Running the Sodium Emission Experiment

Read This Handout Completely Before Coming to Lab

Equipment

Jobin-Yvon Model 1500 monochromator
Jobin-Yvon CIV 301 Controller
Keithley 160 Digital Multimeter
Fluke 405B High Voltage Power Supply
Hamamatsu 9R Photomultiplier Detector
Computer with a National Instruments 12 bit A/D board running LabView5.1 Data Acquisition Software.

The equipment listed above will be used to determine the emission lines of a sodium lamp. The monochromator will first be calibrated by scanning a known emission source. Instrument control and data acquisition will be performed with the computer program called LabView.

The mechanical indicator on the monochromator gives only the approximate wavelength setting of the instrument. An accurate determination of the various positions of the monochromator over the wavelength range in which we are interested requires a spectral calibration lamp, and we will use a mercury lamp. The positions of the strongest mercury emission lines over our region are:

3125.67 Å  3131.55 Å  3650.15 Å  4046.56 Å
4358.33 Å  5460.74 Å  5769.60 Å  5790.66 Å

The monochromator can be moved to a desired wavelength in three different ways:

1. Manually, by rotating the hand crank. The CIV 301 must be turned off or the MAN button on the CIV 301 must be depressed before manual operation or damage to the motor drive will occur.

2. Through the CIV 301 control using the front panel buttons.

   Depress the SENS button to scan to a higher wavelength or release the SENS button to scan to a lower wavelength. The λ+ and λ− lamps will indicate which direction is selected.

   Depress the P.P (French for pas à pas “step by step”) button for slow speed or depress the RAP (“rapid”) button for fastest speed.

   Depress and hold the AV button to advance the monochromator. Release the AV button to stop.

3. Through computer control, using LabView software and its program CIV 301 Interface2001.vi (the “vi” stands for “virtual instrument”). When the INT/EXT button on the CIV 301 is depressed all local controls are disabled and the monochromator is controlled through the computer. This is how you will control the monochromator during most of the experiment.
Equipment set-up

1. Preliminary:

Power up the sodium lamp and allow it to warm up as you set up the other equipment. 
Power up the high voltage supply. Set the output to –1000 V. 
Power on the Keithley 160 multimeter, and set the dial selector to the 1 μA range. 
Power on the CIV 301 controller and the mercury pen lamp.

2. Initial scan settings:

There is a large variation in the intensities of the emission peaks of both the mercury and sodium lamps. 
In order that the output of the photomultiplier detector does not go above or below the rather narrow 
allowable limits of our data acquisition hardware, you will scan the spectrum at two different sensitivity 
settings and with different exit slit widths (which controls, in part, both the amount of light reaching the 
detector and the spectral resolution). The optical path will have been adjusted before you come to lab to 
ensure proper alignment and monochromator illumination; be careful during lab that you do not disturb the 
positions of the lamps, mirrors, and lens.

You will begin your scan at 3000 Å and end at 6200 Å, scanning at 50 Å min⁻¹ for a total scan time around 
1 hr. Start your run by manually adjusting the wavelength to somewhat less than 3000 Å then slowly 
approaching 3000 Å from below. Stop when the red line on the mechanical wavelength dial is right at 
3000. Be sure that the MAN button on the CIV 301 is depressed.

3. Starting LabView:

Click on in the lower left corner of the computer screen. Click on .

Click on Open VI in the following window:
The following screen is displayed:

Click on CIV 301 Interface2001.vi, then click OK. Wait a few moments as the program loads. The program starts, and the following screen is displayed.
4. Running the scan:

In the box, type the name of the file in which you will store your data as follows: C:\chem81\yourfile where “yourfile” is a name you choose.

Enter 3000 in the Start Wavelength box:

Depress the INT/EXT button on the CIV 301 front panel.

Be sure the button marked 10 is also depressed. This sets the scan speed at 50 Å min⁻¹.

Type Ctrl+R to start the program.

Set Direction to UP; Speed to scan; and Record to ON:

When you are ready to begin the scan, click to start the stepper motor and begin collecting data.

Whenever you want to pause, stop the scan by clicking . The program is still running and keeping track of the position of the monochromator. Check that the mechanical wavelength indicator and the computer display are still in close agreement after each pause.

When you have made adjustments after a pause (according to the schedule below), click on to resume the scan.

When the scan is complete, click on to terminate the program and save your data in a file you can blitz to yourself and your partner.
5. Scan Schedule:

To accommodate the varying intensities of the Na and Hg lines, and to keep extraneous Hg lines out of your spectrum, you will run the scan in three sections, and you will turn on the Hg lamp only when you expect one of our calibration lines to appear. Keep the list of lines on the first page close at hand. Keep the lamp on from the first of the scan until you see the two lines at 3125 and 3131 Å, then switch off the Hg power supply. As you approach each line, switch the power supply on when you are about 10 Å below the expected wavelength. Once you see the line appear, wait another 5 or so Å, and switch it off.

The conditions for the three separate sections of your scan are shown below. Note that you should have the instrument already set for the first segment, and note that all you change is the Multimeter Scale.

<table>
<thead>
<tr>
<th>Segment</th>
<th>Wavelength Range</th>
<th>Multimeter Scale</th>
<th>Exit Slit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3000–4200 Å</td>
<td>1 µA</td>
<td>50 µm</td>
</tr>
<tr>
<td>2</td>
<td>4200–5400 Å</td>
<td>100 nA</td>
<td>50 µm</td>
</tr>
<tr>
<td>3</td>
<td>5400–6200 Å</td>
<td>1 µA</td>
<td>50 µm</td>
</tr>
</tbody>
</table>

The exit slit is adjusted via a micrometer knob. As you turn the knob clockwise looking down on it, the knob will move down and the slits will widen. One full turn changes the slit spacing by 50 µm, and there are marks on the micrometer body every 50 µm. The diagram below shows the slit at 50 µm.

![Micrometer Knob Diagram]

When you reach the end of a scan section (and you need be only within a few Å of it), click the LabView Enable button to pause the scan, adjust the multimeter scale, then click Enable again to continue. Record in your notebook at each pause what you changed and both the monochromator’s mechanical wavelength reading and the program’s current wavelength indication.

At any point in the scan, you can adjust the full-scale setting of the on-screen display to any value between 0.1 and 10 volts full scale. Use the control shown below (you don’t need to pause to adjust it) to keep your current signal visible, to see peaks that go off scale, etc.
6. Completing the scan:

Once your scan is complete (press the Stop button at the end), you can use another LabView program, DataPlayback, to review your data file and explore your peak positions.

Type Ctrl+O and then click on Data_playback.vi. You will see the following screen:

Type Ctrl+R to start the program. Do not select Open File from the File menu.

Click the file you want to open from the dialog box that appears next. Your entire spectrum should appear.

You can magnify any segment of the display using controls described below.

First, select the magnification icon from the Tools palette.

Next, select “zoom by rectangle.”

Using the magnifying tool cursor, hold down the mouse button, and draw a rectangle around the peak region to be examined. Release the mouse button, and the expanded peak will be displayed.
Click on the cursor display and select “Bring to Center.”

Select the Crosshairs from the Tools palette.

Use the button palette to move the cursor to the apex of the peak, and read the wavelength on the cursor display.

Select “Undo Zoom” to display the original trace.

Repeat the above procedure for each significant peak.

The same section of the chart can be “zoomed” several times. However, “undo zoom” works only once. You can use the Crosshairs tool to “grab” the cursor and drag it back and forth across your entire spectrum. Whether you use this method or the Excel method outline below to measure your peak positions is not important, but you should probably explore your data with DataPlayback at least a bit before leaving lab.

Using Excel

You should Blitz your data file to yourself (and your lab partner!) so that you can open it in Microsoft Excel at home. Unless you use the Windows extension “.xls” at the end of your filenames, you will not be able to double-click a data file and have it open automatically in Windows Excel. On a Mac, your files will appear as generic icons, and again, double-clicking will not open them automatically in Excel (unless you have one of the most recent Mac OS systems and use the .xls filename extension).

To open it, therefore, it is best to launch Excel on either platform and open the data file from within Excel. On a Mac, you will need to use the “All Files” option in the “List Files of Type…” drop-down menu in the file open dialog box. Select your file, and when the “Import Wizard” (in Excel 98—other versions may open differently) appears, just click on “Finish.” The data files are “tab-delimited” text files, and Excel should easily give you a column of wavelengths and an adjacent column of associated signal values. Use “Save As…” to save your data file as an Excel file (under the old name or a new one, as you wish, but be sure to select “Excel Workbook” under “Save File as Type:” in the save dialog box) once you have opened it. This will ensure that subsequent saves include any graphs or calculations you have embedded in your worksheet.
Probably the first thing you should do is graph your data. Click at the top of column A (on the “A” itself, not in the first cell) and drag over to column B so that all of both columns are highlighted. Then click on the “Chart Wizard” button in the Excel toolbar. Select “XY (Scatter)” with lines but no points, and click “Finish.” This will give you a good basic graph that you can edit or embellish as you wish. You can adjust the graph scales (or select data peak-by-peak and plot a series of new graphs) to zoom in on a peak. Excel’s cursor can display interactively the (x,y) coordinates of any data point you point to once you click on a graph to select it.

7. Bogus Peaks:

Since there are impurities in the Na lamp that emit wavelengths in our range, here, to save you time, are approximate air wavelengths of the peaks you are likely to see. They vary in intensity, and you may not see them all (or you may see a few more than these!), but this list should be a big help to you. These values have been rounded to the nearest Å, which should be sufficient for their identification.

<table>
<thead>
<tr>
<th>3342</th>
<th>4191</th>
<th>4260</th>
<th>4316</th>
<th>4425</th>
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<td>4300</td>
<td>4366</td>
<td>4554</td>
<td>4935</td>
<td>5496</td>
<td>5651</td>
</tr>
</tbody>
</table>

In addition, you may see two or three Hg lines just past the 3650 Å Hg calibration line. They fall in the 3655–3665 Å range and can be ignored.

The most intense lines in your spectrum, by far, are the Na “D lines” described in the theory handout for this lab. The monochromator can produce exquisitely narrow and precisely located signals for these two lines, but to do so, one has to narrow both the entrance and exit slits quite a bit to increase the resolving power of the monochromator. Your spectra will probably show very rounded or off-scale (saturated) peaks for these lines, which makes assigning precise wavelengths to them difficult. To save you the misery of trying to pick a center wavelength for these peaks, here are their precise air wavelengths: 5889.950 Å and 5895.924 Å.