Experiment 7
Synthesis and Analysis of a Complex Iron Compound

Part 3: Spectrophotometric Determination of Iron Content

CH 204 Spring 2006

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Quick Recap of Last Week

We converted our $Fe(C_2O_4)_3^{3-}$ to $Fe(phen)_3^{2+}$, made a lot of dilutions, and then used a spectrophotometer to measure how much light this solution absorbed at 510 nm.

This week we will see how much light is absorbed by solutions that have known $Fe(phen)_3^{2+}$ concentrations, and we will then make up a calibration curve, aka standard curve aka working curve.

What remains to be done

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

Lab Procedure, Part 2

2.1 Get 10 ml of the standard iron solution – record the concentration on the bottle in your notebook.

2.2 Pipet 5 ml into a 25 ml volumetric flask. Add NH_2OH, sodium acetate, and phenanthroline, and bring up to the mark with water.

2.3 Swirly, swirly, swirly. Let it sit for 20 minutes for the color to develop. Practice covering a flask with Parafilm if you didn't master the art last time.

Some quick vocabulary

STANDARDS are solutions that we know the concentrations of. These are made by diluting a **STOCK SOLUTION**. Iron is the **ANALYTE** in this lab, the thing we're analyzing for. Both our sample and our stock solutions contain the analyte.

A **STOCK SOLUTION** is a more concentrated solution that we know the concentration of, and it's what we start with when we're going to make a series of standards.

An **ALIQUOT** is a measured portion of a larger volume.

Our **SAMPLE** is the solution that we don't know the concentration of, and that's what we're trying to figure out.

Let's make our standards

3.1 Get five test tubes and label them 1, 2, 3, 4, 5. Write directly on the glass with your marker.

Using a graduated pipette, add that many milliliters of the $Fe(phen)_3^{2+}$ stock solution (this is the 25 mL volumetric flask full of orange solution that you prepared in Part 2) to each test tube.

Using the graduated pipette again, fill each test tube to 5 mL total by adding 4, 3, 2, 1, and 0 mL of deionized water to test tubes 1-5 respectively. Mix each one thoroughly.

A whole lotta dilutin' goin' on!

When we mixed up the five standards in the test tubes, each one was diluted by a different factor:

1 was diluted 1 to 5
2 was diluted 2 to 5
3 was diluted 3 to 5
4 was diluted 4 to 5
5 was not diluted at all in this step.

Correcting for dilutions

So when you calculate the actual concentration of each of the standards measured in the spectrophotometer, they will be:

Concentration on the bottle × 1/5 × test tube dilution factor

1: Conc. × 1/5 × 1/5
2: Conc. × 1/5 × 2/5
3: Conc. × 1/5 × 3/5
4: Conc. × 1/5 × 4/5
5: Conc. × 1/5 × 1

Measure Absorbances

Two cuvettes to a customer!

Remember to rinse each cuvette twice with the solution you plan to measure. Wipe off the sides, no air bubbles, you know the routine.

After you have your data

Go to the computers, make sure you have calculated all the dilutions correctly, and enter the absorbance and concentration values into Excel.

Plot a graph of Absorbance (y-axis) versus concentration (x-axis). Include 0,0 as a data point – that is your blank.

You should get a straight line plot, and the slope of the line is your molar absorptivity, ε, in units of $M^{-1}cm^{-1}$. Get a least squares fit so you have an equation for the line.

Don't forget about those dilutions

Enter the actual concentrations of your standards into the spreadsheet. Remember, each standard will have a different dilution factor!

When you determine the concentration of your unknown from the graph, remember that you have to back calculate through all of the dilutions you did last time in order to figure out the original concentration you started with.

Looking ahead

- The final three labs (Thermochemistry, Acid-Base Equilibria, and Kinetics) will be done in pairs.
- Pre-Lab 8 is longer and more involved than any previous pre-lab. It's chock full of calculations that you will need in order to do the lab write-up.
- Start on this pre-lab EARLY!