Lab # 6 - SPECTROPHOTOMETRIC ANALYSIS OF Mn

Summary:

The objective of this experiment is to determine the % Mn in your unknown Mn Ore sample.

You are responsible for knowing the theoretical fundamentals of spectrophotometry and Beer's Law.

I. Preliminary Preparations:

Dry your unknown Mn ore at 110°C for one hour. Cool the sample in a desiccator before weighing.

II. Week I

Dissolve your unknown Mn ore and store the resulting solution

Dissolving the Unknown Mn Ore:

1. Weigh out **TWO** 0.2 gram samples (±0.1 mg) of your unknown Mn ore into 250 mL beakers (labeled).

2. Add 50 mL 6 M HNO_3, cover with a watch glass and gently boil on a hotplate IN THE HOOD (remember to put a screen under the beaker).

3. To dissolve the sample, add superoxol******,** one mL, every five minutes over a period of 30 to 45 minutes. The mixture should be held near boiling or at simmer (slow boil) during this period.

Do not allow your solution volume to go below 10 to 20 mL; add more acid if necessary!

Remember that **ALL** DARK PARTICLES MUST GO INTO SOLUTION! Check your sample for complete dissolution by examining it against a white background. After the sample has dissolved, cool the solution, cover with a watch glass or parafilm and store until the next lab.

*******CAUTION :** Superoxol, 30% H_2O_2, is a strong oxidizing agent and causes severe burns!

III. Week 2

Convert both the known and unknown Mn^{+2} solutions to MnO_4^-, and then measure their absorbencies (all of the absorbance measurements can be done in about 10 minutes).

The Spectro Master Model 415 spectrophotometers will be used to measure your absorbencies. The spectrophotometers should be turned on at the start of the lab period so they have time to warm up and stabilize.

Preparation of Known MnO_4^- Solutions:

1. Obtain 20.00 mL of a known 0.1000 g/L solution of Mn^{+2} in a clean 400 mL beaker.

2. Add 60 mL distilled water, 5 mL 85% H_3PO_4 and 0.4 grams KIO_4 (don't confuse KIO_4 with KIO_3, which were used in the last lab).

3. Cover with a ribbed glass and gently boil the solution for 5 minutes on a hotplate in the hood (remember the screen under the beaker).

The solution should slowly turn purple due to the formation of MnO_4^- (if a brown precipitate appears, consult your instructor).

4. After boiling for five minutes, allow the solution to cool below boiling and then add an additional 0.4 grams KIO_4.

5. Gently reboil the solution for ten more minutes. **This is a critical step!**

ALL of the Mn^{+2} (colorless) MUST BE CONVERTED to MnO_4^- (purple). Don't try to rush here. Boil for the full ten minutes!

6. Allow the solution to cool and quantitatively transfer it to a 100.0 mL volumetric flask (**BE CAREFUL**, your volume is already over 80 mL). Dilute to the mark and **MIX THOROUGHLY!!** This will be solution # 1, your stock solution.

7. Clean, rinse and drain four 25.0 mL volumetric flasks. Label these flasks : # 2, # 3, # 4, and # 5.

8. Drain your buret and rinse it three times with 5 mL portions of the stock solution (# 1). **DON'T WASTE THE STOCK SOLUTION !!!** You will need most of it.

9. Fill the buret with the stock solution. Add the following volumes of stock solution from your buret into the **25.0 mL volumetric flasks**. **Dilute to the mark** and MIX THOROUGHLY!

soln #	vol of stock soln (# 1)	vol of water
# 2	18.00 mL	7.00 mL
# 3	12.00 mL	13.00 mL
# 4	6.00 mL	19.00 mL
# 5	3.00 mL	22.00 mL

Absorbance Measurements of the Known MnO_4^- Solutions:

1. Obtain a cuvette (absorption cell) from your instructor. It must be scrupulously clean. IT IS IMPORTANT TO USE THE CORRECT CELLS FOR EACH INSTRUMENT. The Spectro Master uses square cells.

2. With the wavelength control at 525 nm, fill a cuvette with distilled water, gently wipe it off with a Kimwipe and place it in the cuvette holder. This is your blank. Adjust the meter to 100% transmittance (0 absorbance).

3. Rinse the cuvette with three small portions of sample # 1. Fill the cuvette 2/3 full with sample # 1, wipe it off with a Kimwipe and measure its absorbance (and transmittance).

4. Repeat this process and measure the absorbance (and transmittance) of the other four samples.

Preparation of the Unknown MnO_4^- Solution:

1. Take your **cooled** unknown Mn^{+2} solution and quantitatively transfer it to a 500 mL volumetric, fill with H_2O, and **MIX WELL**!

2. Place a 10.0 mL aliquot (use a pipet) of this solution into a 400 mL beaker, keeping the original just in case.

3. Add 70 mL H_2O and 10 mL 85% H_3PO_4. Cover with a ribbed watch glass and gently heat on a hot plate IN THE HOOD (remember the screen).

4. When the solution reaches 70 to 80°C add 0.4 grams KIO_4. Stir the solution with a glass rod to insure complete dissolution.

5. Cool the solution below boiling, add an additional 0.2 grams KIO_4, then reboil for five more minutes.

6. After reboiling for 5 minutes, cool the solution and then quantitatively transfer it to a 250.0 mL volumetric flask. Dilute to the mark and MIX THOROUGHLY!!

Absorbance Measurement of the Unknown:

1. Transfer approximately 20 mL of the unknown MnO_4^- solution to a 100 mL beaker and add several drops of concentrated HCl . (Warm if necessary)

2. When the purple color has disappeared, use this solution as your blank, exactly as in the measurement of the knowns.

3. After using the blank to calibrate the instrument at 525 nm, measure the absorbance (and transmittance) of the unknown MnO_4^- solution (remember to rinse the cuvette first). The final absorbance reading of the unknown should be between 0.2 and 0.8.

If the absorbance of the unknown is above 0.8, make an accurate dilution (1:1, 1:2, 1:3 as appropriate) and again measure the absorbance (and transmittance). Keep an accurate record of any dilutions that are made!
IMPORTANT Remember to dilute the blank as well as the unknown and to rezero the instrument.

For the Report:

1. Calculate the MnO_4^- molarity in each of the five known solutions.

2. Prepare two graphs:

   (A) transmittance vs concentration MnO_4^-

   (B) absorbance vs concentration MnO_4^- (Beer's Law plot).

3. Use the Beer's Law plot and the computer to obtain an accurate value for the concentration corresponding to the absorbance reading of the unknown.

4. Calculate the percent Mn in your unknown ore sample.

5. Using the results from those knowns that are in the linear region of the Beer's Law plot, calculate the molar absorptivity of MnO_4^- (the cell length is 1.00 cm).

6. Think about the following questions:

   (A) Why was HCl not used to dissolve the unknown ore?

   (B) Why was H_3PO_4 added?

   (C) How was the wavelength of 525 nm selected?

   (D) Why did you prepare a different blank for the unknown?