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Cost-Effective Teacher

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A New Cost-Effective Diode Laser Polarimeter Apparatus Constructed by Undergraduate Students

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The construction of a modular apparatus by undergraduate students gives them an understanding how it works and how a physical chemistry property is measured. The students choose the parts to assemble and evaluate the price and quality of the parts, which gives them valuable experience for their professional future. By using a modular apparatus, the undergraduate students understand the physical basis of the measurements as opposed to a commercial instrument that it is often viewed as a black box. The construction of this laser polarimeter apparatus is described. It is then used to obtain the rotation angle values for the acid hydrolysis of sucrose, which are in good agreement with values obtained by a commercial instrument and those reported in the literature. The construction took one semester to complete, including testing and development of the classic sucrose hydrolysis experiment adapted for this apparatus.

The laser polarimeter uses a diode laser as a light source, which is more powerful and has a narrower bandwidth than a high-intensity LED. In addition, the diode laser is less expensive than the narrower bandwidth helium–neon laser *(1)*.

The reaction chosen to test this modular apparatus was the hydrolysis of sucrose. It was the first reaction to be scientifically studied by chemical kinetics, by L. Wilhelmy *(2)* in 1850.

$$\underset{\text{(sucrose)}}{(+)\text{-}C_{12}H_{22}O_{11}} + H_2O + H^+ \rightarrow \underset{\text{(fructose)}}{(-)\text{-}C_6H_{12}O_6} + \underset{\text{(glucose)}}{(+)\text{-}C_6H_{12}O_6} + H^+$$

This reaction has been mentioned in this *Journal* as a practical example for testing the construction of inexpensive polarimeters *(1, 3–10)* and as a case study for chemical kinetics *(11–14)*. The angle of rotation α is given by

$$\alpha = [\alpha]_\lambda^T \ell c \qquad (1)$$

where $[\alpha]_\lambda^T$ is the specific rotation of a chiral compound at a temperature T and for an analyzing light with wavelength λ, ℓ is the optical path length (in dm), and c is the concentration of the chiral compound (in g/mL). To standardize the values of the specific rotation, T is 20 °C and the analyzing light is the sodium D line, at 589 nm wavelength. At $t = 0$,

$$\alpha_0 = [\alpha_S]_\lambda^T M_S \ell [S]_0 10^{-3} \qquad (2)$$

and at "infinite time" for a complete reaction,

$$\alpha_\infty = ([\alpha_G]_\lambda^T M_G + [\alpha_F]_\lambda^T M_F) \ell [S]_0 10^{-3} \qquad (3)$$

where M_S, M_G, and M_F are the molecular masses of sucrose, glucose, and fructose, respectively, and $[S]_0$ is the initial molar concentration of sucrose. The time dependence of the angle of rotation, α_t, for the reaction can be expressed as

$$\alpha_t = \alpha_\infty + \alpha_0 e^{-k't} - \alpha_\infty e^{-k't} \qquad (4)$$

which leads to

$$\ln(\alpha_t - \alpha_\infty) = \ln(\alpha_0 - \alpha_\infty) - k't \qquad (5)$$

where t is the reaction time and k' is the pseudo-first order rate constant. Another way to calculate the rate constant without the need to measure the α_∞ is to apply the Guggenheim method, reading α at equal time intervals *(15)* or to use an exponential function regression for the experimental data by means Solver application from Excel or equivalent computer software.

Experimental Setup

The experimental assembly is shown in Figure 1. The housing is an aluminum box painted black with a lid on the top side and is attached to a 20 cm × 25 cm × 15 cm optical mount breadboard. A 1 in. Polaroid filter, mounted in a retaining ring, and the polarimeter cell support (two parallel stainless steel cylindrical bars) are inside the housing box. The windows of the polarimeter glass cell are aligned with the two holes on the opposite sides of the housing box. Outside of the housing and aligned with the hole and the internal Polaroid filter is a 635 nm, 2 mW diode laser from Edmund Optics, with emission centered on 637 ± 5 nm. Aligned with the opposite hole is a 1 in. Polaroid filter in a precision rotation mount and a Si detector (DET36A, Thorlabs). The Si detector is connected to a digital voltmeter to measure the optical signal output. Using the Drude expression *(14)*, the angle of rotation for sucrose, $[\alpha_S]_{635}^{20}$, is 56.3°.

The polarimeter glass cell (Figure 1 and Figure 2) was made by a glass blower and has a length of 100 mm and a diameter of 50 mm and two acrylic caps with 1 cm width and a diameter greater than 50 mm. The acrylic caps have a 50 mm diameter circular groove that fits the glass cylindrical glass wall. The ends

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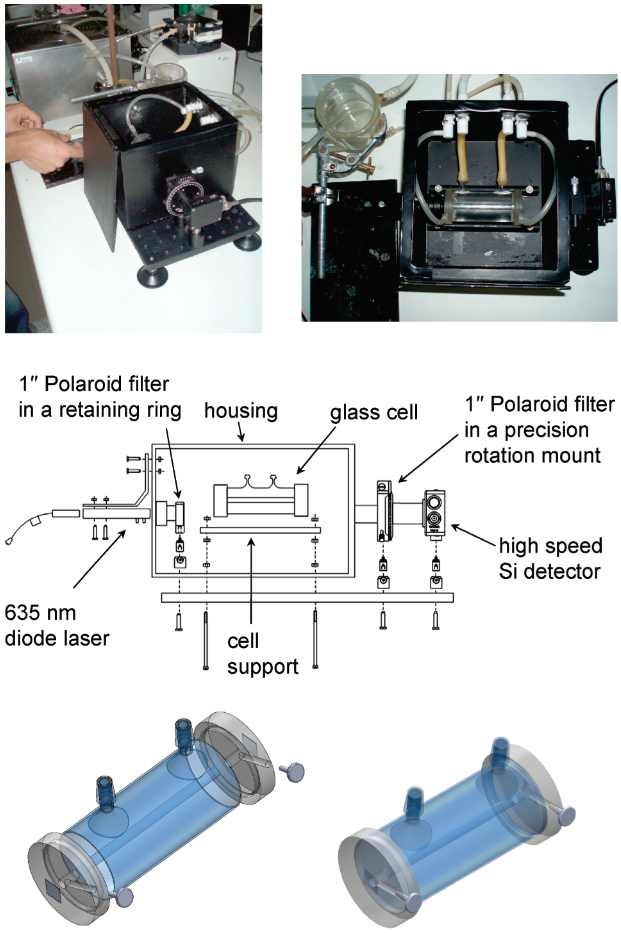

Figure 1. Above on left, the overall laboratory-made diode laser polarimeter apparatus; above on right, the housing with the glass cell; at the center, the schematic diode laser polarimeter apparatus; below on left, the assembly of the cell; below on right, the polarimeter cell.

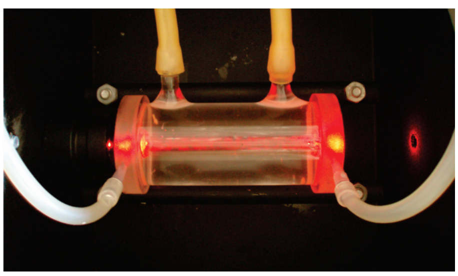

Figure 2. The polarimeter glass cell.

of the glass cell are glued into the grooves in the acrylic caps with an epoxy resin. At the center of each acrylic top is a 3 mm diameter hole with a 6 mm slot around each hole that fits a 6 mm diameter glass tube. The 6 mm diameter glass tube is 100 mm length and is the optical path of the cell. It is glued into the acrylic caps with an epoxy resin. Microscope glass covers are glued over the central holes in the acrylic tops to close off the 6 mm glass tube. A second hole in each acrylic cap was made along the radius of the cap, connecting to the central hole. The chiral solution is fed into the optical path of the cell through these holes. There is no dead space near the windows with this configuration.

Table 1. Description of the Components Used To Build The Polarimeter

Component	Source	Model Number	Price/€
Breadboard	Thorlabs	MB2025/M	121
Polarimetry cell	Made by a glass blower	-	-
Housing	Made by the students	-	-
2 Polarizers 1 in.	Edmund Optics	47316	272
Photodiode detector	Thorlabs	DET36A	98
Diode laser, 635 nm 2 mW	Edmund Optics	59084	158
Precision rotation mount	Thorlabs	PRM1	247
4 Quick connections and 4 stopper valves	RS Amidata	-	50
Precision voltmeter	In the lab	-	-
Peristaltic pump	In the lab	-	-
Temperature-controlled bath and water pump	In the lab	-	-

The polarimeter glass cell lies on two horizontal parallel stainless steel cylindrical bars, which are adjustable in height via four positioning screws that fix them to the optical breadboard. The cell is positioned in the laser beam with its windows perpendicular to the direction of the laser beam.

The 50 mm diameter glass cell is fitted by the glass blower with two connectors used to circulate water from a temperature-controlled bath outside the housing. The thermostated circulating fluid provides constant temperature in the polarimeter cell within ± 1 °C. There is a sample cell outside the housing box that is also connected to the thermostated circulating fluid. The sample cell is a double wall beaker connected in series with the polarimeter glass cell, as shown in the left upper corner of the right upper Figure 1. The temperature-controlled fluid circulates from the bath to the polarimeter glass cell and then to the sample cell before returning to the bath again by a pump.

The sucrose/acid solution is placed in the sample cell outside the box. A peristaltic pump takes the solution from the sample cell at a 32 cm^3/min flow rate to the polarimeter cell, thus, ensuring the stirring of the solution. Both sucrose/acid solution and water connections in the polarimeter cell are made by quick connectors with stopper valves.

The cost of this modular laser polarimeter apparatus (not including the common lab accessories such as the peristaltic pump, ~500 €; the temperature-controlled bath, ~400 €; the water pump, ~15 €; and the precision voltmeter ~150 €) was about the cost of a single commercial polarimeter cell, around 1000 € ($1300), as can be seen in Table 1. The polarimetry cell was made by a glass blower. It was a simple glass work, which took around 2 h to accomplish. The two acrylic tops were also made in 30 min by the glass blower; however, they can be made by an ordinary craftsman or even by a student. The housing, an aluminum box painted black with a lid on the top face, was made by students in one week. If it was not possible to have the help of a glass blower, one alternative solution is to use a water jacket connected to a simple polarimeter tube, as suggested in this *Journal (16)*.

We include the details of how a measurement is preformed with this apparatus in the supporting information. We also comprise some results obtained in a range of sucrose and hydrochloric acid concentrations. An Arrhenius plot for given experimental conditions shows the dependence of the kinetic constant with temperature that permits the calculation of the activation energy of the hydrolysis reaction of the sucrose.

Hazards

Hydrochloric acid is corrosive and causes burns to all body tissue.

Conclusions

A low-cost laboratory-made polarimeter has been developed and used for measuring the rotation angle of a sucrose solution during its hydrolysis reaction with hydrochloric acid. Undergraduate students built this apparatus during one semester as a physical chemistry project and the results obtained for the hydrolysis reaction are consistent with those published in literature or measured in commercial polarimeters, demonstrating that this diode laser polarimeter apparatus is a reliable piece of equipment when applied to the study of either hydrolysis of sucrose or polarimetry studies in general.

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Supporting Information Available

Details for the construction of a diode laser polarimeter apparatus; experimental details of the acid hydrolysis of sucrose. This material is available via the Internet at http://pubs.acs.org.

