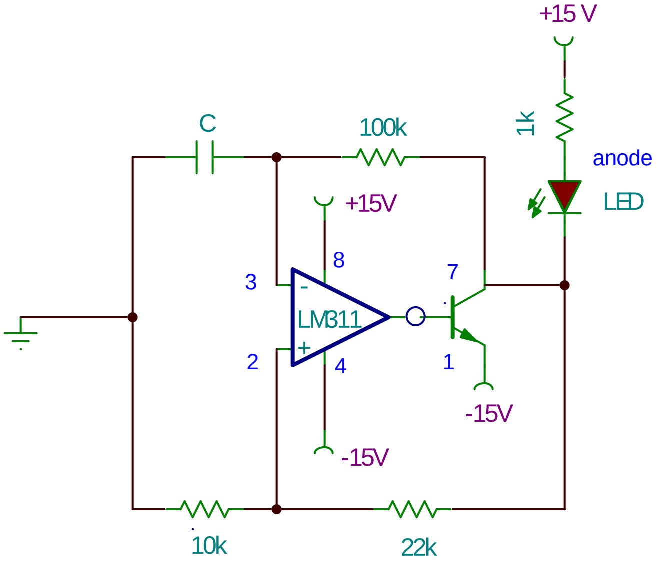
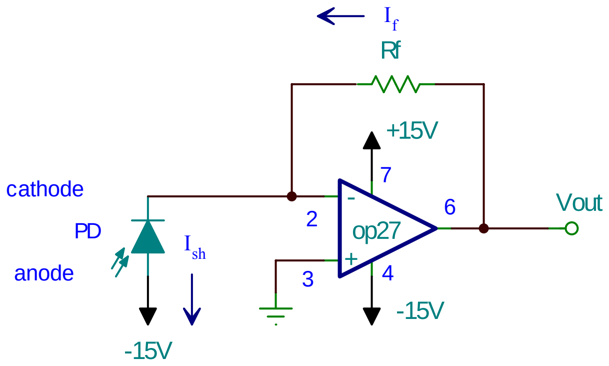
University of Wisconsin
Chemistry 524
The LED Spectrometer*

As you will see throughout the semester, the instruments used for chemical analysis can be quite sophisticated, complex, and expensive. Regardless of how complex an instrument appears, you should always keep in mind that these instruments are based on the fundamentals of chemistry, light, optics, and electronics. If you learn these fundamentals, you will not only be able to understand the operating principles behind the expensive commercial instruments, but you may even be able to design your own instruments for certain applications. In this experiment, you are going to build your own spectrometer with a light emitting diode (LED) source and photodiode (PD) detector. You will interface the spectrometer to a computer using an analog to digital converter (ADC) and write your own acquisition software with LabView. Finally, you will use your spectrometer to measure the absorbance of a grape Kool-Aid solution and quantitatively determine the amount of Blue #1 food dye in grape Kool-aid powder.

The LED Light Source Construct the following circuit to light the LED. In this circuit, an LM311 comparator integrated circuit is used to make an oscillator that will turn the light on and off at a specific frequency. Functionally, the LM311 is an inverting open loop operational amplifier connected to the base of a bipolar transistor as shown in the diagram. Construct the circuit with a 1 μF capacitor for C so that the light goes on and off at a slow enough frequency for your eyes to see the flicker. Pay attention to the polarity of the LED because it will only light when forward biased. Once you have it working, replace C with a 4700pF capacitor that will increase the oscillation to about 1.5 kHz.

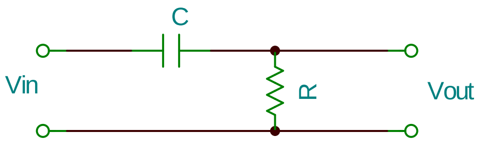


The Detection Circuit Build the following detection circuit for the photodiode. The detection circuit (a current to voltage converter) will give an output voltage proportional to the current passing through the photodiode, which is dependent on the radiant power hitting the photodiode. Notice the photodiode is reversed biased. This helps with the time response of the photodiode and also makes the response more linear. Be careful with this connection since you can destroy the photodiode if you forward bias it. Monitor the output voltage of this circuit with the oscilloscope as you direct the light from the LED on to the photodiode. Verify that your detector is working properly. The voltage of the signal should be proportional to the light power hitting the detector.



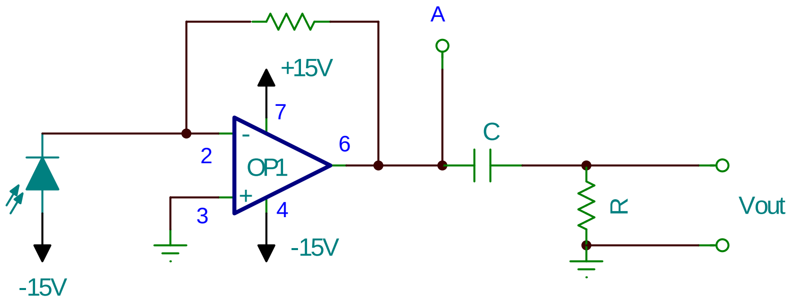
When your photodiode is completely blocked from incident light, your output should be very near zero volts. This small signal is called the background. The dark current in the photodiode and imperfections in the electronics contribute to the background signal. You should notice that room light also contributes to your measured signal. This is called stray radiation. In the next section you will eliminate the room light by modulating the LED and adding a high pass filter.

The High Pass Filter On a clear section of your breadboard, build a high pass RC filter using a 47 K resistor and a 0.10 μF capacitor.

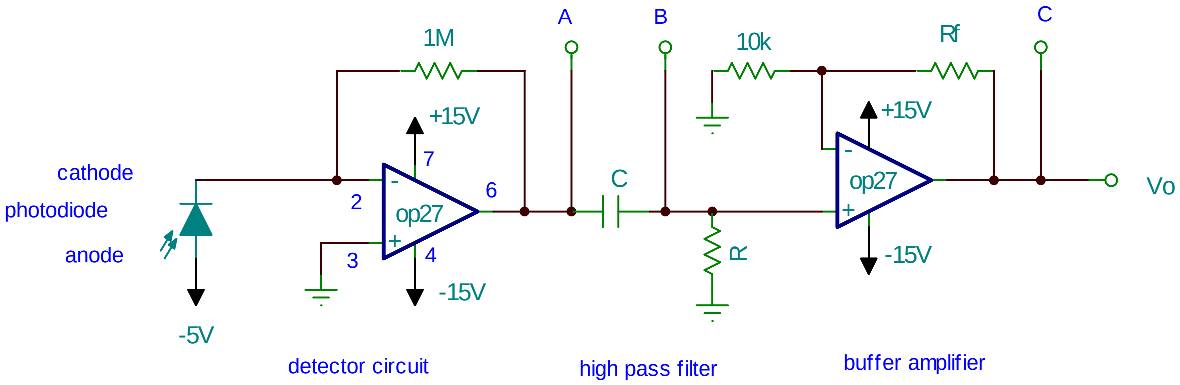


Use 1 V p-p sine wave from the function generator as V_i, and measure the peak to peak V_{out} and V_{in} at several frequencies between 5000 and 1 Hz using your oscilloscope. Display both the input and output signals on your oscilloscope and look at the relative phase of the two signals. **Use Excel to plot your data as V_o/V_i vs. frequency. Be sure to get enough data points to clearly define the curvature of the function. Also use Excel to generate a theoretical plot of the filter characteristics. How well do your values match the theoretical plot?**

Now add the high pass filter to your detection circuit as shown in the diagram. With the LED on, look at the detector signal before (A) and after (Vout) the high pass filter. The signal after the filter should be due to only the LED light. **Why?**



The Buffer Amplifier Add a buffer amplifier to your detection system according to the following diagram. Start with a 10 K resistor for R_f in the buffer amplifier. The buffer amplifier, also known as a non inverting amplifier takes advantage of the high input impedance of the op amp to prevent the loading of the circuit by the A to D converter. Use your oscilloscope to measure the signal at pts A, B, and C. **Sketch these waveforms in your notebook**. V_{out} can be measured with your oscilloscope using its rms measurement capability. Verify that V_{out} changes with changes in the LED intensity. If necessary change the gain resistor in the amplifier to get V_{out} near ±5 V. If the waveforms show "ringing" of the op amp at point A, add a small capacitor, about 10 pF, to the feedback of your I to V converter.



The Computer Interface You will now make the spectrometer more user friendly by interfacing it to the computer via a USB-6008 analog to digital converter and using a LabView program to collect the voltage data and calculate the transmission and absorbance for you. Attach Vout to AI0+ and ground to AI0- of the USB-6008. Complete the attached LabView Tutorial for an introduction to data acquisition using LabView .

Measurement of Absorbance Now use your LED/detection system to measure the absorbance of a series of blue #1 solutions. The red LED has a maximum emission wavelength near 630 nm and the blue dye strongly absorbs light at 630 nm (see Figure 1). Obtain the standard Blue #1 solutions and record the concentrations. Cover the LED and measure the background Vout with the LED spectrometer program. This small background voltage will be subtracted from each of the subsequent voltage measurements. Uncover the LED and measure the reference Vout with DI water in the sample cuvette. This is the reference measurement. Measure the sample Vout for each of the standard Blue #1 solutions in the sample holder, and record %T and Absorbance of each. **Generate a calibration plot (A vs conc.) for the standards.**

Determination of Blue #1 in Grape Kool-Aid Prepare a Kool-aid solution by accurately weighing 1 g of Kool aid into a 1000 mL volumetric flask and diluting to the mark with water. Measure the absorbance of the Kool-aid solution and determine its concentration from the calibration curve. **Determine the mass percent of Blue #1 in the powdered Grape Kool-Aid. Estimate the uncertainty in your reference measurement and use this to estimate a detection limit for the Blue#1 concentration in your instrument.**

Adding an Interference Filter You probably noticed that your calibration plot shows deviations from Beer's Law at higher absorbencies. Polychromatic radiation of the source is one the factors that contribute to Beer's law deviations (see section 13B-2 in your text). As shown in Figure 2, the LED output has a bandwidth of about 40 nm. Place the small interference filter in front of your photodiode. This filter reduces the bandwidth of the source to about 10 nm.

The filter also attenuates the light, so increase the gain resistor in the buffer amplifier to again get the signal near ±5V.

Use your spectrometer to measure the absorbance of the blue #1 standards and make a new calibration plot. Remember to measure the background and the reference voltages before the sample voltages. **Has the filter improved the plot? Explain.**

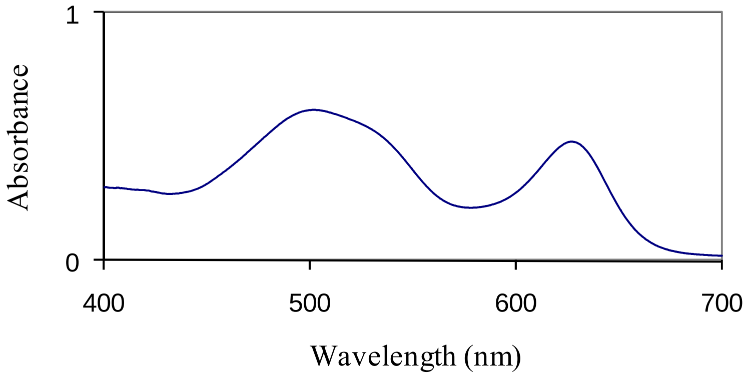


Figure 1. Absorbance Spectrum of Grape Kool-Aid

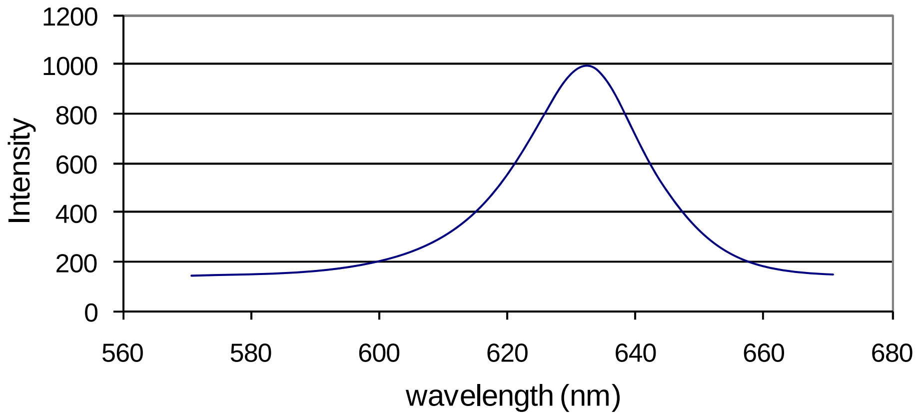


Figure 2. Output Spectrum of Red LED

LabVIEW Tutorial

Introduction The following LabVIEW tutorial will provide you with an introduction to the LabVIEW environment. LabView (Laboratory Virtual Instrument Engineering Workbench) is a graphical programming based development environment designed for building software applications used for communications with commercial or home built electronic instrumentation. After going through the tutorial, you will be able to use the computer to measure the voltage from your LED spectrometer.

Starting LabVIEW From the Start Menu launch National Instruments LabVIEW 8.0. Select **Blank VI**. Notice that there are two windows for working within the LabVIEW environment. The first window, the front panel, is the graphical user interface of the virtual instrument, vi. The front panel collects user input in the form of buttons, dials, switches, etc (called controls) and displays program output in the form of meters, graphs, etc. (called indicators). The second window, the block diagram, is the source code of your vi. You can move between the diagram and panel from the window menu or by using Alt-tab.

Controls and Indicators You will start the tutorial by programming a simple switch. From the **View** menu on the front panel, click on **controls palette**. Find the Boolean menu within the controls and place a push button switch on your front panel. Use the text pointer from the tools palette and label the switch as "measure voltage". Right click on the button and change its mechanical action to "switch until released"

The Case Structure You are now going to use your push button to control a case loop. From the block diagram, be sure you can see the functions palette. If you do not see the function palette, use the **View** menu to show the **functions palette**. From the functions palette, choose **Programming**, **Structures**, **Case Structure** and place a case structure next to your pushbutton.

The Wiring Tool Now that you have the push button and case structure in place, go to the block diagram and wire the button to the ? on the structure. The wiring tool may take some getting used to. Do not be afraid to play around with it, you can always edit the wires.

Analog to Digital Conversion Recent versions of LabView have attempted to make Data Acquisition easy. You will be using the DAQ Assistant to set up communication with the NI USB-6008. Find the DAQ Assistant in the measurement I/O menu Drag the DAQ Assistant into your case structure. Set up the DAQ to read voltage from device 1 channel ai0. You can now see some of the features of the data acquisition for the board. Set the max voltage to 10 V and the minimum to –10 V. Choose N samples and collect 1000 samples at a rate of 10k, this is the maximum rate for the USB-6008. Make sure you are set to use the differential input of the USB-6008.

You can now wire directly to the DAQ Assistant icon in your program. A right click on the icon allows you to look at the help menu for the icon. The help window will describe the connections to the icon.

The DAQ output is accessed through the right side of the side of the icon. Add the "mean" vi within the structure and connect the DAQ and the mean with a wire. Also create an indicator by right clicking on the mean output so that the mean voltage it is displayed on the front panel.

Change the precision of the display to show 3 decimal places and label it as "average voltage (V)". Run the program from the front panel using the run continuously mode. Your program should now be collected the voltage signal from your LED spectrometer. (It is actually measuring the voltage 1,000 times and calculating and displaying the average, which may be close to 0 for the AC signal.)

Add two controls to the left of the DAQ icon. Use one control to control the number of data points acquired, and the other to control the acquisition rate. Also add a graph indicator by right clicking on the data out of the DAQ Assistant so that a plot of the data is displayed on the front panel.

Run your vi to verify it is working. Set up the collection parameters so you can see the acquired square wave from the LED spectrometer on the chart. Save your vi.

Calculate the RMS You now want to calculate the rms value of the LED signal voltage. You will do this using the mathematical functions in LabView. To get a good RMS value, the input must have no DC component. Use the subtraction function to remove the average value from the array of data points. Use the squaring function to square your data. Use the mean function to average the squares. Use the square root function to take the square root of the mean. Display your rms value on the front panel. Run your vi and compare the calculated rms value of your LED signal to the rms value measured by the oscilloscope.

The Spectrometer Program To complete the spectrometer program, make 2 additional data acquisition routines like the one you just wrote. Label the acquisitions "collect background", "collect reference" and "collect sample". Use the mathematical functions to calculate %T and Absorbance. Use the display functions to show these results on the front panel.

Test your program using the Blue #1standard absorbance solution to confirm that you can make correct absorbance measurements with your instrument.

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[*] R. McClain original developed in June 2002. Modified in February 2010.