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Laboratory Experiment

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Build Your Own Photometer: A Guided-Inquiry Experiment To Introduce Analytical Instrumentation

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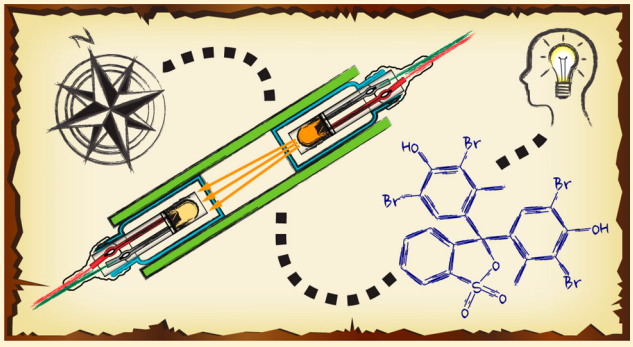
Supporting Information

ABSTRACT: A guided-inquiry project designed to teach students the basics of spectrophotometric instrumentation at the second year level is presented. Students design, build, program, and test their own single-wavelength, submersible photometer using low-cost light-emitting diodes (LEDs) and inexpensive household items. A series of structured prelaboratory assignments guide students through the processes of researching background information, designing a photometer device, and developing their own procedure to test the performance of the device. Students also learn basic skills of data acquisition by programming an easy-to-use LabVIEW interface for their device. Using a colorimetric indicator dye, students use their photometers and LabVIEW interfaces to determine the endpoint of an acid−base titration and compare the linear response of their device against that of a commercially available spectrophotometer. Students who completed the experiment indicated that the experience improved their understanding of spectroscopy, as well as their critical thinking skills and research ability.



KEYWORDS: *Second-Year Undergraduate, Upper-Division Undergraduate, Analytical Chemistry, Laboratory Instruction, Hands-On Learning/Manipulatives, Inquiry-Based/Discovery Learning, Laboratory Computing/Interfacing, Instrumental Methods, UV-Vis Spectroscopy*

INTRODUCTION

Modern analytical chemistry is a dynamic discipline with a strong emphasis on the utilization and development of scientific instruments and technology. Unfortunately, few introductory analytical chemistry curricula share this level of emphasis. One of the challenges of teaching introductory analytical chemistry is incorporating modern methods and experimental skills into the introductory laboratory curriculum while maintaining a level of challenge suitable for second-year undergraduate students. To this end, we have developed a guided-inquiry laboratory experiment that provides students with a hands-on introduction to analytical instrumentation and method design in the context of UV−visible photometry.

There are many examples of low-cost spectrophotometers, photometers, and fluorimeters that can be built for use in undergraduate laboratories.[1−5] In addition, several educators have developed upper-year undergraduate laboratory experiments where students themselves build simple analytical instrumentation. For example, light-emitting diodes have been combined with simple detectors and sample containers to create colorimeters;[6,7] LEDs and transmission gratings have been used with LEGO[8,9] or cell phone cameras[10] to build spectrophotometers; and LEDs, optical filters, photodiodes, optomechanical components, and either LabVIEW or microcontrollers have been used to build fluorimeters[11] and photometers.[12] These experiments quickly dispel the surprisingly common but erroneous notion that instruments are "black boxes" that imperceptibly convert a sample into an infallible result.[13] Moreover, students gain skills in assembling, operating, and troubleshooting instrumentation that are important for their employability and research ability.

The "build-your-own-instrument" experiments cited above typically use a traditional structured inquiry format, in which the experimental problem, theory, background, procedures, and design are provided to students.[14] While this format can help prevent cognitive overload for students who are learning complex new skills, they do little to develop students' skills of scientific inquiry, such as performing background research, developing and refining a procedure or design, and testing hypotheses. To bring the benefits of "build-your-own-instrument" experiments into our second-year analytical chemistry laboratory while also helping students to develop skills of scientific inquiry, we created a guided-inquiry experiment in which students, research, design, build, and test a submersible photometer probe that can monitor the progress of an acid−base titration in real-time. Students construct the photometer using common household items and inexpensive LEDs, then interface their device to a computer using a simple LabVIEW program and low-cost data acquisition module. A series of carefully designed prelab assignments guide students through

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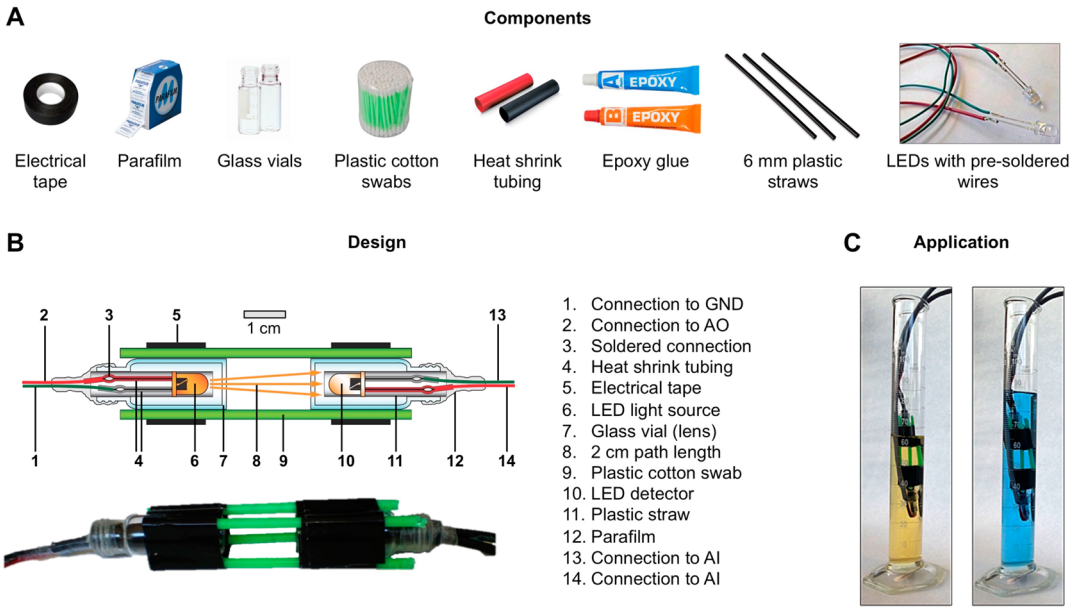

Figure 1. (A) Components available to students for construction of a submersible photometer. (B) Schematic of the basic photometer design (drawn approximately to scale) and a photograph of an assembled photometer device. Students build devices similar to this design. (C) Photograph of the photometer submerged in aqueous solutions of bromocresol green at acidic pH (left) and basic pH (right) during an acid–base titration. The DAQ is not shown.

Table 1. Weekly Prelab Assignment Tasks

Week	Tasks[a]	Grading	Cognitive level[14]
1	Differentiate between photometers and spectrophotometers	Immediate	Understand
	List the essential components of a photometer	Immediate	Understand
	Provide an example of a monochromatic light source	Immediate	Understand
2	Background reading on absorption and emission of light by molecules and semiconductors	None	-
	Draw wiring diagrams for the emission and detection modes of an LED	After feedback	Apply
3	Derive an equation for calculating absorbance values from voltages measured by the detector LED	Immediate	Analyze
	Rank indicator dye candidates based on their absorption spectra and K_a values	Immediate	Evaluate
4	Design a photometer instrument from a list of simple parts*	After feedback	Create
	Create a detailed procedure for acid/base titration*	After feedback	Create

[a]Students worked in pairs for tasks marked with an asterisk.

the process of research in a step-by-step fashion, making the lab suitable for an introductory course in analytical chemistry. Student feedback indicates that the experiment engages students in the process of research, challenges them to think critically and creatively about instrumental design, and improves their understanding of important course concepts.

PHOTOMETER DESIGN AND CONSTRUCTION

The photometer is designed around two low-cost LEDs and a USB data acquisition module (DAQ; National Instruments, Austin, TX). One LED is connected to an analog output channel on the DAQ and used as a light source; the other LED is connected to an analog input channel on the DAQ and used as a photodetector. A simple LabVIEW program is used to control the output voltage that powers the source LED and to measure the input voltage from the detector LED. Using the common household and laboratory materials shown in Figure 1A, students construct a water-tight body around the LEDs with a design similar to that illustrated in Figure 1B. Pictures of the photometer submerged in acidic and basic solutions of bromocresol green to make measurements during an acid–base titration are shown in Figure 1C. A detailed list of materials and construction tips are provided in Appendix I of the Supporting Information. The estimated cost per photometer, not including the DAQ, is less than $5.00. Moreover, most of the components are reusable, such that costs over multiple terms should be much lower.

STUDENT ACTIVITIES

The laboratory activity includes four weekly prelab homework assignments, which students complete in addition to their regular lab schedule, two 3-h lab periods, and a final report. Detailed experimental procedures are available in the Supporting Information. The Results and Discussion section addresses pedagogical aspects of these activities.

Prelab Assignments

Prelab assignments, summarized in Table 1, guide students through the initial steps of inquiry: background research, instrument design, and development of an experimental procedure.[14] Most students report spending between 20–60 min per assignment. The full activities are provided in Appendix 2 of the Supporting Information. All of the prelab tasks are completed before the first laboratory session.

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Laboratory Session 1

Students work in pairs to build a photometer based on a corrected version of the design that was developed as part of their fourth prelab assignment. Most students spend 10−20 min building their photometer. Students also set up a LabVIEW program to control the output voltage (2−4 V) to the source LED, and measure the input voltage from the detector LED. The programming stage requires ≤2 h. Prior to leaving the laboratory, students perform a practice titration of HCl (aq) with NaOH (aq) using bromocresol green (BCG) as an indicator dye. The raw detector voltage is monitored while NaOH (aq) is added to ensure that the device and LabVIEW program are working as expected. Step-by-step instructions for assembly of the photometer design in Figure 1 are provided in Appendix 3 of the Supporting Information, and diagrams for the LabVIEW program are provided in Appendix 1.

Laboratory Session 2

Student pairs perform a colorimetric acid−base titration. The photometer is connected to the DAQ, and dark and blank voltages recorded. The device is then submerged in 30.0 mL of 0.50 M HCl (aq) with ca. 30 μM bromocresol green. This solution is titrated with 1.0 M NaOH (aq) and the voltage from the detector LED is recorded after each addition of base. Students perform a second titration using their choice of indicator dye (e.g., cresol red, phenolphthalein, bromophenol blue, or methyl violet). Finally, students prepare five dilutions of bromocresol green (3−30 μM) in 2 mM NaOH. For each solution, students record measurements with their photometer and with a commercial spectrophotometer (Genesys 20, Thermo Scientific, Waltham, MA). Each titration takes 30−50 min, and the calibration curve measurements take 40−60 min. Since students work in pairs, one student is in charge of performing the titration while the other student prepares the solutions for the calibration curve.

Postlab

Following the laboratory sessions, students prepare a short report that consists of calculations (concentrations of standards, absorbance values, molar absorptivity of the indicator dye), calibration curves, and answers to a set of discussion questions. The complete instructions provided to students are available in Appendix 2 of the Supporting Information. Teaching assistants assign four or five discussion questions from a larger set. The discussion questions generally require students to justify steps in their experimental procedure or rationalize differences in experimental results obtained under different conditions. Students submit their reports 1 week after completing the experiments.

■ HAZARDS

Acids and bases are corrosive. A lab coat, chemical resistant gloves, and safety goggles should be worn when handling these reagents. The DAQ and computer should be protected against spills. The analog output from the DAQ does not pose any special electrical hazard. Standard undergraduate laboratory precautions for electrical equipment should be observed.

■ RESULTS AND DISCUSSION

Prelab Assignments To Scaffold Higher Levels of Inquiry

In many traditional analytical chemistry experiments, students follow a set procedure to determine the concentration of an analyte in an unknown sample. While this format can be beneficial for teaching students specific laboratory skills and techniques (e.g., proper use of volumetric glassware),[14] tasks of this nature can be completed with only a superficial understanding of the underlying concepts and little or no critical thought as to the suitability of the method used for analysis.

In contrast, our primary goals for the photometer experiment were for students to engage with the important concepts of photometry and to practice scientific inquiry. Provided with a problem (i.e., build a photometer) and some basic resources, students were required to perform background research and develop their own procedure and instrument design. A series of structured prelab assignments gradually increased the level of cognitive demand to guide students through this process. Table 1 lists the tasks, grading, and cognitive level (Bloom's level, based on a revised version of Bloom's taxonomy[15]) for each prelab assignment. The Bloom's level of the prelab tasks gradually increased from understanding basic concepts (level 2) to creating an experimental procedure and instrument design (level 6, the highest on the taxonomy). The specific tasks in each prelab assignment structured the inquiry process so that students could successfully complete and learn from the activities as their cognitive demand increased.[16]

The more challenging prelab tasks provided regular opportunities for constructive feedback prior to grading. For example, the second prelab required students to predict how an LED could be wired to a battery or voltmeter in order to emit or detect light (the information required to answer this question was not included in the background reading). Instead of grading the predictions immediately, students had the opportunity to test their predictions using a battery pack, a voltmeter, and two LEDs and revise their answer based on their observations. Students received full marks if their final answer was correct and a bonus mark if their initial prediction was correct.

Similarly, in the fourth prelab, students' detailed procedures for the acid−base titration and calibration curve of BCG were not graded. The laboratory director met with each pair of students to provide formative feedback on their procedures. On the basis of the feedback they received, students were expected to revise their procedure as needed. Teaching assistants graded the revised version before students performed the experiment. Students' designs for the submersible photometer were graded only for logical design of the instrument. For example, the light emitter, sample, and the detector must be placed in a logical order; the path length must be held constant; etc. All groups received feedback on their designs before construction. The most common design errors were technical in nature rather than conceptual. These errors were quickly resolved with the opportunity for hands-on experiments, and students gained a new appreciation for the technical details of instrument design.

Device Performance

As noted above, students complete two activities to test their photometer: a strong acid−strong base titration with BCG and another indicator dye; and a comparison of calibration curves between their photometer and a commercial spectrophotometer. Figures 2 and 3 show representative results from these activities. The titration experiment (Figure 2A) is characterized by a sharp transition from a high detector signal (low absorbance) to a lower detector signal (higher absorbance) near the equivalence point of the titration, when the conjugate acid of the indicator is deprotonated to yield its conjugate base

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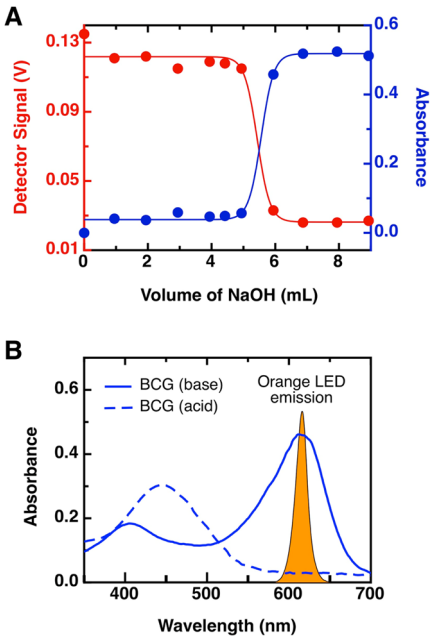

Figure 2. (A) Representative curves for the titration of HCl (aq) with NaOH (aq) using BCG as an indicator dye. (B) Absorption spectra for the conjugate acid and conjugate base forms of BCG, overlaid with the emission profile of the orange LED light source.

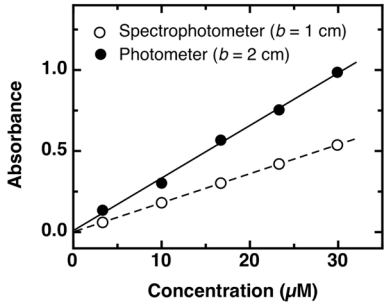

Figure 3. Representative calibration curves for BCG in basic solution for the submersible photometer in Figure 1 and a commercial cuvette-based spectrophotometer. The path length, *b*, for each device is noted.

form. The conjugate base of BCG absorbs the light from the orange LED whereas the conjugate acid does not (Figure 2B). In the calibration curve experiment (Figure 3), both the photometer and a commercial spectrophotometer produce linear calibration curves for increasing concentrations of BCG in basic solution. The slopes of the calibration curves differ in proportion to the difference in path length (a factor of ca. 2 since the commercial spectrophotometer uses standard cuvettes with a 1 cm path length and student-made photometers have a 2 cm path length). This difference provides a useful discussion point for students and an additional connection to the Beer–Lambert law.

Student Perceptions

This experiment was piloted in the fall 2013 term with a subgroup of 32 students from 115 enrolled in the course. Participants were recruited on a volunteer basis, and the photometer experiment replaced two traditional structured inquiry experiments from their lab schedule. A week after completing the photometer experiment, volunteers were invited to participate in a short interview to discuss their impressions of the new experiment. Twenty students participated in interviews conducted by an independent researcher (E.J.M.) who had no involvement with the grading of the lab or the course. The interviews revealed that students' overall attitudes toward the experience were very positive. In particular, students indicated that they enjoyed building and testing an instrument that they had designed themselves. Several students cited the opportunity to explore, experiment, or "do research" as a favorite aspect of the experience. When asked specifically about the guided-inquiry prelab assignments, students indicated that they felt well prepared for the in-lab activities after completing the prelabs, and that the spaced-out timing of the assignments made the process more manageable. For example:

> *I actually liked that we did prelabs week by week and I thought it was really useful for [preparing]. I think it was really nice because after all the prelabs were done, and right before starting the actual lab, I felt like I had all the information I needed to connect the dots and see exactly what we were doing, so it was really helpful.*

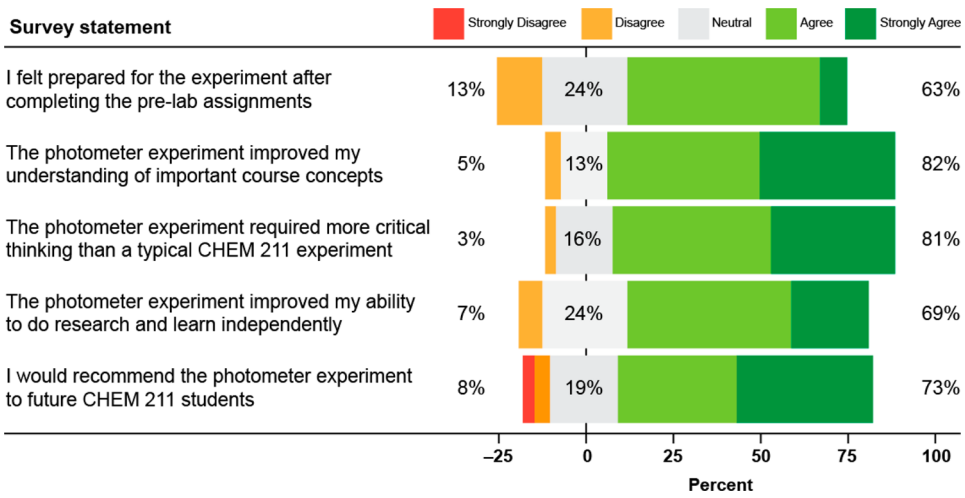

Figure 4. Student perceptions of the photometer experiment as collected in a feedback survey. Percentages on the left and right of each distribution represent the percent of negative (disagree or strongly disagree) and positive (agree or strongly agree) responses to each question; $N = 62$ (2014 winter, 21 students; 2014 fall, 20 students; and 2015 winter, 21 students).

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Some students found the independent research to be challenging and requested additional guidance on how to proceed or where to find reliable resources. When asked what they learned from the experiment, students overwhelmingly agreed that the experience had reinforced or improved their understanding of concepts from class. Several students also commented that, compared to traditional experiments, the photometer experiment required more critical thinking, problem solving, and creativity, which they considered to be valuable for their future studies or careers. One student remarked:

> *[The lab activity] sets you up to understand how you should go about designing certain procedures and, when you're unsure how to make an educated guess, as to how to approach certain problems [...] It's a very good learning experience.*

When asked how the experiment could be improved, several students indicated that the instructions and process for creating the LabVIEW software were too long or complicated for the time available. As a result of this feedback, we simplified the programming procedure for future implementations of the experiment.

The photometer experiment was again offered on a volunteer basis during the winter 2014, fall 2014, and winter 2015 terms, with 22, 21, and 24 students participating, respectively. We collected feedback on those students' experience using an anonymous survey based on the common themes from the pilot interviews. As shown in Figure 4, the survey responses indicated that the photometer experiment was a valuable learning experience for a strong majority of students who participated. Many students also remarked that they felt a keen sense of satisfaction when they succeeded in making measurements with a device they had built themselves. As instructors, we also noted that many students were more personally invested in this laboratory experiment than other experiments that lacked the do-it-yourself component and guided-inquiry model.

CONCLUDING REMARKS

This build-your-own-instrument, guided-inquiry experiment introduces students to analytical instrumentation in a research-like format. With guidance from prelab activities, students generate their own background information, design a photometer, and propose a procedure to use and test their own device. Students' photometers are able to detect the end point of an acid−base titration when using BCG as an indicator dye, and the photometer response to increasing concentrations of BCG is linear in basic solutions for absorbance values between ca. 0.1 and 1. Students regard this experiment as a valuable learning experience that improves their understanding of spectrophotometry, enhances their research ability, and develops their critical thinking skills.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00426.

Information on the photometer components, design, software, calibration and titration procedures, and relevant absorption and emission spectra (Appendix 1) (PDF, DOC)

Instructions that are provided to students for the prelab assignments and the lab report (Appendix 2) (PDF, DOCX)

Step-by-step instructions and photographs for the assembly of a photometer device (Appendix 3) (PDF, DOC)

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Notes

The authors declare no competing financial interest.

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