Reflectance spectroscopy lab

Read these instructions (including the Appendix) thoroughly and carefully before you get started. Please cite any references you use, or discussions with classmates. However, as always, your write-up of what you turn in for grading must be *your own work*, *in your own words.*

We have discussed reflectance spectra of meteorites and asteroids, but not much about what has to be done to obtain those spectra. Naturally, you learn most if you try to do it yourself. In this assignment, you will be taking a reflectance spectrum of a powdered rock sample that I will provide. You will check out the Ocean Optics Spectrometer and the rock sample (from Bert Orosco in the LPL main office). You will do your experiment in the lab room 330. Several computers in that room have the Ocean Optics software installed. In one of the cubbies near the south door, there is a light bulb, complete with dimmer switch; it can serve as your light source. There are also lab stands, with a variety of connectors, either in the cubby or next to the computers.

I suggest working in groups of two or three. It is not required, but it is a smart choice to get a better result, because you will have more hands (for handling the sample and equipment) and more brains (for problem solving). These instructions are deliberately not very detailed, in order to maximize chances that you will exercise your ingenuity.

Your assignment is to (a) obtain a reflectance spectrum of the crushed rock sample[1], and (b) write a report to explain (i) how you obtained it (in enough detail that if I really like the spectrum, I can go in and reproduce the conditions accurately), and (ii) discuss the spectrum you obtain, answering such questions as:
What mineral or minerals seem to be present?[2] What did you do to guarantee that you are looking at the reflectance of the sample, and not reflections off whatever you put the sample on? If you wanted to do this right ("professionally"), what improvements would you want in your detector or light source? And anything else you would want to do differently.

A description of how to operate the Ocean Optics Spectrometer is given in the appendix. Read that appendix for several cautions **BEFORE** you get started. Take care that the light you are recording is reflected from the sample, and is not the light bulb shining directly on the fiber or the room lights shining either directly or indirectly on the fiber.

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[1] The green mineral sample is what I expect analyzed for this assignment. But there are additional samples provided to you, including a meteorite sample; experiment with some of those and include their analyses in your report for extra credit.

[2] There is a good spectral library at http://speclab.cr.usgs.gov/ or http://speclab.cr.usgs.gov/spectral.lib06/ds231/datatable.html (the first links to the second). Obviously, I won't inform you what the sample is, but I will tell you that it is a silicate, so you can concentrate on the silicates in the library: olivine, pyroxene (various pyroxenes are listed as augite, bronzite, diopside, enstatite, hypersthene, and pigeonite), and feldspar (albite, anorthite, bytownite, microcline, oligoclase, and orthoclase).

Appendix: Ocean Optics Spectrometers

To take reflectance spectra, you will need to use an Ocean Optics spectrometer, with the Ocean Optics software running in a Windows-based computer. Advanced computer skills are not required. However, if no one in the group is familiar with a Windows-based environment, try to rearrange your group so that your group has someone who is.

First, you'll need to put the electronics together. There should be a total of three items, a box and two cables. The box is the spectrometer. One of the cables connects the spectrometer to the computer (there's a USB port on one end of the cable, and a connector on the other end that will only attach to the spectrometer in one place). The other cable is a fiber optic, or light pipe. It should screw into the other opening on the spectrometer. Now, light going into the tip of the fiber optic should reach the spectrometer. Note: Fiber optics are meant to be bent, and you will definitely need to bend the fiber optic. However, fiber optics are NOT meant to have sharp turns in them. If you try to put a sharp crease in the fiber, you will break the fiber (though it may not be obvious at first). So take care that all turns are curves, not sharp bends.

To start the software, click the icon that says "Ocean Pacific" or "OOIBase32". The screen should then display a graph.

On the x-axis (horizontal axis) is wavelength. At the start, the wavelengths run from about 340 nm to 1020 nm (you'll learn to adjust this range later). For comparison, visible light ranges from about 400 nm to 700 nm. So at the left edge of the screen, the computer is displaying ultraviolet wavelengths, and much of the right half of the screen is infrared.

On the y-axis (vertical axis) is intensity. When you start the program, this should go from 0 to about 4000. This is the count of the number of photons at a particular wavelength.

If you lightly cover the business end of the fiber optic, you should see something very much like a flat (horizontal) red line near the bottom of the graph. There should be very little light getting to the detector right now, but because there might be a few light leaks, and because the electronics are not perfect[3], you may see a few short vertical spikes here and there from time to time. If you point the fiber optic at the ceiling lights, you should see a spectrum on the screen; this spectrum will change as you move the fiber optic around.

Use the movable stand with adjustable clamps to clamp the fiber optic, if you wish. For many tasks, this will work very well (it keeps the spectrum on the screen from jittering too much), but you can take the fiber optic out to point at things. Once again, the fiber optic is very flexible, as long as it is curving, but don't try to crease it or make a perfect right angle turn without a bend.

Note: The electronics can only count to about 4000. As you point at the room lights (or if the fiber optic is too close to the light source), you will probably find situations where you're getting more and more counts at most wavelengths, but the lines that were the strongest never go any higher than 4000 (actually 4,096). What's happening is that the electronics is **saturating** at 4096. You'll usually want to keep the number of counts less than 4000. One way to do this is to change the "integration" (near the top of the screen) to a smaller number, which reduces the

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[3] They occasionally send a pulse even when there is no incident light; this is called "dark counts" and you'll deal with it later.

length of time over which the software counts the photons (which is usually a tiny fraction of a second).

A good way to get started is just to play around with the setup, while pointing the fiber optic at the room lights. Even when the fiber optic is clamped to the stand, the spectrum may still be time-variable or jittery, making it hard to make any measurements. What you need is a stationary spectrum. To get this, first get a spectrum where the highest intensities are near the top of the screen, then take a “snapshot” with the software. To take a “snapshot”, find the icon near the top left that looks like a camera. When you have a spectrum that you like (the highest spikes get almost, but not quite, to the top of the screen), click this icon; this will freeze the screen. If you click the same button a second time, the screen will unfreeze again, allowing you to take other spectra. When you have a snapshot you like, you are ready to move on. You should be able to print out “snapshot” spectra on the printer. If something there is not working, then just save any spectra you want to files, but make sure you give the files unique names that you will be able to identify for printing later.

There are two ways to extract the exact wavelength of a particular line.

One way is to zoom in on a particular line. Use the icon that is a cross with arrows in all four directions. If you click on this, you can set the scale for both axes. To find the wavelength of a line, just keep setting the scale closer and closer to it. You should be able to find the wavelength to within 1 or 2 nm.

The other way to find the wavelength is to use the cursor: Click on the “Toggle Cursor” icon (near all the right and left arrows at the top of the screen). You should then see a vertical green line on the screen. In the lower left corner of the screen, it will tell you the wavelength the line it located at (something like “657 nm”), as well as the number of counts at that wavelength (for example, 3200). There is a number in between those two that you should ignore. To move the cursor, you can use the yellow arrows that will now be highlighted in the toolbar at the top, or you can use the keyboard arrows, or use the mouse cursor to click where you want to move the green line.

Reflectance spectra: To get a reflectance spectrum, you have to know the spectrum of the light that is being reflected. Since we want to have a light source that covers a fairly broad spectral range, we will use a highly sophisticated piece of equipment – a light bulb. You should find a light bulb in a cabinet in the southwest corner of the lab. The light bulb is connected to a dimmer switch; so you have another to adjust the amount of light the spectrometer receives (other than moving the fiber optic around). However, if you do adjust the dimmer switch, it changes the temperature of the light, Wien’s Law kicks in, and the light bulb’s spectrum changes.

To generate a reflectance spectrum, we will need to take the ratio of “the light reflecting off the sample” to the “incident light”. For the “incident light” we probably want to use the light reflected off a plain white surface, such as a white sheet of paper. Also, any electronics will have some “dark” counts – it will read a few counts even in the dark. So we will need to take three spectra.

1) With just a white surface, and a spectrum that you like (peak near the top of the screen), go to the menu along the top, click “Spectrum” and then click “Store Reference”. That will store the spectrum that is on the screen as the one to which you will compare the others.

2) **Without moving the fiber optic or the bulb**, turn off the power to the bulb and/or cover the end of the fiber optic. This should give you a line near the bottom of the screen. This represents the "dark" counts (if the room lights are on, the spectrum of those may show up, too, but that's probably OK). Click "Spectrum" and then click "Store Dark". That will store the spectrum that's on the screen as the background that gets subtracted out of every measurement.

3) Finally, place your sample on the white surface so that you are able to get the reflected light off it. Click the blue "T" (for Transmission, but the calculations for reflectance are identical, so it works fine) underneath the word "Spectrometer". The computer will now display reflectance spectra. What you are now measuring is the fraction of the original light that gets to the detector at each wavelength. If **S** is the number of counts at some wavelength in the sample spectrum, **R** is the number of counts in the reference spectrum at that same wavelength, and **D** is the number of counts at the wavelength in the dark spectrum, then the number now displayed is 100*[(**S**-**D**)/(**R**-**D**)]. Change the y-axis scale, using the icon with the cross-hairs, to have a maximum somewhat larger than 100%, for example, 150% (otherwise, it is hard to see things that are at 100%). If there is nothing other than the original background, you should see a flat line very close to 100%. It may get very jagged at the edges, because there isn't much light coming from the light bulb there, so the statistics are pretty bad.

Some things to consider:

None of these are particular requirements, they're just ideas:

If you want to get a better spectrum at the UV or IR end of the spectrum, you can do the whole procedure again with a longer integration time, but just remember that the middle of the visible will presumably now be saturated, so the data there will be meaningless.

When figuring out how much sample to use, and where to put it, remember that you want to fill the fiber optic's field of view with the sample. You may want to find a way to use a dark object to map out the area that you need to cover with sample.

The rock powder source is limited(!), so please return as much of it as possible to the container when you are done (this is another reason to put it on a piece of paper).