

Spectrophotometric Determination of Trace Iron in Solution

Measure the concentration of iron using a calibration curve

Introduction

Background

The ability to measure the concentration of iron in aqueous solutions in a quick and efficient way is important to many industries. Manufacturing industries where metal parts need to be cleaned may need to determine the level of iron in their waste streams for environmental compliance. Governments at all levels have an interest in testing wastewater, natural waters, and drinking waters to determine iron content to ensure compliance with the law and to ensure the safety of the water supply for wildlife and the human population.

Well-equipped, modern laboratories may perform iron content analysis by atomic emission spectroscopy in an inductively coupled plasma (ICP) spectrometer. Flame atomic absorption spectrometry also can be used, but iron solutions are notorious for clogging the burner with iron oxide when the concentration exceeds a certain level. Both of these techniques require a significant investment in instrumentation and a sustained laboratory infrastructure involving compressed gases and control of exhaust vapors. Fortunately, the solution chemistry underlying a colorimetric determination of iron content is simple enough to be reduced to kit form and performed in the field with hand-held equipment or in the lab with a low-cost visible spectrophotometer and simple glassware.

Preparing the colored iron complex

The colorimetric determination of iron content involves the measurement of the ferrous ion (Fe^{2+}) when it forms a complex with three molecules of 1,10-phenanthroline, also called *ortho*-phenanthroline or abbreviated as phen.



The chemical structure and numbering scheme for 1,10-phenanthroline is shown in Figure 1.

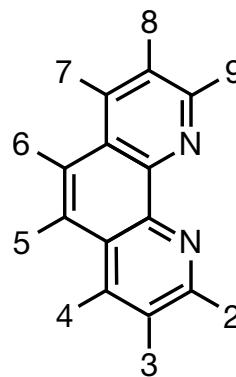


Figure 1. Numbering scheme for positions in 1,10-phenanthroline

The complex formed by 1,10-phenanthroline and Fe^{2+} , ferrous tris(1,10-phenanthroline)iron(II) or $[\text{Fe}(\text{phen})_3]^{2+}$, is a bright orange color. A 3D model of the structure of the complex is shown in Figure 2.

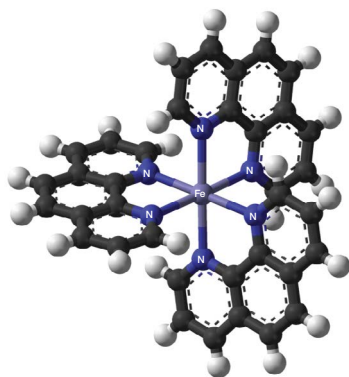
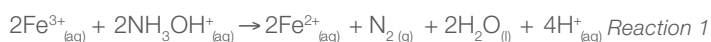


Figure 2. 3D model of the $[\text{Fe}(\text{phen})_3]^{2+}$ complex

In addition to its Fe^{2+} (ferrous ion) form, iron also can exist in a Fe^{3+} (ferric ion) form. To perform a *total iron* measurement, it is essential to *reduce* any Fe^{3+} in solution to Fe^{2+} before adding the phenanthroline to form the complex. The chosen reducing agent in this experimental protocol is hydroxylamine hydrochloride, which reacts with Fe^{3+} by *Reaction 1*:



Upon adding the phenanthroline, *Reaction 2* occurs:



Using Beer's Law to determine the concentration of an unknown

To determine the concentration of iron in an unknown solution, we must first *calibrate* the method with the spectrophotometer using Beer's Law:

$$A = \epsilon bc \quad \text{Beer's Law}$$

- where
- A** = the absorbance reported by the spectrophotometer
 - ϵ** = the extinction coefficient, a value that describes how strongly the particular compound absorbs photons at the particular wavelength, typically with units of $(\text{L}\cdot\text{cm}^{-1}\cdot\text{mol}^{-1})$
 - b** = the pathlength of the cuvette in cm, where typically a 1 cm pathlength cuvette is used
 - c** = the concentration of the solution in mol/L ($\text{mol}\cdot\text{L}^{-1}$)

A *Beer's Law plot* can be constructed by preparing a series of solutions of known concentration and graphing

the absorbance of each solution on the y-axis versus concentration on the x-axis:

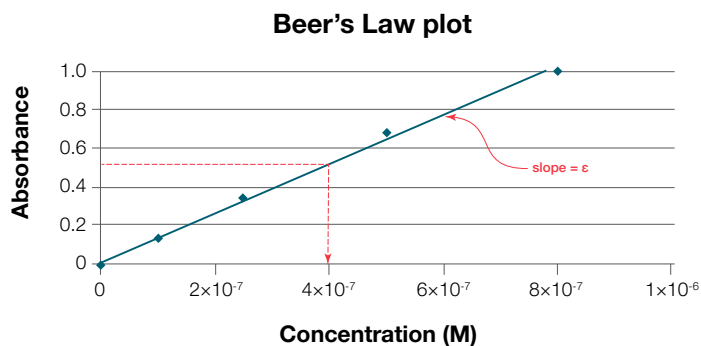


Figure 3. Example of a generic Beer's Law Plot

Comparing the equation for Beer's Law to the plot, we see that the slope of the line is equal to ϵ . We can use the plot to calculate the concentration of an unknown solution in one of two ways:

1. Use the plot itself to select the point on the y-axis representing the measured absorbance. Trace a horizontal line from that point to the plot line, then draw a vertical line straight down to the concentration on the x-axis. The point where this intersects the x-axis represents the concentration of the unknown solution.
2. Use the equation of the line. If $A = \epsilon bc$ and $b=1$, then $c = A/\epsilon$. Use a spreadsheet program or a graphing calculator to plot your data and determine a best-fit line (trend line) to calculate the slope of your line. This slope equals ϵ . Divide the measured A value for the unknown by ϵ to calculate the concentration of the unknown solution.

Experimental

Purpose

In this experiment, you will perform an analysis of an iron-containing solution with an unknown concentration by reducing all the iron in solution to its ferrous form, determining λ_{max} , and creating a standard curve of absorbance versus concentration to calculate the concentration of the unknown solution.

Reagents

- Ammonium iron(II) sulfate, hexahydrate
- Hydroxylamine hydrochloride
- 1,10-phenanthroline
- Sodium acetate

Part 1. Prepare the reagents and standards

- Prepare solutions prior to beginning this experiment, using deionized water for all dilutions.
 - 100 mg/L iron solution:** Dissolve 0.7022 g of ammonium iron(II) sulfate, hexahydrate in water in a 1 L volumetric flask.
 - 10 mg/L iron working solution:** Pipet 5 mL of the 100 mg/L iron solution into a 50 mL volumetric flask and fill to the mark with water.
 - 0.3 M hydroxylamine hydrochloride solution**
 - 0.25 % 1,10-phenanthroline solution:** Stir the solution to ensure that all solids have dissolved, using heat if necessary.
 - 1.0 M sodium acetate solution**
- Set up six 50 mL volumetric flasks and pipette reagents into them as follows:

Flask no.	10 mg/L iron solution (mL)	0.3 M hydroxylamine hydrochloride solution (mL)
1	0.0	1.0
2	2.0	1.0
3	5.0	1.0
4	8.0	1.0
5	14.0	1.0
6	20.0	1.0

Stopper each flask, then invert repeatedly for 2 minutes to allow the reaction to complete.

- To each flask, add:
 - 5.0 mL of 1.0 M sodium acetate solution. Stopper and invert the flask to mix.
 - 5.0 mL of 0.25% 1,10-phenanthroline solution. Stopper and invert the flask to mix.
 - Add deionized water to the mark. Stopper and invert several times to mix.

Part 2. Determine the proper analytical wavelength

- Pipet 3 mL of the solution from Flask 1 into a cuvette. This solution will be your blank.
- Wipe the outside faces of the cuvette with a laboratory tissue and place the cuvette into the square cuvette stage of the GENESYS UV-Vis Spectrophotometer sample compartment.
- Close the lid of the GENESYS UV-Vis Spectrophotometer. Select the Scan method and use the settings shown in Figure 1. Select Continue and then select Blank to record a baseline.
- Once the blank measurement has completed, open the lid, remove the cuvette with the blank solution and set it aside.
- Pipet 3 mL of the solution from Flask 6 into a cuvette. Wipe the outside faces of the cuvette with a laboratory tissue and place the cuvette into the square cuvette stage of the GENESYS UV-Vis Spectrophotometer sample compartment. Close the lid and press Measure to record the scan.
- Once the scan is completed, use one of the cursor lines to select the wavelength corresponding to the largest absorbance value. Record the wavelength of maximum absorption, known as λ_{\max} in the Lab Report (Figure 2).
- Print the spectrum if your GENESYS UV-Vis Spectrophotometer is equipped with a printer (Figure 3).

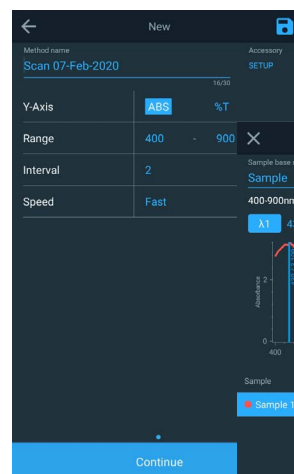


Figure 1: Scan method settings



Figure 2: Scan results

Figure 3: Export or print

Part 3. Prepare the Beer's Law plot

1. Calculate the concentration of iron in mM in each of Flasks 1 through 6 and enter the values in the Lab Report.
2. Select the Quant method in the GENESYS Software using similar settings to what is shown in Figure 4. Set the analysis wavelength at the λ_{max} as determined in Part 2. Enter the concentration values for the iron standard solutions you calculated in the previous step.
3. Use a cuvette with blank solution from Flask 1 to record a blank value.
4. Prepare and measure the absorbance of the cuvettes containing the solutions from Flasks 2 through 6. Record the values in Data Table 1 in your Lab Report.
5. The software will automatically generate a Beer's Law plot from the entered concentration data and measured absorbance values. Record the slope of the line in your lab report as ϵ (Figure 5). Use this plot to complete Part 4.

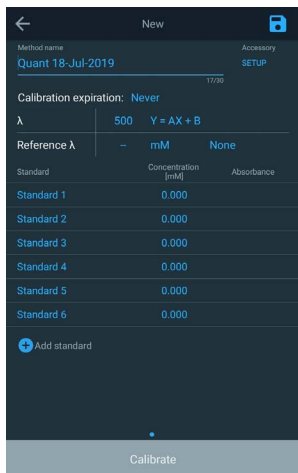


Figure 4: Quant method settings

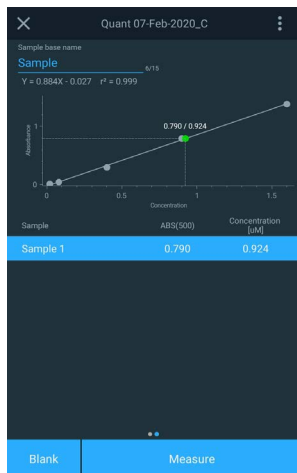


Figure 5: Beer's Law plot

Part 4. Determine the iron concentration in an unknown sample

1. Clean out one of the volumetric flasks.
2. Pipet 5.0 mL of an unknown iron solution into the flask. Follow the procedure used in Part 1, steps 2 and 3, to prepare the solution for measurement.
3. Measure your sample using the calibration curve obtained in Part 3.
4. Record the absorbance of the unknown iron sample.
5. Use the iron concentration reported by the software to calculate the actual concentration of your unknown iron sample. Remember to include the effect of diluting 5 mL of the unknown to 50 mL before you made your measurement when you calculate the concentration of the unknown.

Disposal of chemicals:

Check with your instructor before discarding any solutions. All solutions can be poured down the sink and rinsed with lots of water to dilute. Discard solids in the trash.

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Lab Report

Spectrophotometric Determination of Trace Iron in Solution

Name: _____

Date: _____

Section No. or Lab Period: _____

Questions

1. What is the wavelength of maximum absorption for your iron standard solutions?

$$\lambda_{\max} = \underline{\hspace{2cm}} \text{ nm}$$

Data Table 1

Flask no.	Fe ²⁺ concentration	Measured absorbance
1		
2		
3		
4		
5		
6		

2. What is the extinction coefficient for your iron standard solutions?

$$\epsilon = \underline{\hspace{2cm}}$$

(don't forget to include the units)

3. What is the measured absorbance of the unknown [Fe²⁺] solution?

$$A = \underline{\hspace{2cm}}$$

4. What is the concentration of the unknown [Fe²⁺] solution?

$$C = \underline{\hspace{2cm}}$$

(don't forget to include the units)

5. What is the absolute error between your value for the unknown [Fe²⁺] solution and the true value obtained by your instructor?

6. Calculate the percent error from question 4.

7. Why was it necessary to add hydroxylamine and sodium acetate to the solution used to record the blank?

8. Many concentrations in this experiment were given in units of mg/L. Express 10 mg/L iron using units of mol/L.

Remember:

- Staple hand-drawn or printed graphs to your lab report



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