

Spectrophotometry

Danial J. Neebel, Joseph R. Blandino, and David J. Lawrence,
College of Integrated Science and Technology
James Madison University

Instructor's Portion

Summary

In this experiment, data collection is simplified to allow students to focus on the measured phenomena. They will measure the transmittance as a function of wavelength for three different materials, and calculate the absorption coefficient as a function of wavelength for two of them.

LabVIEW facilitates the data taking process by recording the measurements and doing some simple mathematics. In essence, the use of LabVIEW converts older analog spectrophotometers into digital spectrophotometers with manual dials and setup. This is the best of both worlds; students get hands-on experience with the equipment without being burdened by hand recording of data.

The lab uses a VI written to work with a spectrophotometer that has an analog voltage output corresponding to the transmittance (signal range is from 0 to 1V).

Uses

The spectrophotometry lab has applications in electrical engineering, mechanical engineering, chemistry, and physics. Chemists may want to measure the transmittance through chemical samples to determine the sample elements.

Equipment List

- Computer running Windows, Macintosh, Linux, Sun, or HP-UX (visit http://www.ni.com/labview/lv_sysreq.htm for requirements specific to your operating system).
- Breadboard Connector Starter Kit from National Instruments (part number 777448-40)

- LabVIEW Full Development System
- PCI-6024E Data Acquisition Board
- SC-2075 Breadboard Connector
- SH68-68-EP Shielded Cable
- Spectrophotometer (i.e. Hitachi 100-10 UV/VIS)
- Silica, glass, plastic, color filter, and sunglass (green, gray-brown, metallic, and polarized) samples.
- A micrometer or calipers used to measure the thickness of the materials.
- Adhesive tape for securing samples to the sample holder of the spectrophotometer.
- A piece of black construction paper used for calibrating the spectrophotometer.
- Websites
 - Hitachi — www.hitachi.com
 - National Instruments — www.ni.com

Setup

Follow the steps listed to prepare the workstations for this experiment. The instructions assume you are using the equipment list shown previously.

Note: Most of the manuals that are referred to ship with National Instruments hardware and software. If you can't find your hardcopy of the manuals, you can get them online at <http://www.ni.com/manuals>. If you encounter problems during setup, contact technical support at <http://www.ni.com/support>.

Before the Day of the Lab

1. Install LabVIEW (see the *LabVIEW Release Notes* for your version of LabVIEW).
2. Install your PCI-6024E board (see the *6023E/6024E/6025E User Manual*).
3. Configure the SC-2075 Breadboard Connector (see the *SC-2075 User Guide*).

4. Cable the PCI-6024E to the SC-2075 Breadboard Connector with the SH68-68-EP.
5. Configure the PCI-6024E board (See the *NI-DAQ Release Notes* for your version of NI-DAQ).
6. Create the following directory in your LabVIEW folder:
\\LabVIEW\Experiments\Spectrophotometry.
7. Copy **Spectrophotometry.llb** into the Spectrophotometry folder you just created.
8. Connect the analog output of the spectrophotometer to analog input channel 0 (CH0) on the SC-2075 Breadboard Connector.
9. Any other setup is specific to your own spectrophotometer.
10. Conduct a run-through of the lab procedure the students will perform.

On the Day of the Lab

1. One of the materials the students test in this lab is silica (SiO_2). Measure and record the thickness of the silica sample.
2. Because of the brittle nature of this material, install the silica sample into Position 2 on the spectrophotometer before the students arrive for lab.
3. Prepare other sample materials at the workstations.
4. Turn on the computers.

References

- John A. Allocca, *Transducers: Theory and Applications*, Reston Pub. Co.
- Wheeler, Anthony J. and Ganji, Ahmad R. (1996), *Introduction to Engineering Experimentation*, Prentice-Hall Inc. Englewood Cliffs, NJ.

Student's Portion

Introduction

In this experiment, you will use a pre-written LabVIEW program to display and record the transmittance data from a spectrophotometer. You

will compare the characteristics of several transparent materials over the wavelength range from the near ultraviolet to the near infrared.

Objective

- To gain experience with spectrophotometry as a technique for measuring the optical properties of materials.
- To compare the transmittance of several transparent materials (such as silica, borosilicate glass, polyethylene, and plastics used for sunglasses) over the wavelength range from the near ultraviolet to the near infrared.
- To determine the absorption coefficient for borosilicate glass and plastics used for sunglasses.

Theory

No matter how transparent a material may appear, in general it does not transmit all wavelengths (colors) of light equally. A spectrophotometer is an instrument that measures the fraction of incident light that passes through a sample as a function of the wavelength of the light. This fraction is called the transmittance and is an important material property. Some spectrophotometers can also measure reflectance, which is the fraction of the incident light that is reflected. A spectrophotometer contains one or more light sources to provide the incident light. In the Hitachi spectrophotometers you are using, a tungsten lamp provides light in the visible and infrared portions of the spectrum, and a deuterium lamp provides the ultraviolet wavelengths. A diffraction grating breaks the light from these lamps into its spectra. By rotating the grating with a knob, you control which wavelength (color) goes through a slit opening and hits the sample. Any light that gets through the sample is detected by a photodiode, which converts the transmitted light to an electric current. This electric current is amplified and displayed on a meter.

In this experiment, rather than using the analog meter on the spectrophotometer, you will use a LabVIEW VI to acquire and display your data. This will make data collection substantially easier. You will compare the transmittance of several transparent materials (such as silica, borosilicate glass, polyethylene, and plastics used for sunglasses) over the wavelength range from the near ultraviolet to the near infrared. In this way, you will be able to determine whether the sunglass samples block all the “harmful” ultraviolet light.

Pre-Lab Preparation

- Read the theory section in this experiment.
- Bring the following items to lab:
 - This experiment.
 - A virus-free, formatted 3.5-inch floppy disk.

Workstation Details

- A computer with National Instruments LabVIEW software.
- National Instruments DAQ board (inside computer).
- National Instruments SC-2075 Breadboard Connector.
- A Hitachi 100-10 UV/VIS spectrophotometer or similar spectrophotometer.
- Silica, glass, plastic, color filter, and sunglass (green, gray-brown, metallic, and polarized) samples.
- A micrometer or calipers.
- Adhesive tape.
- A piece of black construction paper.

Lab Procedure

1. Turn on the spectrophotometer power.
2. Turn on the tungsten lamp (switch labeled W).
3. Turn on the deuterium lamp (switch labeled D2).
4. Launch LabVIEW.
5. Open the **Spectrophotometry.llb**. This file is located under \\LabVIEW\Experiments\Spectrophotometry.
6. Open the sample chamber and remove the four-position metal sample holder. Position 1 will be left uncovered. Position 2 should already be covered with a piece of silica (SiO_2 , pure quartz glass). If the silica is not present, ask your instructor for assistance.
7. Select two other samples for measurement. **At least one of these should be a portion of a sunglass lens.**

8. Attach these two samples over openings 3 and 4 of the sample holder with adhesive tape. Do not block any of the openings with tape and make sure that the samples and tape do not interfere with placing the sample holder into the sample chamber. If the samples extend too far below the openings, you may experience problems.
9. **Record which sample is in each position on your data sheet**, identifying which type(s) of sunglass sample(s) you are characterizing. Four types of sunglass samples are available: green, gray-brown, metallic, and polarized. Do not place the sample holder in the sample chamber yet.
10. Select the **%T** mode on the spectrophotometer.
11. The photodiode detector is in the sample chamber, on the left side. Cover the photodiode with black paper, close the sample chamber cover, and adjust the **0%T Adj** knob on the spectrophotometer so that the analog meter reads zero. **From now on, do not change the 0%T Adj knob setting.**
12. Remove the black paper, place the sample holder in the sample chamber, and close the cover. The sample chamber cover must be closed whenever you are making measurements.
13. Make sure that the **Filter** lever is in the **open** position.
14. There are three controls on the spectrophotometer that you will need to use as you collect your data. The **Spectrophotometry VI** will prompt you to adjust them as necessary. These controls are the **Wavelength** knob, on the top, the **UV/VIS** lever, on the back, and the **100% T Coarse Adjust** knob on the right side.
15. Run the **Spectrophotometry VI**. The VI should prompt you to “Set new wavelength on both the spectrophotometer AND on this panel and click on ‘Enter Wavelength.’” Begin your measurements by setting the **Wavelength** knob to 200 nm and entering 200 nm into the **Wavelength** control on the **Spectrophotometry VI** front panel. You can then click on **Enter Wavelength** as instructed. The VI will instruct you on what to do next. *It is important to keep track of which position the sample holder is in and which sample is in each position of the sample holder.*

If you have any questions, ask your instructor for assistance. The **Spectrophotometry VI** front panel has a table display and a graphical display. The table column in the table labeled **Ref** and the plot on the graph labeled **Reference** give the “raw” spectrophotometer output when the sample holder is in position 1 (the open position). This value is divided into the spectrophotometer output when the sample holder is

in positions 2 (silica), 3, and 4 to determine the transmittances of the samples in those positions, which are also displayed and labeled **T2**, **T3**, and **T4**.

16. After you use the **Spectrophotometry VI** to acquire data for $\lambda = 200$ nm, repeat the process for wavelengths of 225 nm, 250 nm, 275 nm, 300 nm, and so on, up to $\lambda = 950$ nm. *As you collect your data, it is important to keep track of which position the sample holder is in and which sample is in each position of the sample holder.*
17. After collecting all your data, be sure to save it on a floppy disk by pressing the **Save Data** button on the Front Panel of the **Spectrophotometry VI**. Open the file with Excel and check your data. The first column contains wavelength data; the second column contains **T2**, the transmittance of the silica sample in position 2; the third column contains **T3**; the fourth column contains **T4**; and the fifth column contains the “reference” data, which is the “raw” spectrophotometer output when the sample holder is in position 1 (the open position).
18. Turn off the **Spectrophotometry VI**, the W lamp, the D2 lamp, and the spectrophotometer.
19. Leave the silica sample in place, but remove the other two samples.
20. Use calipers to measure the thickness of your sunglass sample(s).

Lab Report

You must submit an informal laboratory report. Your report should also contain the following information:

- Prepare a table of the data collected by LabVIEW.
- Graph your data for silica and the other two samples with the transmittance on the vertical axis and the wavelength on the horizontal axis.
- You know that the refractive index varies from material to material, and for a given material, the refractive index varies with wavelength. Because you lack sufficient data, ignore these effects and assume that the refractive index of your samples is 1.458 at all wavelengths. (This is the actual value for silica at $\lambda = 589$ nm.) Use this number and your transmittance data to estimate the absorption coefficient, α , for the silica sample and *one* sunglass sample over the 200 nm to 950 nm wavelength range. Perform the required calculations in new columns in your spreadsheet. You will need to know the sample thicknesses.

You measured the thickness of the sunglass sample, and you can obtain the silica thickness from your instructor. Some values may be impossible to compute due to insufficient resolution in your data. Do not plot values for these data points, but do discuss them in your report.

- Prepare a graph of your absorption coefficient data for silica and one sunglass sample, with α on the vertical axis and the wavelength on the horizontal axis.
- Do your sunglass sample(s) block all of the UVA (315-400 nm) and UVB (280-315 nm), as promised by the manufacturer? How do you know?