Chemistry 150L, General Chemistry Lab I

Name_____
Lab Time_____

DEVELOPING LABORATORY SKILLS
PIPETS AND BALANCES
(Individual Exercise)

Developing pipetting skills is critical to success in the next experiment, Analysis of Iron Ore. Therefore, each student needs to do this exercise. Obtain a 10 mL pipette from the rack of pipettes in the lab. Determine whether the pipette is clean by following the procedure described in the lab lecture. Fill a clean 250 mL beaker with distilled water. Using the techniques discussed in the lab lecture, pipette 10.00 mL of distilled water into a pre-weighed 125 mL Erlenmeyer flask. Weigh (±0.001 g) the flask and water. Record the information for trial #1 in the table below and calculate the mass of water that was delivered by the pipette. Perform additional trials until you and your lab instructor are satisfied with the precision of your measurements. You should be able to obtain a precision of ±0.02 g. Try to get this precision in only three or four trials. (Time saving hint: Simply taring before each addition allows you to get away with filling in the last column only.)

Trial	Mass of flask (g)	Mass of flask & added water (g)	Mass of water delivered (g)
1			
2			
3			
4			
5			
6			
7			
8			

QUANTITATIVE ANALYSIS OF IRON ORE FOR Fe

INTRODUCTION:
Color is one of the easier properties of a solution to monitor. The color that is observed by the eye is dependent upon the wavelengths of light that are either absorbed or transmitted by a material, known as the spectrum of a solution. In addition to the wavelengths that are absorbed, it is also important to understand how efficiently a solution absorbs light. This can be quantified by a value called the extinction coefficient (also called molar absorptivity) and Beer's Law:

$$A = \varepsilon \cdot l \cdot c$$

where: A = absorbance
ε = extinction coefficient
l = path length
c = concentration

The measured absorbance of a solution depends upon not only the extinction coefficient, but also on the concentration of the solution and the amount of solution the light must pass through (the path length).

In this experiment you will use a colorimetric method to analyze the %Fe by mass in a sample of iron ore. Samples of ***known concentration*** of the red-orange colored iron phenanthroline complex will be prepared and the absorbance of these solutions will be measured. These solutions are referred to as the ***standards***. Once the relationship between the concentration of this complex and its absorbance is established, *measurement* of the *absorbance(s)* of a similarly prepared iron ore solution(s) can be used to *calculate* the *concentration* of Fe in the prepared solution(s) and ultimately the %Fe in the iron ore sample.

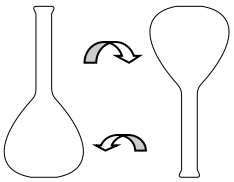
SAFETY CAUTION: Care should always be taken when working with acids. If any acid is spilled on your skin, wash immediately. Any acid spills on the lab bench should be cleaned up immediately. Always use a rubber bulb to fill pipettes. Dispose of all colored solutions in the appropriate waste bottle.

PART I: Preparation of stock and standard solutions of Fe^{2+}.
The preparation of the standard Fe^{2+} solutions and the graphing of the calibration curve relating the Fe^{2+} concentration to the respective absorbance may be performed by students working in-groups; **however, each student must analyze his or her own unknown ore sample.**

The instructor will prepare 500.0 mL of a stock Fe^{2+} solution by weighing the necessary amount of iron (II) sulfate heptahydrate, $FeSO_4 \cdot 7H_2O$, to make a solution that is approximately $9x10^{-4}$ M in $Fe^{2+}(aq)$ (**record the exact concentration in your lab book**).

Prepare four samples that contain 2.00 mL, 4.00 mL, 6.00 mL and 8.00 mL of the stock Fe^{2+} solution in a 50.0 mL volumetric flask. Using the appropriate glassware, add the following reagents to **each** of the standard solution preparations including the blank.

2.0 mL of 10% hydroxylamine hydrochloride (keeps iron in the 2+ state)
4.0 mL of 1 M sodium acetate (buffer; prevents pH from changing drastically)
10.0 mL of 0.30% o-phenanthroline (produces blood red colored Fe^{2+} complex)



Swirl the solution gently and then dilute to the calibration mark with distilled water. Stopper the flask (Do not use your thumb.) and mix the diluted solution well by turning completely over 10 times and shaking each time while the flask is upside down. Transfer the solutions, as they are made, to clean, dry beakers or flasks so that ***only one volumetric flask is needed***. Prepare all 4 solutions before performing any calculations; the solutions need to react for at least 30 minutes before their color can be measured. During this 30-minute reaction time, calculate the concentration of $Fe^{2+}(aq)$ in each of the samples and prepare a spreadsheet to graph absorbance vs. concentration of your samples. You must also prepare a blank solution that contains all of the above reagents but with no added Fe^{2+} solution. *This blank will be used to calibrate the spectrophotometer*. A single blank solution can be shared between multiple lab groups to minimize waste.

PART II: Generation of the Visible Spectrum and Calibration Curve

Based upon previous experiments, *predict the approximate wavelength* of λ_{max}. Make a hypothesis. After your solutions have reacted for at least 30 minutes, record the spectrum of all 4 solutions. To ensure that the pathlength does not change, you must use the same cuvette in the same orientation for every spectrum. Start with the least concentrated sample and rinse the cuvette multiple times with each successive sample before recording the spectrum. Use Stop→ Collect → Store Latest to keep the spectra of all solutions on screen. You should save and/or export this data to a file for reference later. Do not save it only to the lab computer because it probably won't be there next week when you need it. Save the cuvette in your lab drawer for the measurements you will make next week.

Choose the wavelength of maximum absorbance, λ_{max}, for the most concentrated solution and record in your lab notebook both this wavelength and the absorbance for each of the standard solutions at this wavelength. Prepare a plot of Absorbance vs. Concentration for the four standards. Determine the equation that best represents the observed trend in the data and include the best-fit equation and R^2 value on the graph. Lab work for the first week may stop at this point.

► Why is it usually considered better practice to start with the lowest concentration and go in order to the highest concentration when taking a series of measurements?

PART III: Preparation of iron ore sample

A. Obtain a numbered vial containing the iron ore sample. This is your unknown. **Record the unknown number in your lab notebook and be sure to include it in your report.** Weigh the full vial to ±0.001 g. Transfer the ore sample to a clean, dry, 50 mL beaker (*do not use a beaker larger than 50 mL*) without sending ore dust all over the sides of the beaker. The best way to do this is to place the vial up inside the upside-down beaker and then invert the vial and beaker together. Reweigh the empty vial to obtain the mass of the ore transferred. Record the mass in your notebook.

In the fume hood, add 3 droppers (several milliliters) of concentrated HCl to the iron ore sample. Heat the sample gently on a hot plate for about 30-60 seconds until the dark solid has completely dissolved. The length of time needed is somewhat dependent on the temperature of the hot plate. It may take longer than 30-60 seconds but **do not allow the solution to boil or overheat**. You may add some additional concentrated HCl if necessary to dissolve the **dark**

solid. If some **white** solid is present, it should dissolve when the water is added. When the dark solid is completely dissolved, add about 10 mL of distilled water quickly. Notice that this is not the normal safe protocol. Typically, when diluting a concentrated acid, the acid should be added to water to aid in dissipating the heat generated from the dilution. Water should not be added to acid as it may cause the acid to splatter. In this particular procedure, because we are using a small amount of acid and adding a large amount of water, there is not much risk of acid splashing if the water is added smoothly and quickly. Hot HCl gives off dangerous HCl fumes! Do not remove the beaker from the fume hood until it has been diluted.

NOTE: Your iron ore sample is contained in a "vial", a noun meaning "a small container, typically cylindrical and made of glass". The fumes given off by hot acid could be described as "vile", an adjective meaning "extremely unpleasant". These words are not interchangeable.

B. Transfer this solution to a 100.0 mL volumetric flask being careful to rinse the beaker very well in order to get all of the iron into the volumetric flask. Dilute the solution to the calibration mark carefully and mix it thoroughly. This solution contains all of the iron that was in your transferred ore sample.

C. Pipette 1.00 mL of the iron ore solution prepared in the prior step into a series of three 50-mL volumetric flasks. Add the same reagents as in part I.B, mix the solutions, dilute to the calibration mark, and mix again. Allow at least 30 minutes for the characteristic color to fully develop. Note that each of these solutions contains only one hundredth of the amount of Fe in the original sample solution. **This fact is important to keep in mind when doing your calculations.**

D. Record the full spectrum of each of these samples. Use the same cuvette and the same spectrophotometer that you used in the first part of this experiment. Note the absorbance of all three samples at the exact same λ_{max} you chose when you measured your standard samples last week.

PART IV: Calculations

Use the best-fit equation determined from the known samples (week 1) to calculate the concentration of the three prepared samples of Fe^{2+}. A visual inspection of the graph should give an estimate. It is usually considered a better practice to calculate the concentration of each sample and *then* take the average of the results, rather than to take the average absorbance at the beginning of the process and only do the concentration calculation once. How will either of these processes affect the way you determine the error in your reported value?

How many moles of Fe^{2+} are/were present in the 50.00 mL of sample that you prepared?

How many moles of Fe^{2+} are/were present in the 1.00 mL of solution that you diluted to 50.00 mL?

How many moles of Fe^{2+} are/were present in the original 100.00 mL sample you prepared from your sample of iron ore? How many grams of iron does this represent?

Calculate the %Fe by mass in your ore sample (solid). Report your answer as the average of your individual calculated results and with an absolute error.

In your hand-in, you must show all the calculations associated with making the stock and standard solutions and all the calculations associated with determining the final answer for the %Fe in your ore sample.