

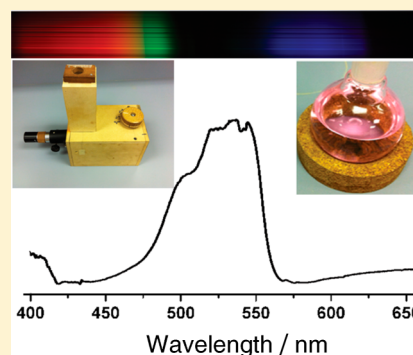
Developing Tools for Undergraduate Spectroscopy: An Inexpensive Visible Light Spectrometer

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ABSTRACT: The design and implementation of an inexpensive, high-resolution Littrow-type visible light spectrometer is presented. The instrument is built from low-cost materials and interfaced with the program RSpec for real-time spectral analysis, making it useful for classroom and laboratory exercises. Using a diffraction grating ruled at 1200 lines/mm and blazed in the first order, the spectrometer was found to have a resolution ($R = \lambda/\Delta\lambda$) of 7500 (0.07 nm) at 525 nm allowing for detailed spectroscopic experiments such as an analysis of the iodine vibronic spectrum or multiplet line splitting in the solar spectrum. The simple design of the spectrometer makes it possible to exchange gratings of different lines/mm easily. The instrument provides a means of incorporating macroscopic experimental observations of visible spectroscopy into the undergraduate chemistry program in a practical hands-on way while avoiding the “black-box” nature of many modern spectrometers. Special attention is given to the possibilities such an instrument presents for the introductory chemistry curriculum and example demonstrations and experiments are provided.

KEYWORDS: First-Year Undergraduate/General, Second-Year Undergraduate, Demonstrations, Physical Chemistry, Laboratory Instruction, Hands-On Learning/Manipulatives, Dyes/Pigments, Laboratory Equipment/Apparatus, MO Theory, UV-Vis Spectroscopy



Spectroscopy is an integral part of modern chemistry, allowing chemists to study the structure and properties of atoms and molecules. Unfortunately, many undergraduate students are not typically introduced to the fundamental principles that lie behind these techniques until the last years of their education. Some educators have identified this as a potential problem and suggested in depth discussions of different spectroscopies be introduced at an earlier stage in an undergraduate chemistry education.¹ For example, a recently published introductory chemistry textbook, *Chemistry: Human Activity Chemical Reactivity*,² integrates basic types of spectroscopy early and connects them to ideas of molecular structure. This educational strategy is part of a larger trend moving away from an atoms-first (models-first) approach to an experimental-facts-first approach, which emphasizes the experimental methods and results before the atomic and molecular theories. Not only does this approach emulate the scientific process more accurately, but it also means that interesting chemistry can be introduced earlier in a program, allowing students to see the beauty and depth of chemistry at a time when they are deciding on their educational path.^{3–5} Understanding the macroscopic properties of molecules and atoms also contributes to a more robust understanding of chemistry. An important model for chemical education suggests that chemistry can be best understood when students can connect the symbolic, the molecular, and the macroscopic.⁶ Spectroscopy can provide the macroscopic observations needed for students to connect theory to the physical world.

One challenge to introducing spectroscopy early in an undergraduate chemistry curriculum is the lack of experimental resources available to students. This is particularly true for introductory chemistry courses, where class sizes are often large and the many laboratory sections limit the availability of spectroscopic tools. Attempts have been made to provide inexpensive spectroscopic tools that allow undergraduate students to understand how the instruments function,^{7–14} as well circumvent the need to have direct access to instruments via remote access.¹⁵

A simple spectrometer is developed that can be used in both a laboratory environment and in classroom demonstrations to expose students to visible spectroscopy.^{16–18} Visible spectroscopy plays an important role in chemistry and biology as a technique for measuring the energy of electronic excitations and determining concentrations of dye molecules in solution. Visible spectroscopy can also be a powerful way to teach basic principles of spectroscopy because it makes use of the part of the electromagnetic spectrum that students are most familiar with. However, visible spectrometers that can achieve spectral resolutions below 0.1 nm are typically expensive, \$5000 CDN or more, limiting the number of spectrometers available to students. Lower-cost commercial spectrometers and other home-built spectrometers such as the periscope-type DVD spectroscopy can provide tools appropriate for many undergraduate investigations;¹⁴ however, the availability of a

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spectrometer that can achieve resolutions greater than 0.1 nm for spectroscopic investigations is also desirable. A spectrometer is described that can:

- Be assembled by students, allowing them to visualize the basic operation of a spectrometer while achieving resolution greater than 0.1 nm for more advanced spectroscopic experiments.
- Connect the observation of the sample color to the spectral image and to an absorbance plot of the spectrum, that is, connect macroscopic observation to symbolic representation.
- Be created from inexpensive materials so that students can have access to the instrument even in resource-limited contexts.

The spectrometer was built by an undergraduate student and constructed for less than \$250 CDN (excluding the digital cameras or software). The resolution and ease of use make the spectrometer ideal for laboratory experiments and classroom demonstrations.

■ DESIGN, CONSTRUCTION, AND CALIBRATION OF THE SPECTROMETER

A simple prototype spectrometer was constructed based on the Littrow design (Figure 1).¹⁹ The materials required for the construction are listed in Table 1 and shown in Figure 2, along with the approximate prices of each component in Canadian dollars.

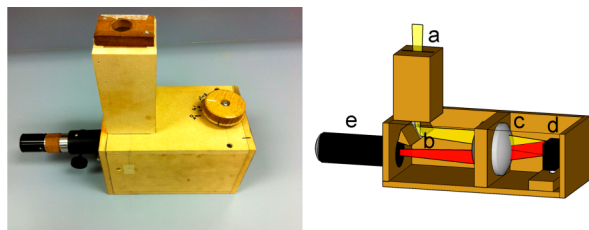


Figure 1. The Littrow spectrometer: (left) the finished spectrometer and (right) a diagram of the Littrow spectrometer showing the key optical pieces (a) the slit, (b) pickoff mirror, (c) lens, (d) diffraction grating, and (e) eyepiece.

Table 1. A List of the Materials Used To Build the Spectrometer and Approximate Prices

| Material | Purpose | Cost/Canadian dollars | Availability |
|----------------------|------------------------------|-----------------------|--------------------------------|
| Diffraction Grating | Diffraction Grating | \$105 each | Edmund Optics |
| Photocopier Lens | Collimator and Focusing Lens | \$10 | Photocopying Surplus Stores |
| Front Surface Mirror | Pickoff Mirror | \$5 | Department or automotive store |
| Razor Blades | Slit | \$2 | Department Stores |
| Wood | Frame | \$10 | Department Stores |
| Screws, Bolts, Nuts | Construction | \$5 | Department Stores |
| Camera | Detector | \$200 | Department Stores |
| Eyepiece | Detector | \$30 | Astronomy Stores |
| Focuser | Detector Mount | \$30 | Astronomy Stores |
| T-Ring | Detector Mount | \$15 | Astronomy Stores |
| T-Adapter | Detector Mount | \$16 | Astronomy Stores |

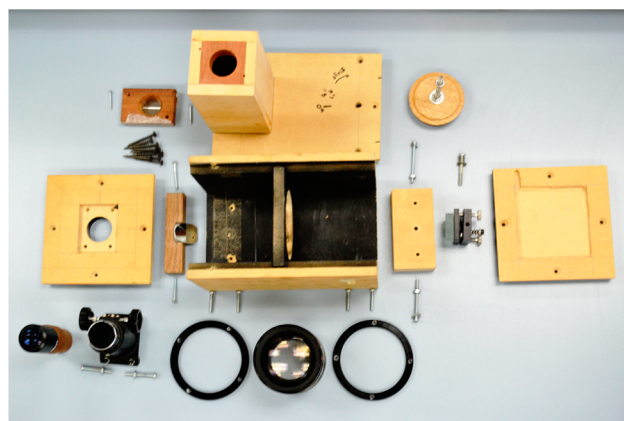


Figure 2. The disassembled spectrometer showing the basic components used (see Table 1).

The spectrometer includes a pickoff mirror, lens, and diffraction grating (Figure 1). The pickoff mirror reflects incoming light toward the diffraction grating that separates light into the visible spectrum and reflects it toward the detector. The lens serves two purposes: it collimates the light reflected from the pickoff mirror so that the light projects an image of the entrance slit on the grating, and focuses the light reflected from the diffraction grating on the detector. The Littrow design's use of the lens as both a collimator and a focusing lens allows the spectrometer to be more compact and reduces its cost. In this spectrometer, a front surface mirror and a photocopy surplus lens with a 260 mm focal length were used. Two diffraction gratings were purchased from Edmund Optics: a 30 mm × 30 mm grating blazed at 500 nm with 1200 lines/mm (part number A 46077) and a 25 mm × 25 mm grating blazed at 500 nm with 300 lines/mm (part number NT64-403). The 300 lines/mm grating was used to collect images of the entire visible spectrum whereas the 1200 groove/nm grating was used for higher-resolution quantitative analysis. These gratings can be switched with ease and can be purchased depending on the desired applications.

The wooden frame includes a removable front, back, and top (Figure 2). The top includes a hollow pillar that provides an entrance for light into the spectrometer, whereas the front contains an opening over which a detector can be mounted. The inside of the spectrometer contains the pickoff mirror, lens, and diffraction grating, all placed on wood mounts. The mirror mount can be rotated to adjust the pickoff angle and direct the light from the slit to the collimating–focusing lens. The diffraction grating can be rotated to permit different parts of the spectrum to be directed toward the detector. The grating can also be rotated by a small amount along the vertical axis to help align the spectrometer. A standard telescope focuser was used as a detector mount.

To align the spectrometer, a helium–neon laser was pointed directly into the column on the top of the spectrometer. The pickoff mirror and grating angles were adjusted until the laser was centered on the detector. Focusing is done by adjusting the position of the detector to produce a sharp image of the spectrometer slit when viewed in the zeroth order.

The width of the slit can also affect the spectrometer's performance. The slit consists of two razor blades; the slit was optimized by adjusting the positions of razor blades by hand and observing the image of the slit through the detector until a sufficiently narrow line was observed. A simple single slit

diffraction pattern was produced using a helium–neon laser to determine the location of first- and second-order antinodes. From this, a slit width of $13\ \mu\text{m}$ was determined. After aligning the spectrometer, focusing the detector, and adjusting the slit width, the resolution of the spectrometer was determined. The resolution of the spectrometer with the 1200 line/mm diffraction grating was 7500 (0.07 nm). This was calculated by measuring the full-width half-max (fwhm) of a weak (unsaturated) spectral line due to Fe I at 516 nm. Figure 3

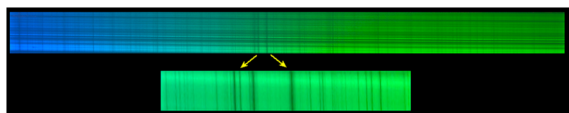


Figure 3. Calibration spectrum (top: 485.0–557.5 nm) with enlarged view of the magnesium triplet (bottom).

shows a test image taken of the solar spectrum using a Nikon D3100 as detector (discussed below). The top image shows the entire region between 485.0 and 557.5 nm. The hydrogen beta line (486.1 nm) is visible on the left edge of the spectrum with the magnesium triplet near center. The bottom image is a portion of this spectrum centered on the magnesium triplet line. Figure 4 shows the normalized intensity spectrum for this

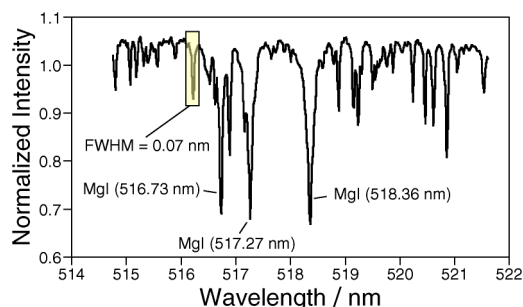


Figure 4. Normalized absorption profile for the magnesium triplet in the solar spectrum. fwhm measurements for the Fe line at 516 nm shown in yellow box.

region and also reveals the high resolution of this instrument. In addition to the magnesium triplet numerous other lines are present (many of which are Fe absorption lines.)

Different detectors can be used to capture images. The most basic detector is an eyepiece through which an individual can observe the spectrum directly. This detector is useful for quick observations and calibrations of the spectrometer, but cannot be used to capture images. Alternatively, a camera can be mounted onto the spectrometer as a detector. The choice of camera depends largely on the application. Commercial-grade cameras, such as the Nikon D3100, provide larger detectors that can view a wider range of the spectrum than lower-resolution cameras. Live-streaming cameras such as webcams can be used for class demonstrations and real-time experiments. The size of the pixels in the CCD detector affects the resolution: generally smaller is better. Additionally, most consumer cameras contain ultraviolet and infrared cutoff filters, which decrease the spectral range of the instrument.

Two cameras were used as imaging sources for the spectrometer: a Nikon D3100 and an Imaging Source DMK 21AU04. Each camera has a pixel size of $5\ \mu\text{m}$ on a side. The Nikon D3100 has a 4608×3072 pixel detector and has internal

IR and UV filters which limit the spectral coverage to the visible region only. It can save images or record videos. To mount the camera on the focuser, an appropriate T ring and T adapter were attached to the camera in place of the camera lens so that it could be interfaced with the spectrometer. The Imaging Source DMK has a 640×480 pixel detector and does not include any internal cutoff filters. It cannot save images or videos, but it can stream images directly to the analysis software for real-time analysis. Because the Nikon D3100 has a larger field of view and can save images, it is preferred for spectral analysis. The live-streaming capabilities of the Imaging Source DMK make it an ideal detector for live demonstration. Less-expensive cameras (such as webcams and even some cell phone cameras) were also used successfully, but images captured with these cameras are not presented here.

■ INTERFACING WITH RSpec OR OTHER SOFTWARE PACKAGES

The conversion of a spectral image into a transmittance plot can be achieved using the program RSpec.²⁰ This real-time spectroscopy software was developed by Tom Field primarily for applications in astronomy. RSpec can be used to process spectral images into absorbance spectra, either from image files or as a live-feed from the camera. This is a commercial product that currently sells for under \$100 USD. The package VSPEC²¹ is a freely available spectroscopic reduction program and is very similar to RSpec in capability. Another powerful and free program capable of displaying and analyzing spectra is ImageJ.²²

To correctly process a spectrum, RSpec must be calibrated using known emission lines, such as lines from a neon emission lamp. RSpec contains a library of emissions data for both stars and elements, which simplifies the calibration process. To achieve the best results for a calibration, the lines used for calibration should span the width of the spectrum. Although higher-order calibrations can be made, a 2-point linear calibration results in an accurate spectrum.

■ EXERCISES AND DEMONSTRATIONS

Spectroscopic experiments using the spectrometer described above can be performed by placing various samples between an incandescent light source and the spectrometer. The sample cell used for these experiments was a sealed 250 mL round-bottom flask (Figure 5). This simple experimental setup allows

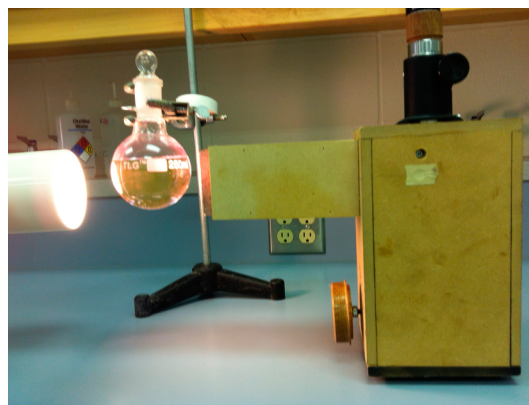


Figure 5. The experimental setup used to measure the visible absorbance of the dyes and vibronic spectrum of I_2 .

students to identify the main components of a visible spectrometer. The demonstrations and laboratory exercises described below were performed in an introductory chemistry class and in a second-year physical chemistry laboratory.

Constructing a Spectrometer

An important part of understanding any spectroscopic technique is to understand the way the spectrometer works. Although classroom discussions of spectrometer operations are useful, there is no substitute for hands-on experience with the different parts of a spectrometer and how they are put together. The spectrometer described here is simple enough to allow students to disassemble and reconstruct the spectrometer in a laboratory environment. Only the diffraction grating requires careful handling so that students do not damage the grating surface. Although this process is more involved than using a commercial spectrometer, it accesses a deeper level of student learning, emphasizing that an understanding of how the instrument works is as important to chemistry as the acquisition and analysis of the spectra.

Starting from a disassembled spectrometer, similar to Figure 2, students can be shown the key components of the spectrometer and guided through the assembly of the instrument. The assembly process can be completed in as little as 45 min, but may take longer depending on students ability and the amount of support available. A subsequent experiment, such as acquiring an atomic emission spectrum from a discharge tube, would demonstrate to the students that their "home-built" spectrometer is of high enough quality to reproduce the spectra they observed in class or in their textbooks. Further exercises to calibrate the spectrometer and determine the resolution can then be performed.

Absorbance of Dyes

Many introductory courses cover atomic structure and an introduction to molecular orbital theory. When teaching atomic structure, an experiment-first approach that is often used is an observation of the spectra of discharge tubes and the colored flames of methanol salt solutions. This connection of atomic structure to color is a useful way of grounding the complex theory in experimental observation. A similar technique can also be useful for discussing the importance of molecular orbital (MO) theory. Often, learning objectives for an introduction to molecular orbital theory are to make students aware that (i) molecules contain different orbitals than atoms, (ii) molecular orbitals are delocalized over the entire molecule, and (iii) electrons fill the molecular orbitals from the lowest energy up. When MO theory is taught using an experiment-first approach, experiments such as the paramagnetism of O₂ or the photoelectron spectrum of methane are presented. Although these experiments are excellent examples of the need for molecular orbital theory, they are not easily connected to the experiences of introductory students. Alternatively, the color of organic dyes can provide an experimental platform on which to build simple ideas of MO theory at the introductory level by connecting the theory to student experiences.^{23–28} By investigating the electronic absorbance of various organic dyes and how MO theory explains them,²⁹ students will comprehend the need for MO theory, particularly ideas of frontier orbitals and the HOMO–LUMO gap.

A demonstration of visible light absorption by food dyes can be performed as a demonstration or laboratory exercise using the spectrometer described. Different dyes can be prepared in 250 mL round-bottom flasks and placed in front of the visible

light source.²⁵ By observing the effects of placing the dye in front of the light, students can clearly understand the concept of light absorbance and its relationship to color. For example, when the food dye FD&C blue #1 is used, an absorbance in the red region of the spectrum can be observed (Figure 6A,B), and

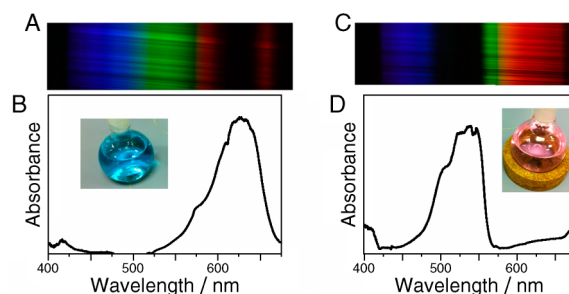


Figure 6. The dye absorption experiment. (A) The observed absorption spectrum and (B) absorbance spectrum for FD&C blue #1. (C) The observed absorption spectrum and (D) absorbance spectrum for FD&C red #28.

when FD&C red #28 is used (Figure 6C,D), absorbance in the green region of the spectrum can be seen. Students can clearly see that only a small quantity of the red or green light needs to be absorbed for the dye to be blue or red, respectively. This observation can overcome a misconception many students have that a blue dye will absorb all colors other than blue. Finally, it can be shown that the differences in absorbance are related to the molecular structure of the dyes by comparing the absorbance of FD&C blue #1 and FD&C green #3, which differ only by the presence of an alcohol functional group. Using these dyes as examples, students can see that the inclusion or exclusion of even a single functional group has an effect on the molecular orbitals of a molecule and hence the wavelength at which the molecules absorb. This exercise was performed as a demonstration in an introductory chemistry course as part of the lecture for 40–60 students.

The macroscopic observation and molecular understanding of spectroscopy can also be connected to the symbolic ideas behind the technique. If a background spectrum is collected, an exercise can be undertaken to process the spectra and create both transmittance and absorbance spectra. The student can instinctively see that the molecules in the dye are absorbing in the visible region. They can also see that the light source is not uniform across the visible spectrum when they acquire a background spectrum. By performing a background correction students can convert their light intensity data into a transmittance spectrum, $T = I/I_0$. They can then convert their spectrum into corrected absorbance plot, $A = -\log_{10}T$. The process of mathematically performing these conversions will provide a much richer learning experience than simply clicking the absorbance–transmittance button on a spectrometer.

Although the dye absorbance exercise can be performed in the laboratory, it can also be presented as a demonstration during a lecture using the live-streaming capabilities of a video camera or webcam interfaced with RSpec. With this demonstration, students are able to see the dye under investigation, the resulting spectrum of light shown in RSpec, and the absorbance plot shown in RSpec all at the same time. Different dyes can be swapped in and out easily to show the different absorbance properties of the molecules.

Vibrational Spectrum of I₂

The study of the visible light absorption spectrum of I₂ provides an example of the overlap of vibrational and electronic excitation and has been incorporated into many undergraduate physical chemistry courses.^{30,31} Although a complete analysis of the I₂ visible spectrum is best left to upper-level chemistry courses (see below), the spectrum does present opportunities to avoid misconceptions in the introductory chemistry class. Vibrational spectroscopy at an introductory level is typically taught while focusing on IR spectroscopy as a technique for identifying functional groups in organic chemistry or to help explain the properties of greenhouse gases. IR spectroscopy requires molecules to have a changing dipole moment during a vibration to absorb the radiation. This requirement may give students the false impression that only molecules with changing dipole moments have vibrational energy levels (Raman spectroscopy is not usually discussed at an introductory level). By showing students the electronic absorbance spectrum of iodine, one can eliminate this misconception while at the same time showing students that different types of energy transitions, that is, electronic and vibrational, are not mutually exclusive.

For more advanced experiments in an upper-level physical chemistry lab, the spectrometer provides high enough resolution for the detailed analysis of an iodine spectrum. The experiment offers a chance to explore spectrometer calibration, acquisition of spectra, conversion to transmittance and absorbance plots, and the numerical analysis of the spectrum to obtain vibrational parameters. First, the spectral window must be set up and the calibration spectra acquired; mercury and neon emission spectra can be used, ensuring that the emission lines at 546.0 nm for mercury and 585.2 nm for neon can be detected without moving the diffraction grating. Next a 250 mL round-bottom flask, equipped with a thermometer, containing a small quantity of I₂(s) can be placed between the slit of the spectrometer and a tungsten light source and heated with a heat gun to 70 °C. The resulting spectrum can be observed using the eyepiece and captured using the digital camera (Figure 7); a background spectrum of a 250 mL round-bottom flask should also be collected. All four images are transferred to RSpec, calibration using the mercury and neon emission images is performed, and the I₂ and background spectra are saved. By performing the background correction, the iodine spectrum can be presented as either transmittance or absorbance as discussed above. This exercise took approximately 1 h of experiment time and was performed as part of a second-year physical chemistry course.

The resulting spectrum (Figure 7) can be used to determine physical constants for I₂, including the fundamental frequency of oscillation, force constant, anharmonicity constant, and dissociation energy using a modified procedure described by Snadden.³⁰ It is helpful to provide students with a reference peak, that is, $\nu' = 28$ peak near 541.5 nm, to simplify the analysis and compensate for the fact that the spectrometer does not have a large enough field of view to observe all the vibrational peaks.³¹

A subsequent exercise can be used to show the temperature effects on the Boltzmann populations of the different vibrational bands. As shown in Figure 7, vibrational bands originating from $\nu'' = 0, 1$, and 2 (green, orange, red lines, respectively) can all be detected. As the temperature is increased, the peaks from the $\nu'' = 1$, and 2 bands increase in intensity, making it obvious that the number of molecules in

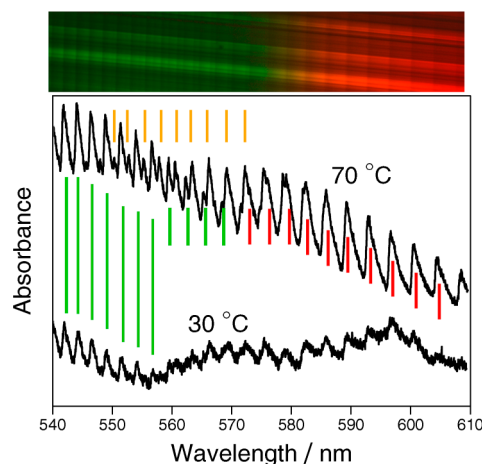


Figure 7. (Top) The iodine spectra obtained using the home-built spectrometer when the sample was heated to 70 °C. The spectra represent the absorbance plots of the sample at 70 °C (top) and 30 °C (bottom). The colored lines indicate the peaks from the different vibrational bands. The green lines indicate excitations from the $\nu'' = 0$ vibrational state, the orange lines from $\nu'' = 1$, and the red lines from $\nu'' = 2$. The nonlinear baseline at 30 °C arises from slight differences transmittance due to solid iodine depositing on the surface of the flask.

the excited vibrational states is increasing. Careful analysis can relate these increases to the Boltzmann equation. Such an effect can be shown in a live demonstration in the classroom or as an experiment in a physical chemistry course.

CONCLUSIONS

An inexpensive Littrow-type visible spectrometer has been constructed and found to be a useful tool for teaching spectroscopy to undergraduate students. Its high resolution allows for an accurate analysis of the iodine spectrum while still being versatile enough for use in classroom demonstrations. The inexpensive nature of the device, costing an order of magnitude less than comparable commercial devices, makes it an appealing option for supplying spectrometers to large introductory chemistry classes for use in laboratory experimentation or in resource-limited educational settings.

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Notes

The authors declare no competing financial interest.

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