 C in the lab manual.

The isolated limonene left over was dissolved in 3 mL of ethanol. The solution was then shaken to get rid of the cloudiness and get a clear solution. Using a Pasteur pipette, the solution was transferred to a 10 mL volumetric flask. The Erlenmeyer flask was rinsed several times with 1 mL portions of ethanol and transferred to the volumetric flask. The flask was filled up to the calibration mark, with ethanol. The flask was inverted several times to mix the solution thoroughly. The solution was handed over to a TA to measure the observed rotation and the observed rotation was recorded.

Table of Chemicals: Formula: C10H16 Molar Mass: 136. $24 \mathrm{~g} / \mathrm{mol}$ Melting Point: -74oC Boiling Point: 175-177oC Density: $0.844 \mathrm{~g} / \mathrm{mL}$ Refractive Index: n20 D 1. 47 Solubility: insoluble Vapor Density: 4. 7 Vapor Pressure: < 3 mmHg Toxicity/ Hazards: Extremely Hazardous in case of eye contact or ingestion. Flammable Formula: CH2Cl2 Molar Mass: $84.93 \mathrm{~g} / \mathrm{mL}$ Melting Point: -97oC Boiling Point: 39. 8-40oC Density: $1.325 \mathrm{~g} / \mathrm{mol}$ Vapor Density: 2. 9 Vapor Pressure: 24.45 psi Solubility: 20g/L Toxicity/ Hazards: Contact can severely irritate and burn the skin and eyes.

Possibly a carcinogen. Formula: Na2SO4 Molar Mass: $142.04 \mathrm{~g} / \mathrm{mol}$ Appearance: white crystalline solid Density: $2.664 \mathrm{~g} / \mathrm{cm} 3$ Melting Point: 884oC Boiling Point: 429oC Solubility: 4.76 g/L Toxicity/ Hazards: Minor skin/ eye irritant Formula: C2H6O Molar Mass: $46.07 \mathrm{~g} / \mathrm{mol}$ Appearance: colorless liquid Density: . 789 g/cm3 Melting Point: -114oC Boiling Point: 78. 37oC Toxicity/Hazards: Avoid strong oxidizing agents, peroxides, acids, acid chlorides, acid anhydrides, alkali metals, ammonia. Flammable, causes blindness, narcosis, and irritation.

