

## **Chemistry 61: Instrumental Analysis**

### **Building and Operating Your Own Diode Array Spectrometer**

#### **Part 0: Preliminaries**

Unlike the electronics lab earlier this semester, you are required to finish this project. This will involve completing each of the parts outlined in this handout, including the assembly of the spectrometer, use of your spectrometer to perform an experiment, and writing a report on your work. (Each team should turn in one report). Keep notes on your trials, tribulations, and successes throughout this project. You need not write down every last thing you do (that's what the detailed instructions here are for), but do note significant events, such as alterations in the procedure outlined here. These notes will be the basis for the Procedure section of your report (more details in Part V).

Start by going through the checklist of items for the experiment. This will give you an opportunity to figure out what everything is—especially the optical parts. Note that you will not use all the items in the kit.

#### **Part I: The Source and Monochromator**

The first part of the lab involves setting up the light source and the monochromator for the spectrometer. The source is a tungsten-halogen desk lamp with a post already attached. Secure the post to the post holder attached to the ring stand base. (It may be a good idea to secure the base to the table using a C-clamp.) The lamp has an energy density distribution that closely resembles that of a typical blackbody radiator. As we have discussed in class, such a source is fairly intense in all parts of the visible region.

The next optical element is an achromatic lens which is used to focus an image of the source onto the entrance slit. An achromatic lens is a compound lens (it contains more than one element) which has been designed to correct for coma and spherical aberration normally encountered in typical lenses. Achromatic lenses are necessary for transmitting all wavelengths of source light equally well. Make sure the lens is clean. If not, use a piece of lens paper and a drop of methanol to clean off each side. (Ask me for help with cleaning optics.) You are encouraged to clean all your optics except for the grating. You should **never** touch the surface of your grating with anything!

If the source is placed approximately 30–50 cm away from the breadboard, the first lens may be placed 3–5 inches in from the edge of the breadboard (orient the breadboard lengthwise). There is a special mount for lenses—try to figure out how to secure the lens in it. The lens mount is, in turn, secured to a post, which is secured to a post holder, which is secured to a base. (These intricacies are meant to maximize both the mechanical stability and positional flexibility of your optical components.) Use a pair of L-shaped clamps to “tie down” the base to the aluminum breadboard.

Next, use a sheet of white paper to find the image of the source behind the lens. The distance of the image from the lens and the size of the image depend only on the focal length of the lens and the distance from the object to the lens. An easy way to find the focal length of the lens is to use a distant source (like a window or an overhead room light) and project the image of that source onto a piece of paper. If the object is at infinity (or at least very far away), the image will be focused when the paper is exactly one focal length away from the lens.

Secure the pre-mounted slit in another “lens” holder, and position the holder behind the lens in the approximate position of the image you projected onto the paper. Rotate the slit so that it is in the same orientation as the filament in the lamp. Carefully adjust the position of the slit so that the filament’s image is both focused at the slit and at the same height as the slit. This will maximize the flux from the source entering the monochromator. (Question: Why use a slit that is only 25  $\mu\text{m}$  wide?) Again, secure the slit to the breadboard using the L-shaped clamps and screws.

The next step is to collimate the beam of source light coming from the slit. (As we mentioned in class, a collimated beam is one in which the light rays travel parallel to one another.) This is achieved by using a lens identical to the one used to focus the source. (Question: At what distance from the slit would you want to place the lens in order to collimate the beam?) A good way to check to make sure that the outgoing beam is collimated is to hold a piece of paper behind the collimated lens and slowly move the paper away. If the cylinder of light rays stays the same size as the paper recedes (even a few meters), then the beam is collimated. If the rays get focused, or if they diverge, then you need to re-position your lens.

As we have discussed in class, we want to collimate the light for the next component in the monochromator, the diffraction grating. Make sure that the collimated

beam falls entirely on the grating and that the grating is nearly perpendicular to the beam. Off to the side of the grating you should be able to see the zeroth and first order spectra. The zeroth order is simply a reflection of the incoming beam, but in first order, the spectrum shows the entire rainbow of visible colors. The blaze wavelength of the grating is such that the first order spectrum gets most of the light flux. (The higher-order spectra are nearly invisible.) The distance from the grating to the collimating lens is not really important; you just want to make sure that everything fits onto the breadboard and that you will have enough room to maneuver the camera lens, the final component in the monochromator.

The camera lens will allow you to focus the spectrum onto a detector. (A camera lens is used because it is specifically designed to focus many colors of light onto a flat plane (e.g., a piece of film). Normal single-element lenses allow dispersion to occur. Dispersion is caused by the fact that the speed of light in a medium varies with the wavelength. Thus, a normal lens will not focus all wavelengths of light onto the same plane. The many elements of the camera lens diminish this effect and produce a nice planar focus (and clear photographs). So even though the lens is meant to be mounted on a camera, it works very well for our purposes.)

Mount the camera lens in the home-built aluminum mount, align it with the first-order spectrum, and focus the spectrum sharply. The anti-dispersive elements of the camera work best when the object being focused is about the same distance from the lens as the image. This distance should be about one or two inches. If you have difficulty placing the lens without clipping part of the collimated beam hitting the grating, you can turn the grating slightly away from the perpendicular in order to increase the angle of reflection for the first-order spectrum.

## **Part II: The Beam Profiler (Detector) and the Oscilloscope**

Now that you have assembled the monochromator, you are ready to measure and digitize the resulting spectrum with a detector connected to a digital oscilloscope. Since our instrument has no mechanical apparatus for continuously changing the angle of the grating, we need some way to measure simultaneously the intensities of the wavelengths of light you have dispersed. A detector with a linear array of photodiodes (your device has 2048 diodes) is a very convenient way to make this measurement. Note that

recording the entire spectrum at once makes an exit slit unnecessary. (The detector provided is actually called a beam profiler, and it is really intended for measuring the spatial profile of a low-intensity beam of light. Beam profilers are often used to measure the transverse electromagnetic modes from a laser.)

We have talked some about how diodes work. The basic idea is that each of the 2048 diodes arranged along a line inside the detector can measure the intensity of the radiation (or number of photons) impinging upon it and translate that to a voltage. All of these voltages are compiled and sent out of the detector to be read by another device. The diodes are very sensitive and can be damaged by high-intensity light. Room light should not damage them, but keep the neutral density filter in front of the photodiode array slot unless you have a reason to remove it. (You may also want to insert one or more of the 2" neutral density filters somewhere along the optical path to attenuate the light intensity. Be aware, however, that these filters can cause the beam to "walk" slightly, requiring some realignment of the optics.) Using the magnetic mount, a post holder, and a post, align the detector so that the focused spectrum is at the same height as the opening in the camera's housing. The magnetic mounts will not adhere to the breadboard itself (it's made of aluminum), but the rectangular piece of steel provided can be mounted onto the breadboard into an appropriate position to allow you to use the magnetic base. Move the detector so that the spectrum is focused just as it falls onto the front filter (notice that the diodes lie a short distance inside of the detector's housing for their protection). Fine focusing will be done in the next step, as you watch the beam profile on the oscilloscope.

The leads protruding from the back of the detector each serve a specific purpose. The video BNC jack is what contains the actual signal, *i.e.*, the voltage levels at each of the photodiodes. The lead labeled "to scope" contains the trigger pulse, and the cable labeled "clock" carries the clock pulse (no surprise). The other cable is for externally triggering an instrument, and it will not be used in our experiment. Finally, there is the jack for the detector's power supply.

Now it's time to turn on the detector and oscilloscope and connect all of the cables. The switch on top of the detector should be set to "SCOPE." Use one of your BNC cables to connect the video jack to Channel 1 on the front panel of the scope. The "to scope" cable should be connected to Channel 4. The clock pulse goes into the back

of the oscilloscope into the port labeled “external clock.” Depending on your oscilloscope’s initial settings, you may or may not see anything on the screen. There are a lot of menus and settings on the digital oscilloscope, and even a very experienced user doesn’t necessarily know what they all do, so don’t be intimidated.

The first thing to note is that the oscilloscope needs to be triggered; that is, you need to tell it when to start acquiring electronic signals. That’s what the “to scope” cable is for. Go to the trigger menu and find the source of the trigger. Set the source to Channel 4, and then press “Channel 4” near the display screen. Set the level to TTL. Also, in the horizontal menu, set the trigger position to 0%. You should see a series of pulses ~24 ms apart. Zoom in on the pulse by turning the horizontal scale knob. Keep zooming in until you can see the individual trigger pulses that are being sent. Use the cursor function to find the width of each pulse. You’ll notice that the pulse is basically a square wave, and each time the voltage rises or falls (depending on how you’ve set it), the scope begins acquiring data. The camera’s trigger pulse is synchronized with the signal so that the scope begins acquiring data at the same time the detector sends it. All 2048 pulses are sent in the interval between each trigger pulse, and those voltage levels are plotted versus time on the oscilloscope screen.

(If you cannot see a trace on the screen, you can try adjusting a number of scope settings. You can increase the voltage/division (using the vertical scale knob), adjust the height of the trace, and finally, using the vertical menu, you can adjust the voltage offset. These techniques will be particularly useful when you start looking at Channel 1.)

Besides the main trigger, the scope also needs to know when to take individual data points. The internal clock normally tells the scope when to do this, but for special applications such as this one, an external clock pulse must be provided. Connect the “Clock” lead into the port at the back of the oscilloscope, and use a T-shaped BNC adapter to look at the clock pulse on Channel 3. (The “waveform off” button will let you clear the Channel 4 trace.) If you zoom in horizontally, you’ll see that the signal is just a square wave with a period of about 6 microseconds, which is about the time it takes each individual diode to send its voltage level. It turns out, for reasons unknown, that the clock pulse from the detector is not sufficient to trigger the correct acquisition of data. To remedy this, a low-pass filter (remember your *RC* circuits!) should be placed between the clock output from the detector and the BNC jack on the back of the scope. The filter

will slightly delay the trigger, which enables the signal to be read at the correct time. Examine the Channel 3 trace and see how the RC circuit affects the shape of the pulses.

After examining the various signals, zoom out again, and change the clock to external in the horizontal menu. Make sure the maximum clock rate is set to 10 MHz. The time domain label should now read “M 250c” and the horizontal scale knob should no longer have any effect.

Finally, expose the detector to the light source. Keep the neutral density filter in front of the photodiode array slot, and turn off the room lights. Make sure that the input impedance (set in the vertical menu) for Channel 1 is 1 M $\Omega$ , not 50  $\Omega$ . You should see a signal which rises and falls from left to right. Go the Measure menu. Choose the measurement “Peak to Peak” (on the fifth page of measurements). The oscilloscope screen will now display the voltage difference between the baseline level and the peak voltage of the signal. If the oscilloscope reads “Clipping Positive”, try increasing the number of volts per division. If that doesn’t solve the problem, it is likely that the detector is saturated, and you need to use one or more neutral density filters to decrease the intensity of the source.

Typically, we will want to acquire the average of several spectra. To set this, go to the acquire menu, choose the average option, and set (using the upper left dial on the scope) a reasonable number of averages (like 25).

If you see no signal or one that is not what you expect, make sure that the spectrum is focused on the detector. If that is not the problem, then the source of your difficulty is probably the oscilloscope settings. Try adjusting the horizontal, vertical, and trigger settings, but if the signal still does not look right, ask someone for help.

### **Part III: Acquiring Data with LabVIEW**

Before you start using LabVIEW (the merciful acronym for Laboratory Virtual Instrument Engineering WorkBench), let’s consider some basics of computerized data acquisition. This experiment uses a GPIB (General Purpose Interface Bus) to transfer data from the oscilloscope to the computer. A GPIB is a programmable device which can be given instructions by a LabVIEW program about how and when to acquire data. The GPIB can then give commands to the oscilloscope, or to another type of instrument, which configure the scope or tell it to send out data. The GPIB comes as a card that is

inserted into a slot in the back of the computer. There are several other types of data acquisition options available--for example, the calorimetry experiments in the Physical Chemistry I lab use DAQ boards. However, the GPIB is one of the fastest and most reliable for somewhat complicated experiments. (I used GPIB for my spectroscopy experiments throughout my grad student career.)

Connect the thick GPIB cable from the back of the computer (it should be obvious where to make the connection) to the back of the oscilloscope. Then double-click the Chemistry 61 icon on the computer desktop. The display that initially comes up is called the front panel. The front panel is the user interface of the LabVIEW virtual instrument (vi). The commands that make the front panel run are in the diagram. Go to Windows->Show Diagram. You should see a frame that is made to look somewhat like a filmstrip. This is called a sequence, and by clicking on the arrows on the top of the frame, you can scroll through the various sequences. Sequences allow the program to execute in a particular temporal order. LabVIEW is based on a "data-flow" format, so unless you tell it to do otherwise, it executes commands as soon as elements of data are available. Data will be available as long as the input wires into the command are connected to something that has a value, whether it be a string of characters, a number, an array, or whatever. Within each sequence a particular operation is being performed. Go to Help->Show Help. Now a window pops up that gives a description of each structure as you place the pointer over that structure's icon. The first three frames are commands which talk to the GPIB, giving the oscilloscope some set-up commands. The next frame is where the actual data acquisition begins. Notice the small box that says "Read WFM". This is called a sub-vi, meaning that it is a separate program within the larger program. If you double-click on the icon (using the pointer only), you can see the panel and the diagram for this sub-vi. "Read WFM" is a driver for the oscilloscope, and it gives commands to the scope in the oscilloscope's own programming language. The scope sends back waveform data, and "Read WFM" parses these data and formats them into something that can be plotted. The output data, called an array, are then sent out into the rest of the main program. Go through the rest of the plotter.vi diagram and see if you can figure out what is going on in each part of the program (using the Help window, of course).

One of the basic tenets of the LabVIEW language is that it allows you to build “virtual instruments” which can be specialized to deal with the particular experiment at hand. LabVIEW is general enough so that if no company sells the data acquisition instrument or software that you need, you can quickly and easily build your own with LabVIEW. If there are some structures that you don’t understand, consult the “LabVIEW for Everyone” handbook or ask an experienced user.

It would be a good idea at this point to test communication between the oscilloscope and the computer. To do this, we will record the spectrum from the W lamp. Under Scope Setup, choose “average” (as opposed to “sample”) and set the number of averages to something different from what you set on the scope. Press the play button on the LabVIEW interface to start data transfer from the scope. The scope display should blink momentarily, and have its number of averages (as depicted in the acquire menu) changed to whatever you just set in LabVIEW. By default, the first spectrum LabVIEW records is assumed to be that of a reference cuvette. It will then prompt you to insert the sample cell. Proceed with collection of the second spectrum. When LabVIEW is finished, you should see virtually identical green and purple traces in the left panel (corresponding to sample and reference), and their difference (a noisy trace centered at zero) in the right panel.

(One problem you shouldn’t encounter, but just in case: If there does not appear to be communication between the scope and the computer, first make sure the GPIB cable is securely connected to both devices. Next, check the configuration of the scope’s GPIB port. Access this by hitting SHIFT-Utility. Next, choose the system button at the lower left of the scope display. Select the I/O option. Then, at the upper right of the display, set the Talk/Listen address to 1.)

#### **Part IV: Calibration**

Like any instrument, the spectrometer you have assembled needs to be calibrated. We will use the emission lines from a mercury discharge lamp for this purpose. You may remember from class the three most intense visible lines: purple, green, and orange (the latter is actually a doublet). Look up accurate values for the wavelengths of these lines in the CRC Handbook.

We start our calibration process by replacing the tungsten-halogen lamp with the mercury lamp. Carefully focus your new source onto the slit. Make sure that all three lines are passing through the camera lens, and are focused onto the entrance of the detector. It will be necessary to remove the neutral density filter at the front of the detector, at least at first. Make sure the room lights are off when you remove the filter, and use the flashlight in your kit for light. Adjust the position of the detector carefully until you see at least three lines on the oscilloscope. The big challenge is to see any peaks on the scope in the first place. Once that hurdle is surmounted, it is easier to tweak (carefully!) the grating tilt, camera lens, and especially the distance between the detector and the camera lens, to optimize both the intensity and resolution of the Hg spectrum. (Also, be sure to figure out which end of the scope trace corresponds to which end of the visible spectrum!) With very accurate focusing, you may be able to see six or seven lines.

Once you have obtained an acceptable focus and you are satisfied with the spectral range, lock down the detector and capture the spectrum with LabVIEW as described above. Then zoom in on the spectrum to find the pixel number of each of the spectral lines. Write down the pixel number ↔ wavelength correspondences, and enter these numbers along with their corresponding wavelengths into the arrays. Under the LabVIEW Operate menu, choose the setting “Make current values default” and save a new version of the vi.

Note that your calibration notwithstanding, any spectral data files written are only one-dimensional arrays containing absorbance values. The row number of each datum corresponds to the pixel number. To analyze any data, you will need to use Excel to insert a column with the pixel number ↔ wavelength calibration. So be sure to write your calibration data down!

Note also that your calibration holds only as long as you do not adjust any part of your monochromator. If you move any of the optics or the detector, you will need to re-calibrate.

### **Part V: Experiments and Reports**

Once you have calibrated your spectrometer, test it by taking an absorption spectrum of a colored solution. Mount the cuvette holder and place it onto the breadboard directly

in front of and as close as possible to the slit. You may need to adjust some of your optics to accommodate the cell holder (if this is so, remember to re-calibrate). Turn the lamp on and make sure that the beam width at the point of entry into the cell is small enough so that most the light goes through the cell. Now you're ready to place the cell in the holder and take a reference spectrum. Enter the data points from the calibration step into the wavelength and pixel number arrays. Also choose the average option and the number of averages you wish to take (the more averages, the longer it takes, but there will be less noise in the spectrum). Run the VI. The program will configure the oscilloscope and then take the reference sample (you will not see it on the screen yet). Then place the cell with the colored solution into the holder and press the "OK" button to take a sample spectrum. The reference and the sample spectra will appear on the same plot, and the program will automatically calculate an absorbance plot. This option may be useful if you want to change the order in which you calibrate, take the reference spectrum, and take the sample spectrum.

When you save any of the data, it is automatically written to spreadsheet files (called "ref" and "sample") on the hard drive; these can be opened and modified in Excel. Note that these are the default file names. You must manually re-name these files after they have been created; else, a subsequent run will write over your old files. Try obtaining the spectrum again if you are not satisfied with your results, and then compare it with the results from a spectrum of the same solution taken with the Beckman UV-Vis diode-array spectrometer in Olin-Rice 387.

The rest of this project is now up to you. There are many experiments to choose from involving spectroscopy in the visible and near-infrared regions of the spectrum (you may want to test the spectral range of your spectrometer and find out which components are providing the limits before you choose an experiment). Consult physical or analytical lab textbooks, or back issues of the *Journal of Chemical Education* for ideas.

When you are done with your experiment, you are required to turn in a report on your work. Follow the standard format for scientific papers you learned in Physical Chemistry I lab, providing an abstract, introduction, procedure, results and discussion, and conclusion.

(originally designed and written by J. Johnson and T. D. Varberg, Summer 1998)

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