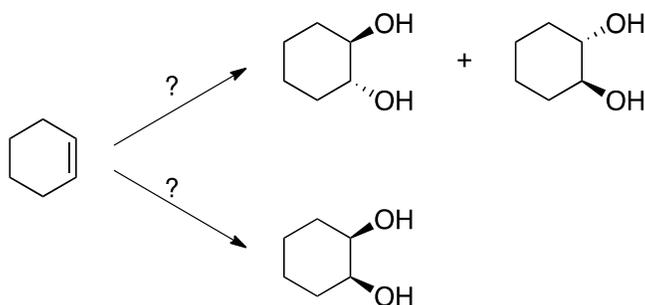


S343 Experiment: Dihydroxylation

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, KMnO_4 . 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 dimethyldioxirane.



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 KMnO_4 in 4.0 mL of 0.1 M NaOH solution in a small Erlenmeyer flask. Cool the KMnO_4 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 4 assignment: Turn in a hardcopy of your lab notebook (carbon copy or photocopy) and a hardcopy of a formal introduction section. Submit an electronic copy of the formal introduction through Turnitin.com

- Due at the beginning of lab next week
- 25 pts based on in-lab performance, completion of all sections, correctness of content, formal introduction
- The main purposes of an introduction section are to discuss previous work, summarize the current understanding of the field, and/or point out why the current study is important in light of what is already known/not known. There are a number of ways that you could frame this introduction. You could write about the importance of diol formation. You could write about the importance of stereochemical control in synthesis. You could write about how an understanding of mechanism is essential to synthesis. You could write about the importance of developing complementary reagents to accomplish slight differences in synthesis. At this level, the introduction should be about 2 paragraphs.
- For the introduction section, you need to cite current literature. This is difficult for beginning students, so I have given you some papers to choose from. Refer to two of the following papers in your introduction. You must access them through the IU Chemistry Library page or in the chemistry library. While you do not have to understand everything in them, you should not read one sentence and cite the source—it is easy to tell if you have read enough to understand the main point of the paper before citing it.
 - J. Org. Chem. (2011) 76, 9997-10004.
 - A review in a book: See “Comprehensive Organic functional Group Transformations II, pp 581-600 in the chemistry library. Call number QD262.C534 2005 v.1
 - A theoretical paper: European J. of Organic Chemistry (2003) (5) 833-839.
 - J. Molecular Catalysis A: Chemical (2008) v285(1-2), 111-119.
 - J. Org. Chem. (1991) 56(15) 4585-4588.