Experiment 7

Synthesis and Analysis uff das Same Green Crystals vat ve made tvo veeks ago

Part 3: Spectrophotometric Determination of Iron Content

CH 204 Fall 2007

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Last week

Redox Chemistry

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

Experiment 7 in a nutshell

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

Make some solutions with known concentrations of $Fe(phen)_3^{2+}$ compare the absorbance of our sample to these standards to determine how much $Fe(phen)_3^{2+}$ is in our solution.

The Plan for Today

1) Part 1 (free up Fe^{2+} from sample)... until the hot solution has to cool
2) Part 2 (make up standard solution)... until the solution has to sit for 20 minutes
3) Finish Part 1, start Part 4 (make up sample solution)... until the solution has to sit for 20 minutes
4) Do Part 3 (measure standards)
5) Finish Part 4 (measure sample)

Lab Procedure, Part 1

1. Weigh out 0.15 g of green crystals and dissolve in deionized H_2O. Transfer the dissolved sample to a 25 mL volumetric flask. Dissolve it right there in the weighing boat!
2. Add 8 mL of 6 M H_2SO_4, and fill to the line with deionized water using a disposable pipette.

Your sample is now dissolved in 25 mL of solution.

Part 1, continued...

3-4. Pipet 5 mL into a 30 mL beaker, add about 10 mL deionized water, heat and stir.

5. Add $KMnO_4$ dropwise until the solution turns light pink. This might take about 50 drops.

6-8. Transfer the solution to a clean 25 mL volumetric flask.

Let the sample cool and go on to Part 2.

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 0.0185 grams of Fe per liter

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

Finish Part 1

9. The sample has cooled off in a 25 ml volumetric flask, and needs to be filled to the mark.

The sample has now been diluted 1 to 5 from the original concentration.

On to Part 4

1. Pipette 5 mL of your sample from part 1.9 into a 25 mL volumetric flask.

   Add 1 mL of hydroxylamine, NH_2OH

   2 mL sodium acetate, and

   8 mL 1,10 phenanthroline

Swirl and mix, and allow it to sit for 20 minutes to let the reaction proceed.

After 20 minutes is up...

...FILL TO THE MARK WITH PHENANTHROLINE!!!

In Part 2 (making the standard) you used water.
In Part 4 (working with your sample) use phenanthroline to fill up the 25 ml volumetric flask.

Part 3 – Make Individual Standards

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 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.

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

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

Next Step: Spectrophotometry!

Spectrophotometry!

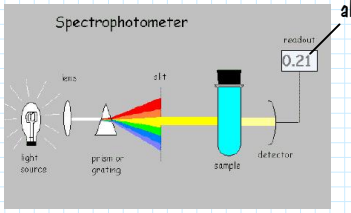
Spectrophotometers are the most widely used analytical instruments in the world except for the analytical balance, and they're about as easy to use as an analytical balance.

"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.

Lather, Rinse, Repeat

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.

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.

$A = \varepsilon cl$

Sometimes written as $A = \varepsilon bc$

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). Include 0,0 as a data point – that is your blank – and set the 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.

Don't forget about those dilutions

When you determine the concentration of your sample from the graph, remember that you have to back-calculate through all the dilutions you made in order to figure out the original concentration you started with.

Zwei Important Warnings!

1) Make all sample dilutions 5 to 25 ml, and write it down in your notebook every single time you make one.

   Part 1, step 9

   Part 4, step 1

   Plus as many dilutions as necessary in Part 4 step 2

2) Record every absorbance measurement you get, even if it is out of range.

Looking ahead

- The final three labs (Thermochemistry, Kinetics, Acid-Base Equilibria) will be done in pairs.
- Pre-Lab 8 is longer than previous pre-labs.
- Chock full of calculations that you will need in order to do the lab write-up.
- Start on this EARLY! Be finished by Friday if possible.

Next Week's Quiz

Beer's Law

Dilutions

Questions about the procedure from lab today