Oxidation of Isoborneol to Camphor

Measure out 2.5 grams of isoborneol and fully dissolve it in 6 mL of glacial acetic acid in a 125 mL Erlenmeyer flask. Add 25 mL of chlorox bleach , slowly, dropwise over five to ten minutes maintaining the temperature between 5-15 degrees celcius using a cold bath.

Add a stir bar and stir the reaction for one hour at room temperature. During this time you can gather in room 180 to work on your worksheet.

After one hour, the solution should appear colorless with a precipitate. It may still be yellow if it has excess bleach. To test for excess bleach , dip a glass rod into the solution and test the solution with KI-starch test paper. If you have excess bleach, the following reaction will occur on the test paper.

$$NaOCl + KI + 2H^+ \longrightarrow NaCl + I_2 + H_20$$

The resulting iodine complexes with the starch that is embedded in the paper and forms and inky blue/black complex. The dark color is an indication that there is bleach present. If this is the case, you need to add sodium bisulfite to destroy the bleach on a larger scale.

$$NaHSO_3 + NaOCl \longrightarrow NaCl + NaHSO_4$$

Add this reagent in small increments (ca. 1 mL) with stirring until you observe a negative starch-iodide test (no color – white).

Take the reaction mixture and add it to 50 mL of cold saturated NaCl solution. The compound is solid and will appear as a precipitate. Add 30-40 mL of dichloromethane. Using a 250 mL separatory funnel, partition the camphor and any un-reacted isoborneol into the dichloromethane. The precipitate should dissolve into the organic layer.

Separate the layers and extract the aqueous layer with an additional 20 mL of dichloromethane. Combine the organic layers and wash them with 2, 10 mL volumes of saturated sodium bicarbonate (watch out for gas, use your 250 mL separatory funnel). Then rewash the organic layer with 10 mL of saturated sodium chloride. Dry the organic solution over dry sodium sulfate. Decant the liquid into a tared 100 mL roundbottom and rotavap it to dryness with gently heat (ca. 50 ^oC).

Obtain the mass of the compound and measure the IR an melting point. See your TA/Instrutor about running the IR.

Please fill in the following worksheet and give it to your instructor. This will count as your lab report. Please finish it during lab if possible.

Name:_____ Date:_____

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1. Write the overall reaction for the Oxidation of Isoborneol to Camphor.

2. Write a reasonable mechanism for the Oxidation of Isoborneol to Camphor.

3. Mass of Product:

4. Percent Yield of Product:

5. Melting point of Product:

6. Interpretation of IR (attach IR):

7. Isoborneol can be oxidized using Chromic Acid (H_2CrO_4). The product is Camphor and Cr^{+3}. Try to balance this Redox Equation.

8. Given the fact that many oxidations are eliminations, please explain why ketones and tertiary alchols do not react readily with Clorox or chromic acid.