

CHEMISTRY 133

CHEMICAL INSTRUMENTATION

LABORATORY MANUAL

Spring 2017

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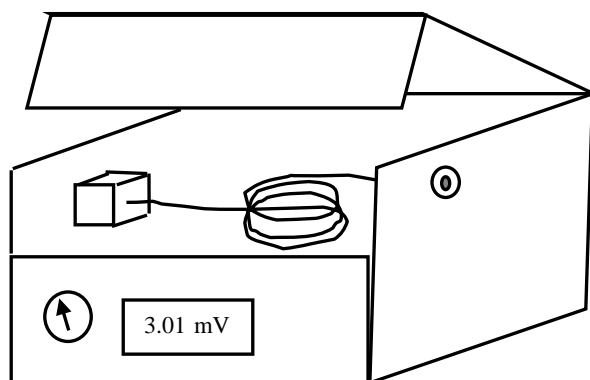


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Introduction and Laboratory Format

Chemical analysis is of great importance in chemistry and other fields. The goal of this laboratory course is to introduce the use of instruments for chemical analysis. Instruments can be used in a number of different ways from repetitive use for chemical analysis of a single component in one type of sample, to more exploratory research where numerous instrumental parameters must be changed to optimize qualitative or quantitative analysis for a variety of different samples or analytes. Some of the experiments covered in this course will include some degree of instrument optimization, as this teaches more about how the instrument functions and the instrument's potential applications.

The laboratory grade (excluding the term project) will be based primarily on laboratory reports. It is up to individual students and student groups to work productively in the laboratory and to record data of sufficient quality to be able to turn in high quality reports. The following steps will aid in working productively in the laboratory.

1. **Read the entire laboratory experiment** before beginning work on an experiment. In some cases, supplemental information will be passed out to add to the experiments here.
2. In a number of cases, **calculations will be needed** before a laboratory procedure can be carried out. **These should be done before coming to class.** As an example, in the fluorescence experiment you will need to determine how much NaOH to weigh out to prepare a buffer with the proper pH.
3. Work with laboratory partners at the start to **define who will be in charge of each aspect of the experiment.** Each student in a group should learn how to use an instrument and will be required to write their own report, but some of the sample preparation steps in the

laboratory procedure and data analysis can be divided between group members if desired. **Make sure all experiment activities indicated are completed.** Some of the steps are not needed to determine the concentration of the unknown (a key goal of many lab reports) but are needed for other parts of the report. Be sure to read the section of the lab manual for the experiment dealing with things to put in the report. For example, any time the lab report asks for a calculation of the detection limit, the uncertainty of a blank will need to be determined. It is painful to realize that you need to collect more data the day before the report is due!

4. **Use a bound laboratory notebook to collect primary data.** Leave room at the beginning for a table of contents and a page for useful reference information. Primary data includes information on how standards or samples were prepared or treated and operating conditions for instruments (unless recorded on the instrument output). Because you often need to change instrument parameters from those initially given, it is critical that instrument parameters are recorded. In this class, much of the data will be on instrument outputs (e.g. spectra or chromatographs, generated with computers or simpler recorders). You should save this data in a Chem 133 folder or your own folder on the instrument's computer (many of the instruments have thousands of data files so it is possible to 'lose' data by not keeping track of data file names). This data should then be transferred to a memory device (thumb drive or floppy drive on older instruments). Hardcopies of data should be photocopied, distributed to group members, and stored safely (e.g. pasted into the laboratory notebook or stored in a three ring binder). It is usually necessary and extremely important to note in your lab notebook what instrument 'runs' are associated with data that has been collected and stored in a computer file; otherwise no record of these conditions exist once the instrument is used by the next person. The laboratory notebook can be used during laboratory practicals if they are offered.

Data Processing and Statistics

In this class, it is a common requirement for laboratory experiments that quantities be calculated from the collected raw data. Many instruments contain software allowing them to be used for determination of analyte concentrations, while other instruments do not. However, in most cases we will use Excel to carry out calibration. Use of Excel has a number of advantages over using instrument software, such as the ability to perform the same operation for any linear least squares line fitting, rather than needing to learn different software for each instrument. There are a number of data processing operations that are commonly used in this class that are described below. You may need additional instructions to perform these operations for specific instruments.

1. Transfer of raw data to a text file.

One of the simplest ways to store data is in a text file. For instruments such as the Shimadzu fluorescence spectrophotometer where the software does not easily produce well documented data plots, it is often advantageous to save the raw data in a more convenient form. In these examples, the most basic data is a response as a function of time (chromatograms) or signal as a function of wavelength (spectra). If this data can be kept, it is easy to transfer it to a spreadsheet program such as Excel where plotting may be improved.

The simplest data files consist of a column of data (such as the FID signal from a GC with each row corresponding to a new measurement made 0.2 s after the previous measurement if operating at 5 Hz). Many of the instruments we use have an option to save data in a text format. As an example, when using Chemstation software with the Agilent 1100 HPLC, you can click on the window for Report → Specify Report. In the menu box, you can click on the boxes under destination for file (as opposed to printer) and click on txt format. Ask your instructor for help with other instruments. In the electronics experiment, we will be storing data in a text format using LabView. Because of their simplicity, one can look at text files using simple programs such as "Notepad" (found as an accessory program on PCs).

Another feature of text files is that they can easily be imported into Excel. To input this data into Excel, start Excel, and go to "File/Open". Where it says "Files of type" in the dialog box, select "All Files" or "Text Files" and find the text file of interest. Excel will then give you a series of dialog box for converting the text file to an Excel file. You will need to know if the data consists of one or multiple measurements (e.g. one column of data or more than one column such as time and response) and how the data in row is segregated (usually with a comma or a space). For example, data consisting of 2 columns and 25 rows in a text format will fill 2 columns and 25 rows in an Excel program. Once the conversion in Excel is done, it is a good idea to rename the file. Note, that for some data to be useful, you may need to know other information. For example, a program that collects data at 5 Hz (data collected every 0.2 s) but only retains the measured voltage will not display the time. However, one can deduce that the first data point corresponds to 0 s, the next data point is at 0.2 s, etc. Thus, one can make a column of time data by typing 0 in the first cell, 0.2 in the next cell lower, and then clicking and dragging over those two cells to automatically increment the number by 0.2 s for each subsequent cell (see the instructor if confused by these directions).

2. Transfer of plots to word documents.

While it may be possible to transfer raw data from a computer to Excel and to make a plot

using Excel, often one wants to use the plot provided by instrument software package. For example, chromatograms prepared through instrument software normally will show how peaks are integrated, which will not be displayed on an Excel plot. There are generally two ways to transfer plots (besides transferring the raw data and making an Excel plot). One way is to use functions in the instrument's software. For example, when using Chemstation software with the Agilent 1100 HPLC, you can go to the Graphics menu and then click on copy to clipboard. Then, you can paste the plot directly into a Word document. A second way is to use the SHIFT → Print Screen key on the keyboard which will form an electronic picture of the monitor. This can then be pasted into a Word document and edited to remove any parts beyond the plot.

3. Raw data filtering.

Many instruments allow data that is collected to be processed in various ways. Among the processing we will see in the lab (and discuss in more detail in the lecture) are low-pass filtering, Fourier transformation, integration, and linear to log transformation. While this type of processing is typically done with instrument software, it is also possible to use Excel to accomplish this data processing. One example of a simple data processing method that can be done with Excel is to plot raw data and use a moving average trendline which will reduce high frequency noise in the raw data. Additionally, once in Excel, it is easy to determine the standard deviation or data for noise estimation by using the STDEV function.

4. Calibration.

While a typical instrument "response" value consists of processed data, additional calculations are needed to convert from an unknown sample analyte response to a concentration. A number of possible calibration methods are needed for different laboratory experiments. This includes external standard methods using linear or non-linear calibration, internal standard calibration and standard addition calibration. You will be expected to know how to use linear external standard models, including determining uncertainties (see Harris sections 4-7 to 4-9), and how to use standard addition (see Harris section 5-3) and internal standard calibration (see Harris section 5-4). If you took Chem 31 here, the lab manual will also cover use of Excel to perform linear least squares regression (this is similar to Harris Section 4-9).

Electronics Laboratory Experiment

Originally, this laboratory experiment was designed to be a multi-part experiment with the first part consisting of a number of experiments putting together and testing electronic circuits. The remaining parts, which have not been developed and will not be conducted, were to construct and test simple microchip capillary electrophoresis instruments. This is mentioned to emphasize the connections between the electronics exercises that follow and electronics used in chemical instruments.

The purpose of the electronics section of the laboratory experiment is to introduce the student to electrical circuits and their role in instrumentation. The topics covered in this experiment include voltage divider circuits, RC circuits, light transducers, instrument response, noise and electrical measurements including digitization. Because of the variety of topics, a set of experiments will be performed. These will include direct current (DC) experiments (construction of voltage divider circuits for lighting a light emitting diode and detecting light with a photodiode) and pulsed voltage circuits for testing response time behavior, for measuring noise, and finally measurement of the pulse sequence of a flashing bicycle light. These experiments also cover measurement of electrical quantities using digital voltmeters (DVMs), oscilloscopes, and computers with a data acquisition (DAQ) board. While in some of the experiments you will be given step-by-step instructions, in other experiments, you will be asked to design experiments yourself to accomplish the objectives.

This part of the laboratory description is organized in the following manner: in part A, electrical devices used in this experiment are described; in part B, the experiments to be performed are listed; and in part C, information on how to report your results is given. Read all parts before beginning.

A. Getting Started - Components Used in Experiments

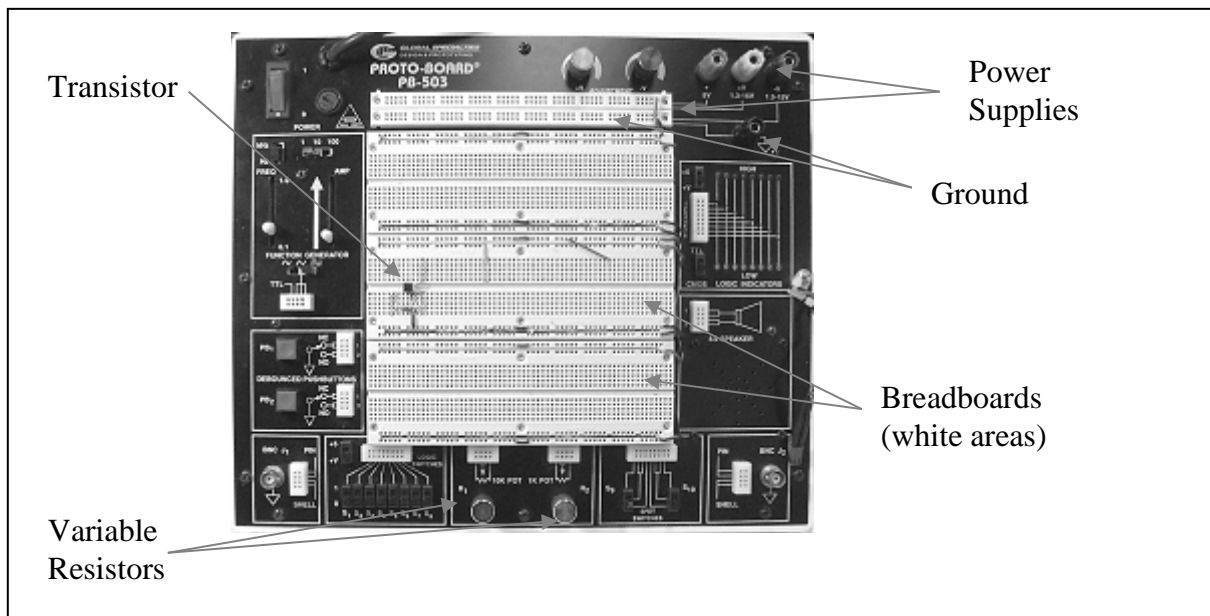
1. Prototype Boards

Prototype boards are built for testing circuits before constructing electronics board. They consist of a 5V and variable DC voltage power sources, a function generator for producing square waves (as well as other waveforms) of various frequencies and amplitudes, three 'breadboards' in the center for making connections in test circuits, and other switches, lamps, and connectors. An example is shown in Figure 1. Only solid core wires of the correct diameter should be used with the breadboards (there is a large set of short wires in some of the bins). **Do NOT attempt to force overly thick wires or stranded wire into the protoboards!** Also, hold devices (e.g. photodiodes) by their wires when connecting and disconnecting these devices. Two problems that can occur are poor contact (most common with thin wires in slots) and wires breaking off (most common with thick wires. Many devices can be plugged into sockets that are then plugged into boards. The breadboards consist of top rows currently wired for 5V and ground on the bottom. In the middle set, the columns are all connected together on the top and bottom halves but not connected across the two halves. Circuits are built by placing resistors, diodes, chips, etc. into the central part (often with leads extending across the central divider). Connections are made by placing connecting wires in the holes of the same column as the device (e.g. resistor, photodiodes, etc.) leads.

The function generator in the Proto-Boards can not provide very much power. The

function generator is normally used in the square wave form. Because of the lack of power, it will be necessary to connect the function generator through a transistor (see Figure 1) to be able to provide enough power for the device being used. The use of the transistor will be explained in class. A demonstration of circuit design using the prototype boards may be provided by the instructor.

Figure 1. The Proto-Board.



2. Resistors

Resistors are located in the bins marked with the resistance range in ohms. Resistors with narrow wires can be connected directly into the breadboard. Resistance values for resistors can be read off the resistors (for higher quality resistors), by reading the color code and using the coding instructions in the Appendix, or by measuring the resistance with the DVMs (described below). Please return resistors to the correct bins. At the bottom of the Proto-Boards, there are variable resistors to provide a resistance between 0 and 1 k Ω or 10 k Ω (plug wires to one outer and one inner hole). These are then adjusted by turning the knobs

3. Capacitors

There is a set of ceramic capacitors with values listed in pF that can be used. The wires are thin for the breadboards so use care when inserting into the protoboards.

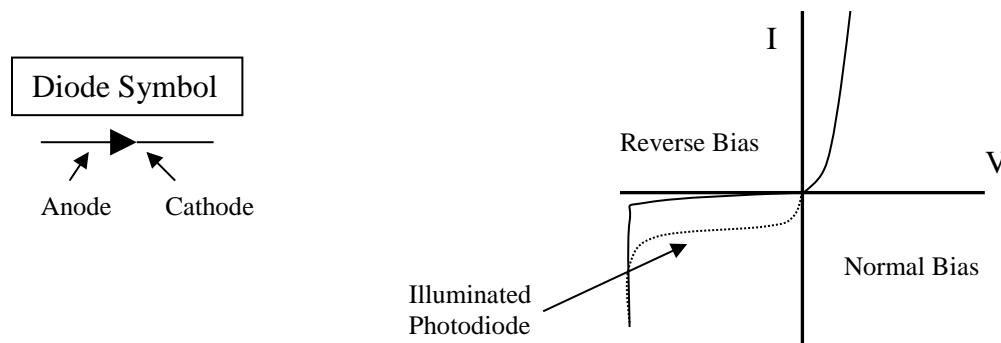
4. Light Emitting Diodes (LEDs)

These are diodes; thus you need to remember that they have an anode and a cathode (represented on the diode symbol in Figure 2 by the base and tip of a triangle, respectively) and are not symmetrical with respect to current flow. If the anode is connected to a higher potential than the cathode, this is considered to be 'normal biased' and the resistance across the diode is small. On the other hand, connection of the cathode to a higher potential (in other words a negative voltage drop from the anode to the cathode) results in 'reverse bias' and high resistance to current. This is demonstrated by a current vs. voltage diagram in Figure 2. As

shown in Figure 2, diodes are non-linear devices, meaning that the ratio of voltage to current is not a constant. In LEDs light is emitted as current flows through the LED under normal bias conditions. LEDs emit light in a quantum process and the light is fairly monochromatic.

The LEDs with leads (wires) attached (as well as the photodiodes) should be color coded so that the red lead is the anode (connected to positive under normal bias). (This may be changed if the LEDs are plugged into sockets instead of using soldered leads). Since the resistance is small under normal bias, **some LEDs** (yellow and red ones) **should be used only in series with a**

Figure 2. Diode Diagram and Current (I) Vs. Voltage (V) Curve for a typical diode



resistor to avoid overheating (and permanent failure). Some of the LEDs are colored to indicate which light they emit, while others are clear (the high intensity, narrowly focused LEDs). Make sure that the LEDs are returned to the proper bins

5. Incandescent lamps

These are similar to standard light bulbs, except that they are designed for 5 VDC and are much smaller in size. There is no polarity as opposed to the LEDs, and white light is emitted from heating the filament.

6. Light Transducers

We will be conducting tests with both photoconductivity cells (cells with two red leads) and with photodiodes (cells with red and black leads corresponding to the anodes and cathodes, respectively or alternatively, with some indicator of the anode or cathode on the body of the device). With both of these light transducers, a potential is applied across the device and the resistance decreases with increasing light intensity. Thus, light results in an increase in current through the transducer (also shown for the photodiode in Figure 2). Again, **there should be a resistor in series** or an operational amplifier at one of the ends so they do not overheat from too much current flow. For the photoconductivity cell, it does not matter which lead is attached to positive and negative, while **the photodiode should be reverse-biased**. Sensitivity to light and response rate is considerably different between these two types of devices.

7. Transistor

A transistor will be set up by the instructor in order to provide more power for certain circuits using pulsed voltage than is available from the clock on the prototype boards. The

instructor will indicate how this will be used with the pulsed voltage source.

8. Digital Voltmeter (DVM)

The DVM can be used for measuring voltage as well as current and resistance. Decide first what quantity you want to measure and select the correct setting (for resistance, current, or voltage). Most of the measurements we will be making will be using the voltage or the resistance settings, with most current measurements made indirectly by measuring the voltage drop across a resistor. Resistance measurements are best made by attaching the DVM probe leads only to the resistor(s) of interest (while they are disconnected from the rest of the circuit) and making sure that the dial is set to resistance (given by the Ω symbol – see Figure 3). Voltage is measured by setting the voltage button to V for voltage (the one shown in Figure 3 for DC currents), and placing the red probe at the higher voltage location and the black probe at the lower voltage location. **Care should be used in measuring current since there is very little impedance in using the DVM for current measurement. For this reason, the DVM should be placed in series with a resistor or other device having impedance when measuring current. Never try to make current measurements in parallel with devices as this will short-circuit the device.** Additionally, the positive probe position in the DVM needs to be switched for current measurements, and the dial should be set to A (right most position shown in Figure 3). **Make sure you switch the dial and probe position back before making other measurements.** For these reasons and because the DVM can not measure less than 1 mA, we will be measuring current indirectly by measuring the voltage across resistors in series with LEDs or light transducers in most cases.

Figure 3. The Digital Voltmeter



Move red (grey in picture) electrode to 'A' plug to make current measurements

9. Oscilloscope

The oscilloscope is a useful tool for measuring frequency or voltage associated with alternating current circuits. Groups will need to use both the oscilloscope and the computers

(see below) for the pulsed electronics experiments (although not at the same time). The power to the oscilloscopes is turned on with the scale illumination knob in the upper left-hand corner. Measured voltage goes into the x channel input (in the lower left hand corner). **Note that the secondary lead (alligator clip) is actually grounded – unlike the DVM - so the oscilloscope can only be used for measuring voltages relative to ground.** The intensity knob should be increased until the spot on the cathode ray tube (CRT) is bright enough to be seen but not too bright. The y-axis position of the spot is adjusted by turning the x-channel V/cm knob, while adjustment of the s/cm knob sets the sweep rate.

10. Computer with data acquisition board and software

There are three computers available with National Instrument 12-bit data acquisition (DAQ) boards and with LabVIEW software for data acquisition, data processing, and instrument control. The computers will be used along with the oscilloscopes for pulsed voltage data collection. The combination of a DAQ board with software allows the implementation of many of the data collection, data processing, data display, and instrument control functions that are available with most modern analytical instruments. However, LabVIEW programs must be written and debugged for these functions to work successfully. The computers and current programs will provide a time profile of the voltage in real time and can store data (unlike the older oscilloscopes). The ability to store data to disk is an advantage for recording and processing the data.

Because LabVIEW software requires some expertise to become proficient at programming, you will be expected only to use LabVIEW programs written by instructors. There are two different sets of programs to use. One is the 'Oscilloscope Sim' program, which is designed to replicate an oscilloscope. This program has more and better features than the original LabVIEW programs named "SCREEN" and "STORAGE". Like a real oscilloscope, the Oscilloscope Sim program allows pulse triggering, which means that a replicated waveform will be shown the same way regardless of the start time. The Oscilloscope Sim program does have one disadvantage – if the pulse triggering is not working (e.g. for low amplitude or noisy waveforms), no data will be displayed. In that case, it is better to use the "SCREEN" program to "find" the pulse so that the pulse triggering amplitude can be set better. Then, either the Oscilloscope Sim program or the "STORAGE" program can be used to store data to disk.

Before starting the program, be sure to connect the wiring correctly. The Chem 133 computers are equipped with National Instruments 12-bit data acquisition (DAQ) boards and LabView software that can be used to manipulate the data that is collected through the DAQ board (and send data out through the DAQ board). The interface between the DAQ board (which is located inside the computer) and the outside environment is the green terminal board that should be attached to each computer via a thick data cable. A red and a black wire should be attached to two of the screw terminals on this board. We have been using the DAQ board in a "single-ended" input mode (a dual-ended input mode is probably more common). In this case the terminal connected to the red wire corresponds to an analog input for the DAQ board. The black wire is connected to a ground terminal. An analog voltage signal coming from your experiment can be transmitted through these wires to the DAQ board where it is converted to a digital signal that can be read by the computer. Therefore the other end of the red wire should be connected to the signal that you wish to observe and save. The black wire should be connected to ground. This type of data

conversion (analog to digital or A/D) is universal in modern instrumentation that takes advantage of microprocessors.

Start the software by double clicking on the 'Oscilloscope Sim' program icon. To begin observing data click on the white arrow icon located in the upper left hand portion of the screen. When the program is running, the arrow icon should appear black. The program is designed to work much like an oscilloscope. The two virtual knobs located to the left can be used to adjust the x axis of the graph, which corresponds to time (units of seconds) and the y axis, which corresponds to voltage (units of volts). The setting on the knobs corresponds to the delimitations between grid lines on the graph. To turn the knobs place the pointed finger mouse cursor over the knob, hold down the left mouse button and drag the knob in a circular manner, just as if you were turning it. The actual value of the knob displays in the box below each knob (you cannot enter a specific value in this box, you must turn the knob). Once you make an adjustment to a knob, wait a few seconds for the data displayed in the graph to adjust before making any further adjustments. Depending on the time axis setting (x-axis) a prefix may appear after each number on the axis scale. No prefix corresponds to seconds, an 'm' prefix corresponds to milliseconds, and a 'u' prefix corresponds to microseconds. If the average value of the signal you are trying to observe is not zero, you may have to adjust the trigger. Adjust the trigger by clicking on, and dragging the control located below the data display. Drag the control slowly until you observe a signal. Adjust the control knobs until the data is displayed to your liking; when you save the data, you will save exactly what you observe on the graph.

If you wish to stop the program without saving data make sure that the switch located to the lower left is in the 'off' or down position. The text, 'Don't save data on stop' should be visible. Placing the pointed finger cursor over the switch and left clicking the mouse will operate the switch. Then left click on the 'Stop and save data' button. The program will stop collecting new data from the DAQ board and no data will be saved. The start button should turn white, and to begin the program again, simply press the start button with the mouse cursor.

To save data you must make sure that the switch in the lower left is in the 'on' or up position. The text, 'Save data on stop' should be visible. Before pressing the 'Stop and save data' button a file path and file name should be entered into the text box located along the bottom of the screen. A specific path and file can be chosen by clicking on the file icon to the far right of this text box. If no path and filename is given, a standard Windows dialog box will open when the stop button is pressed that will prompt you for a file path and name.

Next you will want to work with the data in Microsoft Excel. See the instructions in the lab manual on saving raw data in a text format for instructions on how to transfer the data to an Excel file. In the LabVIEW software data saved using the 'Oscilloscope Sim' program is delimited, so make sure this radio button is chosen and click on 'Next' at the bottom of the dialog box. Specifically, the data is comma delimited, so make sure the 'comma' box is chosen. You should now have a spreadsheet with two columns. Column A is time in seconds. Column B is voltage in volts. When plotted (voltage versus time) the data in the spreadsheet should be the same data that was displayed in the 'Oscilloscope Sim' program when you pressed the 'Stop' button.

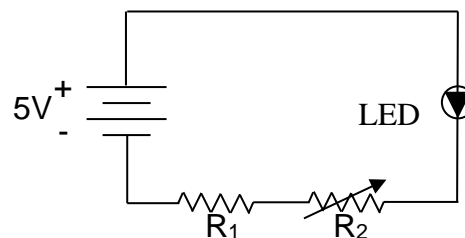
B. Experiments

1. DC Experiments

a) LED Circuit

The first experiment will be to build a circuit for lighting an LED. Lighting an LED may be necessary for something as simple as an indicator light on an instrument panel, or an LED could be used as the light source in a spectrometer. By using various resistors in series with the LED, the intensity of the light emitted can be controlled. It is also imperative that we do not allow too much current to flow through an LED or we could destroy it. Construct the circuit shown in Figure 2.

Figure 2. LED Circuit



This can be done using a relatively small resistor for R_1 (e.g. $40\ \Omega$) and the 0 to $1\ \text{k}\Omega$ or 1 to $10\ \text{k}\Omega$ potentiometers for R_2 on the ProtoBoard. If you wish to use resistance values greater than this, use a single resistor in place of $R_1 + R_2$, and replace that resistor with resistors of different values. If the LED does not emit light, check to make sure that its polarity (bias) is correct. The other possibility is that there is too little current passing through the LED if the total resistance of $R_1 + R_2$ is too large. Change the resistance of R_2 three or four times and note the effect on the intensity of the light. Make a table in which the resistance ($R_1 + R_2$) and the voltage drop across $R_1 + R_2$ is measured so that the circuit current can be calculated. Measure the current once by directly using the DVM (in current measurement mode) instead of measuring the voltage drop across the series resistors. Does the directly measured current agree with that determined from the voltage drop? Note: in all other cases, we will measure the current by measuring the potential drop across a resistor.

b) Construction of the photodiode/photoconductivity cell circuit

Photodiodes and photoconductivity cells (photo resistors) are two common light transducers used for detecting a light signal. In this experiment we will learn how to use the voltage drop across a resistor in series with the light transducer to determine how much light is illuminating the transducer. We will also discover that the range of the voltage reading can be adjusted by using resistors of different values. The circuit for the photodiode and photoconductivity cell should be the same as for the LED except that 1) the photodiode (when used) should be reverse biased, 2) a single non-variable resistor (e.g. R_1) may be desired in place of a single and a variable resistor ($R_1 + R_2$), and 3) resistors with larger R values will be needed. First put together the circuit and then measure the voltage across R_1 or $R_1 + R_2$ with the light transducer pointed toward the fluorescent lamps (on the ceiling). Change the resistance so that the voltage drop across the resistors is in the range of 0.5 to $2.5\ \text{V}$ when the light transducer is aimed at the fluorescent lamps (make sure the transducer is illuminated). Record the voltage drop and resistance values for 3 different R_1 (or combined) values for the photoconductivity cell and the photodiode (only one voltage drop needs to be in the range 0.5 to $2.5\ \text{V}$). Also measure the “dark current” when no light is shining on the light transducer.

2. Pulsed Voltage Experiments

The following experiments involve pulsed voltage sources. Pulsed voltages are common in chemical instrumentation; they often are used for noise reduction. Additionally, the use of pulsed sources allows the evaluation of the time response of different devices. In many instruments, a detector will need to respond quickly because the compound may pass through

the detection window quickly (e.g. in capillary electrophoresis where the sample passes through the detector in milliseconds). The DVM used in the DC experiments cannot be used for the measurements performed here because the voltage will be changing faster than the DVM can display (or an eye can read) values. Thus, data collection will require use of either an oscilloscope or a computer with a data acquisition (DAQ) board. All groups should use both the computer and the oscilloscope, starting with one and then switching to the other. Experiment 2a should be done twice: once with the oscilloscope and once with the DAQ board, experiments 2b and 2c can be done with either the oscilloscope or the DAQ board, while experiments 2d and 2e should be performed using the DAQ board.

a) Display of an Unmodified Square Wave Pulse.

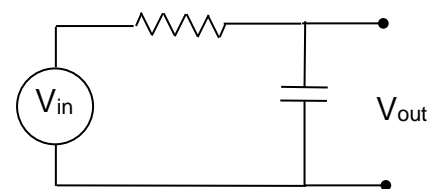
This experiment is just to show the student how to use an oscilloscope or DAQ board for displaying a pulsed voltage output. For the oscilloscope, attach the clock (or function generator) output directly to the positive probe on the oscilloscope and attach the probe ground to the proto-board ground. For the DAQ board, attach the clock output to the number 0 channel (pin 68) on the DAQ interface board and attach the Proto-Board ground to the DAQ ground (pin 67). If using the oscilloscope, set the pulse frequency on the Proto-Board to between 100 and 10,000 Hz, and adjust the sweep rate and amplitude knobs on the oscilloscope until several pulses can be observed and fill most of the screen. If using the DAQ board, set the Proto-Board to between 1 and 10,000 Hz, and then use the DAQ LabVIEW Oscilloscope Sim program in a similar manner. The sampling frequency of the LabVIEW program must be at least 10 times greater than the pulse frequency to avoid artifacts. The following experiments involve more complex circuits and measurements. However, the oscilloscope and DAQ board should be operated in the same manner as described here with only the attachment of the positive probe/DAQ terminal changing position.

b) The Low Pass RC (resistor-capacitor) circuit

Low pass RC filters are useful for reducing high frequency noise and are commonly applied to raw data generated in chemical instrumentation before the data is displayed to the user. The goal of this experiment is to determine what a pulsed wave-form looks like following passage through an RC filter and to test methods for measuring τ (the response time) for the filter. The instructor will cover various methods of calculating τ . Figure 3 shows the RC filter we will be making with voltage measured across the capacitor.

Using the same pulse frequency used in part a, select a resistor and capacitor so that $R \cdot C = 0.1 / (\text{pulse frequency})$ to $10 / (\text{pulse frequency})$. It is better to use relatively high resistor values (e.g. 10 k Ω) than a large C value to achieve this range. Sketch (if using oscilloscope) or plot data showing how the square wave is changed by the RC filter (shape and amplitude). Then change the pulse frequency or RC values and sketch how the filtered square wave is changed. Record at least three different pulse frequency/RC values. Your goal should be to find pulse frequency/RC filter combinations for which the square wave

Figure 3. Low Pass RC Circuit



signal is slightly, moderately, and completely filtered. Additionally, you can check to see if calculated τ values match $R \cdot C$.

c) Time Response of Light Sources and Light Transducers

In many instrument applications, light transducers must respond quickly as well as being sensitive to light. For some instruments, it may be desirable to pulse or chop the light source to modulate a signal in order to remove low frequency noise. The goal of this part of the experiment is to estimate the response times of two light sources (LED and incandescent lamp) and two light transducers (photodiode and photoconductivity cell). You will need to come up with experimental setups to determine response time on your own. These experiments should be carried out using black boxes to house the light sources and light transducers (to eliminate room light). The light source can be pulsed by using the clock output from the prototype board (or through the transistor) or by using a DC source to the light (continuous light beam) and positioning a fan between the light source and the light detector. The fan will periodically block the light reaching the transducer with a frequency related to the spin rate. Use of an electrically pulsed light source will only produce a square wave transducer signal if both the light source and the light transducer respond fast enough, and a "filtered" response may be due to slowly responding sources or transducers. On the other hand use of the fan will determine the response speed of light transducer only. Consult with the instructor to make sure the experiments you devise will be able to give you sufficient information. Be sure to use the same types of circuits constructed in part A. (Hint: One of the light sources and one of the light transducers respond "fast" while the others respond "slow". Also, the fast sources/transducers may respond too quickly for their time constants to be accurately estimated).

d) Measurement of Noise

In data collection, an important activity is to measure the "blank" signal. This can be done using the DAQ board and one of the light transducers with all light to the transducer blocked off. The goal is to record a period of time (e.g. 50 to 500 ms) of noise and to interpret the results (see the questions below related to this experiment for how to interpret the results).

e) Measurement of the Pulse Sequence of a Flashing Bicycle Light

Now that you have become an expert at making measurements of pulsing lights, use an appropriately fast responding light transducer circuit to determine one of the pulse sequences of the flashing bicycle lamp. Describe which pulse sequence is used in your measurement (if the light has more than one sequence). Measure the length of the "on" period and the "off" periods (this is best done using the DAQ system and storing acquired data).

C. Report

DC Circuits

LED Circuit:

-For the LED circuit, produce a table giving the series resistor sum resistance ($R_1 + R_2$), the potential drop across $R_1 + R_2$, and any observations about the LED output (e.g. changes in light intensity and/or color). Calculate the current and the resistance across the LED for each series resistance value.

Light Transducer:

-For the light transducers circuits, give a table similar to that for part a) showing the different R1 values used, the voltage drops across R1, and the current and resistance across each of the transducers each time R1 was changed.

Questions:

- Did the resistance across the LED stay constant or change with the LED current? Is this expected?
- Which transducer gives greater absolute sensitivity (larger current for the same intensity)?

Pulsed Voltage Experiments

Display of An Unmodified Square Wave Pulse: - Do not include in report

RC Circuits:

-Give a table showing pulse frequency, R and C values and a sketch or plot of the output wave form.

Light Source and Transducer Response Times:

- List (in a table or in the text) experiments conducted to determine the response times of the light sources and transducers.
- Give an estimate of the response times (time to decay to $1/e$ of the initial value) of the light sources and transducers. If a square wave was always observed in the output, list the maximum frequency used in the experiments.

Questions:

- Which transducer has a better response time?

Bonus question: What was the spin rate (in revolutions per minute) of the fan?

Examination of Noise

-Give a plot displaying the noise.

Questions:

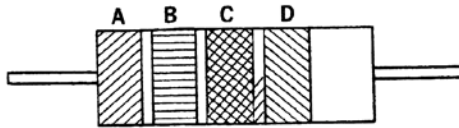
- Are only specific values of voltage observed? Hint: plot your data so that only individual data points are shown (do not connect the data points with a line). If so, is this explained by the DAC board resolution (you need to know the voltage input range of the DAC board to answer this question)? Explain your answers.
- Is there any sinusoidal pattern in the noise? If so, what is the frequency of the noise?

Flashing Bicycle Light:

-Plot the pulse sequence given by the light and determine the times of the pulse period and the time of the period between pulses.

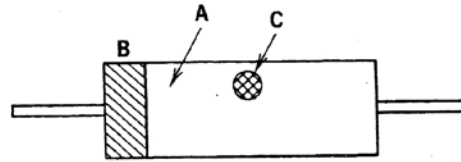
APPENDIX: Resistor Codes

COLOR CODE FOR RESISTORS



Color

- A
- B
- C
- D



Indicates

- First number
- Second number
- Number of zeros
- Tolerance

Color	Number
Black	0
Brown	1
Red	2
Orange	3
Yellow	4
Green	5
Blue	6
Violet	7
Gray	8
White	9
Gold	5% tolerance
Silver	10% tolerance
None	20%

Example:

A 250,000 ohm 20% resistor.

- A red
- B green
- C yellow
- D no color

The resulting value is in ohms