

Extracting limonene from oranges by steam distillation

This experiment demonstrates the extraction of plant oils.

The peel of **oranges** is boiled in water and the oil produced (**limonene**) distilled in steam at a temperature just below 100 °C, well below its normal boiling point. The immiscible oil can then be separated. Direct extraction by heating would result in decomposition whereas **steam distillation** does not destroy the chemicals involved.

The experiment also links for tests for **unsaturation**, and at a higher level, **chirality**.

Lesson organisation

This experiment can be conducted as a demonstration at secondary level as an introduction to some of the ideas about the extraction of plant oils. It can be used to stimulate discussion about the commercial extraction of plant oils – how science works.

As described this demonstration will take a full lesson of approximately 50 minutes.

This can also be conducted as a class practical at key stages 4 and 5. It can stimulate further discussions as to the process of steam distillation where oil with a boiling point of 176 °C is “distilled” at just under 100 °C.

Limonene is an unsaturated hydrocarbon which can be tested for using bromine water or potassium manganate (VII). At a higher level, it is also a chiral compound and the experiment can lead to a discussion of optical enantiomers.

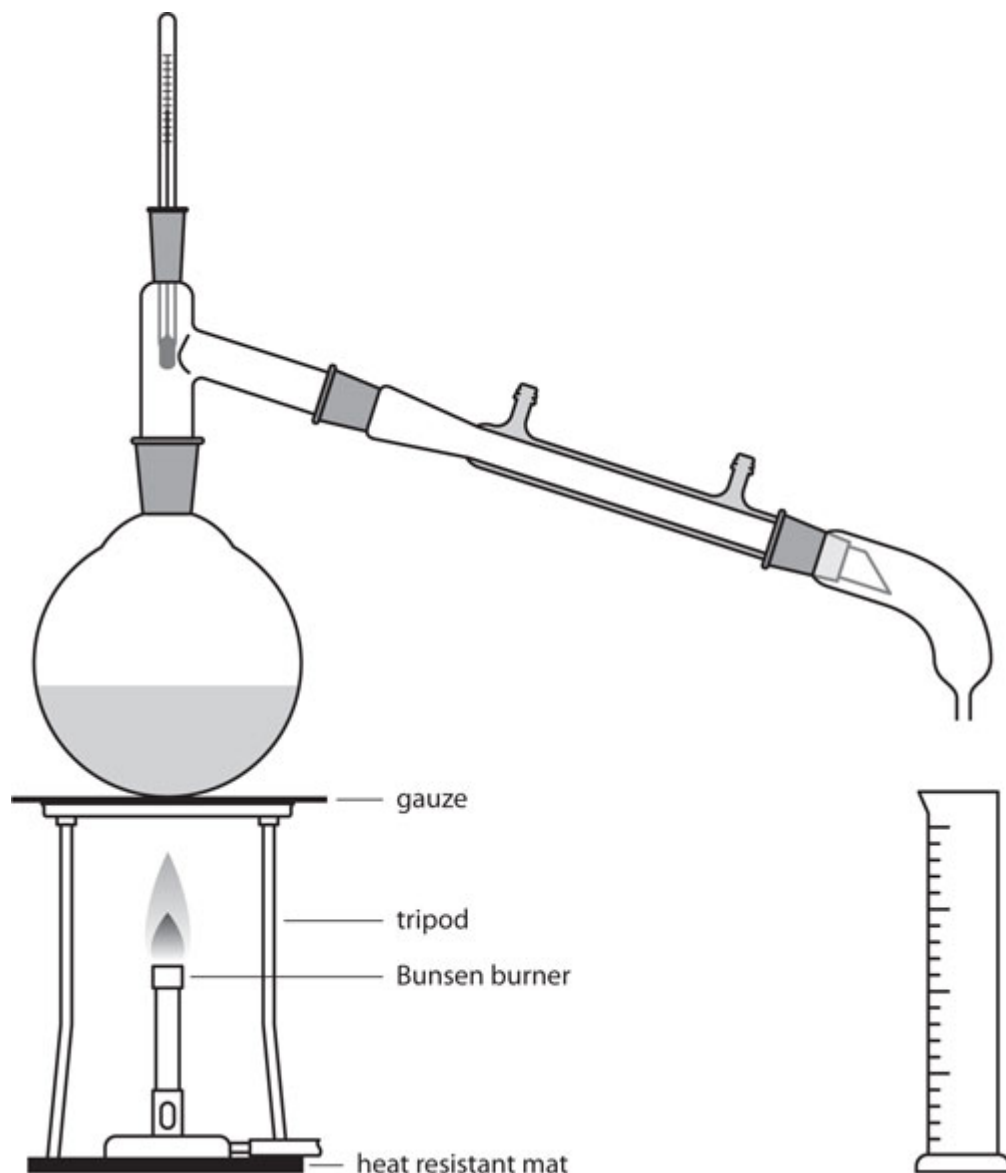
Apparatus and chemicals

- Eye protection
- Grater
- Bunsen burner
- Heat resistant mat
- Tripod and gauze
- Oranges, 2
- 110 °C thermometer
- Measuring cylinder (100 cm³)
- Measuring cylinder (50 cm³)
- Distillation apparatus
- 250 cm³ round bottomed flask
- Still head
- Thermometer pocket
- Condenser
- Receiver adapter
- Test tubes and bungs, 3
- Dropping pipette
- Anti-bumping granules
- Bromine water, no more than 0.2% v/v (**Irritant**)



- Potassium manganate(VII), 0.001M
- Cyclohexene (**Highly flammable, Harmful if swallowed, Toxic in contact with skin**)
- Cyclohexane (**Highly flammable, skin and respiratory irritant**)
- Distilled water, 100 cm³.

Apparatus



Technical notes

1 Cyclohexene is **Highly flammable, Harmful if swallowed** and **Toxic in contact with skin** and cyclohexane is **Highly flammable** and a **skin and respiratory irritant**.

2 Bromine water is **toxic and irritant**. The concentration should not exceed 0.2% v/v.

3 0.001M potassium manganate(VII) solution is **of low hazard**. This is a substitute for bromine water for student use.

Procedure

HEALTH & SAFETY: Wear eye protection and gloves for handling the cyclohexene.

Stage 1

- a Grate the outer orange coloured rind of two oranges and add to 100 cm³ of distilled water in the 250 cm³ round bottomed flask. Add anti-bumping granules to the round bottomed flask.
- b Set up the distillation apparatus as shown in the apparatus section.
- c Heat the flask so that distillation proceeds at a steady rate, approximately one drop per second of distillate. (Note: Take care not to let the liquid in the round bottomed flask boil too strongly).
- d Collect approximately 50 cm³ of distillate in the measuring cylinder. The oil layer will be on the surface.
- e Using a dropping pipette carefully remove the oil layer into a test tube for the next stage.

Stage 2

Odour

- f Cautiously smell the extracted oil by wafting the fumes towards the nose. Do not breathe in directly from the test tube.

Action of bromine water

- g Measure out approximately 1 cm³ of bromine water into each of three test tubes.
- h Add a few drops of the limonene oil to one test tube, a few drops of cyclohexane to another, and a few drops of cyclohexene to the third. Place in the bungs and agitate. If the bromine water is decolourised the molecule contains double bonds.
- i 0.001M potassium manganate(VII) can be substituted for the bromine water for class use. However, students need to know the action of bromine water.

Additional notes

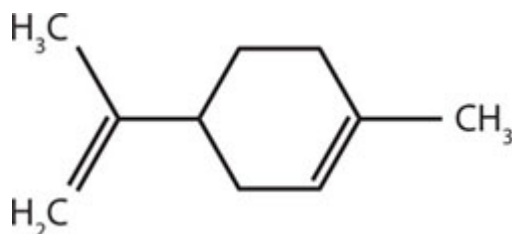
The amount of oil extracted varies considerably with the variety, season and storage of the oranges. However, it is always possible to extract sufficient.

Do not distill more than 50% of the initial volume of water or solid "jam" will form in the flask which is difficult to remove.

Always use a gauze on the tripod or the orange will burn.

Teaching notes

Limonene (1-methyl-4-prop-1-en-2-yl-cyclohexene) is an unsaturated hydrocarbon, classed as a terpene. At room temperature it is a colourless oily liquid with the smell of oranges. Its molecular formula is C₁₀H₁₆ and its boiling point is 176 °C.



Limonene is a chiral molecule with two optical isomers (enantiomers). The major biological form *d*-limonene, the (*R*)-enantiomer, is used in food manufacture and medicines. It is also used as a fragrance in cleaning products, a botanical insecticide, and due to its flammability, a potential biofuel.

The (*S*)-enantiomer, *l*imonene, is also used as a fragrance but has a piney, turpentine odour.

It is possible to allow students to observe the optical activity of chiral molecules by comparing saturated glucose solution with distilled water in a polarimeter.

Reference

This experiment was written by Andrew Thompson on behalf of the RSC

Credits

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