Experiment 7

Synthesis and Analysis of those Same Green Crystals what we made before Spring Break

Part 3: Spectrophotometric Determination of Iron Content

CH 204 Spring 2008

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Two weeks ago

Redox Chemistry

- Oxidation – loss of electrons
- Reduction – gain of electrons
- Balancing redox reactions
- Titration with $KMnO_4$

Today's lab in a nutshell

Parts 2 and 3 of the procedure in the lab manual.

1) Mix up a series of 5 standards by diluting from a stock solution
2) Measure the absorbance of each of the standards
3) Make a calibration curve by plotting Absorbance vs Concentration

Part 2 – make up the standard iron solution

1. Get 10 mL of the iron solution from the hood, and pipette 5 mL into a 25 mL volumetric flask.

   That's a 1 to 5 dilution of the original concentration.

2. Add 1 mL of hydroxylamine, NH_2OH
   2 mL sodium acetate, and
   8 mL 1,10 phenanthroline

3. Fill the volumetric flask up to the line with deionized water using a dropper pipette, then mix it, cap it off and let it sit for 20 minutes for the reaction to occur.

The Iron Solution in the Hood

Is 0.0197 grams of Fe per liter

Convert that to moles/liter before doing any calculations with it.

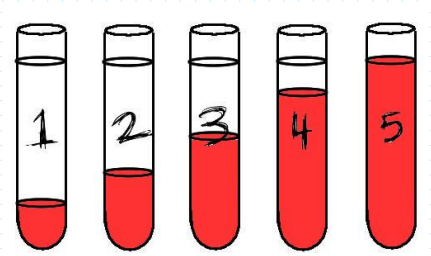
Part 3 – Make Individual Standards

1. Get five small 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 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.

5,000 words about Part 3



A whole lotta dilutin' goin' on!

When we mix up the standards in the test tubes, each one is diluted by a different factor:

1 is diluted 1 to 5
2 is diluted 2 to 5
3 is diluted 3 to 5
4 is diluted 4 to 5
5 is not diluted in this step.

Correcting for dilutions

To find the actual concentration of each of the test tubes, we have to multiply by the dilution factor for each one:

Original Concentration (M) × 1/5 × test tube dilution factor

This dilution was in part 2 ↘ ↙ This dilution is in part 3

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

Spectrophotometry!

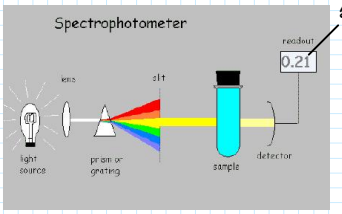
Spectrophotometers are the most widely used analytical instruments in the world except for the analytical balance, and they're about as easy to use as an analytical balance.

"But vat does a spectrophotometer look like?" you are wondering, "Und how does it vork?"

I'm glad you asked!

Looks like this

Works like this



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

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 first standard. The display will automatically read out the absorbance. Record this value.

Again and again ad nauseum

Repeat this procedure for each of your standards and your sample.

Insert the blank before each measurement to make sure the blank reads 0 absorbance units, then insert the next sample.

2 cuvettes to a customer! Reuse the sample cuvette!

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.

But what do these absorbance values tell us?

Beer's Law

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

$$A = \varepsilon c l$$

Sometimes written as $A = \varepsilon b c$

or $A = abc$

Beer's Law plots

When we plot Absorbance versus Concentration, the slope of the line is equal to εl. In our case $l = 1$, so the slope of the line is equal to the molar absorptivity for $Fe(phen)_3^{2+}$.

After you have your data

Enter the absorbance and concentration values into Excel.

Plot Absorbance (y-axis) versus concentration (x-axis). Set the y-intercept equal to zero.

You should get a straight line, and the slope of the line is your molar absorptivity, ε, in units of $M^{-1}cm^{-1}$. Have Excel display the equation for the line on the graph.

When you are done

Rinse your **5** test tubes with deionized water and turn them upside-down in the test tube rack.

Rinse your **2** cuvettes with deionized water and put them in your lab drawer.

Next veek

- Dissolve up some crystals
- Convert them to orange complex ion
- Do lotsa dilutions
- Measure absorbance
- Determine concentration

Quiz Next Week!

Beer's Law

Dilutions

Also Next Week!

Turn in Post-lab 7 next week just like it was a pre-lab.

Quiz This Week!

After today you are 2/3 done with the Final Exam!