Apparatus and Experimental Procedure

Apparatus: Shimadzu scanning UV-Vis spectrometer, model 2101-PC; quartz cuvettes (1.0 cm, 0.20 cm); thermostated cell holder; solid iodine.

Caution: Iodine is corrosive to the skin. Avoid contact with iodine crystals, and wash hands after use.

To obtain the spectrum of gaseous iodine, we will place a sample of crystalline iodine in a covered cuvette, which is then placed in a thermostated cell holder. The cell holder is warmed by recirculating water. The vapor pressure of iodine is such that the iodine should be warmed to **40-50 °C** in order to obtain a nice spectrum (i.e.: a reasonable concentration of I_2 (g) and thus a reasonable absorbance intensity).[9]

This spectrometer is a dual beam instrument, which means that the light from the source is split into two beams, one that passes through the sample, and one which does not. The absorbance spectrum is then calculated by the software from:

$$A = \log\left(\frac{I_o}{I}\right) \tag{23}$$

where I_o is the intensity of the light transmitted through the sample, and I is the intensity of the reference beam. It is common practice to obtain a baseline spectrum even with a dual beam instrument to account for any artifacts. You will obtain your baseline spectrum using an empty cuvette in both sample and reference compartments. The spectrum of iodine will then be obtained relative to an empty cuvette in the reference beam.

Almost all UV/Vis spectrometers are scanning spectrometers (as opposed to Fourier transform instruments, such as an IR or NMR spectrometer). A scanning spectrometer has at least one monochrometer that physically allows only a narrow band of light to pass through. Resolution on a scanning machine is directly related to the slit width of the monochrometer. For this experiment, we will be using a slit width of **0.1 nm**, with a slow sampling speed to improve the signal-to-noise (S/N) ratio. The wavelength range of interest is **450-600 nm**.

With warm water circulating through the sample holder, obtain a baseline spectrum using the appropriate instrument settings. You should always set the monochrometer to start at the low energy (red) end of the spectrum so that it will return there when the scan is finished. This reduces wear on the detector due to high-energy photons. Obtain a sample spectrum using the supplied cuvette containing iodine in the sample beam, and an empty cuvette in the reference beam. Save your data file to the hard disk as **I2F04XXY**, where XX are your initials, and Y is the spectrum number (start with 1 - you may need to take more than one spectrum).

In your notebook, tabulate roughly 12 experimental values for the band origins (approximated as the band heads) in nm. You will need to expand the scale on your spectrum **while still at the UV/Vis computer** to get a reasonable number of significant figures for wavelength. Notice that you cannot use the peak pick function because we are using **troughs**, not peaks, for band

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[9] A spectrum of iodine *vapor* must be obtained in order to be able to resolve the vibrational structure seen in Figure 4. This is because in the vapor phase there are very few intermolecular interactions, which serve to broaden the absorption bands.