

Understanding Polarimetry

A polarimeter is a device that measures the rotation of linearly polarized light by an optically active sample. This is of interest to organic chemists because it enables differentiation between optically active stereoisomers (i.e., enantiomers). Enantiomers, chiral molecules, are molecules which lack an internal plane of symmetry and have a non-superimposable mirror image. One way to tell these molecules apart is to use polarimetry. Polarimetry is also helpful for biological applications because amino acids, nucleic acids, carbohydrates, and lipids are all optically active. Determination of the optical activity of a compound using polarimetry allows the user to determine various characteristics, including the identity, of the specific chemical compound being investigated.

As shown in Figure 1, incident non-polarized light is transmitted through a fixed polarizer that only allows a certain orientation of light into the sample. The sample then rotates the light at a unique angle. As the analyzer is turned, the rotated light is maximally transmitted at that unique angle, allowing the user to determine properties of the sample. A (+) enantiomer rotates the plane of linearly polarized light clockwise, *dextro*, as seen by the detector. A (–) enantiomer rotates the plane counter-clockwise, *levo*.

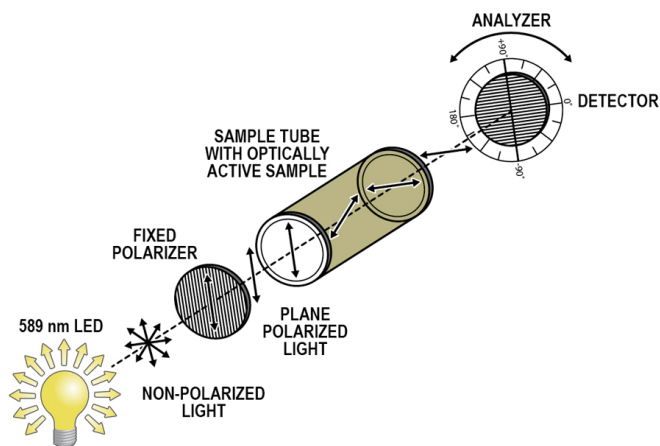


Figure 1 Schematic of a polarimeter

A compound will consistently have the same specific rotation under identical experimental conditions. To determine the specific rotation of the sample, use Biot's law:

$$\alpha = [\alpha] \ell c$$

where α is the observed optical rotation in units of degrees, $[\alpha]$ is the specific rotation in units of degrees (the formal unit for specific rotation is $\text{degrees dm}^{-1} \text{mL g}^{-1}$, but scientific literature uses just degrees), ℓ is the length of the cell in units of dm, and c is the sample concentration in units of g/mL.

This experiment allows you to explore the interplay between these parameters in order to better understand polarimetry and how to use a polarimeter.

OBJECTIVES


- Become familiar with the use of the polarimeter.
- Experience how sample path length and concentration affect observed rotation.
- Calculate the specific rotation for a known sugar sample using Biot's law.

MATERIALS

Chromebook, computer, **or** mobile device
Vernier Instrumental Analysis app
Vernier Graphical Analysis app
Go Direct Polarimeter
polarimeter sample cell
50 mL volumetric flasks
50 mL graduated cylinder
50 mL beaker
sucrose

PROCEDURE

Part I Exploring Path Length

1. Obtain and wear goggles and gloves.
2. Accurately prepare 50 mL of a 10% aqueous sucrose solution. **Caution:** *Treat all laboratory chemicals with caution. Prudent laboratory practices should be observed.*
3. Launch Instrumental Analysis. Connect the Go Direct Polarimeter to your Chromebook, computer, or mobile device.
4. Calibrate the polarimeter.
 - a. Pour distilled water in the polarimeter cell to a height of 10 cm. It is important to read the height to the nearest 0.1 cm. Read to the bottom of the meniscus.
 - b. Place the cell in the polarimeter, and click or tap Finish Calibration.
 - c. When the polarimeter is ready, click or tap Done.
5. You are now ready to add the optically active sample into the polarimeter cell.
 - a. Pour the sucrose solution in the polarimeter cell to a height of 10 cm. Record this value to the nearest 0.1 cm in Table 1.
 - b. Place the sample cell in the polarimeter.
 - c. Start data collection. Data collection will stop automatically and data are stored automatically.
 - d. Empty the polarimeter cell and rinse with a small amount of your next sample.
6. Click or tap Graph Tools, , and use the Statistics or Curve Fit tool to determine the angle closest to 0° where the illumination is at a maximum. This is the observed angle of rotation of the plane of polarized light for the optically active sample. Record this value in Table 1.

7. Repeat Steps 5–6 for 8 cm, 6 cm, 4 cm, and 2 cm. Make sure to measure both the actual height of the liquid and the angle of rotation of the plane of polarized light with each change in volume of sucrose solution. Record these values in Table 1.

Part II Exploring Concentration

8. Prepare 50 mL each of 10%, 20%, and 30% solutions of sucrose in water.
9. You are now ready to add the optically active sample into the polarimeter cell.
 - a. Pour the 30% sucrose sample in the polarimeter cell to a height of 10 cm. Record this value to the nearest 0.1 cm in Table 2.
 - b. Place the sample cell in the polarimeter.
 - c. Start data collection.
10. Record the angle closest to 0° where the illumination is at a maximum.
11. Empty the polarimeter cell and rinse with a small amount of your next sample.
12. Repeat Steps 9–11 for the remaining samples you prepared.

DATA TABLE

Part I Exploring Path Length



Table 1					
	Run 1	Run 2	Run 3	Run 4	Run 5
Sample height (cm)					
Angle of rotation, α (°)					

Part II Exploring Concentration

Table 2			
	10% sample	20% sample	30% sample
Sample height (cm)			
Angle of rotation, α (°)			
Calculated concentration (g/mL)			

DATA ANALYSIS



Part I Exploring Path Length

1. Generate a graph of height of liquid (cm) on the x-axis vs. optical rotation in degrees on the y-axis.
 - a. Save your raw polarimetry data file. Close Instrumental Analysis.
 - b. Launch Graphical Analysis app, and select Manual Entry. You will see a data set with a column named “X” and a column named “Y”.
 - c. To rename the X column, click or tap  in the X column header. Enter **Height** as the name and **cm** as the units.
 - d. In the same manner, rename the Y column. Enter **Angle** as the name and **deg** as the units.
 - e. Enter your data in the appropriate columns. The data are automatically plotted on the graph.
 - f. Continue to Data Analysis Step 2.
2. Click or tap Graph Tools, , and use the Curve Fit tool to determine the relationship between liquid height and optical rotation.
3. Find the best-fit line through the data points. Determine the angle of rotation when the height of the sample is exactly 10.0 cm.
4. Calculate the specific rotation of sucrose using Biot’s law. Compare this value to the accepted literature value and calculate your percent difference.

Part II Exploring Concentration

5. Using the observed angle of rotation and Biot’s law, calculate the exact concentrations of each sample in g/mL. Record these values in the table above.
6. Generate a plot of the calculated concentration values in g/mL vs. optical rotation in degrees, as you did above.
7. Based on your data, what is the relationship between optical rotation and concentration?

EXTENSION

1. Analyze your polarimeter data using the two different methodologies that follow:
 - Statistics: Highlight the peak of interest. Click or tap Graph Tools, , and choose View Statistics. Record the angle value where the illumination is at a maximum.
 - Cosine Squared: Click or tap Graph Tools, , and choose Apply Curve Fit. Select Cosine Squared. Click or tap Apply. In this fit, the x-value corresponding to the maximum y-value is obtained from the negative of the phase shift parameter, $-C$. This is a nonlinear fit which undergoes numerous iterations and has the possibility of not converging, which will result in an unreasonable answer. With all nonlinear fits, it is important to make sure the resulting value is reasonable based on the data presented in the graph.
2. Compare the results from the different fits (Statistics and Cosine Squared). Discuss which one you think is more accurate and why.