



OFF[®] from Oranges??

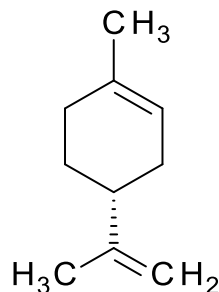
Extraction of Limonene, a Natural Insect Repellent, from Citrus Fruits

The Problem

- SC Johnson, makers of OFF[®], is looking to prepare a brand-new “green” insect repellent.
- Their most famous OFF[®] product uses DEET (N,N-diethyl-m-toluamide). DEET is a man-made chemical that is derived from petroleum products.
- The scientists at SC Johnson found a “natural” insect repellent called limonene that is available right here in the USA
- Limonene is a chemical substance made by citrus fruits
- SC Johnson can get orange and lemon peels from the makers of *Tropicana* at a very low price.
- The problem is, “What peel is better to use?” and “How best to get the limonene out?”

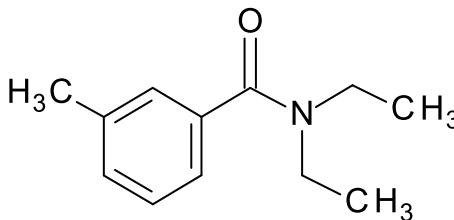
Structures of Limonene and DEET

(D)-Limonene **(4R)-isopropenyl-1-methylcyclohexene**



Molecular Formula = C₁₀ H₁₆ Molecular Mass = 136.23 Boiling Point = 176 °C

N,N-Diethyl-m-toluamide (DEET) **(OFF® Family Care Insect Repellent, 5%, "Standard Product")** **(OFF® Deep Woods , 10%-30%, "Standard Product")**



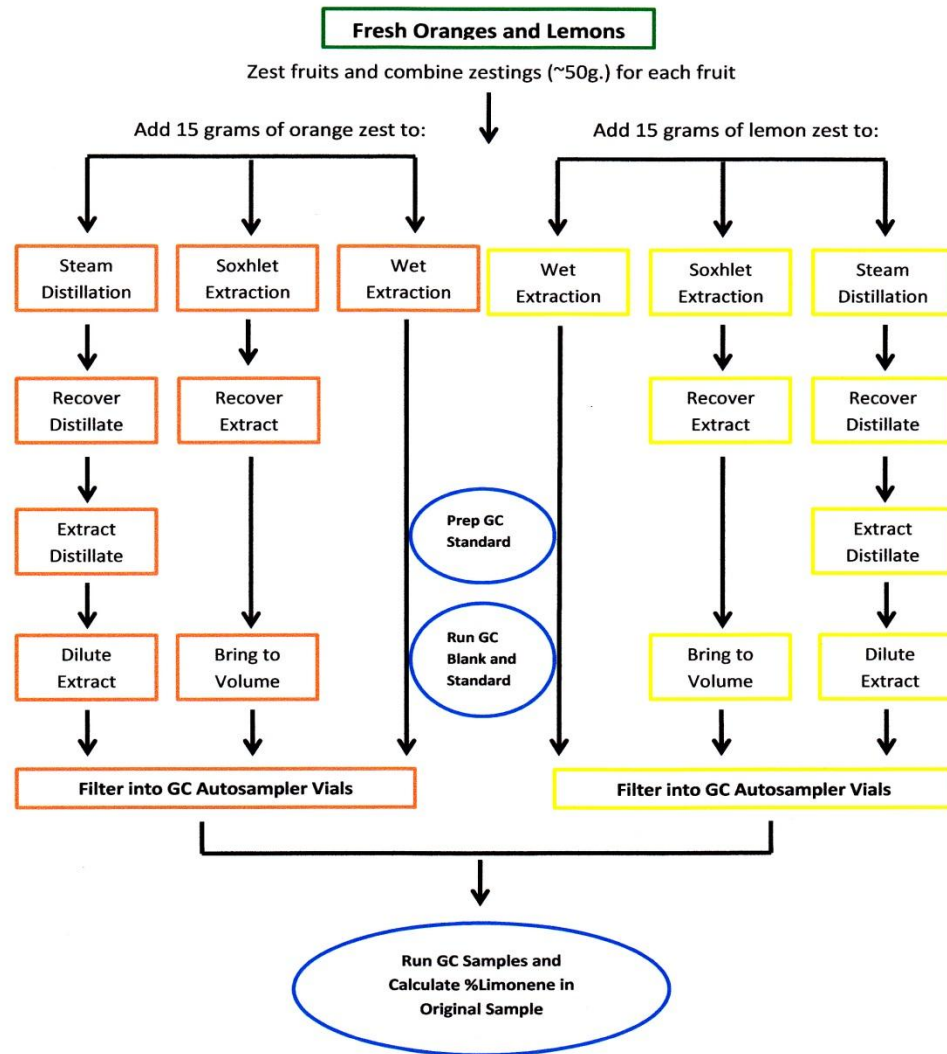
Molecular Formula = C₁₂ H₁₇ NO Molecular Mass = 191.27 Boiling Point = 288 °C

Experimental Background

- (+)-Limonene is found in all citrus fruits to varying degrees.
- The main concentration of limonene is in the zest (skin) of the fruit.
- (+)-Limonene is a terpene (a hydrocarbon) and is not very soluble in polar solvents.
- Common Methods of isolation include
 - **Steam Distillation**
 - **Soxhlet Extraction**
 - **SCF CO₂ Extraction**
- In pure form limonene is a colorless liquid that boils at 173°C.

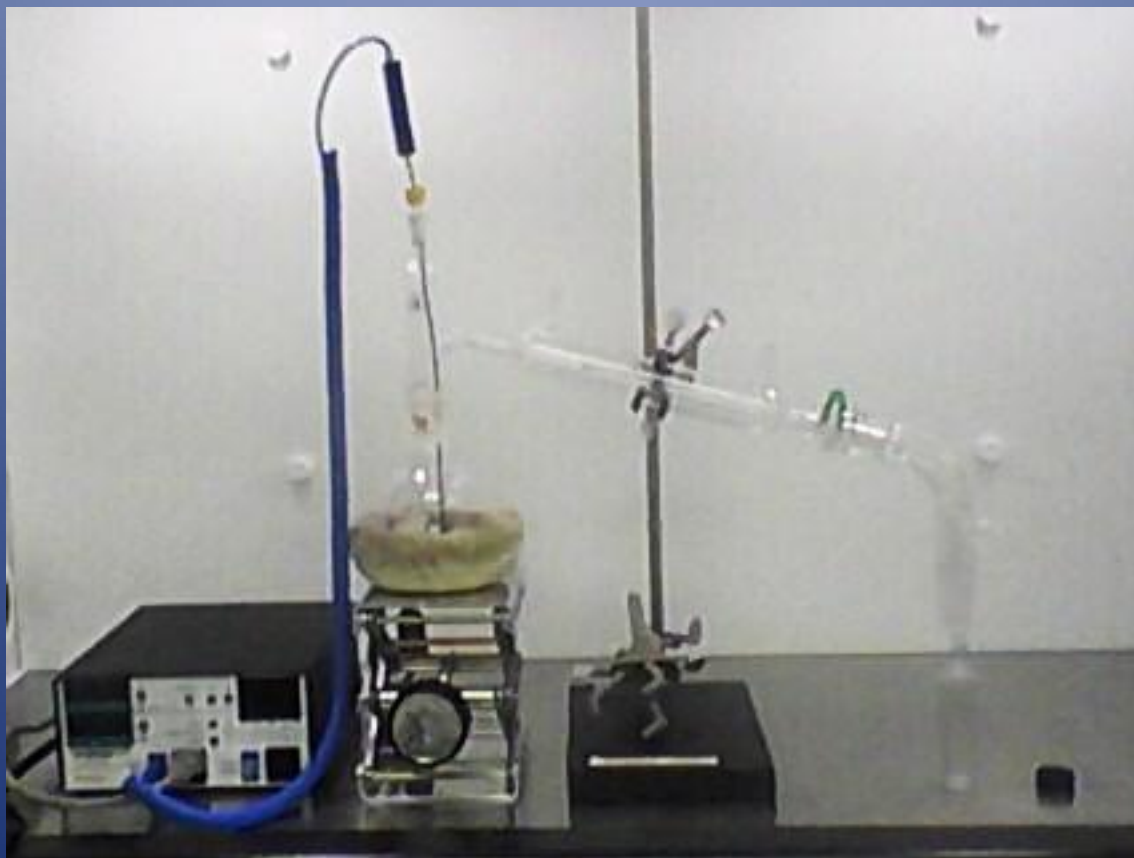
Analytical Scheme

Limonene Extraction/Analysis – Analytical Scheme



Steam Distillation

The zest can be minced and mixed with water and the limonene can then be removed by steam distillation. Because of the Law of Partial Pressures, the limonene can be distilled at a temperature far below its normal boiling point (173C).

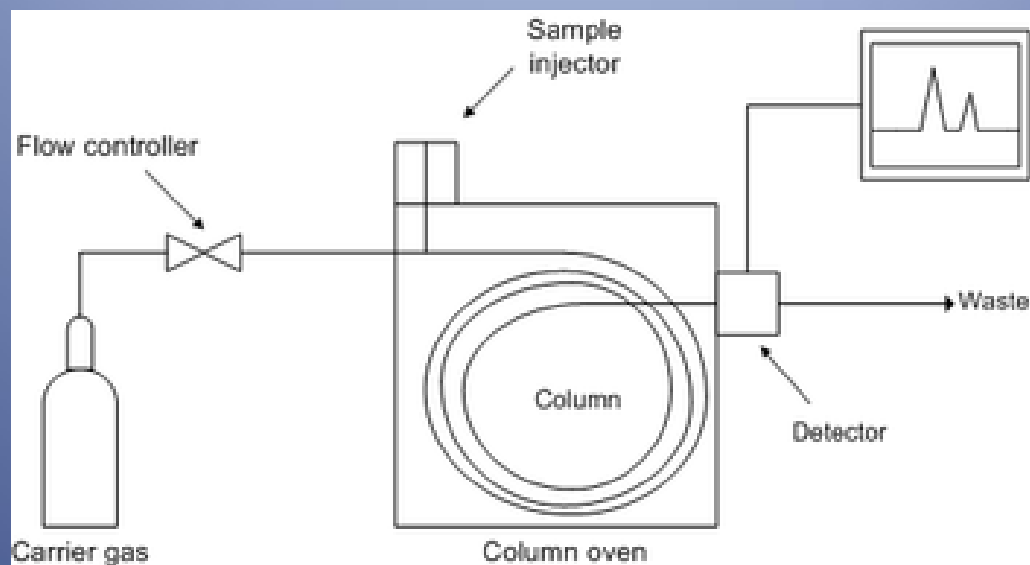


Soxhlet Extraction



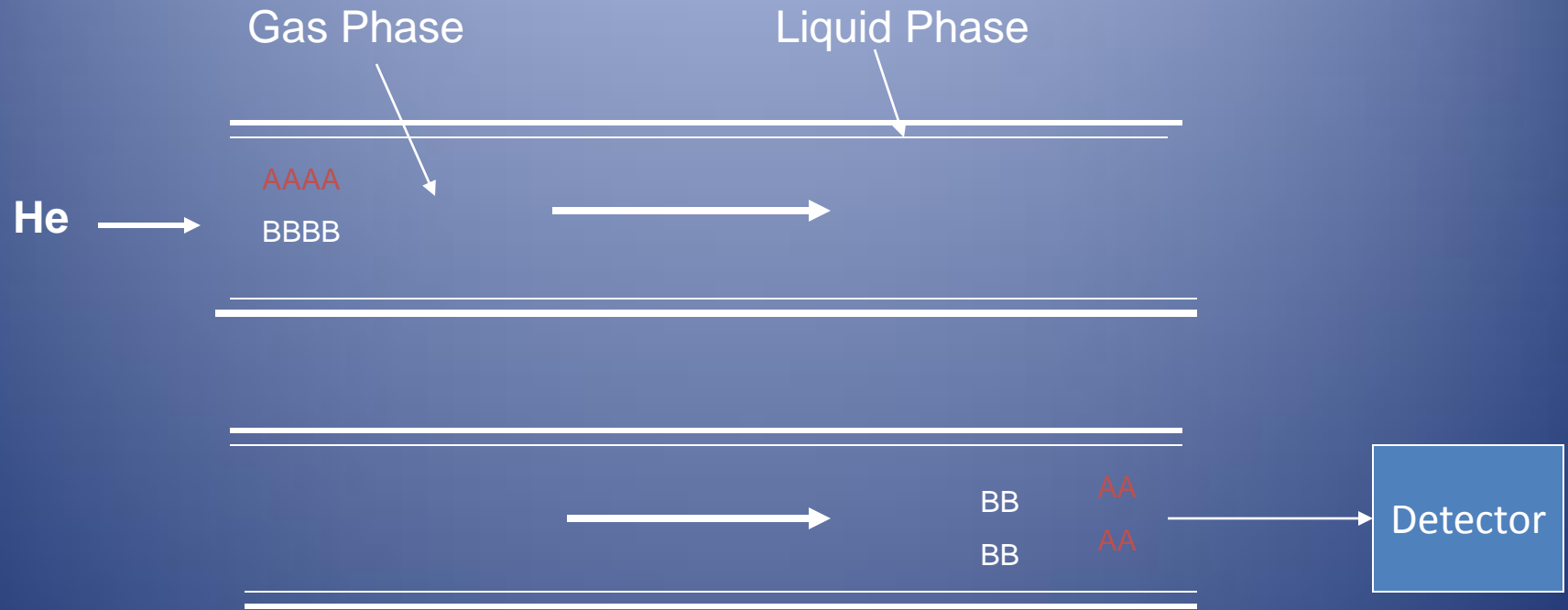
(+)-Limonene can be extracted from zest by Soxhlet extraction. A thimble is filled with zest. The Soxhlet extraction soaks the citrus zest in hot isopropyl acetate. As the extraction proceeds, the thimble fills to overflowing with clean solvent and is then syphoned empty.

Analysis by Gas Chromatography

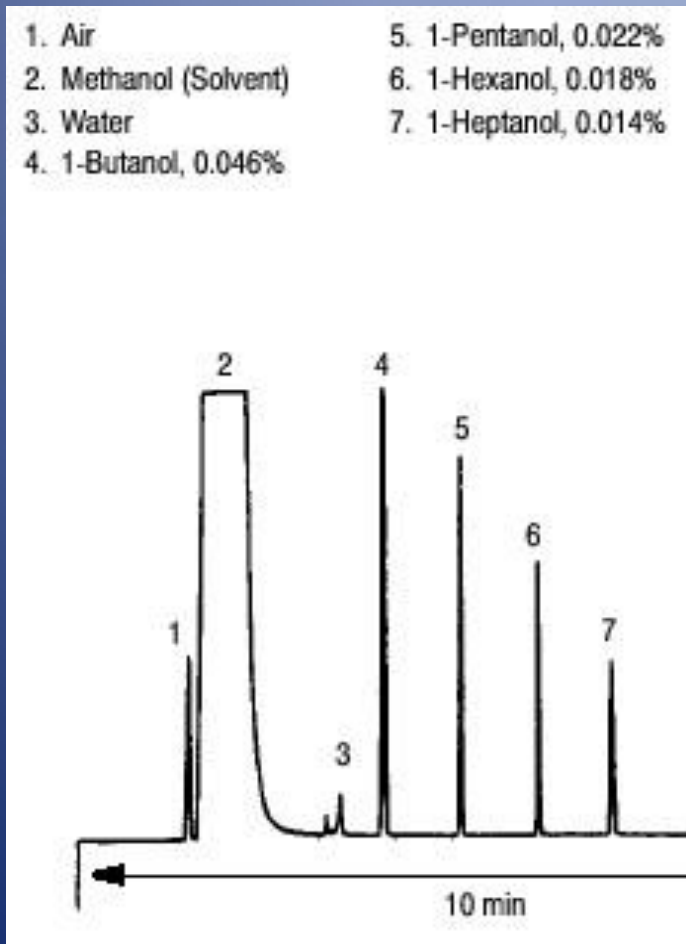


- Amount of limonene isolated from each method will be calculated by comparing isolated samples (extracts) to a known standard by GC analysis.

Illustration of GC Separation



Factors Controlling Separation



- Boiling point or vapor pressure of the molecule
- The strength of the attraction of the molecule to the stationary liquid phase

Things to think about while running the experiment

- Which fruit yields more limonene by each extraction method?
- Which fruit yields higher purity limonene by each extraction method?
- Which extraction method generally gives you more limonene?
- Which method is more efficient (quality and quantity)? Why?
- Which method uses less energy to get maximum limonene?
- Which method uses a safer solvent?
- Which method has “cleaner waste?”
- Which method is greener? Why?
- We will present our data and report to the group!!!