

## Isomerization of (R)-Carvone

**Background:** The essential oils of plants have been extracted from plants for use as perfumes and medicines for centuries. The major components of many essential oils are part of the isoprenoid family, derived from the five-carbon compound, isoprene. For example, the major component of spearmint, (R)-carvone, is biosynthetically produced through a number of steps from isoprene. (Figure 1)

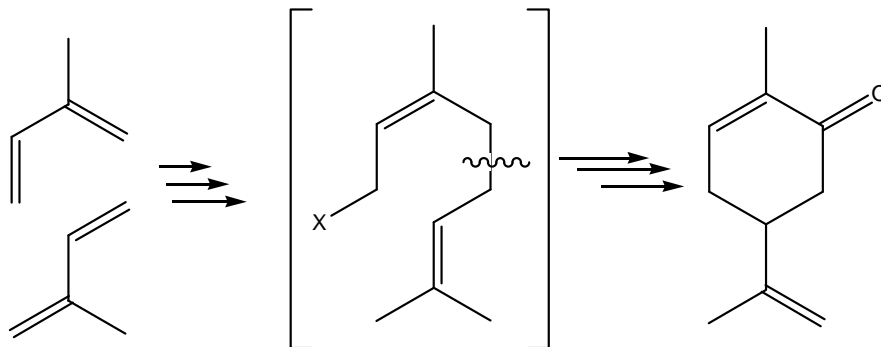


Figure 1. Two isoprene units bond to form a monoterpene skeleton which lead to formation of products such as carvone.

A sub-category of the isoprenoid family are the monoterpenes. Monoterpenes are ten-carbon containing compounds, derived from two isoprene units. A number of other monoterpenes share the same carbon skeleton as carvone (Figure 2).

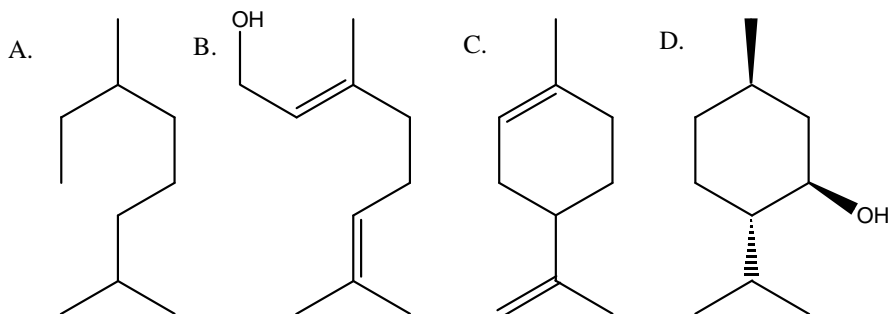


Figure 2. (A.) Base carbon skeleton of monoterpene (B.) geraniol--from roses (C.) limonene--from citrus (D.) menthol--from peppermint

Monoterpeneoids may be interconverted by reactions including rearrangements. In today's experiment, we will use strong acid and heat to catalyze the formation of an isomer of carvone.(Figure 3.) This reaction occurs through a series of rearrangements to form a much more thermodynamically stable product.

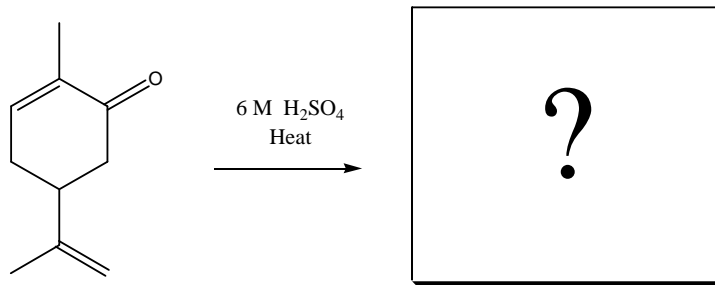


Figure 3. Isomerization of carvone

**Procedure:** Place 1.0 mL of carvone and 10 mL of 6 M sulfuric acid in a 25 or 50 mL roundbottom flask with a condenser. Add boiling chips or use a stirbar if a magnetic stirplate is available. Put a paper towel on top of the condenser with a rubberband. Heat the solution to reflux with stirring, and allow the reaction to boil for 35 minutes. Allow the roundbottom flask to cool to room temperature and pour the contents into a separatory funnel. Extract the crude reaction mixture with two 5mL portions of petroleum ether. Combine the petroleum ether extracts and wash them with 10 mL of brine followed by 10 mL of 10% aqueous sodium bicarbonate. Separate the ether layer into an Erlenmeyer flask and dry with sodium sulfate. Decant the ether solution into a small tared beaker and evaporate the ether with low heat in a sand bath by blowing nitrogen over the beaker. (A rotovap may also be used if available.) Because the final product is not as pleasant smelling as the starting material, make sure to do as much work with the final product as possible in the hood. Obtain a final yield. With the help of your AI, obtain an IR and <sup>13</sup>C NMR of the product.