

Stereochemistry & Polarimetry

In this experiment a solution of an unknown sugar will be prepared, exposed to plane polarized light and the observed optical rotation (α_{obs}) will be measured using a polarimeter. The specific rotation $[\alpha]$ of the material will be calculated, and from this the identity of the sugar will be determined.

A significant amount of lab time will be spent clarifying issues relating to stereochemistry, practicing proper priority assignments around chirality centers, determining the stereochemical configuration, and calculating the composition of an RS mixture. This lab often proves very beneficial to the understanding of concepts presented in lecture.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter of the manual, the lecture notes on the Chemistry Department web site and sections 5.2, 5.3, 5.4 of Klein. Review General Chemistry I, Module 4, learning goal 7 relating to the preparation of solutions from pure materials and water using volumetric flasks. Do the first seven parts of your notebook writeup.

A student who has prepared for the Stereochemistry & Polarimetry experiment should be able to:

1. Define and identify chirality centers.
2. Define plane polarized light, optical activity, observed rotation, specific rotation, racemates, diastereomers and enantiomers.
3. Know the steps involved in making a solution from pure substances using a volumetric flask.
4. Understand how concentration, cell length, observed rotation (α_{obs}) and specific rotation $[\alpha]$ are related.
5. Identify and explain safety considerations for this experiment.
6. Perform the day's experiment safely and successfully.

Quizzes given after the experiment has been performed may also include:

7. If given mass of material, volume of water, length of polarimetry cell and observed rotation, calculate the specific rotation of the compound.
8. If given length of polarimetry cell, observed rotation and the specific rotation of the compound, calculate the solution concentration.
9. If given the specific rotation of a mixture of enantiomers and the specific rotation of a pure enantiomer, determine the R and S composition of the mixture.
10. Identify chirality centers in a molecule.

11. Determine priority of groups around a chiral center.
12. Properly assign R or S designation to chiral centers.

Safety considerations for this experiment:

In this experiment aqueous sugar solutions will be prepared and their optical rotations will be measured. The solutions are nontoxic and the polarimeters have no special hazards. Standard good lab practice of wearing goggles and lab coats applies.

PROCEDURE

In this experiment, a sugar solution of known concentration (c) but unknown identity will be prepared (note the units of concentration used). The observed rotation (α_{obs}) will be obtained by using a polarimeter. These will be used to calculate the specific rotation $[\alpha]$ and the identity of the sugar will be determined.

Obtain an unknown chiral substance. Record the unknown identification in lab notebook. Weigh out approximately 0.5 g of unknown. The mass should not be 0.5000 g but instead some mass between 0.04 and 0.06 and precisely known. Note, if you waste time by measuring exactly 0.5000 g of unknown, your instructor will likely deduct points. Record **all the digits** the balance provides directly in your notebook. In a clean 50ml beaker, dissolve your material in approximately 10ml of deionized water. Swirl the contents until all of the solid has dissolved. Carefully transfer this solution to a 25ml volumetric flask. Rinse beaker with approximately 2 ml of deionized water. Transfer this solution to volumetric flask. Rinse again, transfer solution to volumetric flask.


Carefully and drop wise add deionized water to the volumetric flask until bottom of meniscus is **exactly** on line. If you add too much water and overshoot the line, there is no going back. That solution must be discarded and the entire solution making process repeated. Cap or cover the end of volumetric with a piece of parafilm. Invert the volumetric flask several times to ensure thorough mixing.


Note the final volume in your notebook. Be sure to use the correct number of digits. Calculate the concentration in **g/ml**. Obtain the α_{obs} by analyzing your solution in the polarimeter using the instructions mounted adjacent to machine and reviewed by your instructor. Record all observations directly in lab notebook.

Simplified Polarimeter Instructions

1. Rinse then fill observation tube with sample.
 - a. carefully unscrew one end of tube.
 - b. slightly overfill tube to reduce or eliminate presence of bubbles.
 - c. carefully replace tube cap.
 - d. wipe off end of tube with Kim-wipe.
 - e. invert tube so any bubbles migrate to bulge in observation tube
2. Press “▲RIGHT” or the “▼LEFT” to start from 0.0

3. Look through eye piece and see the lit circle. If nothing is observed, press the “TEMP” button once. While observing the circle, simultaneously press either the “▲RIGHT” or the “▼LEFT” button to equalize the brightness of the two sides of the view. If no circle is observed, briefly press the “SET/TEMP” button.

a. When the left hemisphere is brighter,  press the “▼LEFT” button until both sides are equal brightness.

b. When the right hemisphere is brighter,  press the “▲RIGHT” button until both sides are equal brightness.

4. When both sides are equalized, record the display.

5. Press “▲RIGHT” or the “▼LEFT” to return to 0.0

6. Empty observation tube into waste. Rinse tube with deionized water. Be sure not to drop small glass slide into waste container.

7. Place empty tube and cap in container.

Calculate the specific rotation $[\alpha]$. Compare the calculated specific rotation with the specific rotations of the possible sugars. Identify your unknown.

Possible unknown sugars:

Name (common names)	Specific Rotation $[\alpha]$
D- Fructose (D-Levulose)	-86
D-Glucose	+98
D- Galactose	+ 82
D-Allose	+15
Sucrose	+64.5
Maltose	+118

CLEANUP

Empty any remaining liquid in the volumetric flask down the sink. Rinse the volumetric flask twice with deionized water. Return the volumetric flask to the back bench. Dispose of all Kim-wipes in trash can. Discard used Pasteur pipettes in the broken glass disposal box. Wipe down your work area with a sponge.

POST-EXPERIMENT ASSIGNMENT

Turn notebook pages into your lab instructor. Complete datasheet and turn it into the lab instructor.

Prepare for the Stereochemistry: Polarimetry portion of the next quiz.

References:

1. Juett, A. J, O’Grady, J, *Journal of Antimicrobial Chemotherapy* (1996) 37, 7-32
2. <http://www.chromatography-online.org/topics/thalidomide.html> February 27, 2008

3. T.W. G. Solomon and C. B. Fryhle; *Organic Chemistry*, 8th edition; John Wiley & Sons, New York, 2003, p .
4. L. G. Wade, *Organic Chemistry*, 5th edition; Prentice-Hall, Upper Saddle River, NJ, 2003, p 178-186.
5. K. Williamson, *Macroscale and Microscale Organic Experiments*, 4th edition, Houghton Mifflin, Boston, 2003, p.
6. Cooley, J.H., Williams, R.V., *J. Chem.Ed.*, **1997**, 74(5), 585
7. A.E. Moody, Truman College,
<http://jade6.truman.edu/~patter/organic/procedures/optical-activity.pdf> February 27, 2008
8. DiMaggio, S, Organic Lecture Handouts, 5-1 through 5-23, 2007
9. Microscale Operational Organic Chemistry, J. W. Lehman, Prentice Hall, 2004, 712-716