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Spectroscopy Letters

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597299>

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Online publication date: 24 September 2010

To cite this Article Harvey, David(2007) 'Incorporating Analytical Chemistry into an Introductory Course in Chemistry', *Spectroscopy Letters*, 40: 3, 381 — 394

To link to this Article: DOI: 10.1080/00387010701292924

URL: <http://dx.doi.org/10.1080/00387010701292924>

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Incorporating Analytical Chemistry into an Introductory Course in Chemistry

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Abstract: This paper highlights a new introductory level course entitled “Thermodynamics, Equilibria, and Kinetics” that provides students with an early introduction to incorporating analytical chemistry into their laboratory work. The rationale for creating the course and the course’s format are outlined. Examples highlighting the use of spectroscopy illustrate the development of a student’s ability to think as an analytical chemist.

Keywords: Cooperative learning, laboratory instruction, problem-based learning

INTRODUCTION

Many undergraduate laboratory courses in analytical chemistry have adopted problem-based learning in which students work in small teams to complete a multiple week or a semester-long project that simulates the analytical process and that uses real-world samples.^[1,2–5] Students often enter these courses, however, with a limited exposure to important analytical concepts and with limited opportunities to design their own experiments.

Received 9 August 2006, Accepted 30 September 2006

The author was invited to contribute this paper to a special issue of the journal entitled “Undergraduate Research and Education in Spectroscopy.” This special issue was organized by Associate Editor David J. Butcher, Professor of Chemistry at Western Carolina University, Cullowhee, North Carolina, USA.

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For example, although most students who complete courses in general chemistry have been introduced to Beer's law and the preparation and use of external calibration curves, they are less likely to have been placed in a situation where they are responsible for making the decision to use Beer's law as part of an analysis. This lack of prior experience in making decisions can limit the confidence of some students when they pursue an independent project.

For students to appreciate fully the experience of working on an analytical project-based lab, it is helpful if they have gained in their earlier lab courses both experience in solving problems and a working knowledge of important analytical concepts. This paper describes one approach that has been used successfully in a small, private, selective undergraduate liberal-arts university. The rationale for creating the course and the course's format are outlined. Examples highlighting the use of spectroscopy illustrate the development of a student's ability to think as an analytical chemist.

CURRICULAR DEVELOPMENT AT DEPAUW UNIVERSITY

To place in context this project, which is part of a broader reorganization of the curriculum in DePauw University's Department of Chemistry and Biochemistry, it is necessary to review briefly our motivation for curricular change. Several factors catalyzed our interest in curricular reform, including a desire to counter the vertical nature of the traditional chemistry curriculum with its extensive, linear structure of prerequisites; the necessity of meeting a new requirement from the American Chemical Society's Committee on Professional Training that all chemistry majors receive instruction in biochemistry; the desire to provide a better context for learning chemistry and to provide students with an earlier opportunity to pursue the area(s) of chemistry in which they have the most interest; and a need to adapt to perceived changes in the math and laboratory skills that our students bring with them from high school.

An important feature of our new curriculum is the elimination of the usual sequence of courses for first-year and sophomore students, consisting of 1 year each of general chemistry and organic chemistry. In its place, we now offer four semester-long courses that provide students with introductions to organic chemistry, inorganic chemistry, biochemistry, and physical/analytical chemistry. In addition, students complete a self-paced tutorial course covering stoichiometric calculations. Table 1 highlights some advantages of this introductory core. Although several institutions have adopted a curriculum that eliminates a traditional one- or two-semester sequence in general chemistry, most notably the "organic-first" curricula at The University of Michigan^[6] and Juniata College,^[7] we know of no other institution with an introductory core similar to ours.

Table 1. Advantages of the introductory core in the new chemistry and biochemistry curriculum at DePauw University

There are two entry points into the chemistry and biochemistry majors.

Students may begin with an introductory course in either organic chemistry ("Structure and Function of Organic Molecules") or inorganic chemistry ("Structure and Properties of Inorganic Compounds").

A less rigid prerequisite structure provides students with greater flexibility by allowing them to move more quickly into areas of personal interest.

Introductory courses in physical/analytical chemistry ("Thermodynamics, Equilibria, and Kinetics") and biochemistry ("Structure and Function of Biomolecules") can be taken as early as the second semester. Students with an interest in biochemistry, inorganic chemistry, and organic chemistry can move directly into upper level courses in these areas as early as the second year.

All courses are taught within the context of a disciplinary area.

Incorporating traditional general chemistry topics into disciplinary-oriented courses provides students with a more meaningful context in which to learn and apply chemical principles.

Students completing the core have an early introduction to biochemistry.

With the increasing importance of biochemistry in all areas of chemistry, a wide range of students receive an early introduction to biochemistry.

Students of differing mathematical aptitude and confidence have flexibility in when they choose to take more quantitative courses in chemistry.

The introductory course in physical/analytical chemistry is the only course in the introductory core for which students must complete the self-paced course in stoichiometry. A student with stronger quantitative skills may choose to take the introductory physical/analytical course as early as his or her second semester; other students can delay the course until the fourth or fifth semester.

THE ROLE OF LABORATORY IN CURRICULAR REFORM

An additional motivation for curricular reform is our perception of a decline in the laboratory skills and attitudes toward experimental work that some of our students bring with them from high school. Although many students enter DePauw University with a strong background in laboratory work, we find that an increasing number of our students come from high schools where such work is undervalued. Specifically, some high school chemistry labs meet only sporadically throughout the semester, often for only 45–90 minutes, and do not provide students with many opportunities to become engaged in doing experimental work. Instruction in basic laboratory techniques, data handling, and data analysis often is inadequate. Also of concern is a decreasing appreciation of the importance of experimentation in developing and testing scientific theories. It is not surprising, therefore, that many entering students are uncomfortable in the laboratory, viewing

lab work as a chore to be completed, rather than a significant environment for learning.

An important part of our curricular reform is to strengthen the connection between class and lab and to make the laboratory a more meaningful place for learning. There is a growing body of literature suggesting that shifting the laboratory curriculum from a reliance on “cookbook” experiments to a more student-centered, problem-based laboratory can help create a more effective learning environment.^[8,9] The effectiveness of cooperative learning in analytical chemistry is well-documented^[10] with benefits including improvements in reasoning and critical thinking skills, the ability to use prior experiences to solve new problems, and a more positive attitude toward the subject.

THE COURSE: “THERMODYNAMICS, EQUILIBRIA, AND KINETICS”

The course described in this project, “Thermodynamics, Equilibria, and Kinetics,” is one of four courses in the department’s new introductory core and is required of all chemistry and biochemistry majors. In addition, the audience includes most students who are planning a career in the health sciences and students completing a minor in chemistry. Each section of the course is limited to 24 students and meets for three 60-minute periods each week of a 14-week semester. Lab meets once per week for a 3-hour period.

As suggested by the course’s title, the primary topics for the course are three of the most important concepts underlying much of chemistry. Although the course introduces the principles of physical chemistry (minus quantum theory, which is introduced elsewhere), these topics also are included in the foundational chapters of current quantitative analysis textbooks.^[11–14]

Thermodynamics, equilibria, and kinetics are important components of traditional second-semester courses in general chemistry, but other topics, such as main-group chemistry, coordination chemistry, and nuclear chemistry, also are common in such courses. Narrowing the focus of this course to three key concepts provides time to explore them at greater depth and to make stronger connections between class and lab. Time is spent in class discussing how to experimentally measure or test a concept introduced during class, with an emphasis on analytical topics such as experimental design, calibration, and the uncertainty of measurements. Students also are asked to consider the form the experimental data will take and how it might be processed and analyzed. Where appropriate and feasible, a trial experiment is carried out in class, providing data that students can analyze immediately or as an out-of-class assignment. Because they have participated in designing relevant experiments and considering appropriate data analysis strategies during class, students can be challenged to design and carry out more investigative experiments during lab sessions. Finally, the link between class and lab

is brought full circle when students use the results of their lab work to illustrate concepts during class. This approach is a natural extension of the “discovery”^[15] and “guided-inquiry”^[16] approaches successfully implemented elsewhere.

BASIC DESIGN OF THE LABORATORY

Students work in the lab in teams of three, each equipped with the following suite of instrumentation and software: a Vernier LabPro (Beaverton, OR, USA) data interface running LoggerPro software, Vernier pH, ORP and temperature probes, a Vernier drop-counter for automated titrations, and an Ocean Optics (Dunedin, FL, USA) USB-2000 visible diode-array spectrometer running OOIChem software. The instrumentation is connected to a Dell D600 Latitude laptop computer, which is connected wirelessly to the campus network. Data is stored on a networked drive allowing students access to their data at any time.

During the semester, students complete two blocks of experiments. The first block consists of four 1-week preliminary experiments in which students learn to use the instrumentation and associated software and learn several important analytical concepts. Handouts for these experiments provide detailed instructions, and a portion of each lab period is spent introducing the relevant instrumentation and software. Table 2 provides a brief description of the current experiments along with a list of the instrumentation, software, and analytical concepts that are introduced. Experiments build on each other such that the instrumentation, software, and analytical skills learned in one experiment are used in subsequent experiments. Students are reminded frequently that the purpose of these experiments is to develop a mastery of the instrumentation, software, and analytical concepts so that they can apply this knowledge to later experiments.

In the second block of experiments, students complete four 2- or 3-week projects. Unlike the preliminary experiments, students are not provided with detailed instructions. Instead, students are provided with a statement of goals, in the form of questions to be answered, and a list of issues to consider. Details of the experimental design are left to the students, although consultation with the instructor is encouraged. Table 3 provides a brief description of the current experiments.

PRELIMINARY EXPERIMENT 4: CHARACTERIZING AN OSCILLATING REACTION

In the first of the preliminary experiments, the students prepare three solutions that, when mixed with a fourth solution that is supplied to them, produces an oscillating reaction. The purpose of the experiment, which is adapted from

Table 2. First block of experiments used to introduce students to the lab’s instrumentation and software and to important analytical concepts

Experiment and brief description	Instrumentation, software, and analytical concepts introduced
<i>Preparing Solutions:</i> Students prepare three solutions that, when mixed with a fourth solution, create an oscillating reaction. Students also explore the accuracy and precision with which different types of glassware can dispense 10 mL of water.	• Microsoft Word (for maintaining an electronic notebook) • Microsoft Excel (for data analysis) • Basic statistics • Uncertainty of measurements • The use of volumetric glassware
<i>Newton’s Law of Cooling:</i> After equilibrating a temperature probe in hot water, students monitor the probe’s temperature while it cools in air and then model their data using Newton’s law of cooling. Students extend this analysis to the warming of a probe that has been cooled in an ice-water bath.	• Vernier LabPro data interface • Vernier LoggerPro software • Vernier temperature probe • Applying theoretical models to experimental data • Curve-fitting
<i>Determination of Acetic Acid in Vinegar:</i> Students prepare and standardize a solution of NaOH and use it to determine the concentration of acetic acid in a commercially available vinegar.	• Vernier pH probe • Vernier drop counter • Acid–base titrations • Calibration of pH probe • Primary versus secondary standards • Standardization of reagents • First- and second-derivative titration curves
<i>Characterizing an Oscillating Reaction:</i> Students repeat the first experiment and characterize the oscillations by monitoring absorbance as a function of time. Students also verify Beer’s law.	• Ocean Optics USB-2000 spectrometer • OOChem software • Beer’s law • Preparation of external standards • Calibration curves • Signals and noise • Signal averaging and boxcar filters

Wang,^[17] is to evaluate the students’ ability to perform stoichiometric calculations and to prepare solutions accurately. In the final preliminary experiment, students use visible spectroscopy to characterize the behavior of this reaction, which is the cerium-catalyzed Belousov–Zhabotinsky reaction. Ferroin also is included in the reaction mixture to provide for more vivid changes in color.

For the students, the ostensible goal of the experiment is to monitor the oscillations at wavelengths of their choosing and to prepare a figure displaying

Table 3. Second block of project-based experiments

Brief description of experiment and goals

Decomposition of Hydrogen Peroxide: Students are provided with stock solutions of $\text{Fe}(\text{NO}_3)_3$ and H_2O_2 and a simple cup calorimeter. The goals for the experiment are to determine the value of ΔH for the decomposition of H_2O_2 and to verify that the role of Fe^{3+} is catalytic. Students also must research methods for compensating for the calorimeter's ability to absorb thermal energy.

Solubility of $\text{Ca}(\text{OH})_2$: Students are provided with a saturated solution of $\text{Ca}(\text{OH})_2$. The goals for the experiment are to determine values for ΔG , ΔH , and ΔS for the solubility reaction $\text{Ca}(\text{OH})_2(s) \rightleftharpoons \text{Ca}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq})$ and to determine how temperature affects the solubility of $\text{Ca}(\text{OH})_2$.

Acid Dissociation Constants of Organic Dyes: Students are provided with a paper describing a spectrophotometric approach to determining the $\text{p}K_a$ of acid–base indicators. The goals for the experiment are to use the procedure outlined in the paper to determine the $\text{p}K_a$ of an indicator included in the paper and a species not included in the paper. Students also must research the meaning of ionic strength and how it might affect equilibrium constants.

Kinetics of the Bleaching of Dyes: Students are provided with commercially available blue food coloring and bleach. The goals for the experiment are to determine the rate law for the oxidation of the blue food coloring by OCl^- and to explore how pH affects the reaction's rate.

their results (Fig. 1). The underlying purpose of the lab, however, is to introduce students to visible spectroscopy, to Beer's law and its limitations, to the preparation of external standards and the use of calibration curves, and to the effect of noise on signals and methods for improving the signal-to-noise ratio. These important analytical topics then become part of the repertoire of knowledge that students can draw upon when working on the project experiments.

Those students who have completed the introductory course in inorganic chemistry are already familiar with visible spectroscopy and the relationship between a solution's color and the wavelength range over which it absorbs visible light but not with the application of Beer's law. For other students, however, this experiment is their introduction to visible spectroscopy, although they will already be familiar with NMR and IR spectroscopy from earlier courses.

Because this course focuses on developing theoretical models for studying chemical reactivity, students appreciate that it is the change in a species' concentration during the reaction, not a change in a solution's absorbance, which is most important. This leads to discussions of Beer's law, how to select a wavelength at which to monitor absorbance, and the use of external standards and calibration curves. Limitations to Beer's

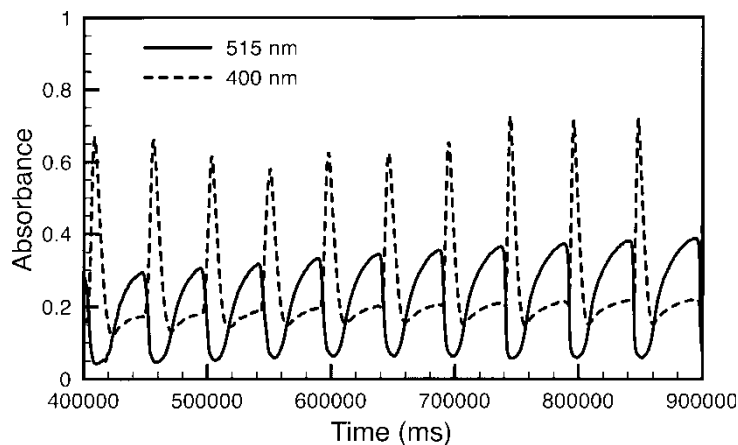


Figure 1. Typical result obtained by students showing absorbance as a function of time for the cerium-catalyzed Belousov–Zhabotinsky reaction.

law, such as curvature when the analyte's concentration is too large, also are discussed.

Of the four solutions used in this reaction, only those containing cerium and ferroin have a visible absorbance spectrum. Students examine the spectra for these two solutions at concentrations similar to that in the reaction, select wavelengths for further work, prepare a series of external calibration standards, measure the absorbances, and prepare calibration curves to validate Beer's law. The selection of a wavelength for ferroin is straightforward, and the advantage of using the wavelength of maximum absorbance (λ_{max}) is evident. Selecting an analytical wavelength for cerium, however, is complicated by the solution's excessive absorbance and by the spectrometer's poor sensitivity below 400 nm, which yields a very noisy spectrum at cerium's λ_{max} . Selecting a wavelength on the absorption band's shoulder provides a suitable compromise.

Because the diode-array spectrometer continuously acquires spectra, the students can see that there is an appreciable uncertainty in the absorbance values. This leads to a discussion of noise and to methods for improving the signal-to-noise ratio. The spectrometer's software allows for signal averaging and boxcar smoothing, and the students are free to explore how these can be used to improve the signal-to-noise ratio while also observing how they might alter the absorbance spectrum.

After completing the fourth preliminary experiment, the students are well versed in the basics of visible spectroscopy and ready to use it as a tool for solving problems in the project experiments. Two examples, one where visible spectroscopy plays a minor role and one where it is the principle analytical technique, illustrate ways in which students demonstrate their ability to think as analytical chemists.

PROJECT EXPERIMENT 1: DECOMPOSITION OF HYDROGEN PEROXIDE

In the first project experiment, students study the decomposition of H_2O_2 in the presence of Fe^{3+} . One of the project's stated goals is to demonstrate conclusively that Fe^{3+} is a catalyst. As the students design their experiments, they are encouraged to verify this catalytic behavior in several ways. The primary experimental technique is calorimetry, and the temperature versus time curves provide convincing evidence for the catalytic behavior of Fe^{3+} . Particularly insightful students, however, also note that the reaction mixture changes color during the reaction and that the solution's final color is similar to that of a dilute solution of Fe^{3+} , a fact that they can demonstrate by preparing a solution of Fe^{3+} of equal concentration and comparing the visible spectra (Fig. 2).

PROJECT EXPERIMENT 3: ACID DISSOCIATION CONSTANTS OF ORGANIC DYES

In the third project experiment, the students are provided with a paper describing a standard spectroscopic approach to determining the pK_a of acid–base indicators.^[18] The method is based on determining the absorbance of a solution of the indicator at several pH levels near the indicator's pK_a value and uses the equation

$$\log \left[\frac{A - A_{\text{In}}}{A_{\text{HIn}} - A} \right] = \text{pK}_a - \text{pH} \quad (1)$$

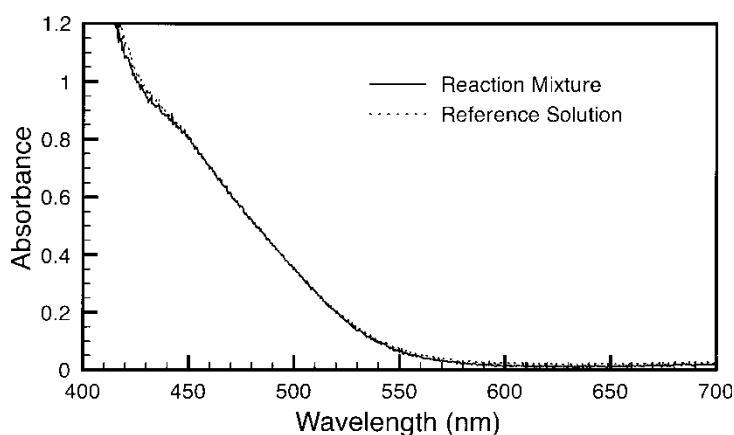


Figure 2. Visible spectra for the reaction mixture after the reaction is complete and a reference solution prepared by replacing H_2O_2 with water. Each spectrum is the average of 16 separate spectra.

where A is the absorbance at a particular pH and A_{HIn} and A_{In} are the absorbance values for the indicator's weak acid and weak base forms, respectively. The paper provides detailed directions for determining the $\text{p}K_{\text{a}}$ for bromophenol blue and outlines general conditions for determining the $\text{p}K_{\text{a}}$ for several other acid–base indicators.

In the weeks preceding this experiment, the students have spent time in class studying acid–base equilibria. Students appreciate that Eq. (1) is a logarithmic form of the K_{a} expression in which the terms $(A - A_{\text{In}})$ and $(A_{\text{HIn}} - A)$ are proportional to the concentrations of the indicator's weak acid and weak base forms, respectively. The students have also studied fractional abundance diagrams and understand that a weak acid will change from its weak acid form to its weak base form at pH levels near the weak acid's $\text{p}K_{\text{a}}$ value. This understanding helps the students appreciate the specific experimental details provided in the paper for the analysis of bromophenol blue and helps them interpret the general conditions provided for other indicators.

After following the procedure outlined in the paper to determine the $\text{p}K_{\text{a}}$ for one of the indicators listed in the paper, students are asked to adapt the procedure to a species not included in the paper. Possible analytes vary from other acid–base indicators to naturally occurring organic dyes. Typical results for the analysis of the anthocyanins in organic cranberry juice are shown in Figs. 3–6.

Having completed a preliminary experiment using visible spectroscopy, students appreciate that Eq. (1) is valid only if Beer's law is obeyed, which they easily verify by constructing a calibration curve. Figure 3 shows a typical signal-averaged spectrum ($n = 12$) obtained after filtering the cranberry juice to remove solids and diluting 30 mL of the juice to 250 mL in a volumetric flask to yield a working solution whose absorbance is not

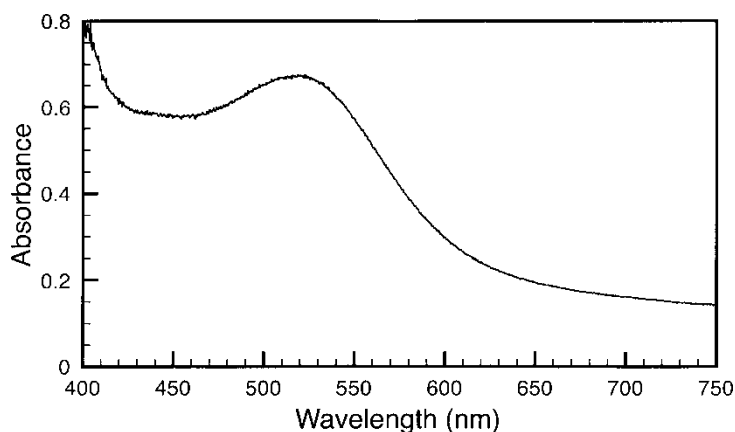


Figure 3. Typical spectrum obtained by students for filtered and diluted cranberry juice. The spectrum represents the result of signal-averaging 12 separate spectra.

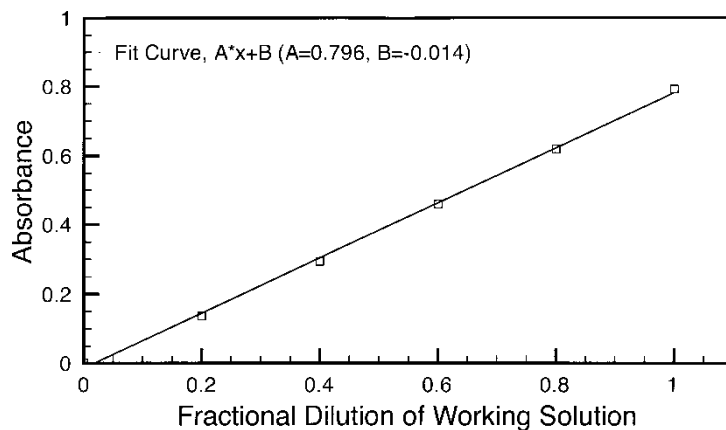


Figure 4. Beer's law calibration curve obtained by students for a working solution of dilute cranberry juice at a wavelength of 520 nm.

too large. Figure 4 shows a typical calibration curve, obtained by a different group of students, measured at a wavelength of 520 nm.

After verifying Beer's law and establishing the appropriateness of Eq. (1), the students proceed with determining their analyte's pK_a . When analyzing a species that is not included in the paper, the students must determine an appropriate range of pH values either by finding an expected pK_a value in the literature or, as shown in Fig. 5, by adjusting the pH of the analyte's working solution to higher and lower values. Here, the students' experience with fractional abundance diagrams helps them identify the maximum and minimum

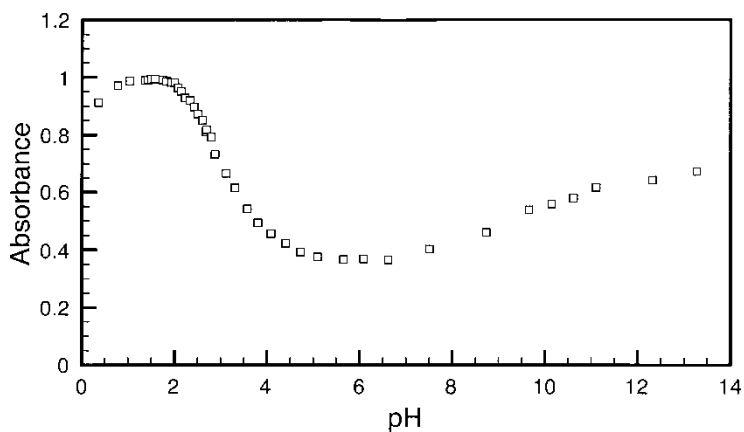


Figure 5. Experimental data obtained by students showing the change in absorbance of a working solution of dilute cranberry juice as a function of pH.

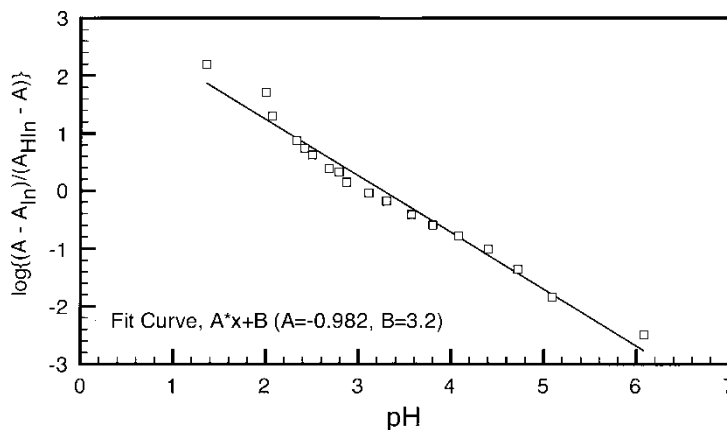


Figure 6. Graphical determination of the pK_a for the anthocyanins in cranberry juice using Eq. (1) and the data from Fig. 4.

absorbances as those for A_{HIn} and A_{In} , respectively. Finally, Fig. 6 shows the graphical determination of the pK_a value for the anthocyanins in cranberry juice. The experimentally determined pK_a of 3.2 is consistent with a published value of 3.1.^[19]

CONCLUSIONS

Implementation of this course has successfully accomplished several of the department's key curricular goals, including countering the vertical nature of the traditional chemistry curriculum, teaching traditional general chemistry concepts within a more disciplinary context, and strengthening the connections between class and lab. Beginning as early as the second semester, students receive a significant and meaningful introduction to important analytical chemistry laboratory skills. Students in the author's advanced analytical chemistry lab course consequently have shown more confidence in pursuing independent project-based experiments. Faculty teaching advanced courses that draw upon the topics covered in this course also have noticed an improvement in the students' retention of and understanding of the course's key topics. For example, a faculty member teaching an upper level course in biophysical chemistry reports that students completing our new core are better equipped to tackle increasingly more complex problems that rely upon prior knowledge of thermodynamics, equilibria, and kinetics.

The most important success of this course, however, has been in strengthening the connections between class and lab and in making the lab a more meaningful place for learning. Comments on the end-of-semester student

opinion forms show that students appreciate the many connections between class and lab; a few representative examples are shown here:

- “I really liked the way we tied the labs in with the class . . . it helped me understand the material.”
- “[The course] bridged the gap between chemistry in the lab and chemistry in the classroom.”
- “Lab was coordinated well with lecture to deepen [the] understanding of lecture topics.”
- “[The lab] gave me a very strong base for thinking through scientific problems.”
- “Labs really pushed my critical thinking and writing abilities . . . I liked the way [the course] flows . . . everything is connected.”

Students also comment that they enjoy the challenge (and freedom) that comes with taking more responsibility for designing their experiments, even though doing so adds substantially to the course's workload. The students are less enamored, however, with group work, particularly when it comes to finding time outside of lab to work on experimental planning and data analysis. For those wishing further information, course materials, including the lab manual, are archived at the course's website at <http://people.depauw.edu/harvey/Chem%20260/index.html>.

ACKNOWLEDGMENTS

Funding for this project was provided by a grant from the Camille and Henry Dreyfus Foundation's Special Grant Program (Award SG-03-041) and DePauw University. The assistance of Nicole Sweet (DPU '04) and Dr. Sharon Crary is gratefully acknowledged.

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