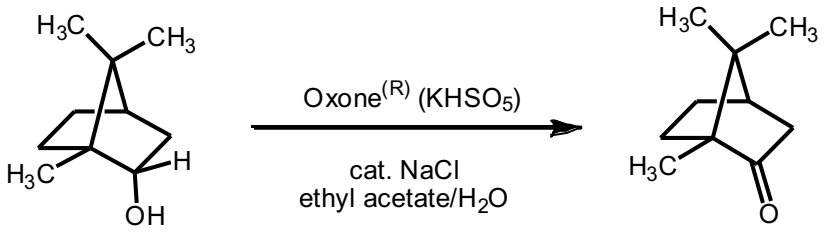
An Oxidation-Reduction Scheme: Borneol, Camphor, Isoborneol. Week 1

Reading The background reading for this experiment is in Pavia (5th edition), Experiment 31, Parts A, B and C, and Technique 17 (Sublimation). The procedure for Part A given below is based on a modified procedure from Wissinger, *et al. J. Chem. Educ*. 2011, *88*, 652-656.



borneol camphor

Prelab

Along with the usual **Name**, **Title**, **Purpose** and **Outline** of the procedure, show the **Chemical Reaction** (above) and a complete **Reagent Tabl**e in your notebook. Be sure to include the theoretical yield of the product.

Prelab Exercise

See the separate handout for the exercise, which covers both Part A and Part B of this experiment. Turn in this exercise on a separate piece of paper, but also put the appropriate information for Part A in your **Reagent Table** in your notebook as well. *Note that the structure shown above for the chemical reaction may (or may not) depict the correct isomer of borneol or camphor.*

Procedure – Modified.

Oxone® is a strong oxidant; do not inhale the dust. The aqueous components of an organic Oxone® reaction are oxidizing and acidic and should be quenched with sodium bisulfite and then neutralized with sodium bicarbonate before disposal.

NOTE: carefully measure the amounts of solvent using a pipette. The rate-determining step of the reaction is second order, so the rate dramatically decreases with an increase in the solvent volumes.

Clamp a 50-mL round bottom flask to a ring stand and position a magnetic stirrer beneath it. Place a medium stir bar in the flask and add 1.0 g of (1*S*)-borneol. Add 4 mL ethyl acetate and begin stirring to dissolve the borneol. Add 2.4 g of Oxone® (which corresponds to 7.8 mmol of $KHSO_5$) to the flask with continued stirring. Then add 0.08 g (1.4 mmol) NaCl, followed by 1.5 mL of deionized water. Allow the reaction to stir at room temperature for 50 minutes. Then add an additional 0.03 g (0.5 mmol) NaCl. Continue to stir for 10 more minutes. Note any changes in color or temperature during the entire procedure.

By the end of this time the oxidation should be complete and excess oxidant can be destroyed. Add 15 mL of deionized water to the reaction and continue stirring to dissolve most of the salts. Slowly add a spatula tip of solid sodium bisulfite ($NaHSO_3$) to reduce the oxidants that remain. Test the aqueous layer (not organic) by dipping a glass rod into it and then touching a piece of starch-iodide paper. A blue-black color (positive test) indicates the presence of excess oxidant; add small amounts of sodium bisulfite if the aqueous layer tests positive, until a negative test is achieved (no color change).

Workup

Carefully transfer contents to a separatory funnel. Add 1 to 2 mL of ethyl acetate to the reaction flask, swirl, and add this wash to the separatory funnel as well. Shake and invert the separatory funnel and separate the layers by draining the aqueous layer from the bottom. Pour the organic layer out of the top into a clean 50-mL Erlenmeyer flask. Extract the aqueous layer twice more, with 5 mL of ethyl acetate each time. Return the combined organic phases to the separatory funnel and wash three times with 5-mL

portions of saturated aqueous sodium chloride solution (brine). Pour the organic phase into a clean Erlenmeyer flask and dry over anhydrous sodium sulfate.

Filter the solution into a tared 50-mL round bottom flask. Use the rotary evaporator to remove the solvent with low heating. Do not leave the flask on the rotary evaporator too long, since your product may begin to sublime. Cover the flask with a Kim-Wipe and rubber band, label the flask with your name(s) and the word "camphor", and store it in the hood until the following laboratory period.

Purify your product by vacuum sublimation using the apparatus shown in the laboratory. Collect your product in a screw-cap vial. Be sure to make a sketch of the apparatus in your notebook. Record the weight of your purified product, obtain a melting point (sealed tube), and record an IR spectrum (use the thin-film method described in Pavia).

To Complete the Experiment – Partial Report

Store your final purified product in a well-sealed screw-cap vial until the next laboratory period. See the handout for Part B for instructions on completing the experimental write-up.