CH 2212 Lab Exploration
Calibration Curves in Spectrophotometry

In this exploration, each student will generate a calibration curve suitable for the determination of the permanganate ion (MnO_4^-) by the external standard method.

In addition, the student will determine the molar absorptivity of MnO_4^- in aqueous solution. **Although you will need to share the spectrophotometers by pairs, each student should work alone and submit his or her own results.**

Background

When a chemist analyzes manganese in steel by a classic spectrophotometric method, one of the steps (after the initial dissolution of the sample) involves the oxidation of Mn^{2+} to MnO_4^-, the permanganate ion. Permanganate is a highly colored ion with an absorption maximum around 530 nm. A calibration plot (concentration vs. absorbance) is typically obtained from dilutions of reagent $KMnO_4$. The absorbance of the MnO_4^- in the dissolved steel sample solutions is compared to the absorbance of the MnO_4^- standards and the corresponding concentration determined. After any dilutions are taken into account, the MnO_4^- (and thus the Mn^{2+}) in each solid steel sample can be calculated.

Safety and Handling

Because MnO_4^- is so highly colored, any residue left in the sink or on the benchtop (or on your clothing) will be readily visible. It is also a relatively strong oxidizing agent. Because of these issues, we will collect all MnO_4^- solutions in a common waste container in the hood. If one is not already available, take an emply acid bottle, label it "Waste MnO_4^- " "CH 2212", date it, place it in a hood and put a funnel in the top. Before starting this exploration, each student should find and download from the web an MSDS for $KMnO_4$. Read it carefully and store it in your folder in the "Quant" folder on your assigned computer. Familiarize yourself with the recommendations for handling, storage and disposal in the MSDS.

Procedure

1) Determine the approximate working (concentration) range for your calibration curve.

a) Your instructor will provide a single weighing bottle containing $KMnO_4$ for the class. Precisely weigh out a mass (0.1 – 0.4 g) and quantitatively transfer to a beaker. Dissolve the $KMnO_4$ and transfer quantitatively to a volumetric flask. Dilute carefully to the mark. Calculate the molar concentration of $KMnO_4$.

b) From this accurate stock solution, prepare approximate dilutions using a graduate cylinder and beakers. You are trying to find a dilution which will give you an absorbance between 0.1 and 1 absorbance unit (A.U.) in your 1-cm cuvette.

c) Set up your Chem2000 diode-array spectrophotomer, if not already done. Configure it to take a spectrum in the visible light range from 400 to 750 nm.

d) Measure the absorbance at the absorbance peak (λ_{max}) for the most dilute solution from b), above. Make dilutions until you get a solution with an absorbance between 0.1 and 1 A.U. at λ_{max}.

e) Now that you know the approximate dilution factor for your stock solution, repeat the dilution of your stock solution with volumetric glassware using your best technique. Verify that the absorbance of this "good" dilution is in the right range.

2) Prepare the Calibration Plot

a) Based on the dilution in 1e), prepare at least four additional solutions of different concentration which have absorbances between 0.1 and 1 A.U. at λ_{max}. This may involve dilutions directly from the stock, or it may involve dilutions from an intermediate strength solution from 1e). In any event, use volumetric glassware and your best technique.

b) Note carefully the dilution steps involved and calculate the molar concentration of MnO_4^- in each solution.

c) Measure the absorbance of each of these solutions. Together with 1e), these five solutions should give you a linear calibration plot. It is always a good idea to sketch a rough plot in your notebook. Any large discrepancies will be immediately noticeable.

d) Use Excel to generate a quality plot. Store the plot in your folder. Have it ready on your screen – your instructor will show each plot to the class at the end of the period.

3) For each of the five solutions of your calibration plot, calculate the **molar** absorptivity of MnO_4^-. Assume the path length of your cuvette is 1 cm. Write your result on the blackboard.