Experiment 8

COMPLEXOMETRIC DETERMINATION OF Mg^{2+} and Ca^{2+}

WEEK 1
REFERENCE: Harris text, Chapter 12

INTRODUCTION

The most common multivalent metal ions in natural waters are Ca^{2+} and Mg^{2+}. It is these ions which give rise to the phenomenon known as water hardness. Hard water originates predominantly from $CaCO_3$ and results in the plugging up of water lines, scum in the sink and bathtub, etc.

In the first week of this experiment you will find the amount of Mg^{2+} in the unknown sample.

In the second week, you will determine the amount of Ca^{2+} and Mg^{2+} in a water sample you bring to the laboratory.

Specifically, Ca^{2+} and Mg^{2+} are titrated with standard EDTA solution using an Eriochrome Black T indicator. You can make a standard Ca^{2+} solution to standardize your EDTA solution using dried, primary standard $CaCO_3$. You need to add a little Mg^{2+} for a sharp endpoint. For every 8 grams of EDTA used, you add about 0.1 g of $MgCl_2 \bullet 6\ H_2O$.

PROCEDURE

PART 1

Preparation of ~0.02 M EDTA Solution. Calculate the amount of disodium dihydrogen EDTA dihydrate ($Na_2H_2EDTA \cdot 2\ H_2O$) needed to prepare 1 liter of ~0.02 M EDTA solution (yes, the two molecules of water should be included in the molar mass).

1. Weigh approximately this amount of disodium dihydrogen EDTA dihydrate and ~0.1 g of $MgCl_2 \cdot 6\ H_2O$ into a clean 400-mL beaker.

2. Dissolve the solids in boiled, deionized water (while hot); transfer the solution to a clean 1-liter bottle (or flask), and dilute to about 1 liter. Store EDTA solution in a plastic bottle as it will slowly leach Ca^{2+} from glass. Mix the solution thoroughly and label the bottle.

(If the solution is turbid, add a few drops of 18 M NaOH solution until the solution is clear.)

Preparation of Standard Solution of Ca^{2+} (Calcium Carbonate). Weigh accurately ~0.4 g of primary standard calcium carbonate that has been previously dried at 110 °C. Transfer the solid to a 500-mL volumetric flask, using about 100 mL of water. Add 6 M hydrochloric acid dropwise until effervescence (bubbling) ceases and the solution is clear. Dilute with water to the mark, mix the solution thoroughly and label.

Standardization of EDTA Solution. Pipet a 50.00-mL aliquot of the calcium carbonate solution into a 250.00-mL Erlenmeyer flask and add about 10 mL of an ammonia-ammonium chloride buffer (pH = 10) solution ***in the hood***. Then add about 5 drops (or less if too viscous) of Eriochrome Black T indicator solution. Titrate carefully with the EDTA solution to the point where the color changes from wine-red to blue. No tinge of red should remain in the solution. The titrated solution can be disposed down the drain. Repeat the titration with at least two other aliquots of the calcium solution.

Titration of Mg^{2+}.

Provide your instructor with a clean 250.00-mL volumetric flask into which a measured volume of an aqueous solution of the Mg^{2+} will be placed. Dilute the contents of the flask to the mark with water and mix thoroughly. Transfer 25.00-mL aliquots to three 250-mL Erlenmeyer flasks. To each of the Erlenmeyer flasks, add 2 to 3 mL of ammonia-ammonium chloride buffer and 3 to 4 drops of ErioT indicator. Titrate with your EDTA solution until the color changes from red to pure blue.

For next week: bring 1-L of water sample that you'd like to test for hardness next week.

Fill out and hand in the Week 1 Results Sheet with the data obtained.

Express the results in ppm (parts per million) Mg in the original 250-mL sample. Calculate the standard deviation and 95% confidence interval for your unknown's Mg concentration.

Complexometric Titration Results Sheet - Week 1

Name: _____

Unknown Number: _____ ppm Mg^{2+} in unknown: _____

Standardization Titrations

Mass $CaCO_3$ _____ mg

Titration Number	$CaCO_3$ Solution Volume (mL)	EDTA Titrant Volume (mL)	EDTA conc. (M)

EDTA Solution Molarity ± Standard Deviation _____

Relative uncertainty in EDTA solution molarity: _____

Unknown Titrations

Titration Number	Unknown Solution Volume (mL)	EDTA Titrant Volume (mL)	ppm Mg^{2+}

Relative uncertainty in unknown titrations: _____

Propagated relative uncertainty associated with overall analysis: _____

ppm Mg^{2+} in unknown ± absolute standard deviation _____

ppm Mg^{2+} in unknown ± 95% confidence interval _____

Grade _____

WEEK 2 (50 pts)
REFERENCE: Harris text Ch. 12, Ch. 28-1

There are no instructions for this part. Choose some water sample to analyze for water hardness.

Based on the results from Part 1, determine how many runs you will need to conduct to achieve a precision of ± 0.5 % at the 95 % confidence interval. Remember that [μ - sample mean] = $e = ts/n^{1/2}$, so $n = t^2s^2/e^2$ where s and e are in relative percent. Thus, given a target e of 0.5 % (0.005), and knowing s from PART 1, n, the number of analyses required, can be estimated. Consult Chapter 28 Section 1 in your text for how to approach this problem.

You will probably need to adjust your procedure to ensure that the titration endpoint lies somewhere between 20 and 50 mL. Be sure to keep track of what you did to achieve this as it will be an important part of the lab write-up.

Calculate the total water hardness of the sample. The hardness is defined as mg Ca^{2+} per L of water. So, assuming that only Ca^{2+} was complexed by the EDTA, calculate the ppm of Ca^{2+} in your sample and use the following chart to assign a hardness value:

Hardness	**ppm Ca^{2+}**
Soft	0-17.1
Slightly hard	17.2 - 60
moderately hard	61 - 120
Hard	121 - 180
very hard	181 +

For Week 2 Lab Write-up:

Results: Fill in the table on the following page with the relevant data. On the back of the page which you will hand in with the results include a brief description of the procedure used so your calculations can be checked. It is critical to include any dilutions made to the EDTA solution prior to titration.

Complexometric Titration Results Sheet - Week 2

Name: _____

Sample Description: _____

Week 1 EDTA Titrant Concentration _____

Week 2 EDTA Titrant Concentration _____

Run #	Sample Volume (mL)	EDTA Titrant Volume (mL)	$[Ca^{2+}]$ ppm

Relative error from Week 1 EDTA Standardization _____

Relative error from EDTA titrant volumes above _____

Overall relative error for above analysis _____

Mean $[Ca^{2+}]$ in ppm for your water sample _____

+/- Abs. std. dev. _____

+/- 95% conf. int. _____

On the "Hardness scale" this water sample would be considered _____