

A Low-Cost Quantitative Absorption Spectrophotometer

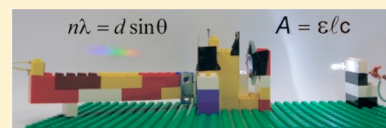
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S Supporting Information

ABSTRACT: In an effort to make absorption spectrophotometry available to high school chemistry and physics classes, we have designed an inexpensive visible light absorption spectrophotometer. The spectrophotometer was constructed using LEGO blocks, a light emitting diode, optical elements (including a lens), a slide-mounted diffraction grating, and a photodiode detector. The photodiode detector was mounted on a rotatable arm for wavelength selection based on elementary laws of diffraction. This simple design demonstrates basic physical principles (such as diffraction and absorption of light) that are frequently lost in commercial “black box” instruments. The homemade spectrophotometer’s performance, as measured by comparison to a commercial spectrophotometer, was shown to be sufficiently quantitative to facilitate experiments in chemistry or physics classrooms.

KEYWORDS: First-Year Undergraduate/General, High School/Introductory Chemistry, Physical Chemistry, Hands-On Learning/Manipulatives, Dyes/Pigments, Laboratory Equipment/Apparatus, Quantitative Analysis, Spectroscopy, UV–Visible Absorption



Absorption spectrophotometry is a commonly taught topic in advanced high school and introductory undergraduate chemistry laboratories. Historically, analog absorption spectrophotometers capable of measuring percent transmission at variable wavelengths (e.g., Spectronic 20) have been employed. More recently, the availability of USB-based visible spectrophotometers (such as those manufactured by Ocean Optics) have made it possible for students to rapidly obtain visible and near-UV absorption spectra and measure absorbance values at specific wavelengths.

Over the past several years, we have led a 4-h module in Cornell’s Institute for Chemistry Teachers (ICT) program, held annually in the summer. This one-week program brings about 25 high school chemistry teachers from around the United States to Ithaca, NY to participate in a week of development projects. The module involving the determination of the pK_a of indicators has been modified from an existing experiment¹ and is intended to be used in high school or introductory chemistry laboratory courses. The existing pK_a experiment may be adapted to the wide range of spectrophotometric equipment available at different schools. The flexibility of this experiment is important because access to spectrophotometers at different institutions can vary widely.

The experiment is carried out by high school teachers participating in ICT program as well as undergraduate students using Ocean Optics USB2000+ spectrophotometers. These spectrophotometers are comparable to research-grade instruments and, when used carefully, allow one to determine pK_a values to within 0.1 unit of literature values. Although the commercially available spectrophotometers provide accurate data, they largely represent a “black box”² that provides the user with little insight into the elementary physics associated with spectrophotometry. Additionally, more than 75% of the high school teachers polled stated that the high cost of modern spectrophotometers was prohibitive for regular classroom use.

The lowest-cost modern spectrophotometers (e.g., Vernier SpectroVis Plus) cost at least \$500 and also require a PC for data acquisition and manipulation.³

To help address this deficiency, we developed a simple, rugged, and low-cost *homemade* visible spectrophotometer. The homemade spectrophotometer could be constructed by high school students in physics or chemistry classes using readily available components. The total cost of the homemade spectrophotometer is about \$25, an eighth of the cost of a similar device⁴ and significantly less than commercial units. The design uses a battery pack, a white light emitting diode (LED) with current-limiting resistor (similar to those in LED flashlights), a lens, a cuvette and cuvette holder, a grating mounted on a slide, a photodiode mounted on a rotatable arm using a simple steel hinge, and a digital multimeter. For ease of alignment and use, we made an “optical table” using half of a 10 in. x 10 in. LEGO baseplate and LEGO construction blocks. It is exciting for students to perform scientific experiments with familiar materials typically thought of as toys.⁵ We note that the University of Wisconsin—Madison Materials Research Science and Engineering Center has published many exciting and educational experiments on “Exploring the Nanoworld with LEGO Bricks”.⁵ A photo of our homemade spectrophotometer is shown in Figure 1.

A number of previous designs have been aimed at inexpensive and robust spectrophotometers. The most notable being the LEGO spectrophotometer by Knagge et al.,⁴ the low-cost LED spectrophotometer by Yeh and co-workers,⁶ Sheeline’s cell phone spectrophotometer,⁷ and a homemade spectrophotometer by Lema et al.⁸ The target audience for Knagge’s LEGO spectrophotometer is first-year graduate students and fourth-year undergraduate students, which make it too complex for typical high school and introductory

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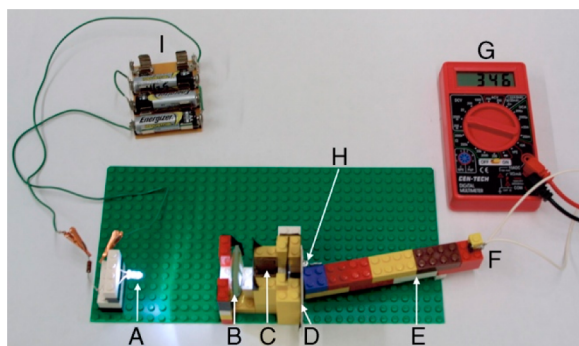


Figure 1. Fully assembled spectrophotometer consisting of (A) LED, (B) lens, (C) cuvette holder, (D) grating, (E) rotatable detector arm, (F) photodiode, (G) digital multimeter, (H) hinge, and (I) power supply for LED.

chemistry classrooms.⁴ Yeh et al.'s spectrophotometer uses a different LED for each wavelength, limiting it to the availability of different wavelength LEDs and the bandwidth of the LED output.⁶ Sheeline's cell phone spectrophotometer focuses, not on the determination of accurate absorbance values at specific wavelengths, but on understanding certain elements of spectrophotometer design such as dynamic range, stray light, and so forth.⁷ Lema's design achieves adequate results for low cost. However, one is not able to select specific wavelengths, only colors, that is, the user can only choose a "red" part of the spectrum, but not specifically 650 nm.⁸ Whereas each of these spectrophotometers has its own merits, our homemade spectrophotometer offers low-cost quantitative absorption measurements at well-defined wavelengths across the visible spectrum. At the same time, it provides the opportunity for students to understand the fundamental physical principles underlying absorption spectrophotometry.

When we demonstrated a prototype of the spectrophotometer to 20 CICT high school teachers, the device received strong approval. We have compared the performance of this device to that obtained using an Ocean Optics USB2000+ spectrophotometer. The quantitative performance of the homemade spectrophotometer makes it a useful tool for a variety of high school or introductory undergraduate laboratory experiments.^{1,9–12}

MATERIALS AND CONSTRUCTION

The primary focus for the instrument development was on simplicity, ease of construction and use, and cost minimization, without sacrificing performance. The entire apparatus was built on a 5 in. × 10 in. LEGO baseplate using LEGO blocks. The light source was a 5 mm diameter white LED and 50 Ω current-limiting resistor, powered by three AA batteries. Although a lens is built into the LED package, a second lens was added to better collimate the light. White light passes through the sample, which is held in a plastic 1 cm × 1 cm cuvette. The transmitted light is dispersed by a 1000 line/mm slide-mounted diffraction grating. The diffracted light was detected with a photodiode mounted on a rotatable arm. Wavelengths were calculated directly from the detector angle using the Fraunhofer diffraction equation. The voltage produced by the photodiode was read using a digital multimeter.

Detailed instructions for constructing the spectrophotometer, construction materials (and their cost) may be found as Supporting Information. Although setting up the apparatus and

getting it to work is a quick and easy task, making the apparatus work well requires additional time and attention to detail. In our experience, the spectrophotometer can be constructed and optimized in approximately 45 min using the instructions provided in the Supporting Information.

EXPERIMENTAL SECTION

Equation 1 is the Fraunhofer diffraction equation:

$$n\lambda = d \sin \theta \quad (1)$$

where the wavelength of light (λ) diffracted at a measured angle (θ) can be calculated. The grating order is n (here $n = 1$), and the grating line spacing d for a 1000 line/mm grating is 1.0×10^{-6} m. According to eq 1, orange light at 589 nm ($\lambda = 0.589 \times 10^{-6}$ m) is diffracted at $\theta = 36^\circ$.

To a reasonable approximation, one can assume the measured voltage is directly proportional to the intensity of the transmitted light, leading to the following equation for the absorbance, A :

$$A = \log_{10} \left(\frac{V_0 - V_d}{V - V_d} \right) \quad (2)$$

where V_0 is the voltage measured with a blank sample (distilled water), V is the voltage measured with the sample of interest, and V_d is the dark signal. The dark signal, which results from stray light that reaches the photodiode, was measured by placing a cuvette containing milk into the cuvette holder.

To take measurements, the angle of the detector was set using a protractor. The cuvette containing the solution was placed into the cuvette holder, and a cardboard box was placed over the setup to exclude room light. The angle and voltage readings were recorded in tabular form and then converted to wavelength and absorbance values using eqs 1 and 2. Additional detail is available as Supporting Information. Figure 2 shows

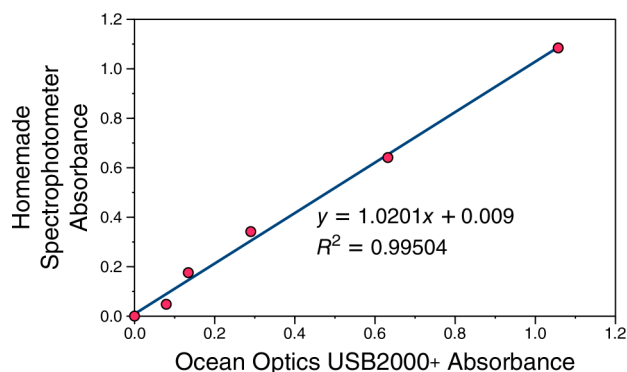


Figure 2. Absorbance at 590 nm recorded using basic bromothymol blue solutions at various indicator concentrations. For each solution, the absorbance measured using the homemade spectrophotometer is plotted versus that measured with an Ocean Optics USB2000+ spectrophotometer. Circles represent absorbance measurements; the line represents a least-squares fit.

calculated absorbance values at 590 nm for basic bromothymol blue solutions at various indicator concentrations using the homemade spectrophotometer versus absorbance values measured using an Ocean Optics USB2000+ spectrophotometer.

A least-squares fit to the data in Figure 2 shows that the absorbance measurements made using the homemade spec-

trophotometer are in close agreement with those made using the Ocean Optics USB2000+ over the typical range of absorbance measurements up to $A \sim 1$. As is usual for absorption spectrophotometry, it is desirable to keep absorbance values within this range as measured signals become comparable to the dark signal (i.e., become $<10\%$ of the maximum values) as A exceeds 1.

To map out the entire visible absorption spectrum, the detector angle was varied in 1° increments, using a protractor to set the angle from 24° to 45° (~ 400 to 700 nm). This enabled the determination of the absorbance of acidic, basic, and neutral bromothymol blue solutions over the visible range. The absorbance values were also determined for the same range with the Ocean Optics USB2000+ spectrophotometer. The results for each are compared in Figure 3. The calculated absorbance values between 420 and 660 nm for the homemade spectrophotometer are in close agreement with those from the Ocean Optics USB2000+ spectrophotometer.

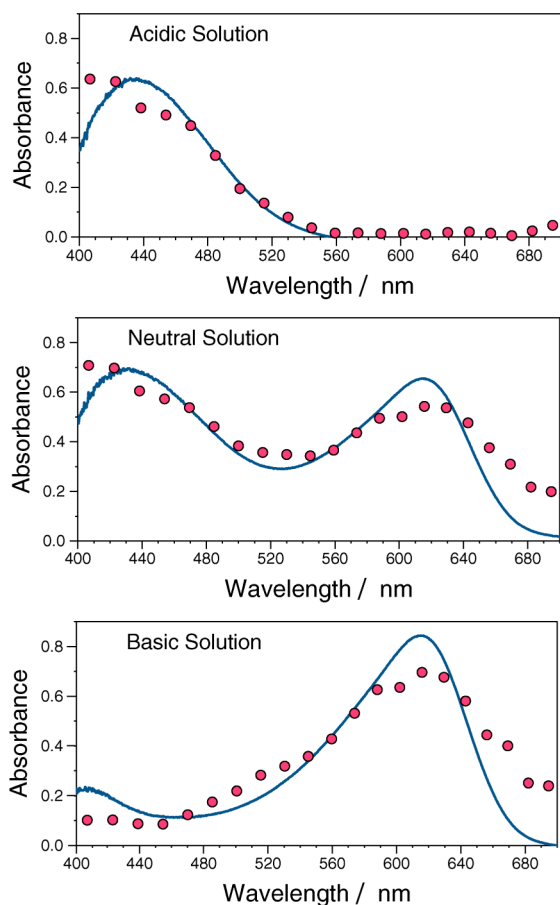


Figure 3. Absorbance of bromothymol blue in various pH solutions. Continuous spectra (solid line) were measured using the Ocean Optics USB2000+ spectrophotometer and data points (solid circles) were obtained using the homemade spectrophotometer.

The calibration of the absolute wavelength is subject to several potential sources of error. These include error in alignment of the rotating arm, error in alignment of the grating, and deviations between the grating line density (1000 lines/mm) specified by the manufacturer and actual line density. To check this, it is convenient to exploit the fact that light that appears orange to the eye spans a very narrow wavelength range near 589 nm and should be diffracted at $\theta = 36^\circ$. This

prediction can be tested by temporarily removing the photodiode and placing one's eye at the location of the photodiode when set to $\theta = 36^\circ$ and looking toward the grating. We found that the predicted diffraction angle coincided exactly with that observed, indicating that the wavelength error is negligible compared to the bandwidth of the spectrophotometer.

The accuracy of the calculated absorbance is dependent upon the absolute signal level and the detector bandwidth. At wavelengths where the LED output is high, calculated absorbance values more closely match true values than those calculated from wavelengths of low output. As shown in Figure 4, the spectrum of the LED is somewhat structured, with a

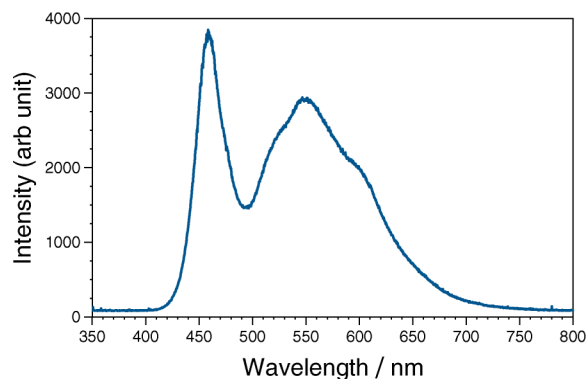


Figure 4. SuperBrightLED RL5-W18015 output as a function of wavelength recorded with Ocean Optics OOIBase32 software.

maximum near 460 nm. The LED intensity is small outside of the 420 – 660 nm range. By using LEDs with different emission spectra, absorbance values could be measured at wavelengths outside of the 420 – 660 nm range.

In the present configuration, the photodiode spans an angular range of about 1.5° , corresponding to a bandwidth of ~ 20 nm. A longer arm would increase the spectral resolution, for example, doubling the length of the rotatable arm would decrease the bandwidth to ~ 10 nm. However, this significantly decreases signal levels. Using the configuration described here, typical voltages measured using the spectrophotometer with blank samples in the 450 – 640 nm range were in the 300 mV range. Because the voltmeter is capable of measuring to within 1 mV, measurements can be made to three significant figures. The substantially decreased signal levels that would result from increased arm length increase the uncertainty in each measurement. This results in increased scatter in the measured data. Most commonly, poor performance results from poor alignment or the presence of excessive scattered light. After some experimentation, the configuration described here and in the Supporting Information provides an optimal balance between signal levels and spectral resolution.

CONCLUSIONS

Construction and use of quantitative homemade spectrophotometers can be a straightforward and inexpensive endeavor. For educational purposes, it is often desirable to sacrifice a small amount of accuracy in favor of simplicity and reduced cost. Considerable educational value is associated with construction of a complete working instrument. The homemade spectrophotometer yields quantitatively acceptable results while allowing the fundamental scientific principles to be

qualitatively apparent. Allowing students to build their own spectrophotometer provides ample opportunity for teaching the underlying physical principles of spectrophotometry, while also allowing for creative variations and improvements in the design.

As scientific instruments become more sophisticated, the conceptual aspects of the relevant physical phenomena are often hidden in a box and controlled via a computer. This spectrophotometer aims to eliminate these hidden features, allowing the user to understand the fundamental qualities of instrument use and design. The low-cost and quantitative performance of the spectrophotometer should make hands-on absorption experiments available to all classroom environments.

■ ASSOCIATED CONTENT

📄 Supporting Information

Detailed instructions and a parts list for constructing the homemade spectrophotometer. This material is available via the Internet at <http://pubs.acs.org>.

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Notes

The authors declare no competing financial interest.

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