

Lab 9

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 the last lab, we looked at the spectra of light emitted from different samples. 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 in. It is usually expressed as a percentage.

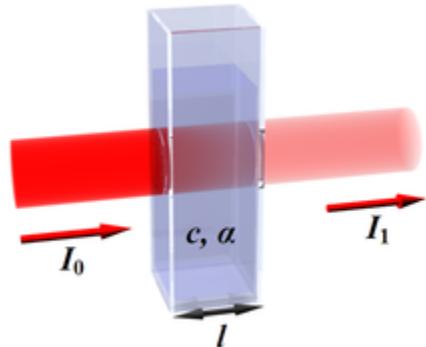
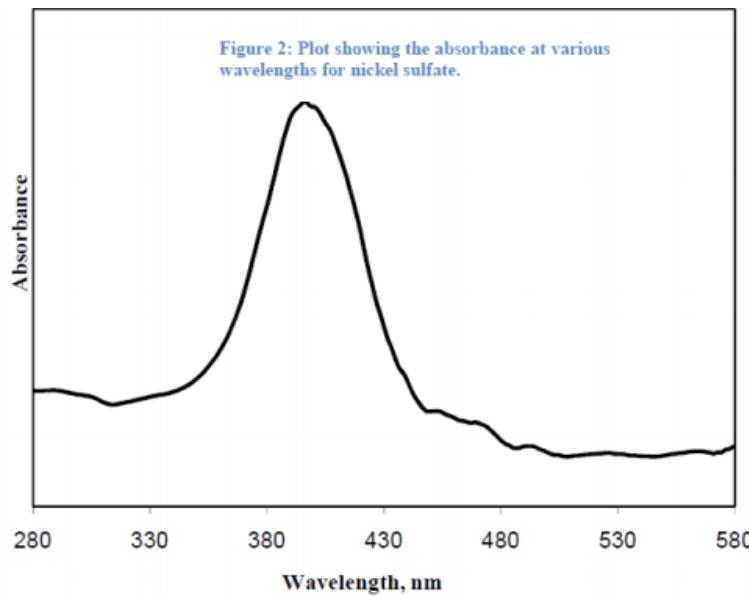


Figure 1. The intensity (I) is reduced as light passes through a sample.

In the previous lab, we observed the emission lines corresponding to electrons transitioning from one orbital to another. In solution, there are many interactions and more energy levels available, 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).

$$A = c \times \ell \times \epsilon \quad (\text{eq 3})$$

ℓ is the length of the tube (*cuvette*)

containing solution (1 cm in this lab) and ϵ is a constant that relates to how intensely colored the solute is. First we will make a nickel sulfate solution, then dilute it to make several solutions of known concentrations. The absorbance of

these solutions will be used to construct a calibration curve.

Procedure: Work in partners.

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. Carefully weigh, to the nearest 0.0001 g, 4 g of solid $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ (mol. wt. = 262.9) on a plastic weight 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 special test tube about $\frac{3}{4}$ full (see Spec 20D instructions for picture of appropriate solvent level.)
2. To use a fixed volume pipet, squeeze the bulb, place it 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.
3. 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

4. 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.
5. Fill the remaining cuvette with deionized water.
6. Once all 7 tubes 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 $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ used _____

Absorbance

Solution 1 _____

Solution 2 _____

Solution 3 _____

Solution 4 _____

Solution 5 _____

Unknown _____ Number of unknown _____