Experiment 7

Synthesis and Analysis of those same old Moldy Green Crystals from before spring break

Part 3: Spectrophotometric Determination of Iron Content

CH 204 Spring 2007

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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 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

Some quick vocabulary

A **STOCK SOLUTION** is a more concentrated solution that we know the concentration of, and it's the solution we start with when we're going to make a series of standards. The concentration of our iron stock solution is 0.0188 g/L Fe^{2+}.

STANDARDS are solutions with known concentrations that are around the same concentration as our sample. We will determine our sample concentration by comparing it with our standards.

Some more quick vocabulary

The **ANALYTE** is the thing we're analyzing for. In this lab the analyte is an orange-colored complex ion $Fe(phen)_2^{2+}$. Our standard solutions and our sample solution contain the same analyte.

Our **SAMPLE** is the solution that we don't know the concentration of, and that's what we're trying to figure out. We'll do this one next week.

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

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.

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 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 in this step.

Calculating final concentrations

To find the final concentration of each of the standards, we have to convert from grams/L to moles/L and then 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!

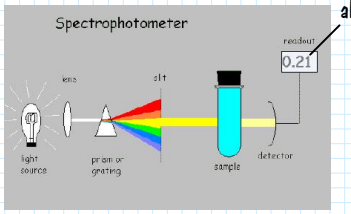
Spectrophotometers are the most widely used analytical instruments in the world, except for the analytical balance, and they're about as easy to use as an analytical balance.

"But what does a spectrophotometer look like?" you are wondering, "And how does it work?"

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 five standards.

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 solution 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.

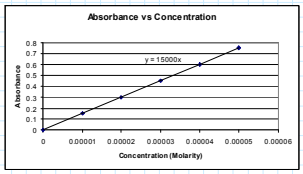
$$A = \varepsilon c l$$

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

You should get a straight line, and the slope of the line is your molar absorptivity, ε, in units of $M^{-1}cm^{-1}$.

Next week

Parts 1 and 4 of Experiment 7 (analyze the sample)

No lab report this week!

Turn in Post-lab 7 at the beginning of lab next week

Quiz next week covering Experiment 7