## NICKEL QUANTIFICATION

Purpose: You will learn how to carry out serial dilutions and relate the amount of visible light absorbed by a solution to its concentration.

Background: In this lab, we will measure the *absorbance spectra* of a sample. Whenever light passes through a sample, some of it is absorbed. There are two different terms to define how much light a sample absorbs. Transmittance (T) is the ratio of the intensity of light that comes out of a sample over what went into it. Transmittance is usually expressed as a percentage.

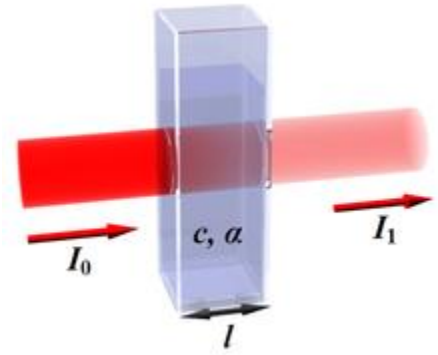



Figure 1. The intensity (I) is reduced as light passes through a sample.

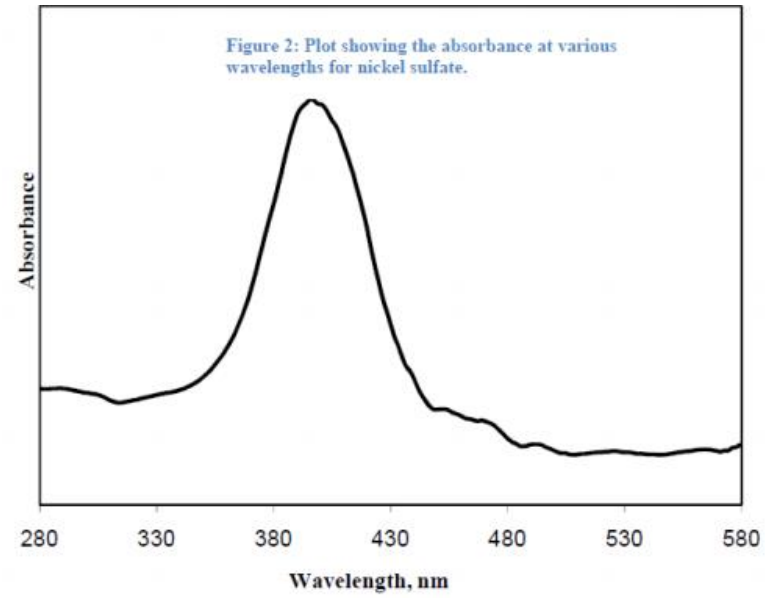
$$\%T = I_1/I_0 * 100 \qquad \text{(eq 1)}$$

The definition of absorbance (A) is given in eq 2.

$$A = 2 - \log_{10}(\%T) \qquad \text{(eq 2)}$$

Since both absorbance and transmittance involve ratios of light intensity, they are unitless quantities.

In solution, there are many interactions and, thus, many energy levels available to electrons transitioning from one orbital to another, so solutions do not absorb at just one specific wavelength. Instead, they absorb at a range of wavelengths, as shown in Figure 2.




Beer's Law (named after August Beer, nothing to do with beverages) relates absorbance (A) to the concentration of solute (c) (eq 3).

$$A = c \times \ell \times \varepsilon \qquad \text{(eq 3)}$$

$\ell$ is the length of the tube (*cuvette*) containing solution (1 cm in this lab), and $\varepsilon$ is a constant that relates to how intensely colored the solute is. First, you will make a nickel sulfate solution. Then, you will dilute it to make several solutions of known concentrations. The absorbance of these solutions will be used to construct a calibration curve.

Procedure: This is what you need to write down. Work in pairs.

Note – Pipets and volumetric flasks are precisely engineered glassware that provide much higher precision than beakers or graduated cylinders. All glassware in this lab must be clean but doesn't have to be completely dry. $Ni^{2+}$ is toxic; do NOT dispose of any of it down the sink or in trash cans. Do NOT put labels on the cuvettes; label the test tube rack instead.

1. Obtain from their designated areas one 100.00 mL volumetric flask, two 50.00 mL volumetric flasks, one 10 mL volumetric pipet, one 5 mL volumetric pipet, a pipet bulb, 7 spectrophotometer cuvettes, and a cuvette rack.
2. Carefully weigh, to the nearest 0.0001 g, 4 g of solid $NiSO_4 \cdot 6H_2O$ (mol. wt. = 262.9) on a plastic weigh boat. With the wash bottle, rinse the sample completely into the 100.00 mL volumetric flask. Fill the flask about half full with deionized water, swirl to dissolve the nickel sulfate crystals, and then fill the flask to the mark with deionized water. Carefully align the bottom of the meniscus with the line on the flask to get accurate results. The resulting solution is "Solution 1". Fill one spectrophotometer cuvette about 3/4 full (see Spec 20D instructions for a picture of an appropriate solvent level.)
3. To use a fixed volume pipet, squeeze the bulb, place the bulb gently but firmly against the pipet, and lower the pipet tip completely into the solution you want to transfer. Slowly release the bulb (don't go too fast or you might splash up into the bulb). Repeat this process until the solvent level is above the mark. When you remove the bulb, cap the pipet with your thumb. Wiggle your thumb to release small amounts of solution until the bottom of the meniscus lines up accurately with the marked line. Then carefully move the pipet to the new volumetric flask and release your thumb. Some solution is *supposed* to remain in the pipet tip. Rinse the pipet with the appropriate solution before each use. If the pipet has graduated marks, be sure to record the initial and final volumes, just as with a buret.
4. Prepare the following solutions in the 50.00 mL volumetric flasks. After diluting to the mark with deionized water and mixing well, fill a spectrophotometer cuvette with each solution. Calculate the concentration (M) of each of the known solutions (1, 2, 3, 4, and 5).

   To make Solution 2: Use 20.00 mL of Solution 1 diluted to 50.00 mL
   To make Solution 3: Use 10.00 mL of Solution 1 diluted to 50.00 mL
   To make Solution 4: Use 5.00 mL of Solution 1 diluted to 50.00 mL
   To make Solution 5: Use 5.00 mL of Solution 2 diluted to 50.00 mL

5. Record your unknown number and carefully dilute the solution to the mark with deionized water. Mix well and fill a spectrophotometer cuvette with your unknown.
6. Fill the remaining cuvette with deionized water.

7. Once all 7 cuvettes have been prepared, use a spectrophotometer to measure the absorbance of each solution. Follow the directions next to the Spec 20D, using 395 nm as the wavelength.

**Example Data Table:**

Mass of $NiSO_4 \cdot 6H_2O$ used _____

| | Absorbance |
|---|---|
| Solution 1 | _____ |
| Solution 2 | _____ |
| Solution 3 | _____ |
| Solution 4 | _____ |
| Solution 5 | _____ |
| Unknown _____ | Number of unknown _____ |

After completing the procedure but before leaving lab, write in your notebook a brief statement (two to three sentences) on the quality and reasonableness of the data you collected. Note what you might do differently if you performed the lab again.