

A3

A3-Vernier Polarimeter

Connection and Setup to LabQuest Interface



Figure A3.1: Vernier Polarimeter (left) connected to LabQuest interface (middle)

LOCATION

Organic Chemistry Laboratory: 2232 DP.

CONNECTING TO LABQUEST

The Polarimeter must be connected to a Vernier interface such as the LabQuest (Figure A1.5). To connect:

1. Insert the **POWER** cord if unplugged.
2. Insert the **CH1 CABLE** into the **CH1 SENSOR PORT** and the **DIG1 CABLE** into the **DIG1 SENSOR PORT** on the LabQuest. If disconnected, these cables should be located in the ring to the right of the LabQuest.
3. Optional (to measure temperature): Connect the Melt Station by inserting the **CH3 CABLE** into the **CH3 SENSOR PORT** on the LabQuest.
4. Press the **POWER BUTTON** at the top left of the LabQuest interface. Observe light coming from the bottom of the polarimeter.

Measuring Temperature

Since specific optical rotation can be temperature dependent, the temperature of the measurement should be reported as well. It is not necessary to turn the Melt Station dial to the on position to observe a temperature reading. If the Melt-Station has recently been used, the temperature may be artificially high. In that case read the temperature from a calibrated thermometer.

COLLECTING DATA ON THE HPLC COMPUTER

The easiest method of displaying and processing data is through the LoggerPro software installed on the HPLC computer. For instructions on how to use the LabQuest interface alone to collect and analyze data, download the manual from the Vernier website at:

<http://www.vernier.com/files/manuals/chem-pol.pdf>.

Automatic Sensor Detection

The Vernier polarimeter is automatically detected by LoggerPro if it is the only connected sensor (CH1 and DIG1) and will load the optimal data collection parameters automatically. If other sensors are connected (e.g., Melt-Station), LoggerPro will not load these parameters. In this case, open the file created by Prof. Landrie as described to the right.

1. Connect the *USB CABLE* to the *USB PORT* on the LabQuest interface.
2. On the HPLC computer, open LoggerPro. Note that while LoggerPro or LoggerLite are running, the LabQuest interface screen cannot be operated directly. To resume using the LabQuest interface, exit the LoggerPro software.
3. From the *FILE MENU* within LoggerPro, open the file *POLARIMETER_A* (Location: *MY COMPUTER* → *SHARED DOCUMENTS* → *VERNIER* → *POLARIMETER_A.PDF*).

Operating the Polarimeter

MEASUREMENT

Equation A3-1: Malus's Law.

$$I = I_0 \cos^2 \theta$$

The resultant intensity (*I*) of plane polarized light passing through two polarizers is proportional to \cos^2 of the angle between their transmission axes (θ).

Phase-Shift Parameter

When plane polarized light passes through an optically active solution, the transmission intensity of light at all angles is shifted. The magnitude of that phase shift is the optical rotation and can be measured by finding the distance between the angles where illumination is greatest in the two waveforms. Those angles are represented by $-C$, where C is the phase shift parameter in the \cos^2 curve-fit generated by LoggerPro.

Equation A3-2: LoggerPro Curve-Fit Equation for a \cos^2 Function.

$$Illum = A(\cos(Bx + C))^2 + D$$

A = amplitude; $B = 1$; x = angle

C = phase shift parameter

Equation A3-3: Determining Optical Rotation from Phase Shift Parameters.

$$\alpha = (-C_{sample}) - (-C_{blank})$$

1. Place a blank solution in the Vernier distance-graduated cylinder, ideally to 10.0 cm (1.0 dm). (This cylinder is graduated in units of centimeters. If volume-graduated cylinders are used, measure the path length with a ruler.)
2. Place the cylinder into the polarimeter retaining bracket.
3. Click the Collect Icon in LoggerPro to begin collecting data.
4. Slowly rotate the analyzer clockwise or counterclockwise until data collection stops (15 s). You should observe a cosine-squared waveform.
5. After data collection stops, click Store Latest Run from the Experiment menu.
6. Optional: From the *DATA MENU*, click *DATA SET OPTIONS* and choose *EDIT DATA SET*. Change the name of the current data set to *BLANK*.
7. Repeat steps 1-6 with your sample solution. You should observe the sample data set waveform superimposed onto the blank data set. Be sure to record the height of the sample solution as accurately as possible. In step 6, change the name of the data set to *SAMPLE*.

ANALYSIS

Generally speaking, the optical rotation is the difference between the two angles immediately after zero at which illumination is at a maximum (Figure A3.2). There are a number of ways to estimate these angles in LoggerPro. However, the most precise method is to fit the data to the true function, \cos^2 . This will reproducibly give optical rotation measurements within $\pm 0.1^\circ$.

1. Make sure that no portion of the graph is highlighted.
2. Choose *CURVE FIT* from the *ANALYZE MENU*.
3. Select the data set you wish to analyze, *BLANK* or *SAMPLE*.

Polarimeter Measurements are Relative

The Vernier polarimeter resets the angle to zero each time the polarimeter is connected regardless of the analyzer position. This means that all measurements are relative and require a blank run. If measurements need to be taken over multiple days, you can manually zero the analyzer when illumination is at its minimum by choosing “zero” from the “Experiment menu” in LoggerPro.

Equation A3-4: Specific Optical Rotation

$$[\alpha] = \frac{\alpha}{l \times c}$$

α = measured rotation

l = pathlength (dm)

c = concentration (g/mL)

4. Select the **COSINE SQUARED** function (also shown in Equation A3-2).
5. Select **TRY FIT**.
6. Select **CLOSE**. The data will be displayed in a box on the graph. The value of interest is the phase shift parameter (C). The angle of maximum illumination is the negative of the phase shift parameter, $-C$.
7. To find the optical rotation (α), subtract the blank's angle of maximum illumination from the sample's angle of maximum illumination (Equation A3-3).
8. To find the specific optical rotation ($[\alpha]_D$) use Equation A3-4. You must know the concentration of your solution as well as the pathlength of the sample cell. If you used the Vernier distance-graduated cylinder, the pathlength is measured in centimeters and must be converted to decimeters (dm) (10 cm = 1 dm).

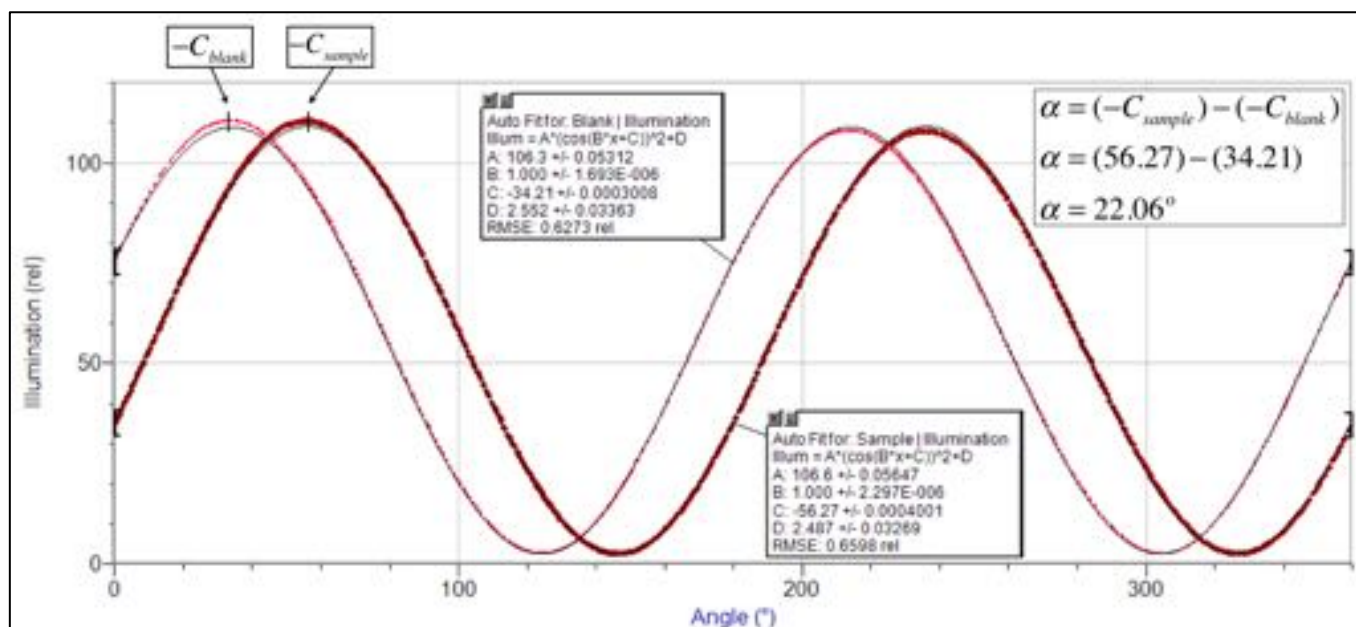


Figure A3.2: Finding Angles of Maximum Illumination from Logger Pro Curve Fit.