

# Honors 203/Chemistry 110

## The Determination of Iron in Water

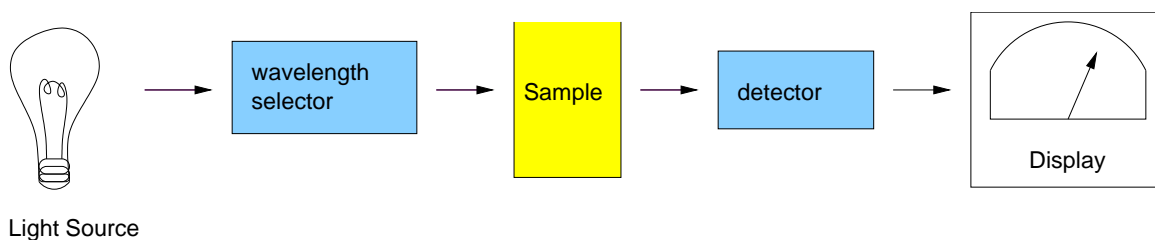
Iron is a common trace substance found in our drinking water. It is not considered a health hazard, however it can make water look and taste unpleasant. Excess iron in water can also cause staining of household fixtures and even your laundry!

Water drawn from underground wells is often naturally high in iron content. Iron is a common element in the earth's crust, and water collected from wells has first passed through many iron containing rocks and minerals on its way to the underground aquifer. Older plumbing systems with deteriorating iron-containing pipes is another possible reason one might find high levels of iron in water.

In this experiment, we will be using an instrument known as a spectrophotometer to determine the iron content of various water samples. Water samples are treated with 1,10-phenanthroline, which forms a bright red-orange complex with iron. This highly colored species absorbs visible light, and thus can be subjected to spectrophotometric analysis to determine its concentration.

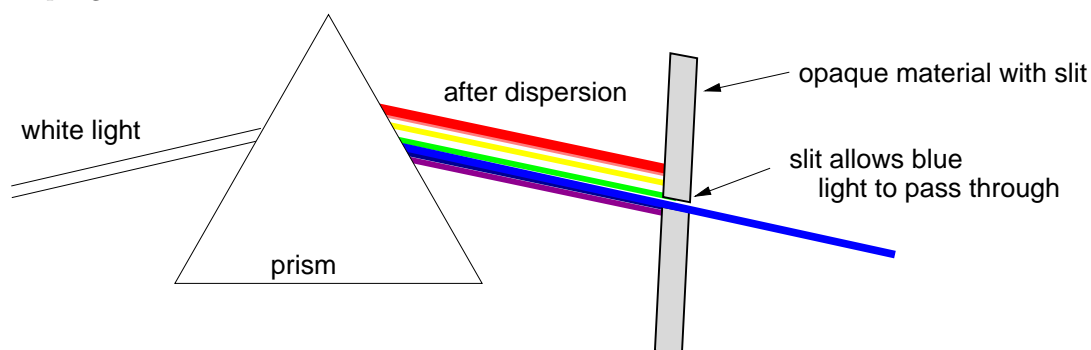
### UV/Vis Spectroscopy

A general scheme for the components of a spectrophotometer is below.



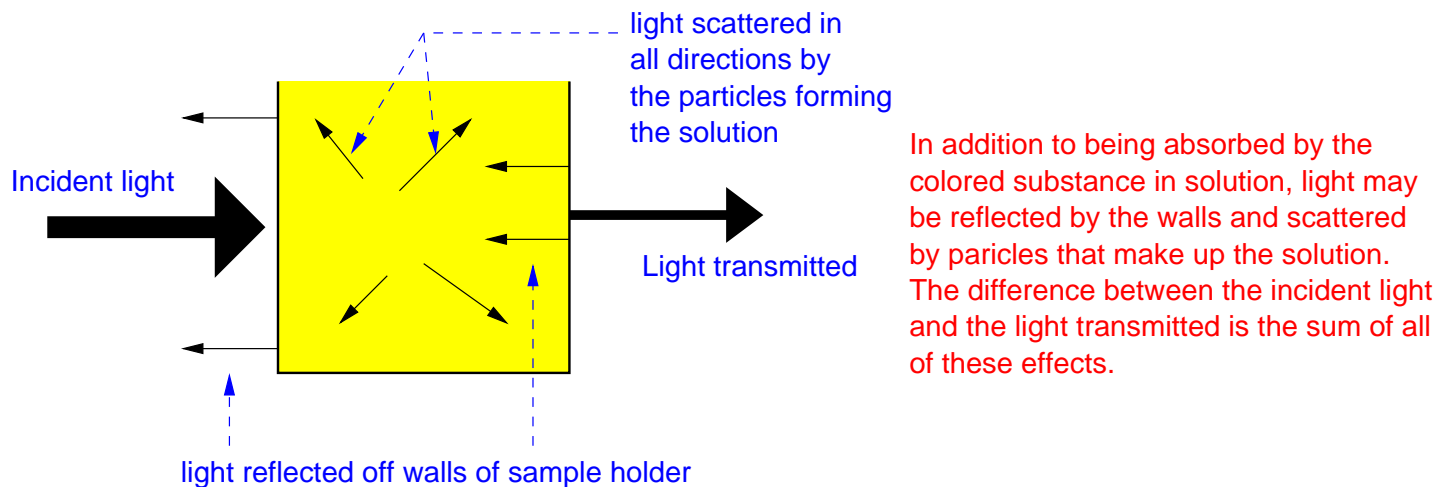
The first thing a spectrophotometer needs is a light source. A tungsten lamp emits light across the entire visible spectrum and is commonly used in spectrophotometers.

We often want to explore how a substance responds to a particular wavelength of light. A colored substance will interact with different wavelengths (different colors) of light differently. For example, a red substance may be very good at absorbing green light ( $\lambda \approx 530 \text{ nm}$ ), but absorb red light ( $\lambda \approx 650 \text{ nm}$ ) very poorly. So, we need a way to separate the wavelength of interest from the rest of the white light from the tungsten lamp. A component called a wavelength selector is used to accomplish this. A wavelength selector selects just one narrow wavelength region out of the entire spectrum. These wavelength selectors are more technically called monochromator. A monochromator includes something that disperses the various wavelengths of light. One such device is the familiar glass prism. White light enters the prism and is dispersed, or spread out, into its various colors. A particular wavelength can then be selected by moving around an optical slit. Another common dispersion device is a grating - a piece of metal or glass with microscopic grooves etched into it.



Next, the light beam passes through the sample. Some of the light may be absorbed, some may be reflected or scattered, while the rest passes through. The light transmitted is measured by a detector. The readings from the detector are processed and displayed for the user.

Because light passing through a sample can be scattered or reflected as well as absorbed, the amount of light transmitted through the sample is influenced by these factors as well.



Thus, it is common to compare the amount of light transmitted by a sample to the amount of light transmitted by a *blank* - a solution containing everything but the colored substance in an identical sample holder. When compared with a blank, the readings from the sample are reported as *percent transmittance* or *absorbance*. These are defined as:

$$\% \text{ Transmittance } (\% T) = \frac{\text{light transmitted by sample}}{\text{light transmitted by blank}} \times 100$$

$$\text{Absorbance} = -\log \left( \frac{\% \text{ transmittance}}{100\%} \right)$$

Beer's Law is a very useful relationship between Absorbance and the concentration of the colored solution (C). Beer's Law is expressed as:

$$\text{Absorbance} = abC$$

Where b is the path length of the spectrophotometer cell, and a is a proportionally constant called absorptivity. The value of a depends on the identity of the absorbing species and on the wavelength of light used. Because Absorbance varies linearly with concentration, measuring Absorbance allows one to determine the concentration of a solution of the species of interest, as long as a and b are known. In practice, this is done by preparing a set of solutions of known concentrations, measuring the Absorbance of these solutions and preparing a calibration curve (graph). Absorbance readings from unknown samples may then be compared to this graph to determine concentration. Your instructor and TA will guide you through the construction of a calibration graph and the data analysis.

### Standard Solutions

1. Clean four 100 mL volumetric flasks with caps using the purified water available in carboys in the lab.
2. Ask the instructor or TA to demonstrate the use of a microliter pipetter. Practice pipetting using water until you feel confident using the pipetter. With a clean and dry tip, add the following volumes of the **iron stock solution** to each of the flasks. Flask 1: 100 microliters; Flask 2: 300 microliters; Flask 3: 500 microliters; Flask 4: 700 microliters. The stock solution has a concentration of 1000 mg/L, and the standards have concentrations of 1, 3, 5 and 7  $\mu\text{g/L}$ , respectively.

3. Add the following to each flask, using a graduated cylinder.

- (a) 10 mL of 6% sodium acetate
- (b) 1 mL of 10% hydroxylamine hydrochloride
- (c) 10 mL 0.1% 1,10-phenanthroline

Fill the flasks 3/4 of the way full with purified water, swirl and allow reagents to mix.

4. Dilute the volumetric flasks to the mark (ask the TA or instructor for help with this the first time). Stopper and invert each flask multiple times to allow thorough mixing. Allow the mixture to react for at least 15 minutes.

5. After 15 minutes, record the Absorbance of each sample using the spectrophotometer.

### Water Samples

Follow this procedure for each water sample to be tested.

1. Clean a 50 mL volumetric flask with cap.

2. Ask the instructor or TA to demonstrate the use of a large glass pipet. Practice with water, then pipet 25 mL of the water to be tested into the volumetric.

3. Add the following to each flask, using a graduated cylinder.

- (a) 5 mL of 6% sodium acetate
- (b) 0.5 mL of 10% hydroxylamine hydrochloride
- (c) 5 mL 0.1% 1,10-phenanthroline

Fill the flasks 3/4 of the way full with purified water, swirl and allow reagents to mix.

4. Dilute the volumetric flasks to the mark (ask the TA or instructor for help with this the first time). Stopper and invert each flask multiple times to allow thorough mixing. Allow the mixture to react for at least 15 minutes.

5. After 15 minutes, record the Absorbance of each sample using the spectrophotometer.

### Spectrophotometer Measurements

1. Your instructor or the teaching assistant will explain how to use the spectrophotometer for this experiment.

- (a) Fill one cuvette about 1/2 to 2/3 full with filtered water. This is called the *blank* or reference solution.
- (b) Set the wavelength selector to 510 nm.
- (c) If using one of the older beige spectrophotometers do the following (others can skip this step): Adjust the zero knob on the spectrophotometer as demonstrated by your instructor or T.A. The zero knob should be adjusted until the dial reads 0% transmittance. This should be adjusted without a test tube in the spectrophotometer and with the cover closed.
- (d) Put your blank solution into the spectrophotometer. For the blue spectrophotometers: push the **0 Abs 100% T** button. For beige: Adjust the 100% transmittance knob until the dial reads 100% transmittance (0 absorbance).
- (e) Place the cuvette containing your sample into the spectrophotometer.
- (f) Record the Absorbance readings in the table provided on the next page.
- (g) Repeat for each sample.

**Data Collection**

Standard Concentration	Absorbance	Notes

Water Sample	Absorbance	Notes

**Calculations**

1. Prepare a calibration curve using your standard solutions. Your instructor or TA will help you with this.

