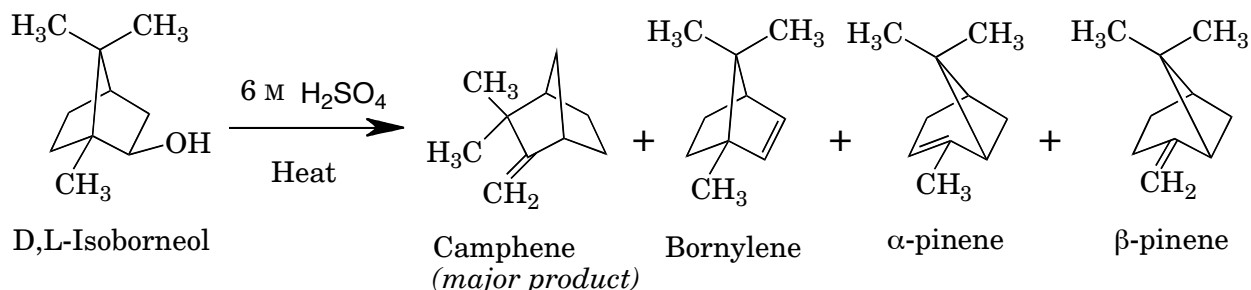


Dehydration of an Alcohol to an Alkene

The dehydration of alcohols is a straightforward, and hence common, method for preparing alkenes. Using this method, one heats the alcohol in the presence of an appropriate acid catalyst. As you know, alcohols undergo dehydration with this order of reactivity: $3^\circ > 2^\circ > 1^\circ$. Mineral acids will dehydrate tertiary alcohols at room temperature, whereas primary alcohols react only at elevated temperatures. The mechanism for the reaction involves formation of a carbocation, so rearrangements are common in this procedure.



Procedure: Preparation of an Unknown Alkene by Dehydration of Isoborneol

Preparing the Alkene

Pour 20 mL of 6 M H₂SO₄ into a 50 or 100 mL round-bottom flask containing a magnetic stir bar. Begin stirring the acid and slowly add 3 g of isoborneol using your powder funnel (*i.e.* the wide neck funnel). Fit the flask for a ***simple distillation*** using a heating mantle. Fit the condenser with a curved vacuum adapter leading into a 25-mL round-bottom flask (you may wish to review the course materials on distillation theory and practice). (Note: do **not** use water to cool the condenser; in fact, it is likely that you will need to heat the condenser to allow the product to reach the collection flask.)

While stirring vigorously with the magnetic stirrer, ***steam distill*** the alkene as it forms (water will co-distill), until only water collects in the receiving flask. This should take 30 to 40 minutes at a 10-drop/minute drip rate. Collect about 10 mL of distillate and quit distilling when only water is condensing in the condenser or when the pot residue turns dark yellow or brown.

The receiver flask should contain a solid organic product and liquid water. Carefully pour the water out of the flask. Use a spatula to scrape the solid product onto a pre-weighed petri dish, while minimizing the amount of water that is transferred to the dish. Spread the product over the surface of the petri dish to maximize the exposed surface area of the product. Cover the petri dish and store the covered product in the laboratory drawer.

Characterization of the Alkene

Measure the mass of product, and calculate percent yield. Use small samples of the product to obtain an infrared spectrum in chloroform, and attempt to obtain a melting point (if any water is still present, avoid transferring the water to the IR salt plate or the melting point capillary).

The product may be further analyzed by gas chromatography/mass spectrometry on a 6' polydimethylsiloxane column at 85°C and compared with standard reference alkenes.

Testing the Product for Unsaturation

Place a small amount of the putative alkene in a small test tube and add a 5% solution of Br₂ in CH₂Cl₂ dropwise (swirl the alkene+bromine solution after adding each drop). How should a positive test look?

Post-laboratory Questions

1. What was the melting point of your product? How does this compare to the literature melting point of camphene?
2. What was the percent yield of the reaction? Please show your work.
3. Include an analyzed IR spectrum of the product, and a published IR spectrum for the compound from SDBS or Aldrich.
4. What product did you obtain? Please justify your answer. Why was this product obtained instead of other possible products?
5. Draw, using a computer drawing program (such as AccelrysDraw or ChemSketch), the mechanism of the dehydration reaction you carried out, showing intermediate carbocations and all possible rearranged carbocations. Predict which products should be present and their relative abundances.
6. Write a mechanism for the reaction used to test for unsaturation in your predicted product.
7. Would you expect the same major product using HCl as the mineral acid instead of H₂SO₄? Explain.