INSTRUMENTATION BREADBOARDING

(VERSION 1.3)

I. BACKGROUND

The purpose of this experiment is to provide you with practical experience in building electronic circuits with common instrumental components such as operational amplifiers, light-emitting diodes, photoconductive detectors, and photodiodes. You will use this circuitry together with optical breadboard components to construct a simple instrument for measuring absorbance. This instrument will be employed to develop a quantitative analysis for methylene blue.

II. OPERATIONAL AMPLIFIER EXPERIMENTS:

1. Construct the voltage follower shown below. Verify that the output voltage equals the input voltage.

   ![Voltage Follower Diagram]

2. Leaving your voltage follower intact, use a second op amp to construct the inverting amplifier shown below.

   a. Choose at least six combinations of $R_f/R_i$ and measure $V_{out}$ for a $V_{in}$ of approximately 1 V. All resistors used should be greater than 100 KΩ.
   b. Repeat this procedure for a $V_{in}$ of approximately 0.5 V.
III. EXPERIMENTS WITH LIGHT-EMITTING DIODES (LEDs)

1. Modify your voltage follower by inserting an LED between the follower output and ground. Verify that the output intensity of the LED varies as a function of the voltage supplied by the follower.

IV. EXPERIMENTS WITH PHOTOCONDUCTIVE DETECTORS:

1. Construct the circuit shown below using a CdS photoconductive detector. Verify (qualitatively) that $V_{\text{out}}$ is related to the light intensity striking the photoconductor.

![Circuit diagram for photoconductive detector]

V. EXPERIMENTS WITH PHOTODIODES:

1. Construct the circuit shown below using a photodiode:

![Circuit diagram for photodiode]

Suggested values for $R_f$ and $R_i$ are 300 kΩ and 100 kΩ, respectively. Using your LED source, change the LED intensity and record the output voltage from your circuit. Use at least five intensity settings.

VI. CONSTRUCTION OF A SIMPLE APPARATUS FOR ABSORPTION MEASUREMENTS

Using your photodiode circuit, construct a device for measuring transmittance. The instructor will supply a diode laser that outputs light at 670 nm along with other optical breadboarding components.
VII. DETERMINATION OF METHYLENE BLUE BY STANDARD ADDITION

Background

Methylene blue is an acid-base indicator that can be used in a variety of chemical analysis applications. Its structure is plotted below.

\[
\text{[CH}_3\text{]}_2\text{N}^+ \quad \text{[N(CH}_3\text{)]}_2\text{Cl}^-
\]

An absorbance spectrum of methylene blue is plotted below from 300-800 nm.

An inspection of the spectrum reveals that your diode laser output at 670 nm is almost optimal for measurement of the methylene blue peak absorbance. In this experiment, you will employ the technique of \textit{in situ} standard addition to determine methylene blue in an unknown.

The technique of standard additions is a common experimental method in spectroscopy. This technique is a replacement for the conventional "calibration curve" approach to quantitative analysis. The standard addition method is based on the concept of using the unknown sample itself as the sample matrix for the construction of calibration standards.

In this approach, a known amount of analyte is added to an aliquot of the unknown. This process is called "spiking" the unknown. The analyte is added by pipetting a fixed volume of a known "spiking solution". The method of \textit{in situ} standard addition uses the serial addition of very small (e.g., \(\mu\text{L}\)) volumes of a concentrated spiking solution to one aliquot of the unknown. The volume of the unknown aliquot is not altered significantly by the addition of the spiking solution. The response (e.g., absorbance of methylene blue) is measured before the first
addition of the spiking solution and after each addition.

The response measurement is then plotted vs. amount of analyte added. Least-squares analysis can be performed on these data, and the slope ($m$) and intercept ($b$) determined. By extrapolating the least-squares line back to the x-axis, the amount of analyte can be determined that was present in the aliquot of the unknown. This amount of analyte gave rise to a response value that was effectively added to the responses corresponding to the spiked amounts of analyte. An illustration of this plot is presented below.

Computationally, we are solving for the amount of analyte ($x$) denoted by a response of zero. If $y = mx + b$ and $y = 0$, then $x = b/m$ (ignoring the "-" sign). This gives the amount of analyte in the specified aliquot of the unknown. Note that this calculation assumes there is no background absorbance (i.e., absorbance = 0 when no analyte is present). If an absorbance background is present, $y = 0$ above becomes $y = y_{bkg}$, where $y_{bkg}$ is the background absorbance. In this case, $x = (y_{bkg} - b)/m$.

**Procedure**

1. With a volumetric pipet, put a 2 mL aliquot of water into the cell. Insert the cell into the beam path and measure the detector voltage. This is your $P_0$ measurement for computing absorbance values.

2. With a digital pipettor, add a 100 µL aliquot of your unknown solution to the cell. Stir the solution and measure the detector voltage. Turn off the laser source between measurements to prevent heating of the detector.

3. Serially add 100 µL additions of the provided methylene blue standard and record the detector voltage after each addition. Be sure to stir the solution after each addition. Make at least five additions.

4. Repeat steps 1-3 two more times to provide replicate measurements.
VIII. CALCULATIONS

1. Using the data acquired from your inverting amplifier, make least-squares plots of $V_{out}$ vs. $R_f/R_i$ for each $V_{in}$ and use the computed slopes and intercepts to evaluate the operation of this circuit. Discuss the shape of the plot (e.g., linear, linear over a finite range, etc.)

2. Make a least-squares plot of photodiode output vs. LED input voltage. Use the computed slope and intercept, along with a visual inspection of the plot to evaluate the detector response. Discuss this response profile.

3. Compute absorbance values from your methylene blue measurements and construct a standard addition plot (with error bars) of absorbance vs. amount of methylene blue standard added. Your first measurement in which the unknown was added to the cell is included in this plot and is treated as zero amount of standard added. Use least-squares calculations to obtain the slope and intercept of this plot. Compute the values of $r^2$ and the standard error of estimate.

4. Calculate the concentration of methylene blue (moles/liter) in the original unknown solution.
Operational Amplifiers

An operational amplifier (op amp) is the basic component of most modern circuits that modify or process analog electrical signals. Modern op amps are integrated circuits that operate at low voltages (12-15 V) and do not require highly regulated power supplies. They exhibit low drift over time and temperature.

Op amps have three properties that account for their widespread use. First, they are capable of high gain. The gain (G) of an amplifier is taken as $V_o/V_i$, where $V_o$ is the output voltage from the op amp and $V_i$ is the voltage fed into the op amp. Gains of $10^4$ are easily obtained. The second useful property of op amps is the provision for external feedback. External feedback involves returning a fraction of the output signal to the input. In practical terms, this is a way of taking the characteristics of the op amp out of the circuit. The feedback circuit characteristics will now depend on the components in the circuit other than the op amp. The third important property of op amps is high input impedance. This means that the op amp draws essentially no electrical current through it.

Schematically, an op amp is depicted as a triangle with two inputs and one output. The inputs are labeled "+" and "-", corresponding to inverting and noninverting inputs, respectively. The "+" input is typically connected to a ground source. This configuration is shown below, with an input voltage $V_i$ and an output voltage $V_o$.

At the top of the next page is shown a typical type of op amp circuit. An input voltage, $V_i$, and a set of resistors and/or capacitors provide the inputs. Taken together, the resistors and/or capacitors define an input impedance, $Z_1$. With the circuit operating, a current, $i_1$, flows through $Z_1$. A feedback loop exists containing other resistors and/or capacitors, together defining a feedback impedance, $Z_2$. The current flowing through $Z_2$ is $i_2$. Since the op amp draws appreciably no current itself, the voltage at the so-called summing point, $S$, is close to 0. This is called virtual ground. Analogously, $i_1 = i_2$. 
An op amp is an electrical device. An op amp requires external power, just as an audio amplifier requires external power before it can amplify an input signal. In actual circuit terms, the simplest op amp configuration has five connections, three corresponding to those mentioned above, and two power connections. The connections for a standard 741 op amp in an 8-pin DIP configuration are shown below. As noted before, the power inputs are typically "+" and "-" 12-15 V.

Laboratory Breadboards

Laboratory breadboards provide a convenient platform for building and testing circuits. These devices combine a low-voltage power supply (5 V, 15 V) with a series of circuit interconnection points. Wires are used to make circuit connections. The connection points on a breadboard are shown at the top of the next page.
**Potentiometers**

A potentiometer is a variable resistor designed to be used with a power supply to adjust a specific input voltage to some lesser value. The connections to a typical circuit-board potentiometer are shown below. The resistance and hence, the output voltage, is varied by turning the screw.

**Measuring Voltage**

Voltage is measured with a voltmeter. Typically, voltage is measured between a test point and ground. The negative (black) lead of the voltmeter is connected to ground, while the positive (red) lead is connected to the test point of the circuit.

**Measuring Current**

Current is typically measured through the use of a voltmeter and a precision resistor. The resistor is placed in series in the circuit in which the current is to be measured. The voltmeter is now placed across (i.e., in parallel to) the resistor. The current flowing in the circuit can thus be calculated via Ohm's Law ($V = IR$).

**Measuring Resistance**

Resistors are often color coded with the resistance value and a tolerance. When a
resistance is reported or used in a calculation, however, the actual resistance value of a particular resistor should be measured with an ohmmeter. When doing this, make sure the leads to the ohmmeter are making the best possible contact with the resistor leads.