Name:_____

Chemistry 117 Laboratory
University of Massachusetts Boston

Volumetric Iron Analysis

LEARNING GOALS

1. Become familiar with complex ion formation
2. Become familiar with EDTA titrations
3. Become familiar with metal ion indicators for complexation titrations
4. Obtain practice with the method of titration
5. Obtain practice using volumetric flasks and dilution

INTRODUCTION

In this experiment we will analyze for the percentage of iron in a sample of an unknown salt containing ferric ion Fe(III). We determine the quantity of Fe(III) by reaction with a complexing agent having the formula $H_2C_{10}H_{12}N_2O_8^{2-}$ that is commonly known by the abbreviation EDTA. The complexing reaction is

$$Fe^{3+} + H_2C_{10}H_{12}N_2O_8^{2-} \rightarrow Fe(C_{10}H_{12}N_2O_8)^- + 2H^+$$

from which we see that complexation is 1 to 1; each mole of EDTA complexes with a mole of Fe(III) forming a mole of EDTA complex.

This reaction is carried out as a *titration* whereby the EDTA reagent is added slowly from a *burette* until an *endpoint* is reached, signaling that the moles of EDTA added equals the moles of Fe(III) in the solution being analyzed. Often an indicator is used to signal when the endpoint is reached. In this experiment the indicator is the salicylate ion, which forms an intensely red wine-colored complex with Fe(III). The Fe-EDTA complex on the other hand is light yellow. So, the actual titration reaction is

$$[Fe(HOC_6H_4COO)_3] + 3H_2C_{10}H_{12}N_2O_8^{2-} \rightarrow [Fe(C_{10}H_{12}N_2O_8)^-] + 2H^+ + 3HOC_6H_6COO^-$$

Intense red-wine color pale-yellow color

The formation of the EDTA Complex is significantly favored over the salicylate complex (it has a greater formation constant, K_f). Our original solution is dark red because of the Fe-salicylate complex. As the titration proceeds the solution retains a reddish color until enough EDTA has been added to react with all the original Fe(III) salicylate complex. At that point (the endpoint) the final traces of red disappears and we see only the pale yellow of the Fe-EDTA. From the known concentration and volume of EDTA solution added at

the endpoint, we calculate the quantity of iron in our solution and in the original ferric salt sample.

Using data given on the PRACTICE EXERCISE on page 4, do the calculations leading to the percent Fe(III) in the sample of ferric salt. The answer is given. The pre-lab quiz will focus on these practice calculations.

PROCEDURE

Standardized EDTA reagent is available from a dispensing bottle. The concentration of this reagent has been measured precisely and this concentration is written on the dispensing bottle. It is important that this concentration not be changed until the EDTA is delivered into the reaction vessel. Take care to avoid diluting the EDTA reagent by water droplets in your glassware.

1. Dispense 60 mL of EDTA reagent into a clean, DRY, 100 or 150 mL-beaker. Cover the beaker with a watch glass until Step 5 below. Record the posted EDTA concentration on your data sheet.

2. Weigh a sample of ferric salt into a tared weighing dish on an analytical balance. The mass should be no less than 1.5 grams and no greater than 2.0 grams. Record your mass to a tenth of a milligram (± 0.0001 g) on your data sheet.

3. Use a stream of water from a plastic wash bottle to transfer ALL the ferric salt to a clean 125-mL Erlenmeyer flask.

4. Dispense 20 mL of 6 M HCl into the Erlenmeyer flask, swirl and gently warm the flask on a hot plate until the salt is completely dissolved. While waiting, continue with Step 5.

The EDTA reagent in your beaker is to be delivered from a burette into the reaction vessel. Any water droplets inside the burette must first be rinsed out with the EDTA solution. Your instructor will demonstrate how.

5. Rinse the burette with EDTA solution from your beaker and then fill to between the 0 and 5 mL lines with the solution. Set the burette in a burette clamp on a ring stand. BEFORE GOING ON, ask your instructor to check your burette for leaks and air bubbles.

6. After you are certain that all the solid ferric salt has dissolved in your Erlenmeyer flask; transfer ALL the solution to a clean 250-mL volumetric flask. Use a funnel in the neck of the flask.

7. Wash the inside walls of the Erlenmeyer flask several times with water and pour these washings through the funnel. Then add just enough water to the volumetric flask so

that the bottom of the meniscus is at the line drawn on the neck. Use a medicine dropper to approach the line.

8. Holding a cap on the flask, mix the solution by inverting several times.

9. Rinse four 200 or 250-mL Erlenmeyer flasks with deionized water.

10. Rinse the inside of a 25-mL pipette with your ferric solution as demonstrated by your instructor. Then pipette 25.00 mL of this solution into each of the four Erlenmeyer flasks.

11. Add the following reagents to each Erlenmeyer flask:
    - about 25 mL of water
    - two small scoops of solid sodium acetate
    - about 5 mL of glacial acetic acid
    - about 5 mL of salicylic acid indicator solution

12. Warm but DO NOT BOIL the Erlenmeyer flasks on a hot plate before titrating. A flask is ready for titration when it feels as hot as a cup of coffee. Proceed as follows:
    - Record the initial burette reading to **two decimal places (±0.01 mL)**.
    - Titrate the sample, swirling the flask continuously as demonstrated by the instructor.
    - Stop as soon as the whole solution turns pale yellow. Further addition of EDTA should cause no change in color. This is the endpoint.
    - Read and record the final meniscus level to **two decimal places (±0.01 mL)**
    - Subtract the initial reading to find the endpoint volume.

Because each titration requires approximately the same volume of EDTA, you can now estimate if there is enough EDTA left in your burette to titrate again without passing the 50 mL line. If there is not enough, refill.

13. Titrate again as above. DON'T FORGET TO RECORD THE INITIAL READING! Record the final meniscus level to two decimal places.

14. Repeat the titration with a third sample. If the endpoint volumes of the three titrations agree to within 0.2 mL, you are finished.

15. If the three titrations do not agree to within 0.2 mL, use the fourth flask and titrate this. If three of the four titrations agree to within 0.2 mL, use these three for the final calculations. If three of the four do not agree, prepare and titrate a fifth flask. Use the three closest endpoint volumes in your calculation.

Name _____ Lab Section _____Partner(s)_____

Volumetric Iron Analysis

Practice Exercise

EDTA reagent concentration					0.02040 M	From step 1
Mass of ferric salt sample weighed out (This sample is dissolved and diluted to 250.0 mL in Steps 6 and 7)					2.1234 g	From step 2
Mass of sample transferred in the 25.00-mL pipette						From step 10
Replicate titrations of samples in Erlenmeyer flasks	Burette readings in mL					From step 12-15
	Final	25.80	24.75	26.10	25.85	
	Initial	1.25	0.80	2.05	1.76	
	Endpoint Volume					
	Circle the three closest endpoint volumes.					

Mean of the three closest endpoint volumes	V =
Millimoles of EDTA in the volume V	
Millimoles of Fe(III) titrated by V mL of EDTA	
Mass of Fe(III) titrated by V mL of EDTA	
Percent Fe(III) in the ferric salt	12.89 %

Name _____ Lab Section _____Partner(s)_____

Volumetric Iron Analysis

Data

EDTA reagent concentration						From step 1
Mass of ferric salt sample weighed out (This sample is dissolved and diluted to 250.0 mL in Steps 6 and 7)						From step 2
Mass of sample transferred in the 25.00-mL pipette						From step 10
Replicate titrations of samples in Erlenmeyer flasks	Burette readings in mL					From step 12-15
	Final					
	Initial					
	Endpoint Volume					
	Circle the three closest endpoint volumes.					

Mean of the three closest endpoint volumes	V =
Standard deviation[1] of the three closest volumes	
Millimoles of EDTA in the volume V	
Millimoles of Fe(III) titrated by V mL of EDTA	
Mass of Fe(III) titrated by V mL of EDTA	
Percent Fe(III) in the ferric salt	

[1] The standard deviation (s_V) of volumes v_1, v_2, v_3 having mean V is:

$$\{[(v_1-V)^2 + (v_2-V)^2 + (v_3-V)^2]/2\}^{1/2}$$

Lab Report

Your lab report will consist of a written abstract and answers to the three questions that follow. The abstract is worth 20 points. The following is a grading rubric for the abstract.

Content

- 2 pts All of the key pieces of data discussed.
- 2 pts The data is interpreted correctly.
- 2 pts The conclusions drawn from the data are correct.
- 2 pts It is evident that the student understands the main points of the laboratory experiment.
- 2 pts It is evident that the student was able to connect the learning goals of the experiment with data obtained in the experiment.

Quality of your writing

- 2 pts It is written in complete sentence(s).
- 2 pts The sentences are comprehendible to the reader.
- 2 pts It summarizes the experiment and the result and puts the results in context of the learning goals.
- 2 pts It is an appropriate length; 3-6 sentences.
- 2 pts It is written in the passive voice with no pronouns or phrases such as "In this lab we".

1. A well-trained technician is expected to perform this titration with a percent relative standard deviation (% RSD) below 1.0 %,

$$\% \text{ RSD} = (s_V/V)\cdot 100$$

Where s_V is the standard deviation of the endpoint volumes and V is the average of the three closest endpoint volumes. How good was your performance? (6 pts)

2. Why is it useful to use an indicator in this titration? What would be the consequence if an indicator was not used? (2 pts)

3. It is critical that the complexation of Fe^{3+} and the indicator is significantly weaker than the complexation between Fe^{3+} and the EDTA. What would be the consequence if an indicator was chosen that forms a stronger complex with the Fe^{3+} than the $FeEDTA^-$ complex? (2 pts)