Experiment 7
Synthesis and Analysis of a Complex Iron Compound

Part 3: Spectrophotometric Determination of Iron Content

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In our last exciting episode:
Redox Chemistry!

How to determine oxidation states

O is usually -2 H is usually +1

How to balance redox half-reactions:

Balance main atom.

Balance O using H_2O, balance H using H^+.

Balance charge using e^-.

Convert to basic solution if necessary.

Experiment 7 in a nutshell

Convert our green crystals to an orangey-pink metal complex, $Fe(phen)_3^{2+}$, then use a spectrophotometer to measure how much light this solution absorbs at 510 nm.

Next week we will measure the amount of light absorbed by some $Fe(phen)_3^{2+}$ solutions with known concentrations.

By comparing how much light our sample absorbs with how much light the standards absorb, we can determine how much iron is in our sample.

We'll do this exbeeriment in 5 parts

1) Convert our sample to $Fe(phen)_3^{2+}$

2) Make up a series of $Fe(phen)_3^{2+}$ standards

3) Measure the absorbances of the standards

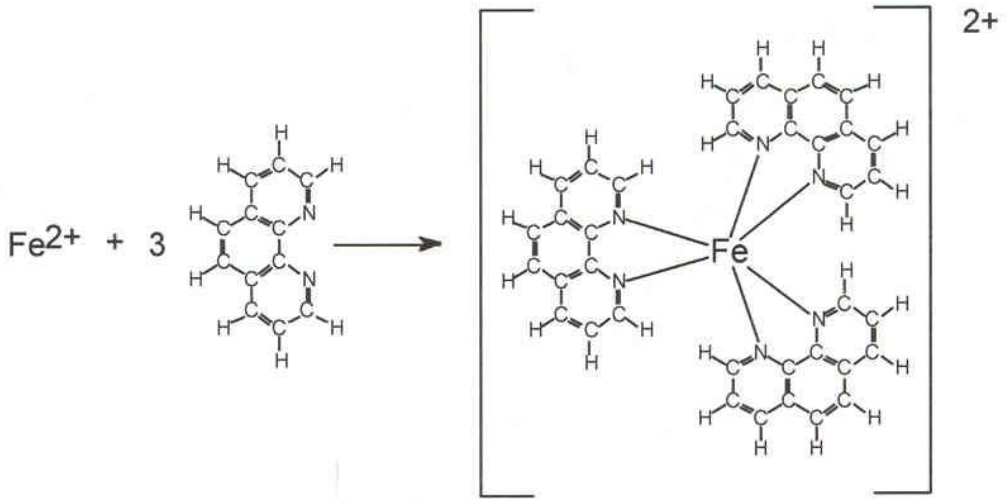
4) Measure the absorbance of the sample

5) Graph Absorbance versus Concentration in Excel to determine how much iron is in our sample

What is $Fe(phen)_3^{2+}$?

1, 10 phenanthroline

Tris(1,10 phenanthroline)iron(II)



Lab Procedure, Part 1

1.1 Start by weighing about 0.1500 g of your sample in a weighing boat. Add deionized H_2O to dissolve the sample. Transfer the dissolved sample to a 25 mL volumetric flask.

1.2-1.3 Add 8 mL of 6 M H_2SO_4, and fill to the line with deionized water using a disposable pipette. Mix the solution then pour it into a small beaker.

1.4 Pipet 5 mL into a 30 mL beaker, add about 10 mL deionized water, and heat to 60-70° while stirring. Heats up fast! Add 10 drops of $KMnO_4$.

Poor man's titration

1.5 Add $KMnO_4$ dropwise until the solution stays just barely pink.

1.6-1.7 Turn off the heat, remove the stir bar, and quantitatively transfer the solution to a clean 25mL volumetric flask.

1.8-1.9. Let the solution cool and bring it up top the mark in the volumetric flask.

So far we have done a 1:5 dilution of our sample, so whatever concentration we get in the end we will have to multiply by 5 to get back to our original concentration.

On to Part 4

4.1 Pipette a 5 mL aliquot of your sample from part 1.9 into a clean 25 mL volumetric flask.

4.2 Add 1 mL of hydroxylamine, NH_2OH

2 mL sodium acetate, and

8 mL 1,10 phenanthroline

4.3 Swirl and mix, and allow it to sit for 20 minutes to let the reaction proceed. The color will deepen.

Hey, that was another dilution!

In Part 4 we diluted our sample 1:5 again.

Since we've done two 1:5 dilutions (once in 1.4 and once more in 4.1), we've now diluted our original sample by 25 to 1!

So the concentration we eventually determine from the graph will have to be multiplied by 25 to get back to its original value.

Spectrophotometry!

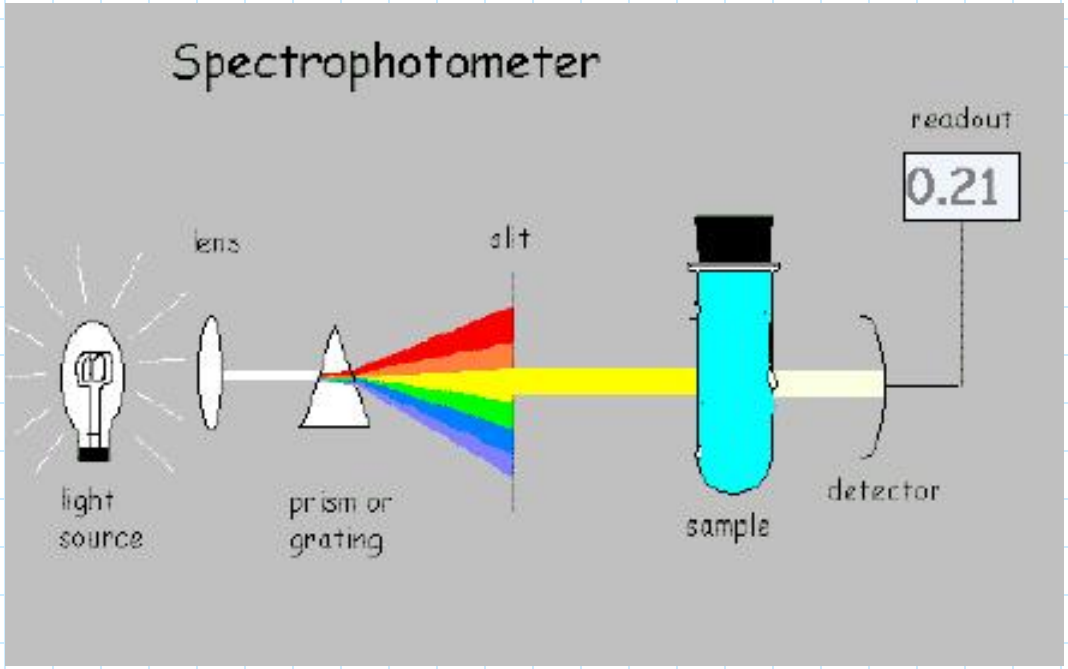
Spectrophotometers are some of the most widely used analytical instruments in the world, and they're about as easy to use as an analytical balance.

What does one look like?

How do they work?

Ours looks like this

Works like this



Anything that is colored has color because it absorbs some wavelength (or wavelengths) of visible light.

Absorbing light

With solutions,

- the more volume you have, the more light it will absorb.
- the more concentrated the solution is, the more light it will absorb.

Some things inherently absorb more light than others. $KMnO_4$, for example, has a very deep color because its electronic structure allows it to absorb a lot of light. We say it has a high *molar absorptivity*.

Beer's Law

Beer's Law says that absorbance depends on three factors: molar absorptivity, concentration, and path length.

$$A = \varepsilon c l$$

Also written as $A = \varepsilon b c$

and $A = abc$

$$A = \varepsilon C l$$

Absorbance equals the molar absorptivity times the concentration times the path length.

Molar absorptivities are usually reported in units of $M^{-1}cm^{-1}$, concentrations are in molarity (moles/liter), and path length is in cm (and is usually 1 cm long).

Using the spectrophotometer

Place a cuvette full of deionized water into the instrument. This is your blank. Press the button that says 0 ABS.

Remove the blank and put in a cuvette containing your sample. The display will automatically read out the absorbance. Record this value on a scrap of paper towel, in your lab manual, or just remember it until you need to use it next week.

How not to screw up this part

1) Rinse the cuvette twice with the sample you are about to measure before you put it in the instrument
2) Wipe the outside of the cuvette clean using Kim-Wipes. No fingerprints, no wetness on the outside.
3) No bubbles in the solution.
4) Fill the cuvettes at least 3/4 of the way up.

Next week

Make standards from a known Fe^{2+} solution and measure absorbances. Lots of dilutions!

Plot a graph of Absorbance (y-axis) versus concentration (x-axis).

You'll get a straight line plot, and the slope of the line is your molar absorptivity, ε, in units of $M^{-1}cm^{-1}$.

Use that number to determine the concentration of the iron in your sample from today, using Beer's Law.