S343 Experiment: Isomerization of (*R*)-Carvone

Lab goals: Use spectroscopy to propose a structure of the rearrangement of carvone under acid conditions

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. A sub-category of the isoprenoid family are the monoterpenes. Monoterpenes are ten-carbon containing compounds, derived from two isoprene units (Figure 1)

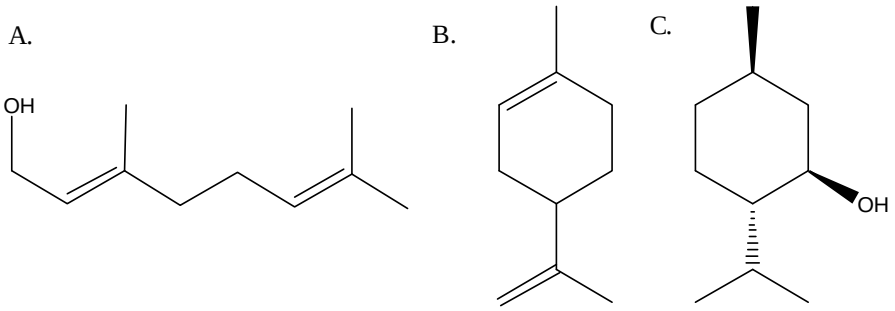
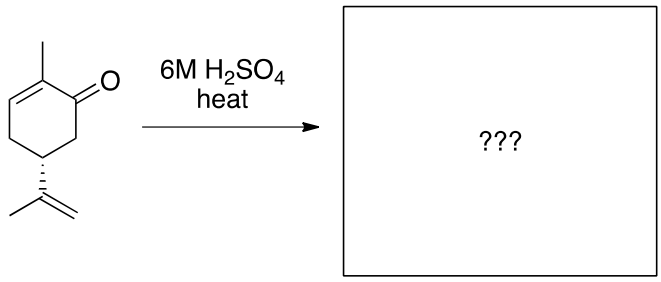


Figure 1. (A.) geraniol--from roses (B.) limonene--from citrus (C.) menthol--from peppermint

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 a monoterpene, R-carvone.(Figure 2.) This reaction occurs through a series of rearrangements to form a much more thermodynamically stable product.



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. (Caution: bumping possible!) Heat the solution to reflux with stirring, and allow the reaction to boil for 35 minutes. (Be sure it is boiling before you start the timer.) 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. (Caution: gas evolution!) 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, IR and ^{13}C NMR. Be sure that your ^{13}C NMR is not too noisy—ask your AI.

Results – Calculate your yield and tabulate all of the characterization data that you used to identify your product (IR, ^{13}C NMR). Your spectra should be taped into your lab notebook, and your tables should reference the page on which the spectra are taped so that your table data can be easily compared to the raw data in the future. Copies of spectra from databases can also be referenced and included in your notebook for comparison.

Discussion—What does your IR data tell you specifically about your rearranged product structure? What does your NMR data tell you about the structure of the product? What is the identity of your product? Does your data suggest that it is pure or impure? Does it contain starting material—and how do you know based on your data? Is your product a 'known' compound (has it been synthesized before, or has it been isolated from a natural source? Use SciFinder Scholar.) Propose a reasonable mechanism for the rearrangement(s).

<u>Lab 8 assignment:</u> Turn in a hardcopy of your lab notebook (carbon copy or photocopy) attached to a full formal report

- Due at the beginning of lab during the week of march 30
- 50 pts based on in-lab performance, completion of all sections, correctness of content
- Find ONE paper using SciFinder Scholar. Use it as a reference in your introduction section.
- Submit an electronic copy through Turnitin.com prior to your lab section, or it will be considered LATE