JOURNAL OF CHEMICAL EDUCATION

Article

pubs.acs.org/jchemeduc

Construction and Characterization of a Compact, Portable, Low-Cost Colorimeter for the Chemistry Lab

Carrie M. Clippard

Bellwood-Antis High School, Bellwood, Pennsylvania 16617, United States

William Hughes

Park Forest Middle School, State College, Pennsylvania 16803, United States

Balwant S. Chohan

Department of Math & Natural Science, Centenary College, Hackettstown, New Jersey 07840, United States

Danny G. Sykes*

Department of Chemistry, The Pennsylvania State University, University Park, Pennsylvania 16802, United States

Supporting Information

ABSTRACT: A low-cost and portable colorimeter was constructed featuring a low-voltage programmable color light sensor-to-frequency converter, a CMOS 8-bit microcontroller, and an LCD display. The instrument has successfully facilitated the introduction and application of spectroscopy to groups of middle school, high school, and undergraduate students. A series of tried and tested laboratory experiments that demonstrate the utility and capabilities of the colorimeter have been developed. These laboratories not only help characterize the colorimeter but also feature challenging problems that allow students to realize the limitations of analytical instruments. The accompanying laboratories enable students to determine the concentration of a mixture of food dyes using simultaneous equations, quantify inorganic phosphate in various aqueous-based agricultural samples, calculate the concentration of salicylic acid in facial acne medication, and elucidate the stoichiometry of an iron salicylate reaction.

KEYWORDS: *Elementary/Middle School Science, High School/Introductory Chemistry, First-Year Undergraduate/General, Second-Year Undergraduate, Upper-Division Undergraduate, Analytical Chemistry, Laboratory Instruction, Hands-On Learning/Manipulatives, Quantitative Analysis, UV−Vis Spectroscopy*

INTRODUCTION

Traditionally, the teaching of analytical chemistry has been carried out in an approach involving formal lectures to transmit knowledge; students receive it passively and must then reproduce this information at examination time. This type of teaching method leads to a surface level approach to learning and an overdependence on the lecturer. In contrast, engaging students with lab-based problem scenarios motivates them to practice higher-level critical thinking skills, develop independent learning strategies, and create relatable learning experiences.[1] We have developed a set of lab-based problem scenarios that help chemistry students acquire disciplinary knowledge as well as higher order thinking skills.[2−5]

One enormous benefit of redeveloping analytical chemistry lectures and laboratories is that it provides an ideal solution to the high cost of modern analytical instrumentation. The analytical sciences have changed dramatically over the past three decades and now involve advanced instruments and techniques that can analyze multiple analytes in multiple samples, occurring in a variety of challenging matrices. However, the high cost of instrumentation (and/or large course enrollments) prohibits many institutions from training students to understand, utilize, and develop these techniques. As a consequence, students either submit samples to the instructor or teaching assistant and receive a copy of the results some time later or are allowed to introduce the sample into the instrument themselves and press a green button to execute a preconfigured optimized method in order to collect their results. In either case, students receive limited real-world hands-on experience in method optimization and validation and the concomitant issues associated with method development, a process which is central to the field of analytical chemistry. Further, students never experience the compromise between theory ("we can analyze anything") and reality ("the limitations in our ability to detect and quantify") with respect to instrument design and operation. Unfortunately, a high-throughput black box approach to student access is often antithetical to the pedagogical aims of the course.

Received: September 11, 2015
Revised: January 15, 2016
Published: February 1, 2016

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241−1248

The SMILE (small, mobile instruments for laboratory enhancement) program was created in response to this pressure and has been used to create low-cost, low-maintenance, and rugged analytical instruments for our chemistry courses.[6−10] In the SMILE curriculum, senior-level analytical students are provided basic information on the chemistry of sample preparation, instrumental methodology, and the common electronic components found in modern analytical instruments. These fundamental elements paired with several lab-based miniprojects provide the scaffolding for the main semester-long problem, where students cooperatively design instruments for specific sample types and analytes. Student teams generate their own strategies to define the analytical and instrumental problems and seek solutions. They hypothesize, build, test, analyze the data, and compare their approach with the instructor and other teams in the lab. Oftentimes, solving these lab questions requires integration of interdisciplinary knowledge and experiences. Students receive prompt feedback and on-demand teaching, thus catering to students' preferred learning modes, empowering students, and building accountability. The teacher intentionally assumes the role of a facilitator rather than knowledge-holder and disseminator. The students in turn assume the role of active problem solvers who then make the majority of all important decisions. This reflects what goes on in the real world, and so encourages active and context-based self-directed learning. SMILE is a value-added component to the analytical curriculum; students continue to receive extensive hands-on experience with commercial research-grade instrumentation.

The SMILE curriculum was introduced in 1993. Since then the number and types of instruments have continued to grow: fluorimeter,[7,8] Karl Fischer,[6,10] cyclic voltammeter,[9] NMR probe,[11] and many unpublished devices, including conductivity meter, dissolved oxygen probe, GC-FID, CE-chip device, diamond-anvil cell, and several others, including the colorimeter described herein. The first four devices listed above and the colorimeter have matured to the point where these instruments are now fully assembled and tested by senior-level analytical students as part of their mini lab project at the start of the semester-long course, and then donated to other science courses (general chemistry, basic analytical chemistry) or to local schools for use in their science laboratories. The colorimeter is the focus of the current manuscript; this instrument is of particular interest because several cohorts of middle and high school students have constructed and assembled the colorimeter as part of STEM initiatives at these institutions.[12] If simply used as a black box, the colorimeter is an effective and sufficiently accurate device for basic spectrophotometric measurements. However, guiding students (middle school to undergraduate analytical) through the construction of the device exposes students to basic principles in electronics, integrated chip programming, and instrument design and operation. The above concepts can be explored in as much or as little depth as the instructor so chooses, making the colorimeter a versatile platform for STEM instruction.[12]

A number of LED-based colorimeters have been described in the literature, but very few of these instruments are specifically devised for educational purposes.[13−21] Some of these instruments incorporate photodiode or phototransistor detectors connected to op-amps for smoothing and amplifying the output current. In some cases optical fiber bundles are employed to guide LED light to the sample cell or the instruments are controlled by software and complex circuitry requiring tedious work to manipulate and display the data.[14−17]

In the study herein we detail our efforts in developing a $40 colorimeter for demonstrating the important spectrophotometric components in analytical chemistry. In addition we provide a series of tried and tested thought-provoking laboratories of varying levels of complexity that address real world problems in chemistry and biochemistry courses; these laboratories have helped students realize the usefulness and limitations of analytical instruments.

EXPERIMENTAL SECTION

Instrument Design

The detailed circuit diagram for the colorimeter is shown in the Supporting Information. The colorimeter is powered by four 1.5 V AA dry batteries, and measures the absorption of visible radiation by a sample in terms of the red (640 nm), green (524 nm), and blue (470 nm) components of light. Light emitted from a low-voltage LED passes through a plastic cuvette containing a colored sample, and the transmitted light is then detected with a tricolor 8 × 8 silicon photodiode array. The instrument features a low-voltage light sensor-to-frequency converter with an easy-to-program CMOS 8-bit microcontroller that generates a signal displayed as percent transmittance on an LCD. The cuvette holder was printed in ABS (acrylonitrile butadiene styrene) plastic, and constructed using a Stratasys 3D printer; the holder could also be printed in nylon. A group of eighth grade technology and engineering students replicated the conventionally manufactured cuvette holder by making careful measurements then drafting a 3D rendering of the holder. The students then converted the drawing to an STL file compatible with the 3D printer.

The custom-made colorimeter is lightweight, rugged, compact, portable, and easy to operate without a need for complex software (Figure 1). Material costs to construct the entire device are less than $40. A complete detailed list of all components and vendors, the 3D printer-friendly STL file for the cuvette holder, and the instructions programmed into the

Figure 1. Custom-built colorimeter.

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241−1248

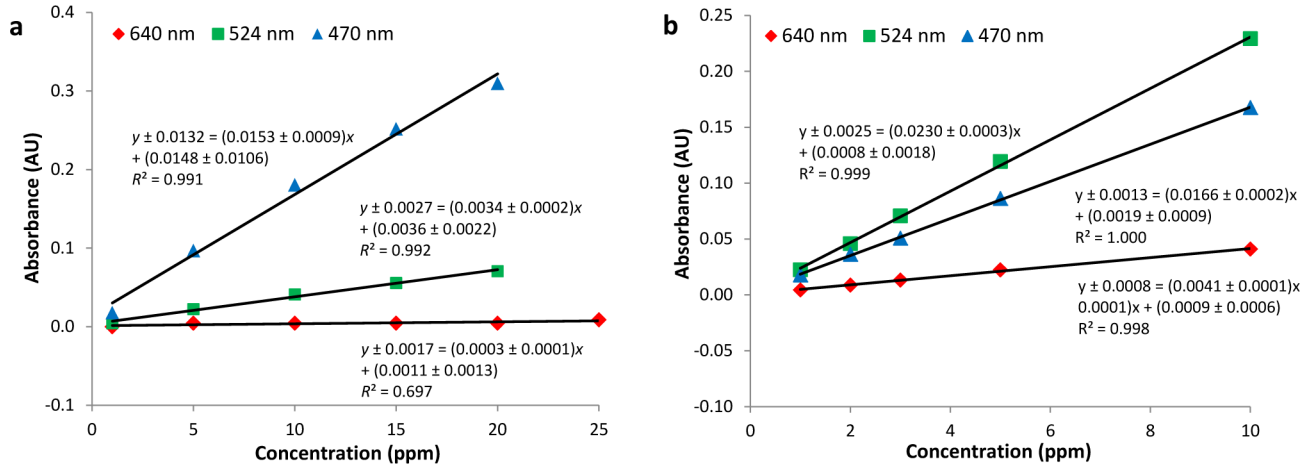

Figure 2. Calibration curves for (a) tartrazine (Yellow 5) and (b) amaranth (Red 2) using the custom-built colorimeter.

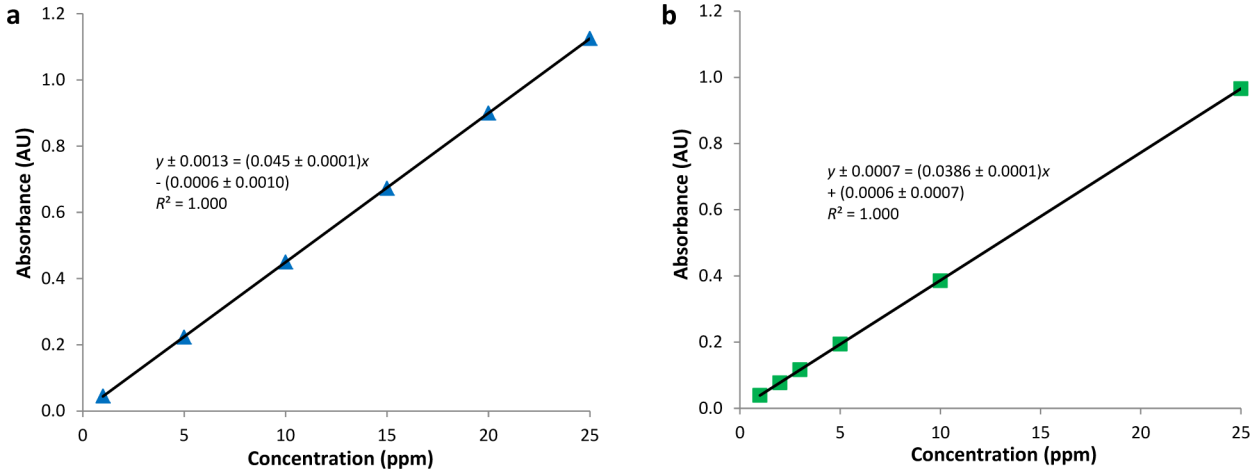

Figure 3. Calibration curves at λ_{max} for (a) tartrazine (427 nm) and (b) amaranth (521 nm) using a Cary-4000 commercial spectrometer.

PIC16F628-20/P chip are provided in the Supporting Information.

General Procedures

All samples were analyzed in triplicate and refrigerated when not in use. The data reported are mean averages. A Cary-4000 UV−vis spectrophotometer (Agilent Technologies) was employed so as to compare data and fully characterize the custom-built instrument. Four experiments of varying complexity were developed for the colorimeter. Each of the experiments can be completed within a 3 h lab period. Detailed experimental protocols are provided as Supporting Information.

RESULTS AND DISCUSSION

The custom-built colorimeter takes about 1 h to construct from kit form, an exercise that school and college students can perform under supervision. Each of the 3 h laboratories was designed to be of varying complexity and sophistication and can be readily altered to meet the aptitude of different student groups.

The instrument is able to quantitatively measure the absorption of visible radiation by a sample in terms of the red (640 nm), green (524 nm), and blue (470 nm) components of light and is designed to allow signals (as percent transmittance) to be read directly from the LCD display, data which can then be transferred to Excel for advanced plotting and analysis.

The custom-built colorimeter provides transmittance values to the nearest 1% with absorbance values reported to two decimal places. The Cary-4000 reports absorbance values to more than four decimal places. For ease of comparison, we report the absorbance data to three decimal places and the calculated concentrations from both instruments to two decimal places. Appropriate standard deviations of regression are included in the figures alongside the R^2 values. Calculated concentrations were derived using a matrix of simultaneous equations, and not from the trendline. All tables and figures are based on student-generated data performed in triplicate. A complete set of tabulated data is provided as Supporting Information. Overall, the colorimeter results were found to be quite comparable in terms of reproducibility and accuracy to the commercial Cary-4000 instrument. Our students have been quite pleased with the instrument and the data it is able to provide; a brief discussion of student survey data and postlab questionnaire is provide in the Supporting Information.

Experiment #1: Determination of a Mixture of Tartrazine and Amaranth

The desired analyte is not always the only compound in the sample that absorbs at a particular wavelength. A time-consuming physical separation of the interfering analyte from solution can often prove to be difficult especially when the interfering compound is chemically similar to the analyte. One

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241−1248

Table 1. Calculated Concentrations (ppm) Using the Experimental Data and the Method of Simultaneous Equations for the Custom-Built Colorimeter and the Cary-4000 Spectrometer

	Unknown Mixture 1[a]			Unknown Mixture 2[a]			Unknown Mixture 3[a]		
Dye	Custom	Cary	Actual	Custom	Cary	Actual	Custom	Cary	Actual
Amaranth	5.10 ± 0.34	5.01 ± 0.07	5.00 ± 0.05	5.51 ± 0.34	5.25 ± 0.08	5.25 ± 0.05	5.65 ± 0.34	5.49 ± 0.07	5.50 ± 0.05
Tartrazine	5.05 ± 0.37	5.01 ± 0.09	5.00 ± 0.05	7.24 ± 0.32	7.25 ± 0.08	7.25 ± 0.06	2.97 ± 0.40	3.00 ± 0.10	3.00 ± 0.03

[a]The uncertainties associated with the actual concentrations are based on the uncertainties associated with the analytical balance (±0.1 mg) and the micropipettors and volumetric glassware (variable). The calculated concentrations (ppm) were obtained using the method of simultaneous equations which incorporated the uncertainties in the ε values and the average and standard deviation of absorbance measurements, in triplicate, of each mixture.

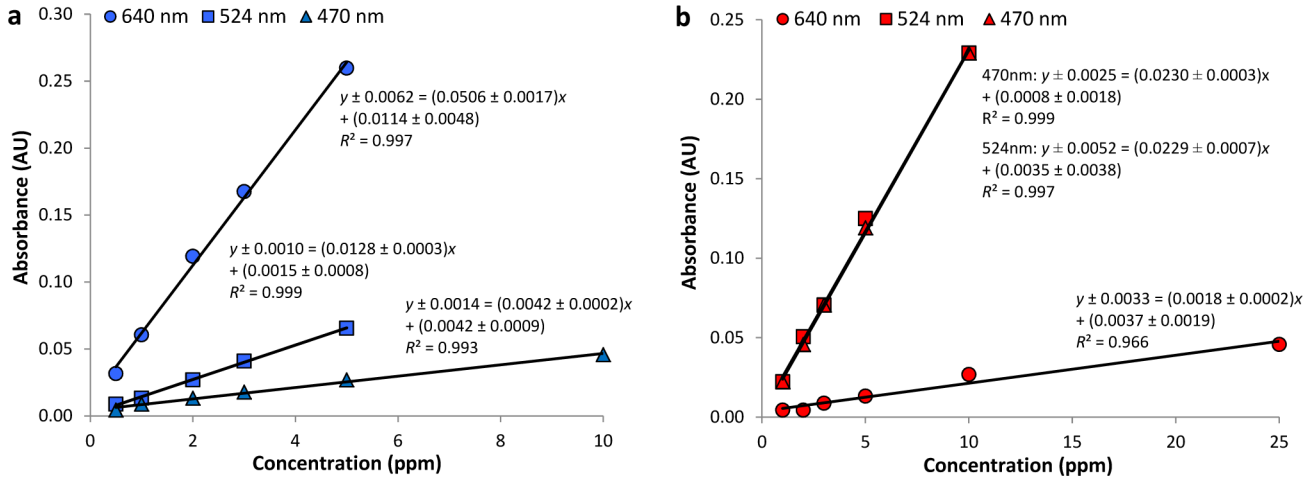

Figure 4. Calibration curves for (a) Blue 1 and (b) Red 40 using the custom-built colorimeter.

technique that can be employed to quantitatively determine the analyte is to select a new wavelength for the analysis, one that is absorbed primarily by the analyte of interest. However, failure to find an appropriate and unique wavelength requires a method that makes use of simultaneous spectrophotometric analysis, where the absorbing solutes absorb independently of each other. Algebraically, the sum of the absorbance of an analyte and an interfering compound is equal to the absorbance of the mixture of the two if the concentration of each is held constant. For a two-component mixture, the following forms of the Beer–Lambert expression can be applied:

$$A_{\lambda 1} = (\varepsilon_A)_{\lambda 1} b C_A + (\varepsilon_B)_{\lambda 1} b C_B \quad (1)$$

$$A_{\lambda 2} = (\varepsilon_A)_{\lambda 2} b C_A + (\varepsilon_B)_{\lambda 2} b C_B \quad (2)$$

where b = path length, C_A and C_B are the analyte concentrations, and (ε_A) and (ε_B) are the molar absorptivity coefficients at wavelengths λ_1 and λ_2. In order to find C_A and C_B, the molar absorptivity coefficients need to be determined by measuring the absorbance at λ_1 and λ_2 for a series of standard solutions of each individual component.

The custom-built colorimeter was used to quantitatively determine the concentration of a mixture of tartrazine (FD&C Yellow 5) and amaranth (FD&C Red 2) anionic organic azo dyes using a method involving simultaneous Beer's law equations. Tartrazine displays two intense absorption bands at 257 and 428 nm, and amaranth displays three intense absorption bands at 219, 322, and 521 nm. A series of standard solutions of tartrazine and amaranth were prepared, and the custom-built colorimeter generated absorbance data for these dyes at the different concentrations to produce the respective calibration curves (Figure 2). These calibration curves are consistent with Beer's law, illustrating that the absorbance is directly proportional to the concentration of the analyte. Data from the commercial Cary-4000 instrument are included in Figure 3.

The detector for the custom-built colorimeter is composed of an 8 × 8 array of photodiodes. Sixteen of the photodiodes have blue filters, 16 photodiodes have green filters, 16 photodiodes have red filters, and 16 photodiodes are clear with no filters. The peak maximum and spectral half-width are fixed for each color region (i.e., each 4 × 4 array). For blue, green, and red, these values are λ_p = 470 nm and $\Delta\lambda 1/2$ = 35 nm, λ_p = 524 nm and $\Delta\lambda 1/2$ = 47 nm, and λ_p = 640 nm and $\Delta\lambda 1/2$ = 17 nm, respectively. As a consequence, absorption measurements do not necessarily correlate with λ_{max} of the analyte; the absorption measurements represent an "effective" or weighted molar absorptivity coefficient (ε_{eff}) centered about λ_p. In contrast, the molar absorptivities calculated from the calibration curves obtained from the Cary-4000 instrument represent ε_{max} for amaranth and tartrazine at their respective λ_{max} values (±1 nm). Data generated using the blue and green wavelengths were then entered into a set of simultaneous Beer's law equations to calculate the concentration of three unknown amaranth–tartrazine samples. Table 1 compares the calculated values for the unknown samples using the custom-built colorimeter to the values obtained from the commercial instrument. For the Cary-4000, the close correspondence between the calculated and the actual concentrations and the similar relative uncertainties suggests that the major source of error is associated with sample preparation. In contrast, the relative uncertainties in the concentrations calculated from the colorimeter data are larger and incorporate both the small differences in concentrations between replicate solutions and the errors associated with using λ_p and ε_{eff}. The overall correlation between the two instruments

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241–1248

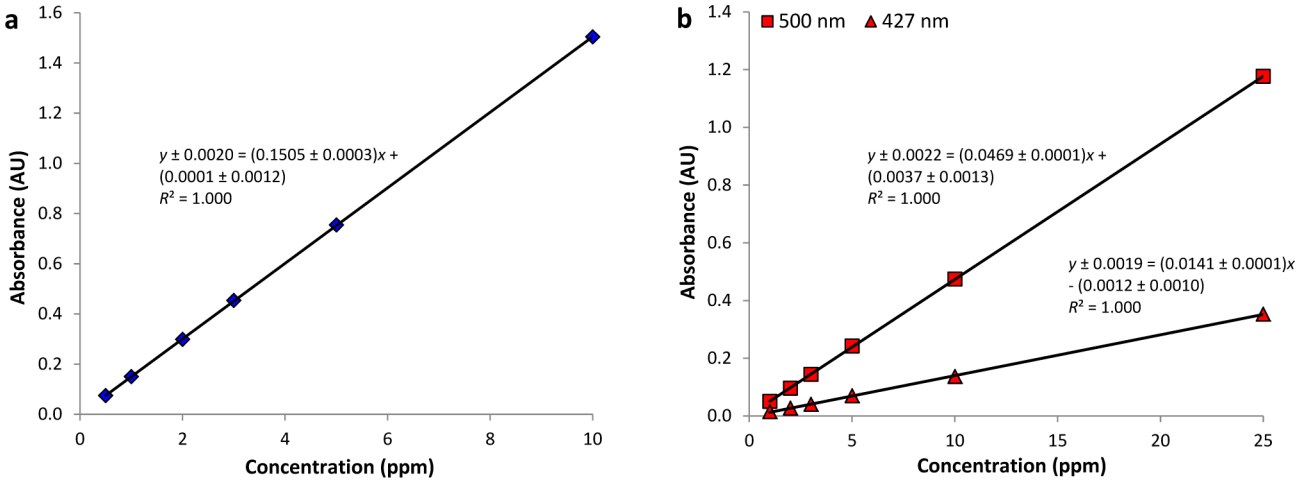

Figure 5. Calibration curves at λ_{max} for (a) Blue 1 (629 nm) and (b) Red 40 (427 and 500 nm) using a Cary-4000 spectrometer.

Table 2. Concentration Data (ppm) for Select Binary and Ternary Solutions Analyzed Using the Custom-Built Colorimeter[a]

Standard Mixtures (ppm)	Actual Concentration[b]				Calculated Concentration[b] (at Wavelength Used)		
	Blue 1	Red 40	Yellow 5		Blue 1 (640 nm)	Red 40 (524 nm)	Yellow 5 (470 nm)
Series 1	1.00 ± 0.02	4.00 ± 0.03			1.06 ± 0.20	4.61 ± 0.34	
	2.00 ± 0.03	3.00 ± 0.03			2.31 ± 0.19	3.18 ± 0.38	
	2.50 ± 0.03	2.50 ± 0.03			2.80 ± 0.19	2.80 ± 0.39	
	3.00 ± 0.03	2.00 ± 0.03			3.33 ± 0.19	2.14 ± 0.37	
	4.00 ± 0.03	1.00 ± 0.02			4.19 ± 0.21	0.96 ± 0.32	
	5.00 ± 0.05	5.00 ± 0.05			5.16 ± 0.22	5.58 ± 0.32	
				Cary data	5.01 ± 0.03 (629 nm)	4.95 ± 0.03 (500 nm)	
Series 4	3.00 ± 0.03	7.50 ± 0.06	8.00 ± 0.06		3.07 ± 0.19	8.58 ± 0.32	9.36 ± 0.32
				Cary data	2.78 ± 0.03 (629 nm)	7.81 ± 0.02 (500 nm)	7.98 ± 0.02 (427 nm)
	5.00 ± 0.05	5.00 ± 0.05	7.50 ± 0.06		4.93 ± 0.21	5.88 ± 0.34	9.21 ± 0.32
				Cary data	4.86 ± 0.03 (629 nm)	5.13 ± 0.03 (500 nm)	7.62 ± 0.02 (427 nm)

[a]Some solutions were also analyzed using the Cary-4000 spectrometer. [b]The uncertainties associated with the actual concentrations are based on the uncertainties associated with the analytical balance (±0.1 mg) and the micropipettors and volumetric glassware (variable). The calculated concentrations (ppm) were obtained using the method of simultaneous equations which incorporated the uncertainties in the ε values and the average and standard deviation of absorbance measurements (in triplicate). The uncertainties are reported at the single-standard deviation.

is very good, showing that the custom-built instrument provides high-quality data comparable to that obtained from the more expensive and more sophisticated commercial instrument.

Experiment #2: Determining the Concentration of Food Dyes in a Mixture

All multicomponent, quantitative dye models are based on the principle that the absorbance of a mixture is equal to the sum of the absorbance of its components. For a two-component mixture, the Beer–Lambert expression in eq 1 and eq 2 can be applied. For ternary mixtures, the total absorbance at any wavelength is equal to the absorbance of the mixture of the three components if the concentration of each is held constant. For a three-component mixture, Beer's law gives the following three equations:

$$A_{\lambda 1} = (\varepsilon_A)_{\lambda 1} b C_A + (\varepsilon_B)_{\lambda 1} b C_B + (\varepsilon_C)_{\lambda 1} b C_C \quad (3)$$

$$A_{\lambda 2} = (\varepsilon_A)_{\lambda 2} b C_A + (\varepsilon_B)_{\lambda 2} b C_B + (\varepsilon_C)_{\lambda 2} b C_C \quad (4)$$

$$A_{\lambda 3} = (\varepsilon_A)_{\lambda 3} b C_A + (\varepsilon_B)_{\lambda 3} b C_B + (\varepsilon_C)_{\lambda 3} b C_C \quad (5)$$

where b = path length, C_A, C_B, and C_C are the analyte concentrations, and (ε_A), (ε_B), and (ε_C) are the molar absorptivities at wavelengths λ_1, λ_2, and λ_3. In order to find C_A, C_B, and C_C, the molar absorptivity coefficients need to be determined by measuring the absorbance at λ_1, λ_2, and λ_3 for a series of standard solutions of each individual component.

The custom-built colorimeter was used to generate calibration curves for three U.S. FDA permitted artificial colorants, namely, Brilliant Blue FCF (FD&C Blue 1), Allura Red AC (FD&C Red 40), and Tartrazine (FD&C Yellow 5). The λ_{max} values are 629, 500, and 427 nm for Blue 1, Red 40, and Yellow 5, respectively. This lab also allowed students to investigate the usefulness and limitations of the "simultaneous equation" method: The best calibration in terms of linearity is often obtained at the wavelength of maximum absorbance. However, the very high correlation coefficients seen in Figure 4 indicate that there is also a strong correlation between absorbance and concentration at the fixed colorimeter wavelengths of 640, 524, and 470 nm. Data from the commercial Cary-4000 spectrometer are shown in Figure 5 at the selected λ_{max} values for comparison. The calibration curves for tartrazine (Yellow 5) at the three fixed wavelengths for the custom-built colorimeter and the Cary-4000 (at 427 nm) were identical to the data displayed in Figure 2a and Figure 3a, respectively.

The molar absorptivity values obtained from the calibration curves were used to calculate the concentrations of various known mixtures via the method of simultaneous equations. Some selected data for various binary and ternary solutions is presented

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241−1248

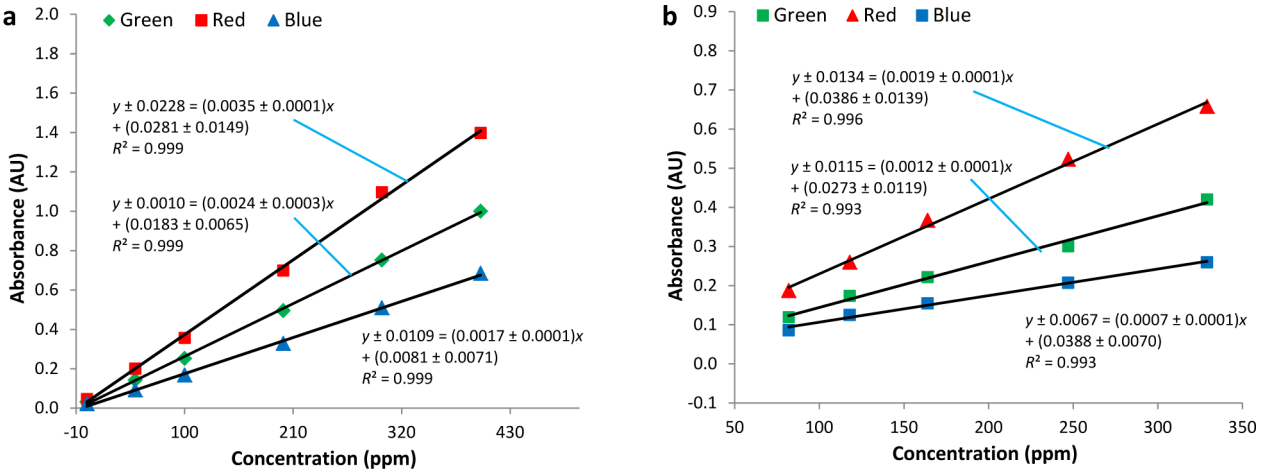

Figure 6. Calibration curves for phosphate using (a) the Murphy−Riley method and (b) the modified Murphy−Riley method, and the custom-built colorimeter.

in Table 2. The complete set of data is provided as Supporting Information.

Discrepancies between the custom-built instrument, the commercial instrument, and the actual values are in part due to the fact that the colorimeter is limited by its three fixed wavelengths, whereas the calculations from the commercial instrument make use of the λ_{max} and ε_{max} values for each dye. In addition the noticeable absorbance overlap around 400 nm between the visible spectra of Red 40 and Yellow 5 leads to poor data. The measurement of Red 40 is particularly problematic because the colorimeter has fixed wavelengths at 470 and 524 nm, which coincidentally occur on either side of the true λ_{max} value at 500 nm. The resulting similar absorptivity values lead to faulty solutions to the Beer's law simultaneous equations. Thus, there are some limitations as to what combination of colored solutions the instrument can measure accurately without some advanced data calibration.

Experiment #3: Determining the Concentration of Inorganic Phosphate

The Murphy−Riley method[22,23] is widely used to determine dissolved inorganic phosphate (Pi, orthophosphate, PO_4^{3-}). Such a method is often used in analyzing environmental samples including soil, manure, and stormwater and agricultural runoff. Phosphates are colorless or pale in solution, so the phosphorus in the test solution is quantitatively converted to a colored compound: Pi reacts with molybdate to form a Mo(VI) complex, which is then reduced to a blue colored Mo(V) with the addition of ascorbic acid at pH = 1. Antimony potassium tartrate is added to increase the rate of this reduction.

Interferences to the analysis include arsenate (AsO_4^{3-}) at concentrations above 100 ppb (μg/L), where the product displays a color similar to that of phosphate and leads to a positive interference. Barium, lead, and silver interfere by forming a phosphate precipitate, but the effect is negligible in natural water. These interferences often require some slight modifications to the standard method. Silica occurs in a variety of forms such as sand, quartz, sandstone, and granite and is widely distributed in asbestos, mica, talc, and lava. All natural water supplies contain some dissolved silicates and suspended or colloidal silica. The interference from silica is the pale-blue color that develops when silica is present in large amounts. Because many of the samples being analyzed include silica as a component, a slightly *modified* version of the Murphy−Riley method was employed that makes use of hydrazine sulfate[24,25] as the reductant to generate a bluish color.

A series of standard monobasic potassium phosphate (KH_2PO_4) solutions were prepared ranging from 1 to 400 ppm. Some students prepared the standard Murphy−Riley reagent with ascorbic acid, and others made use of the modified reagent with hydrazine sulfate. Addition of the test reagent to the phosphate solutions generated a blue color. The standard phosphate samples were then analyzed using the custom-built colorimeter at wavelengths of 470 nm (blue), 524 nm (green), and 640 nm (red) to create calibration plots, as shown in Figure 6a for the Murphy−Riley method and Figure 6b for the modified method. The high quality of data from the custom-built colorimeter is quite apparent given the excellent correlation coefficient values.

Experiment #4: Determining the Concentration of Salicylate via Complexation with Fe(III)

Salicylate and salicylic acid do not absorb visible light, thus creating an analytical challenge. However, reaction with iron(III) results in an intense purple colored solution (λ_{max} = 535 nm), which can be readily detected with a colorimeter[26,27] and so allowing quantification of salicylate in unknown samples utilizing the Beer's law relationships. Under the acidic conditions in this reaction the salicylate is fully protonated and the ferric ion occurs as the hexahydrate species prior to complexation:

$$x\mathrm{Sal}_{(aq)} + y\mathrm{Fe}(\mathrm{H_2O})_6^{3+}{}_{(aq)} \rightleftharpoons (\mathrm{Fe})_y(\mathrm{Sal})_{x(aq)} \qquad (6)$$

Equation 6 shows the complexation of salicylic acid (Sal) with a ferric salt and contains the coefficients as subscripts x and y.

A series of standard solutions of ferric−salicylate were prepared, and each solution was passed through a syringe filter prior to analysis. The custom-built colorimeter was used to obtain absorbance data for these solutions and generate a calibration curve (Figure 7). The calibration curve was then used to determine the concentration of the salicylate analyte in a sample of commercially available acne face wash [Clean & Clear Advantage Acne Spot Treatment]. A mean average of 2.06% w/w was calculated with a standard deviation of 0.20 (n = 32, Fall 2014 semester); this value is essentially identical to the 2.0% w/w value quoted on the commercial face wash sample.

The second and more challenging part of this experiment dealt with the stoichiometry of the reaction using the method of

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241−1248

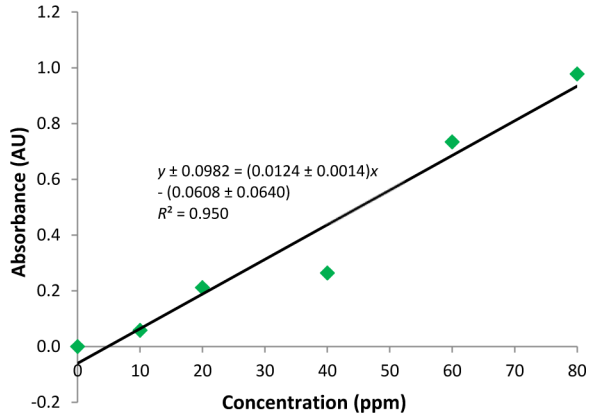

Figure 7. Calibration curves for Fe−salicylate using the custom-built colorimeter at 524 nm. The anomalous data point at 40 ppm is real and a consequence of error introduced by the students during standard preparation; deleting this point gives an R^2 value of 0.999.

continuous variation (Job's method) to determine these quantities for the predominant complex in solution. The absorbance of a series of solutions containing different quantities of salicylate and Fe^{3+} were prepared, where the amount of each reactant was varied, and the total moles of both reagents were kept constant. The absorbance of each solution was measured using the custom-built colorimeter, and a plot of absorbance versus mole ratio indicated a maximum at 0.50 (Figure 8)

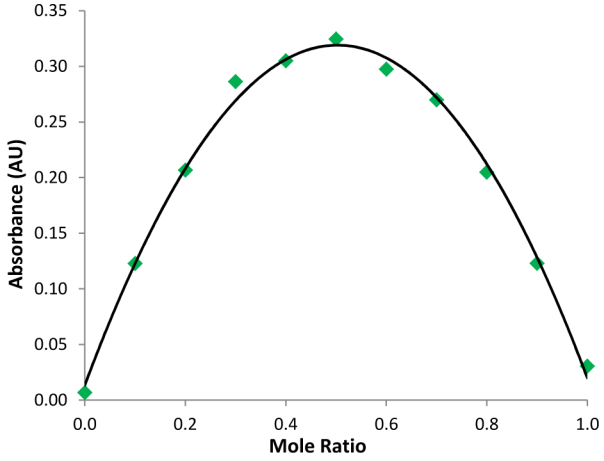

Figure 8. Method of continuous variation plot for the iron−salicylate complex using the custom-built colorimeter at 524 nm.

corresponding to parity in x and y in eq 6, and a stoichiometry of 1:1 for the predominant complex in solution. The quality of data from the low-cost, battery-powered custom-built colorimeter is clearly remarkable.

CONCLUSIONS

We have successfully designed and constructed a lightweight, low-power, portable colorimeter that is capable of generating high-quality absorbance data. The simple circuitry makes the instrument easy to construct and offers an exciting, economical, and practical alternative for the demonstration of colorimetry in the classroom. The colorimeter has been successfully assembled and used by middle school, high school, and college undergraduate students, to teach the fundamentals of spectroscopy and its use as an extremely powerful analytical tool. Feedback from students regarding the colorimeter and the data it can generate has been extremely positive.

The food dyes, phosphate analysis, and salicylate analysis laboratories are low cost and highly rewarding lab experiments for students. Numerous aspects of each of these experiments can be readily customized according to student level, aptitude, and ability. Beer's law calculations showed that the custom-built colorimeter can measure absorbance values for various food dyes within normal ranges of those measured by commercially available instruments. The colorimeter was successfully used to calculate the concentration of monobasic potassium phosphate, as well as salicylic acid found in acne face wash. In addition, the student constructed colorimeter correctly determined the mole ratio of the reaction between salicylic acid and a ferric salt.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00729.

Circuit diagram, parts list, Stratsys figures, CAS numbers and notes on safety and disposal, tabulated experimental data, and questionnaire/survey results (PDF)
Experiment #1 instructions (PDF)
Experiment #2 instructions (PDF)
Experiment #3 instructions (PDF)
Experiment #4 instructions (PDF)
STL files for 3D printing (ZIP)
Instructions programmed into the PIC16F628-20/P chip (ZIP)

AUTHOR INFORMATION

Corresponding Author

*E-mail: dgs12@psu.edu.

Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

We would like to thank the Penn State Schreyer Institute for Teaching Excellence, and the Summer Experience in the Eberly College of Science program (SSECoS) for financial support. The SEECoS program is supported by the Upward Bound Math and Science Center (UBMS) at Penn State and a USDoE TRIO grant. The authors are thankful to the Electronics Research Instrumentation Facility at Penn State, and the effort of our many undergraduate, high school, and middle school students over the past several years who have patiently developed, tested, and piloted this instrument, and provided valuable feedback along the journey.

REFERENCES

(1) Anderson, L. W.; Krathwohl, D. R.; Bloom, B. S. *A taxonomy for learning, teaching, and assessing: A revision of Bloom's Taxonomy of Educational Objectives*; Longman: New York, 2001.
(2) Nendaz, M. R.; Tekian, A. Assessment in problem-based learning medical schools: A literature review. *Teaching and Learning in Medicine* **1999**, *11*, 232−243.
(3) Savin-Baden, M. *Problem-based learning in higher education; Untold Stories*; SRHE and Open University Press: Buckingham, 2000.
(4) Albanese, M. A.; Mitchell, S. Problem-based Learning: A review of literature on its outcomes and implementation issues. *Academic Medicine* **1993**, *68*, 52−81.

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241−1248

(5) Wilson, G. S.; Anderson, M.; Lunte, C. E. Instrumental Analysis at the University of Kansas: An Experiment in Problem-based Learning. *Anal. Chem.* **1999**, *71*, 677A−681A.
(6) Dominguez, V. C.; McDonald, C. R.; Johnson, M.; Schunk, D.; Kreuter, R.; Sykes, D.; Wigton, B. T.; Chohan, B. S. The Characterization of a Custom-Built Coulometric Karl Fischer Titration Apparatus. *J. Chem. Educ.* **2010**, *87*, 987−991.
(7) Wigton, B. T.; Chohan, B. S.; McDonald, C.; Johnson, M.; Schunk, D.; Kreuter, R.; Sykes, D. A Portable, Low-Cost, LED Fluorimeter for Middle School, High School, and Undergraduate Chemistry Labs. *J. Chem. Educ.* **2011**, *88*, 1182−1187.
(8) Wigton, B.; Kreuter, R.; Sykes, D.; Chohan, B. S. The Characterization of an Easy-to-Operate Inexpensive Student-Built Fluorimeter. *J. Chem. Educ.* **2011**, *88*, 1188−1193.
(9) Mott, J. R.; Munson, P. J.; Kreuter, R. A.; Chohan, B. S.; Sykes, D. Development and Characterization of an Inexpensive Portable Cyclic Voltammeter. *J. Chem. Educ.* **2014**, *91*, 1028−1036.
(10) Clippard, C. M.; Nichisti, J. C.; Kreuter, R.; Sykes, D.; Chohan, B. S. The Use of a Custom-Built Coulometric Karl Fischer Instrument for the Determination of Water Content in Chocolate. *Food Analytical Methods* **2015**, *8*, 929−936.
(11) Washton, N. M.; Earnheart, K. C.; Sykes, D. G.; Ucak-Astarlioglu, M.; Mueller, K. T. *Toward the Integration of Liquid- and Solid-State NMR across the Undergraduate Curriculum. Modern NMR Spectroscopy in Education*; ACS Symposium Series; American Chemical Society: Washington, DC, 2009; Vol. *969*, pp 20−35.
(12) Hughes, W. Assembly and Validation of a Colorimeter. *Technol. Eng. Teach.* **2013**, *72* (6), 32−36.
(13) O'Toole, M.; Diamond, D. Absorbance Based Light Emitting Diode Optical Sensors and Sensing Devices. *Sensors* **2008**, *8*, 2453−2479.
(14) Fonseca, A.; Raimundo, I. M. A Multichannel Photometer Based on an Array of Light Emitting Diodes for Use in Multivariate Calibration. *Anal. Chim. Acta* **2004**, *522*, 223−229.
(15) Gros, N. Spectrometer with Microreaction Chamber and Tricolour Light Emitting Diode as a Light Source. *Talanta* **2004**, *62*, 143−150.
(16) Shimazaki, Y.; Watanabe, S.; Takahashi, M.; Iwatsuki, M. A Portable Spectrophotometer Using a White-color Light-emitting Diode and a Charge-coupled Device and Its Application to On-site Determination of Iron. *Anal. Sci.* **2000**, *16*, 1091−1093.
(17) Matsuo, T.; Muromatsu, A.; Katayama, K.; Mori, M. Construction of a Photoelectric Colorimeter and Application to Students' Experiments (Part 2). *J. Chem. Educ.* **1989**, *66*, 848.
(18) Jaffar, M.; Zahid, Q. A Low-Cost Precision Colorimeter. *J. Chem. Educ.* **1988**, *65*, 1099.
(19) Isaacs, N. S. Two Readily-Constructed Instruments for the Teaching Laboratory. *J. Chem. Educ.* **1983**, *60*, 607.
(20) Gordon, J.; James, A.; Harman, S.; Weiss, K. A Film Canister Colorimeter. *J. Chem. Educ.* **2002**, *79*, 1005−1006.
(21) Gordon, J.; Harman, S. A Graduated Cylinder Colorimeter: An Investigation of Path Length and the Beer-Lambert Law. *J. Chem. Educ.* **2002**, *79*, 611−612.
(22) Murphy, J.; Riley, J. P. A modified single solution method for the determination of phosphate in natural waters. *Anal. Chim. Acta* **1962**, *27*, 31−36.
(23) Gales, M. E.; Julian, E. C.; Kroner, R. C. Method for quantitative determination of total phosphorus in water. *Am. Water Works Assoc., J.* **1966**, *58*, 1363−1368.
(24) Ganesh, S.; Khan, F.; Ahmed, M. K.; Velavendan, P.; Pandey, N. K.; Mudali, U. K. Spectrophotometric determination of trace amounts of phosphate in water and soil. *Water Sci. Technol.* **2012**, *66*, 2653−2658.
(25) Ohashi, K.; Enomoto, T.; Yamamoto, K. Dual-wavelength spectrophotometric determination of trace amounts of phosphate in the presence of large amounts of silicate using molybdenum blue. *Bull. Chem. Soc. Jpn.* **1981**, *54*, 1889−1890.
(26) Annino, J. S. *Clinical Chemistry, Principles and Procedures*, 4th ed.; Little Brown & Co.: Boston, 1976.
(27) Reid, K. R.; Meyeroff, M. E.; Mitchell-Koch, J. T. Salicylate Detection by Complexation with Iron (III) and Optical Absorbance Spectroscopy. *J. Chem. Educ.* **2008**, *85*, 1658−1659.

DOI: 10.1021/acs.jchemed.5b00729
J. Chem. Educ. 2016, 93, 1241−1248