S343 Experiment: Dihydroxylation

Lab Goals: Determine the stereochemical outcome of two dihydroxylation reactions by TLC analysis. Propose a reasonable mechanism for each reaction that is consistent with this outcome.

Overview: In this experiment, you will perform two reactions that transform alkenes into diols. While similar in functional group transformation, these reactions may lead to stereochemically different products. The two possible products are diastereomers of one another, and therefore have different physical characteristics. After performing the reactions, you will characterize the crude reaction mixtures to determine if you formed a mix of trans enantiomers, the meso cis product, or a mixture of cis and trans diols. In part A of this experiment, you will use a common oxidizing agent, KMnO₄. In part B, You will use an oxidizing agent that you may not have discussed in lecture called Oxone®. Oxone is a persulfate mix that reacts with acetone to produce dimethylidioxirane.

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Procedures:

A. Oxone Reaction: Dissolve 0.40 g oxone® in 2.0 mL of water by shaking vigorously in a sealed vial. (Caution! Be sure the vial is sealed because oxone is a strong oxidizer.) Dissolve 50 μL of cyclohexene in 2.0 mL of acetone in a small Erlenmeyer flask. Cool the cyclohexene solution in an ice bath, then add the oxone solution dropwise over 5 minutes with swirling in the ice bath. Remove the Erlenmeyer flask from the ice bath and allow the reaction to sit for 15-30 minutes. Add two drops of conc. HCl to the reaction with swirling. Allow the reaction to sit for about 10 minutes, swirling occasionally. Analyze by TLC, and determine your product outcome.

B. Potassium Permanganate Reaction: Dissolve 100 mg of KMnO₄ in 4.0 mL of 0.1 M NaOH solution in a small Erlenmeyer flask. Cool the KMnO₄ solution in an ice bath. Dissolve 50 μL of cyclohexene in 2.0 mL of t-butanol in a separate container. Quickly add the cyclohexene solution into the potassium permanganate. Swirl the flask in the ice bath for 3-5 minutes. Remove the Erlenmeyer flask from the ice bath and allow to sit for 10 minutes. Analyze by TLC, and determine your product outcome.

C. TLC Analysis: Analyze both crude reaction mixtures by TLC, and determine which product(s) is (are) formed in each reaction. Develop the TLC plates in 100% ethyl acetate, and stain with anisaldehyde solution (your AI will help you with this).

IV. Discussion Comments: Which product or products were formed in each reaction? What data supports this conclusion? Include a reasonable mechanism that is supported by the reaction outcome in each case.
Lab 5 assignment: Turn in a hardcopy of your lab notebook (carbon copy or photocopy) and a hardcopy of an experimental section for the experiment (no electronic submission)
- Due at the beginning of lab next week
- 25 pts based on in-lab performance, completion of all sections, correctness of content
- You will need to write a general experimental section, so be sure to get all instrumental/material information you need during lab. You will also need to write separate experimental sections for your two procedures.