Topics in Chemical Instrumentation

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Applied Electronics: Construction of a Simple Spectrophotometer

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Methods of instrumentation are usually introduced to students early in the second year of their undergraduate education. Quantitative analysis and organic chemistry make use of several instrumental tools to determine the structure or quantity of an analyte. Unfortunately, many instruments are still perceived by the students as "black boxes"; samples are introduced into one end and the desired data simply appear at the other. To ensure that students understand signal transduction and manipulation, many programs stress electronics in the instrumental analysis laboratory. For example, Skoog, Holler, and Nieman devoted several chapters to both analog and digital electronics (*1*), Rubinson and Rubinson discussed the fundamentals of electronics (*2*), and Scheeline and Mork detailed a course outline on teaching electronics for scientists (*3*).

It is not difficult to find examples where electronics has been employed in the undergraduate laboratory to create simple instrumentation. Braun made use of operational amplifiers in experiments geared for the chemistry laboratory (*4*). Gruber and coworkers described the construction of a low-cost device to measure polymer conductivity (*5*). Automatic titrators have been designed and constructed by several researchers. Fox and Shaner employed an amplification circuit with their titrator and interfaced their voltage to a computer (*6*). Rocha and Reis performed automated photometric titrations using an apparatus they constructed with a phototransistor and a light-emitting diode (LED) (*7*). Headrick, Davies, and Haegele performed titrations using two differentiator circuits in series to obtain the first and second differential of the pH-titration curve (*8*).

Spectrometers have also been designed and tested by students in the laboratory. Hamilton, White, and Nakhleh built a four-color photometer that is mounted in its own compact case (*9*). Matsuo and coworkers developed a photoelectric colorimeter and applied it toward the study of various chemical systems (*10, 11*). However, these experiments center around the detailed construction of a complete instrument and might be too complex for a student who is just learning electronics.

Overview

In this paper we describe a simple spectrophotometer intended for use as a capstone circuit for an electronics laboratory exercise. Students in the undergraduate laboratory have learned the basic circuitry and are able to construct an instrument that has practical applications in chemical analysis. Figure 1 shows a schematic diagram of the spectrophotometer and its primary components. A simple LED (with peak output wavelengths of 635 or 526 nm) is used as the source and is easily changed to correspond to the species being studied. Detection is accomplished through the use of one of three different signal transducers. A box lined with black felt was used to shield the spectrophotometer from ambient light.

The schematic shows the spectrometer circuit, which employs either a photodarlington, a photodiode, or a photoresistor along with a current-to-voltage converter. These configurations allow students to experiment with different optoelectric devices and to compare sensitivities. Because the entire circuit is built on a protoboard, students are able to visualize the entire process and to make changes easily. Overall, electronics skills are enhanced while the student learns how a signal is obtained and processed.

Experimental Procedure

Advance Preparation. Before the start of the experiment, an unknown containing Ni^{2+} (or any other appropriate metal cation) in concentrations ranging from 20 to 500 mM was prepared. No other chemical preparation was deemed necessary.

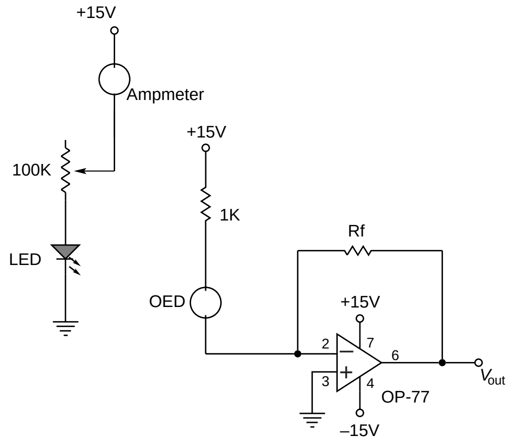

Figure 1. Schematic diagram of the spectrophotometer used to gather absorbance information. The feedback resistor was changed depending on the transducer so that a suitable voltage was generated. For the photoresistor, R_f = 1.0 kΩ; for the photodiode, R_f = 1.0 MΩ; for the photodarlington, R_f = 10 MΩ. The LED was red (635 nm, HLMP 3950) or green (526 nm, HLMP CM15); OED is the photoresistor (Vactec VT935G), photodiode (AdvDetector 22BH18M), or photodarlington (Motorola 2N5780).

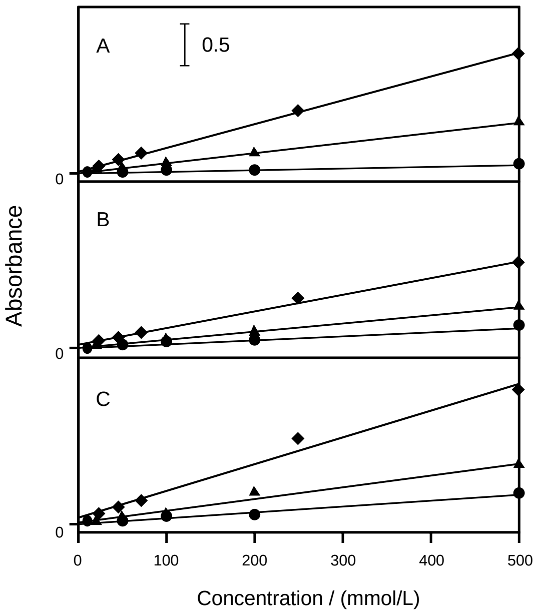

Figure 2. Calibration graphs of absorbance versus concentration for nickel (▲), copper (◆), and cobalt (●) salts using the (A) photodiode, (B) photoresistor, and (C) photodarlington. The red LED (635 nm) was used as the light source for copper and nickel and the green LED (526 nm) was used for cobalt. As concentration increases, the absorbance increases linearly; for all plots $r^2 > .99$.

Because this is a capstone circuit for an electronics laboratory, it was assumed that the students had previously learned the necessary background material that specifically relates to resistor circuits and operational amplifiers. Therefore, laboratory instruction began with a review of basic analog electronics. The lecture included a discussion of voltage, resistance, and current and the basic rules that govern the operational amplifier. From here, attention was focused on the current-to-voltage converter circuit and circuit layout. Finally, a comprehensive discussion of the three optoelectric devices was presented.

Student Involvement. The laboratory exercise continued with the students (in groups of two) building the spectrophotometer based upon the schematic drawing shown in Figure 1. Emphasis during the construction was placed on the layout of the circuit on the protoboard, especially with respect to the placement of the light source and the phototransducer. With the completed circuit, the students worked through a series of evaluation steps that allowed them to explore the sensitivity and utility of their newly created instrument.

The first step for the students was to examine the response of the photoresistor to red and green light so that they could evaluate the spectral characteristics of this transducer. By changing the lamp current and measuring the output voltage, they were able to determine the relationship between light intensity and output for both lamps. Next, they prepared a series of nickel–cation-containing solutions with the goal of creating a calibration curve to analyze an unknown. Using these solutions and a blank, they calculated the absorbance of each sample and the absorbance and concentration of their unknown. Finally, the photoresistor was exchanged for a different transducer. In this step, the students compared the sensitivities of different light-sensing devices.

This procedure was chosen so that the students could learn the basic operation of this analytical instrument without making many changes in the circuit that they constructed. Specifically, the students can qualitatively evaluate the response of the photoresistor at two different wavelengths and then draw conclusions based on this information and the information provided by the data sheets that accompany the photoresistor. Below is a complete evaluation of the circuit for several metals and several transducers.

Hazards

No known hazards are related to this experiment.

Results and Discussion

To gain maximum sensitivity, the wavelength of the LED was selected to correspond roughly to the λ_{max} of the species being studied. In these experiments, copper and nickel solutions were examined with the red LED and cobalt solutions were examined with the green LED. Because of the difference in intensities of these two lamps, the red LED was operated at 1.0 mA, whereas the green LED required only 0.15 mA.

Calibration graphs for the typical responses of the photodiode (graph A), the photoresistor (graph B), and the photodarlington (graph C) are shown in Figure 2. Similar plots were obtained when the lamp current, or LED intensity, was changed. Our procedure to obtain these plots consisted of measuring, in sequence, a blank sample, the calibration standards, unknowns, and a second blank. The spectrophotometer showed exceptional Beer–Lambert law linearity over a concentration range of 20 to 500 mM; regression coefficients for all plots were greater than .99.

Presented in Table 1 are the results from the analysis of the unknown solutions of copper, nickel, and cobalt. The wavelength of maximum absorbance for these species is given

Table 1. Determination of the Concentration of an Unknown Using the Simple Spectrophotometer

Analyte	λ_{max}/ nm	λ_{meas}/ nm	Concn of unknown/ mM	Experimental Results					
				Photodiode		Photoresistor		Photodarlington	
				Concn/ mM	% Error	Concn/ mM	% Error	Concn/ mM	% Error
Cu^{2+}	~800	635	90.2	88.3	2.1	95.5	5.9	105.7	17
Ni^{2+}	710	635	170.2	176.4	3.6	171.2	0.6	155.1	8.9
Co^{2+}	526	526	46.8	34.7	26	47.6	1.7	44.2	5.6

along with the experimental wavelength of peak intensity of the diode. As one can see, the photoresistor gave the smallest error; the photodiode and the photodarlington had significantly larger errors, especially for the cobalt solutions measured at 526 nm.

We attribute the large error obtained when the photodarlington is used as the detector to the fact that at 526 and 635 nm, the relative response of this transducer is between 30 and 40%. This also explains the need for a sensitive current-to-voltage converter (R_f = 10 MΩ). On the basis of the characteristics of the photodarlington, this device should have a gain 1000 times larger than that of the photodiode. In our experiments, the photodiode is 10 times more responsive than the photodarlington and thus requires 1/10th the sensitivity (R_f = 1.0 MΩ). This indicates that the photodiode has a relative response higher than that of the photodarlington at lower wavelengths. However, because the photodiode responds better to red light than to green light (the maximum response occurs at 950 nm), significantly larger errors are noticed for the cobalt solution.

It is apparent from our results that one can achieve good sensitivity over a large spectral range if the photoresistor is employed as the detector for the spectrophotometer; we noticed similar current readings for samples analyzed with the red and green LEDs. This indicates that the relative response for the photoresistor is high for the range that encompasses both 526 and 635 nm.

Summary

Like many spectrophotometers, the instrument described in this paper shows Beer–Lambert-law linearity for an absorbance range of 0 to 1.5. Student results indicate that the photoresistor is the best choice for accurate readings over a large spectral range. However, if one has a specific application where the wavelength will be centered around 950 nm, the photodiode produces more accurate results. For our experiments, we employed solutions of copper, nickel, and cobalt to test the function of the spectrophotometer, but there is no reason why other absorbing species could not be analyzed, so long as the correct LED is used as the source.

The spectrophotometer's simple design allows the circuit (and indeed the entire instrument) to be built and tested during a regular laboratory class period. Students are able to spend time learning the fundamentals of electronics (resistors and operational amplifiers) and are still able to find a constructive use for their newly gained knowledge. Not only does this experiment demonstrate the utility of electronic instrumentation, it also helps the student to understand signal transduction for other advanced instrumental techniques.

WSupplemental Material

A handout for students, a materials and equipment list, and notes for the instructor are available in this issue of *JCE Online*.

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