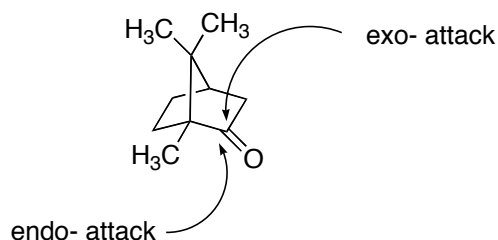
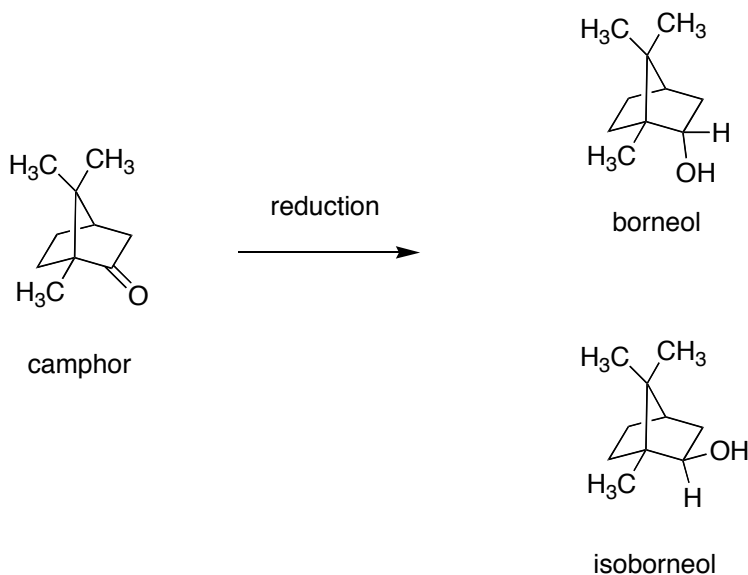


Reduction of Camphor

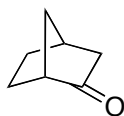
TUD Department of Chemistry

When camphor is reduced with metal hydride reagents such as LiAlH_4 or NaBH_4 , either borneol or isoborneol can be formed depending upon whether the hydride is delivered from “below” (endo-attack) or “above” (exo-attack) the carbonyl group. Statistically, endo- and exo-attack are equally probable and the product should be a 50:50 mixture of borneol and isoborneol. In this experiment, we will reduce, using NaBH_4 , the camphor that we previously synthesized and determine the identity of the product(s).



¹Place 100 mg of camphor in a 10 mL Erlenmeyer flask, add 0.5 mL methanol and stir to dissolve. Cautiously add, in small portions, 100 mg of NaBH₄. After all of the NaBH₄ is added, gently boil the solution (very low hot plate setting) for 2 minutes. Add methanol drop-wise to replace any lost by evaporation. Cool the flask and slowly add 3 mL of ice cold water. Collect the solid using suction filtration and draw air through the solid for several minutes to dry. Return the solid to a 10 mL Erlenmeyer and dissolve in 5 mL of methyl t-butyl ether. Dry the ether with several spatula fulls of anhydrous Na₂SO₄ and then transfer the ether to a clean, dry 25 mL suction flask. Wash the remaining drying agent with a couple of mL of MTBE and add the wash to the previous ether. Evaporate the MTBE using vacuum. Weigh, calculate the %yield, and determine the melting point of the solid. Transfer the solid to a small beaker, label, and store in your drawer covered with an inverted larger beaker.

We will discuss characterization of the isomer ratio of the product for the next laboratory. Before then, make a model of camphor and see if you can predict whether the mixture should be 50:50 borneol:isoborneol or if one or the other compounds should predominate. (Hint: Remember, although we can usually correctly predict the product and visualize the mechanism of borohydride reductions by assuming delivery of a hydride ion, the actual mechanism probably involves approach of the large BH₄¹⁻ ion to the carbonyl group rather than simply the relatively small H¹⁻ ion.) Make a model of 2-norbornanone and predict the predominate reduction product of it.



2-norbornanone

¹ Based upon the procedure in Pavia et al., **Introduction to Organic Laboratory Techniques**, 3rd ed., Saunders College Publishing, 1999, pp. 271-277.