Experiment 7b

The Final Analysis of those Stupid Green Crystals We Made Like a Million Years Ago in Lab

Part 3: Spectrophotometric Determination of Iron Content

CH 204 Spring 2009

Dr. Brian Anderson

Last Week

Made standard solutions from a stock solution

Fun with dilutions!

Spectrophotometry – Beer's Law

'Tis the season...

Throw a pair of sweat pants in your lab drawer if you're going to be wearing shorts around campus in warmer weather.

This week

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.

Use the molar absorptivity determined from the calibration curve to calculate how much $Fe(phen)_3^{2+}$ is in our final sample solution.

Back-calculate through the sample dilutions to determine how much Fe was in the original sample.

Lab Procedure, Part 1

1. Weigh out 0.15 g of green crystals and dissolve in deionized water. (Do this right there in the weighing boat.) Transfer the dissolved sample to a 25 mL volumetric flask.
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...

4. Pipette 5 mL into a 30 mL beaker, add about 10 mL deionized water, heat and stir. Start the reaction right away!
5. Add $KMnO_4$ dropwise until the solution turns pink. (Around 30-50 drops.) Yellow → colorless → pink.

6-7. Transfer the pink solution to a 25 mL volumetric flask and let it cool on the dark part of the lab bench.

8. There is no step eight.
9. Fill the volumetric flask to the mark using deionized water.

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.

The sample has now been diluted 1 to 5 TWICE, or 1 to 25.

Add 1 mL of hydroxylamine, NH_2OH

2 mL sodium acetate, and

8 mL 1,10 phenanthroline

Fill to the mark with PHENANTHROLINE!

Let it sit for 20 minutes.

Measure and record the absorbance

You want an absorbance reading that is between your lowest and highest standard from last week.

If it's too high, do another dilution and test it again.

RECORD ALL DILUTIONS!

RECORD ALL MEASURED ABSORBANCE VALUES!

Make all of your dilutions 5 ml to 25 ml.

"Triplicate" analysis

Once you've got an absorbance reading within the range of your standards, dump the cuvette, rinse it twice with your solution, and make a second absorbance measurement. Then do this again and make a third absorbance measurement. Record all three absorbances in the table on page 59.

Don't forget about those dilutions

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

If all your dilutions are 5 to 25 ml, your total dilution factor will be 5^x, where x is the number of dilutions you made.

Report buried in the procedure

In Part 4 of the experimental procedure, steps 4 – 9 are actually part of the REPORT, not part of the experiment.

These calculations are covered on the cheat sheet from Dr. Leytner. Don't forget to do them!

Unknown Summary Sheet includes oxalate data from Experiment 6 and iron data from Experiment 7.

Busy week for Dr. Anderson

- I will be tied up off-campus most of this week
- No office hours on Friday – rescheduled for Monday morning
- Office hours Monday, April 6, 9:30 – 11 a.m.

Quiz 7 of 9 – the end is near...

The final three labs (Thermochemistry, Acid-Base Equilibria, and Kinetics) will be done in pairs.

Post-lab 8 has lots of calculations. Start on this early.

If your quiz and post-lab averages are low, start on the post-labs sooner. Ask me or the TA's for help. Don't lose any more points on post-labs.

No quiz next week.