

Simple and Inexpensive UV-Photometer Using LEDs as Both Light Source and Detector

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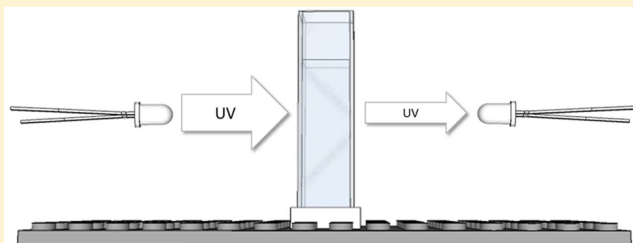
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Supporting Information

ABSTRACT: This article describes how to construct a simple, inexpensive, and flexible UV-photometer using one LED (355 nm) as the light source and another (355 nm) as the detector, one resistor, some wiring, a few Lego bricks, and a millivolt meter. The photometer is used to explore several aspects of molecular absorption spectrometry. As practical applications, the amounts of folic acid in folic acid tablets and cinnamaldehyde in cinnamon were found. The results were in accordance with those obtained using a commercial spectrophotometer.

KEYWORDS: High School/Introductory Chemistry, First-Year Undergraduate/General, Analytical Chemistry, Hands-On Learning, Spectroscopy, UV-Vis Spectroscopy



INTRODUCTION

Spectroscopy is an important part of chemistry syllabi. In practical spectrometry we want to demonstrate basic principles as well as application, however spectrophotometers, like other modern instruments, are black-box systems less suited to conveying fundamental concepts.¹ They are also costly. Colorimetry is a simple method for demonstrating spectroscopic principles; however, from a range of standard solutions the student can often visually estimate the concentration in an unknown sample without the use of a colorimeter. Molecular absorption spectroscopy covers both the ultraviolet (UV) and visible regions, and demonstration in the ultraviolet region has the advantage that all the solutions appear transparent to the naked eye.

In order to measure absorbance in the UV region we have constructed a photometer that uses UV light emitting diodes (LEDs) as both light source and light detector. LEDs increase in price with decreasing wavelength; a red LED costs less than \$1, whereas a 250 nm emitting LED costs about \$300. LEDs in the UV region are expected to drop in price with increasing use and technological progress, but in the meantime they are pricy for many uses. In addition, the shorter the wavelength, the more precautions are necessary from a health, safety, and environmental perspective. We have chosen a 355 nm LED as a light source, which is palatable from the perspectives of both price (at present about \$30) and hazard. Our UV-photometer relies on the fact that LEDs can be used as light detectors for the same and shorter wavelengths than they emit themselves.^{2–4} The photometer is simple and all its functioning parts are visible. In addition, the parts of the photometer can easily

be moved around (as they are made from Lego bricks) making it suitable for exploring the Beer–Lambert Law as described for our colorimeter in a previous article.¹

The UV-photometer (Figure 1) can be assembled in a few minutes and needs only a minimal amount of circuitry (a few wires, a 4.5 V battery, and a single resistor). The resistor is needed to bring the voltage to within the optimal working

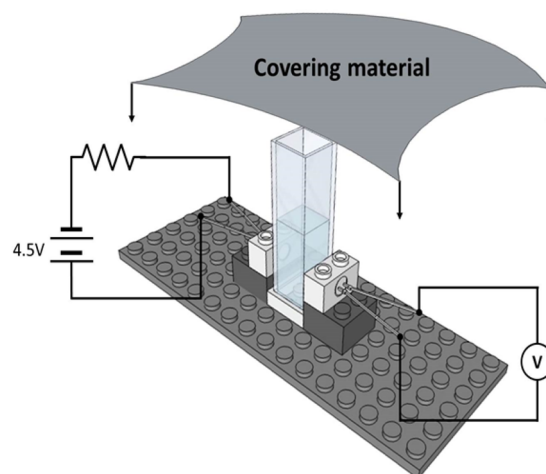


Figure 1. Complete photometer.

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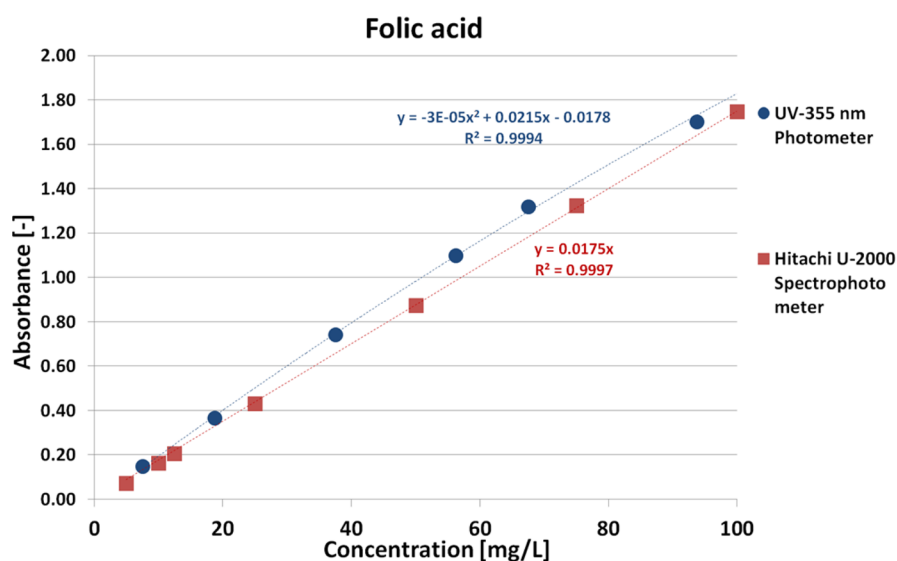


Figure 2. Absorbance of light from a UV LED (355 nm) by folic acid (in 0.10 M NaOH) as a function of concentration, using our UV-photometer and a commercial Hitachi U-2000 spectrophotometer. Fits to the results are also shown, together with R^2 measure of goodness of fit.

range of the LED light source. A few standard Lego bricks provide an accurate and robust alignment of the light source and detector. Their inherent flexibility has previously been exploited to construct model instruments^{5,6} and demonstrate chemical structures.⁵ Another type of enclosure for the LEDs and circuitry is of course possible. For example one made by 3D-printing, as suggested by Anzalone et al.⁷

Small-scale photometers using a LED as a light source were previously reported, both for pedagogical^{1,8–13} and purely analytical^{3,4,14} purposes, but they all operate in the visible spectrum. Photometers using a LED as a light detector use the LED in reverse biased mode, requiring an external voltage and additional circuitry. References to this type of use can be found elsewhere.^{3,4,15,16} Our photometer functions with a LED as detector with no circuitry. The measurement, voltage, is made directly across the LED (see Figure 1). This reduces the number of components and simplifies understanding how the instrument works, an asset many chemistry students and teachers appreciate, and avoids distracting their focus from the science being studied. A literature search in SciFinder and Chemical Abstract did not reveal any report on the use of a LED photometer for the UV-region of such simplicity.

EQUIPMENT SET-UP

Figure 1 illustrates the complete photometer. The top of one Lego brick (2×2) is cut off in order to accommodate a cuvette. Each LED is put into a Lego brick with hole (2×1 with hole). A 4.5 V power source (3×1.5 V batteries) wired in series with a resistance appropriate to the particular LED (typically in the range 10–50 Ω for a 355 nm UV-LED) powers the photometer. The intensity of the emission from a LED depends on the voltage applied and the temperature of the LED. It typically takes 15–30 min for the LED to reach stable conditions. Over time batteries become weaker, which means the voltage applied to the LED diminishes. A solution to this problem is to adapt a mobile charger to provide a stable voltage and thus stable conditions (see Supporting Information (SI)). However, for most uses a battery works well enough.

Measurements of transmittance are made using a millivolt meter (e.g., digital multimeter with internal resistance of at least

10 M Ω) connected directly across the detector LED. Voltages typically range in value from 1–100 mV. If necessary, a piece of black plastic or other material may be used as a cover to avoid stray light.

EXPERIMENTS

Standard Solutions of Folic Acid

Standard solutions of folic acid [CAS 59-30-3] were made in the range 5–100 mg/L in 0.10 M NaOH (see SI for details). Folic acid, for the purpose of this assay, was isolated from folic acid tablets (Nycoplus Folsyre, 130 mg tablets each containing 400 μ g folic acid) and dissolved in 0.10 M NaOH (see SI).

Standard Solutions and Isolation of Cinnamaldehyde

Standard solutions of cinnamaldehyde [CAS 104-55-2] were made in the range 50–1500 mg/L in ethanol/water (50/50%) (see SI for details). Cinnamaldehyde (1–2%) was isolated by steam-distillation in a Hickmann column from both cinnamon powder and cinnamon bark (about 500 mg) and diluted in 50% ethanol (see SI).

Measurements

Measurements were made as transmittance

$$T = \frac{V_{\text{solution}}}{V_{\text{solvent}}} \quad (1)$$

where V_{solution} is the voltage measured when the sample solution is used and V_{solvent} when solvent blank is used.

Absorbance ($-\log T$) is then calculated from transmittance.

HAZARDS

No hazard is involved if the Lego brick is held with pliers when cutting the top off. If deemed necessary, the teacher can undertake this task before class. All the solutions must be handled according to safety instructions. Cinnamaldehyde is harmless in the concentrations used here, but the pure substance is a skin, eye, and respiratory irritant. A solution of 0.10 M NaOH is dilute; nevertheless, it should be handled with care as NaOH is corrosive. Users should use safety goggles. If deemed necessary, wear protective clothing. Ethanol is

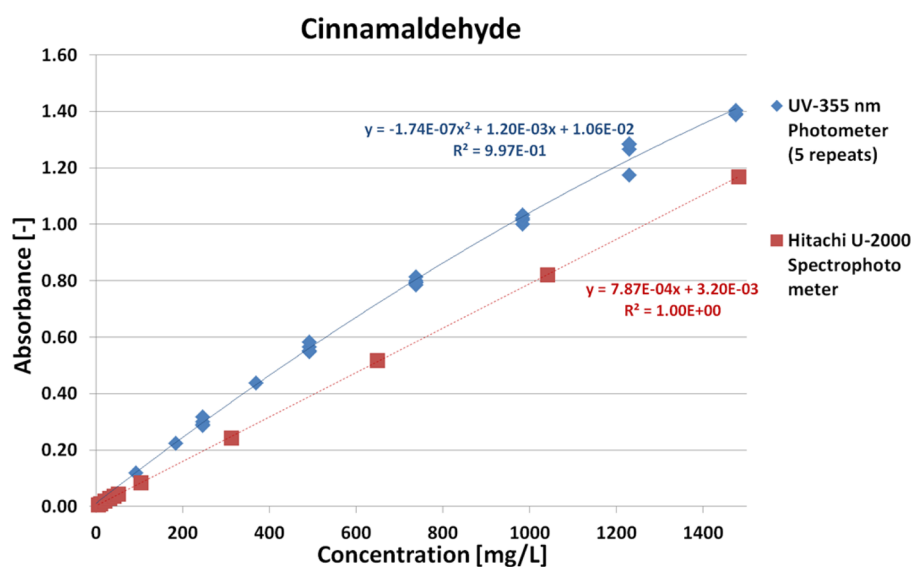


Figure 3. Absorbance of light from a UV LED (355 nm) by cinnamaldehyde (in 50% ethanol) as a function of concentration, using our UV-photometer and a commercial Hitachi U-2000 spectrophotometer. Fits to the results are also shown, together with R^2 measure of goodness of fit.

flammable and should be kept away from ignition sources. A LED emitting light around 355 nm should not be viewed directly. The construction itself, in which the two diodes are placed opposite each other, makes this nearly impossible, but users should be aware of this UV hazard. As this UV radiation (355 nm) is close to visible light this is less of a problem.

RESULTS AND DISCUSSION

Cuvettes: UV Transparent and Solvent Compatible

Spectrometry in the UV region often relies on expensive and fragile quartz cuvettes. However, the experiments described here may be successfully conducted using inexpensive disposable UV-cuvettes or even standard disposable cuvettes, as we have found that neither absorb at 355 nm. The next requirement is that the cuvettes are solvent resistant. Poly(methyl methacrylate) cuvettes work well with dilute aqueous solution but are tarnished by a 50% ethanol solution, the solvent used to dissolve cinnamaldehyde. Disposable BRAND UV-cuvettes are resistant to a range of solvents, including ethanol, and were used for the 50% ethanol solutions.

Compounds and Measurements

For our experiment we sought compounds that absorb light at 355 nm, and with a number of additional requirements to render them useful in a school and nonlaboratory setting. They should be safe, familiar, readily available, inexpensive, and colorless in solution, as well as soluble in nonhazardous solvents. There are few compounds satisfying all these requirements, among which we have chosen folic acid (a vitamin B type compound) and cinnamaldehyde. Folic acid in basic solution has three principal absorbance maxima at 283, 254.5, and 365 nm. Cinnamaldehyde has a single absorption maximum at 295 nm (see spectra in SI) and at 355 nm absorption is very low; nevertheless it is sufficient for this experiment. Alternative compounds that could be used are riboflavin, *para*-nitrophenol, or quinine. Quinine will fluoresce when subjected to 355 nm irradiation, but this will not influence the absorption measurements as the detector LED only responds to wavelengths shorter than about 355 nm.

Folic acid, when dissolved in 0.10 M NaOH, is deprotonated and exists as sodium folate. Folic acid is yellow as a solid, but in the concentrations used here the solutions are colorless. Figure 2 gives the absorption curve for folic acid in the range 5–100 mg/L obtained with our UV-photometer, together with that obtained at 355 nm using a commercial (Hitachi U-2000) spectrophotometer. The slope of the graph (i.e., absorptivity) from our UV-photometer is slightly higher than that obtained from the commercial spectrophotometer. Considering that the peak wavelength of the LED can vary between 353 and 360 nm (according to the product data sheet), this small difference is hardly surprising. In addition the curve from the UV-photometer exhibits a negative deviation from linearity at higher concentrations, as is expected due to the varying absorptivity of folic acid across the 15 nm bandwidth of the UV-LED. Therefore, we choose to use a second order fit to the data to define our standard curve. A linear fit would also give sufficient accuracy for most purposes, especially if concentrations are kept in the range 0–70 mg/L.

This standard curve from the UV-photometer can be used to find the concentration of an unknown sample, and we have found the amount of folic acid in the folic acid tablets (Nycoplus). The results were in accordance with those obtained using the commercial spectrophotometer, which showed around 480 μg per tablet when analyzed at 355 nm and at the two absorption peaks of 283 and 254.5 nm. Our results were some 20% higher than declared by the supplier (400 μg per tablet).

Cinnamaldehyde is the major compound providing the smell associated with cinnamon. Figure 3 shows the absorption curve obtained with our UV-photometer, based on 5 separate analyses of standard solutions of cinnamaldehyde dissolved in 50% ethanol, together with results obtained using the commercial spectrophotometer.

In this case the slope of the graph (i.e., absorptivity) from our UV-photometer is considerably higher at low concentration than that obtained from the spectrophotometer and the negative deviation at higher concentrations is more pronounced than for folic acid. The main reason for these differences is the higher variation of absorption with wavelength around 355 nm

for cinnamaldehyde (see SI). The steeper the absorbance spectrum and the wider the bandwidth of the incident spectrum, the more pronounced are these effects. The 5 repeat calibration curves obtained with the UV-photometer show rather low scatter (see Figure 3) and a second order fit to these data was used to define the standard curve. This standard curve was used to find the content of cinnamaldehyde isolated from cinnamon powder in cinnamon capsules (Solaray), commercial cinnamon powders (2 samples), and cinnamon bark (2 samples). The calculated content of cinnamaldehyde for the 5 samples tested varied between 1.2% and 1.8%, by weight, and was in all cases within 10% of the results obtained using the commercial spectrophotometer. If less time is available, an unknown sample from commercial cinnamaldehyde can be used.

The experiments described here also provide an opportunity to discuss aspects of molecular absorption spectrophotometry, such as structure and absorption wavelength, detection limit, linearity of calibration curve, and negative deviation due to polychromatic radiation and varying molar absorptivity (see SI for details and more topics).

SUMMARY

A simple, flexible, low-cost UV-photometer is made from two UV LEDs (355 nm), a few wires, a single resistor, a battery, a few Lego bricks, and a millivolt meter. This photometer is used to find concentrations of transparent solutions that absorb at 355 nm, i.e., folic acid (from folic acid tablets) and cinnamaldehyde (in cinnamon). Due to polychromatic radiation from the LED, the calibration curves show negative deviation, but nevertheless give quantitative results in line with those obtained with a commercial spectrophotometer.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: [10.1021/acs.jchemed.6b00156](https://doi.org/10.1021/acs.jchemed.6b00156).

Instructor/students notes with supplementary experimental details and pedagogical considerations (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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REFERENCES

- (1) Asheim, J.; Kvittingen, E. V.; Kvittingen, L.; Verley, R. L. P. A Simple, Small-Scale Lego Colorimeter with a Light-Emitting Diode (LED) Used as Detector. *J. Chem. Educ.* **2014**, *91* (7), 1037–1039.
- (2) Mims, F. M., III. *Light Emitting Diodes*; Howard W. Sams & Co.: New York, 1973; pp 118–120.
- (3) Macka, M.; Piasecki, T.; Dasgupta, P. K. Light-Emitting Diodes for Analytical Chemistry. *Annu. Rev. Anal. Chem.* **2014**, *7*, 183–207.
- (4) Lau, K. T.; Baldwin, S.; Shepard, R. L.; Dietz, P. H.; Yezunis, W. S.; Diamond, D. Novel fused-LEDs devices as optical sensors for colorimetric analysis. *Talanta* **2004**, *63*, 167–173.

- (5) Campbell, D. J.; Miller, J. D.; Bannon, S. J.; Obermaier, L. M. An Exploration of the Nanoworld with Lego Bricks. *J. Chem. Educ.* **2011**, *88*, 602–606.
- (6) Albert, D. R.; Todt, M. A.; Davis, H. F. A Low-Cost Quantitative Absorption Spectrophotometer. *J. Chem. Educ.* **2012**, *89*, 1432–1435.
- (7) Anzalone, G. C.; Glover, A. G.; Pearce, J. M. Open-Source Colorimeter. *Sensors* **2013**, *13*, 5338–5346.
- (8) Wang, J. J.; Núñez, J. R. R.; Maxwell, E. J.; Algar, W. R. Build your own Photometer: A Guided-Inquiry Experiment To Introduce Analytical Instrumentation. *J. Chem. Educ.* **2016**, *93*, 166–171.
- (9) Hamilton, J. R.; White, J. S.; Nakhleh, M. B. Development of Low-Cost Four-Color LED Photometer. *J. Chem. Educ.* **1996**, *73*, 1052–1054.
- (10) Crump, J.; Sandwick, R. K. A Simple Microwell Colorimeter for Use in an Introductory Chemistry Lab. *J. Chem. Educ.* **1994**, *71*, A199–A200.
- (11) Gordon, J.; Tye, S. A LED Microtiter Plate Reader. *J. Chem. Educ.* **2005**, *82*, 903–904.
- (12) Gordon, J. S.; James, A.; Harman, S.; Weiss, K. A Film Canister Colorimeter. *J. Chem. Educ.* **2002**, *79*, 1005–1006.
- (13) Mozo, J. D.; Galán, M.; Roldán, E. Application of Light Emitting Diodes to Chemical Analysis: Determination of Copper in Water. *J. Chem. Educ.* **2001**, *78*, 355–357.
- (14) Lau, K. T.; Yezunis, W. S.; Shepherd, R. L.; Diamond, D. Quantitative colorimetric analysis of dye mixtures using optical photometer based on LED array. *Sens. Actuators, B Chem.* **2006**, *114*, 819–825.
- (15) Lau, K. T.; McHugh, E.; Baldwin, S.; Diamond, D. Paired emitter-detector light emitting diodes for measurement of lead(II) and cadmium(II). *Anal. Chim. Acta* **2006**, *569*, 221–226.
- (16) Tymecki, Ł.; Pokrzywnicka, M.; Koncki, R. Paired emitter detector diode (PEDD)-based photometry- an alternative approach. *Analyst* **2008**, *133*, 1501–1504.