University of Wisconsin
Chemistry 524
An LED/CdS Photometer *

In a general sense, a photometer is an instrument that measures the power of a source of light. The measurement of light power is extremely useful in chemistry, since it allows us to measure light that has been absorbed, scattered, or emitted by a chemical sample, which in turn provides information about the structure and the concentration of the molecules in a sample. In fact, the measurement of light power provides the basis for every spectroscopic instrument.

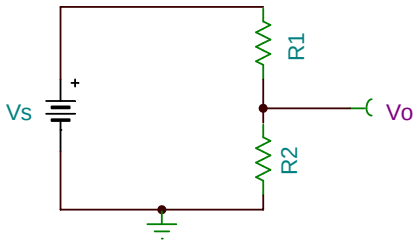
In this experiment you are going to build a photometer with a green LED light source and a CdS photoconductive detector. The detection circuit is a simple voltage divider where the output voltage changes with the resistance of the photoconductor, which is dependent on the intensity of the light striking it.

While setting up this system you will use a Digital Multimeter (DMM) for the measurement of voltage, resistance, and current in the voltage divider circuit. The DMM is an essential tool for analyzing DC circuits.

After your photometer is constructed, you will use it to determine the Fe content in Total cereal. The Fe is analyzed using a colorimetric method where the ferrous ion is complexed with 2,2-bipyridal to form an intensely colored red complex. The red complex has λ_{max} = 522 nm which is close to the λ = 530 nm maximum output of the green LED.

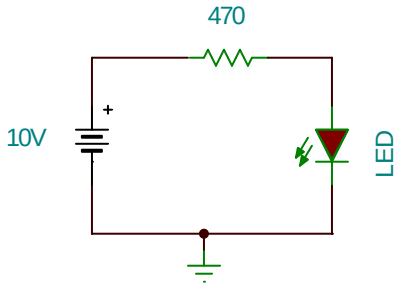
Initial Extraction from Cereal With a mortar and pestle, grind a couple of grams of Total cereal to a fine powder. Weigh out 1 g of the cereal powder into a 250 mL beaker. Add 25 mL of 5% HNO_3 and swirl. Swirl the beaker occasionally while you work on the rest of the experiment.

The Voltage Divider and Use of the DMM You will build and analyze the following voltage divider circuit as an introduction to the DMM. The two resistors provided are nominally 10 kΩ, and 15 kΩ but you should use the DMM to measure their actual values. Use the power supply at +10V for your source and the 10 kΩ resistor for R1. Build the circuit and measure the voltage of the source, the voltage drops across resistors 1 and 2, and the current through the circuit. (Note: When measuring the value of a resistor, the resistor should be removed from the circuit and when using a DMM to measure current, the DMM must be placed in series with the circuit being measured) **Are your measurements consistent with Ohms Law? Include your circuit diagram, your measurements, and your calculations in your lab notebook.**



Now set up the voltage divider circuit as a light detector by replacing R_2 with the CdS photoconductive element. **Measure the voltage across the photoconductor in the dark (block the photosensitive element from light), in room light, and directly exposed to light from a flashlight**. **Calculate the resistance of the photoconductor in each case. (**See voltage divider equation below.)

The LED Light Source Build the LED supply circuit according to the following circuit diagram. The LED is a diode and current can only flow through it in one direction (from anode to cathode). The 470 Ω resistor limits the current to about 21 mA.

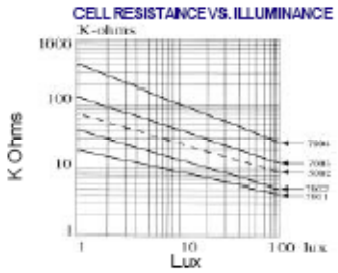


Arrange the LED and detector on opposite sides of the cell holder and direct your LED onto the CdS detector through small glass vial. Verify that your detection system is working with the LED light source.

Calibrating your Photometer Now that your source/detector system is working you can use it to measure the attenuation of the light for samples placed in the cell holder. Start by placing a vial filled with water in the sample cell and measure the detector voltage with the LED off. Shield the system from room light so that you get a voltage reading near 10 V. Now turn on the LED and measure the detector voltage with the DI water in the cell. Finally measure the detector voltage for each of the Fe-bipyridal standard solutions.

To calculate the absorbance of the solution, you need to know the ratio of the intensities of light passing through the sample relative to the DI water. This is not so straightforward since the intensity of the light is related to the resistance of the detector (its actually a log-log relationship, see below) and you are measuring the voltage drop across the resistor in a voltage divider circuit. The relationship between the voltage and the detector resistance is not linear and is given by the equation for a voltage divider:

$$V_m = V_s\left(\frac{R_D}{R_1 + R_D}\right)$$



Calculate R_D for the DI and each of the standard solutions. Your instrument can now be calibrated by plotting $-\log\frac{R_{D(DI)}}{R_{D(sample)}}$ vs $\left[Fe^{2+}\right]$. Create this calibration curve.

Determination of the Iron Content in the Cereal Filter the cereal/HNO_3 slurry through a 2 ply cheese cloth directly into 50 mL volumetric flask. Wash the beaker and slurry with 1 M HCl but do not exceed the 50 mL mark on the flask. After the rinse, dilute the solution to 50 mL with deionized water.

Remove a 1 mL aliquot of this solution and add to a 10 mL volumetric flask. Add in sequence: 0.5 mL 1% hydroxylamine solution, 0.5 mL 2 M sodium acetate solution, and 0.5 mL of 0.1% bipyridal solution. Dilute to the mark with water. Also make a reagent blank using a 1.0 mL aliquot in a 10 mL volumetric and adding all solutions except the bipyridal. Filter the solutions through a syringe filter and measure these in your photometer. **Determine the mass of Fe in the 1 g sample of Total.** The cereal box states that a 30 g serving provides 100% of the daily requirement for Fe, which is 18 mg for a healthy adult.

Preparation of Fe-bipyridal Standards The standard solutions will already be prepared for you. They were made from a 1000 µg/mL Fe stock solution as follows: Place appropriate aliquots of stock solution in 50 mL volumetric flasks to make 0.5, 1.0, 2.0, 5.0, and 10 µg/mL standard solutions. Add in sequence to each flask, 0.5 mLof 1 M HCl, 2.5 mL 1% hydroxylamine solution, 1 mL 2 M sodium acetate solution, and 5 mL of 0.1% bipyridal solution, dilute to the mark with water.

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[*] R. McClain, February 2009.