Experiment 7

Still More Analysis of those Stupid Green Crystals we made a Million Years Ago in Lab

Still Part 3: Spectrophotometric Determination of Iron Content

CH 204 Spring 2007

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Last Week

Made standard solutions from a stock solution

Fun with dilutions!

Spectrophotometry – Beer's Law

This week

Parts 1 and 4:

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 H_2O. (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.
5. Add $KMnO_4$ dropwise until the solution turns pink. (Around 50-60 drops.) Yellow → colorless → pink.
6-7. Transfer the solution to a 25 mL volumetric flask and let it cool.
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.

   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.

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

Remember...

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

Not with water.

Nicht mit der wasser.

¡No con agua, amigo!

[illegible]

Measure the absorbance

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

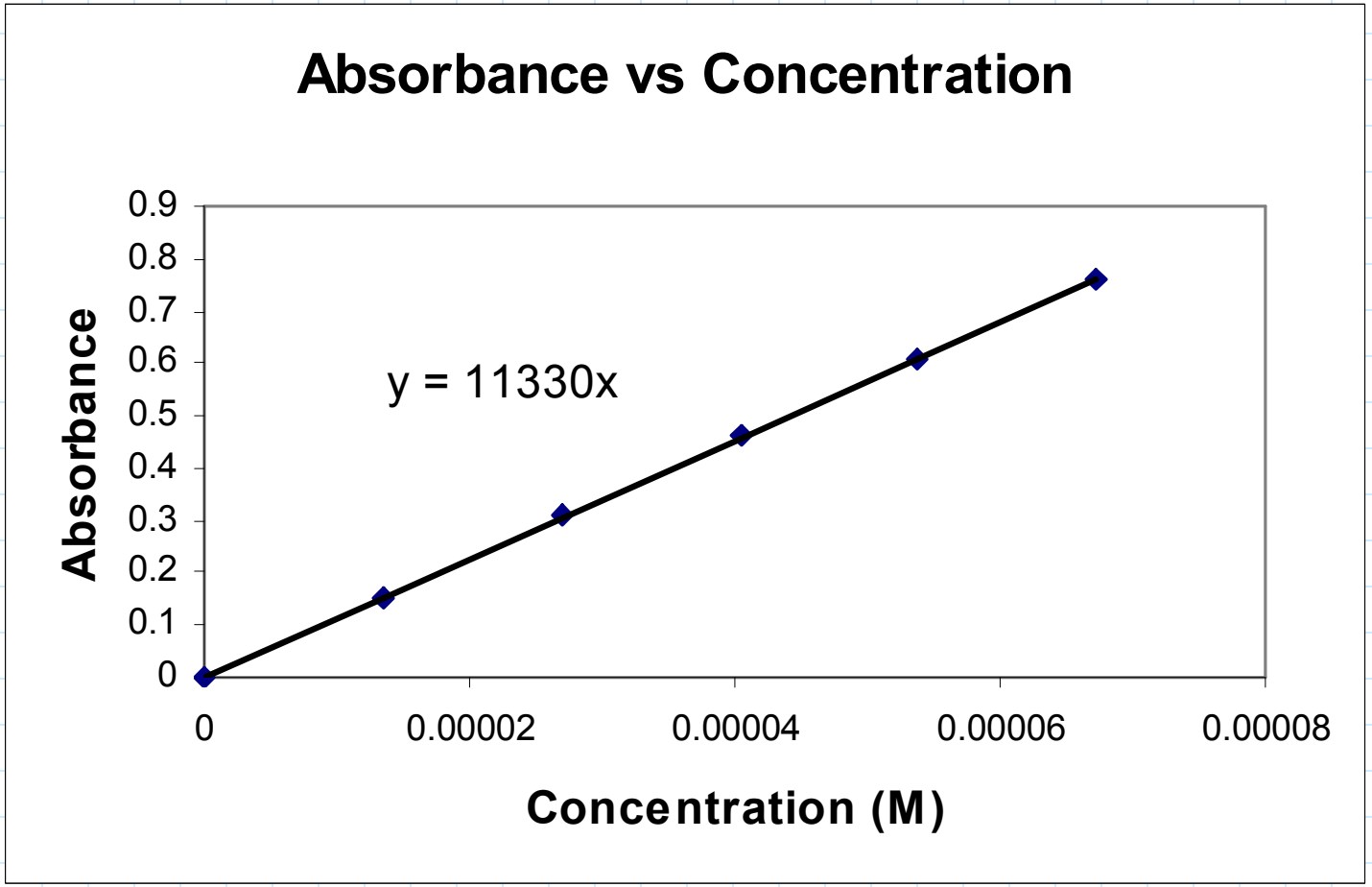
If it's too high, do another dilution and test it again.

Make all of your dilutions 5 ml to 25 ml.

RECORD ALL DILUTIONS AND ABSORBANCE VALUES!

Eventually you get an absorbance within range. Make three separate measurements of your sample. What then?

Last week's graph



Beer's Law

$$A = \varepsilon c l$$

$$c = A/\varepsilon$$

Get some tiny value for c (x 10^{-5} M)

That's the final concentration after all those dilutions.

But what was the original concentration in the very first 25 ml flask?

Dilution calculations

Going forward:

$$\text{Initial } c \times \frac{5}{25} \times \frac{5}{25} \times \frac{5}{25} = \text{Final } c$$

Going backward:

$$\text{Final } c \times \frac{25}{5} \times \frac{25}{5} \times \frac{25}{5} = \text{Initial } c$$

Calculating moles

Once you have your initial concentration from back-calculating through all the dilution factors,

Initial c (moles/L) x initial volume (L) = moles of Fe

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Unknown Summary Sheet

Report moles of Fe per gram of sample

Also report moles of oxalate per gram of sample

(final result from Experiment 6)

Also – don't forget calculations for the report that are buried in Part 4 of the procedure (steps 3-9).

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!