Lab 6
Complexiometric Determination

Partner: Linh Nguyen

10/22/2019

Prelab Assignment

Concentration of EDTA, $[EDTA] = \frac{3.7145\,g\left(\frac{1\,mol}{372.24\,g}\right)}{1.000\,L} = 9.979\times10^{-3}\,M$

At pH 10, EDTA reacts with both Mg^{2+} & Ca^{2+}
At pH 12, EDTA only reacts with Ca^{2+}

pH 10

Titrated volume EDTA = 11.23mL = 0.01123L

mol EDTA = $0.01123\,L\left(\frac{9.979\times10^{-3}\,mol}{1\,L}\right) = 1.121\times10^{-4}\,mol$

since the reaction is 1:1 ratio (between EDTA & Mg^{2+} & Ca^{2+}),

$\Rightarrow$ mol of $\{Mg^{2+}\}$ + mol of $\{Ca^{2+}\}$ = $1.121\times10^{-4}\,mol$

pH 12

Titrated volume = 2.11mL = 0.00211 L

mol EDTA = $0.00211\,L\left(\frac{9.979\times10^{-3}\,mol}{1\,L}\right) = 2.11\times10^{-5}\,mol$

Since the reaction is 1:1 ratio (between EDTA & Ca^{2+}),

$\Rightarrow$ mol of $\{Ca^{2+}\} = 2.11\times10^{-5}\,mol$

mol of $\{Mg^{2+}\} = 1.121\times10^{-4}\,mol - 2.11\times10^{-5}\,mol = 9.10\times10^{-5}\,mol$

$[Mg^{2+}] = [MgSO_4] = \frac{9.10\times10^{-5}\,mol}{0.100\,L} \cdot \left(\frac{120.367\,g}{1\,mol}\right)\left(\frac{1000\,mg}{1\,g}\right) = 110\,mg/L$

$[Ca^{2+}] = [CaCl_2] = \frac{2.11\times10^{-5}\,mol}{0.100\,L} \cdot \frac{2.11\times10^{-4}\,M}{\left(\frac{110.9848\,g}{1\,mol}\right)\left(\frac{1000\,mg}{1\,g}\right)}$

$= 23.4\,mg/L$

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Mass of EDTA (g) → 0.8653 .

Mass of empty beaker (g) → 119.8945

Mass of beaker + EDTA (g) → 120.7598 .

Molarity of EDTA solution (M) → 0.009298 .

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Objective

The purpose of this experiment is to determine the concentration of calcium ions and magnesium ions in a water sample.

Introduction

The concentration of magnesium and calcium ions in the water sample can be determined by titrimetrically mixing a known concentration of EDTA into ~~a sol~~ the water sample containing an indicator. This determination is done in two parts, where. Eriochrome Black T is used as the indicator in the first part and hydroxynaphthol blue is used as the indicator in the second part. The first part will determine the sum of concentration of calcium and magnesium ions in the water sample at pH 10, where the titrant turned blue from red at end point. The second part will determine only the concentration of calcium ions in the water sample, as the magnesium ions have reacted with OH^- to form solid, at pH 12, where the titrant ~~turned~~ retained the blue color for a ~~co~~ long period. From there, the magnesium ions can be calculated from two measured/calculated values.

Procedure

1. Collect a 500mL water sample in a clean, dry beaker.

2. Weigh about 0.9g of sodium EDTA solid to the nearest 0.1mg. & place the weighed EDTA in a 250mL beaker.

3. Dissolve the EDTA in about 100mL of distilled water, which will occurs slowly but can be speeded up by warming the solution gently.

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Eriochrome Black T as indicator

Sample	Reference	1	2	3
Initial buret reading (mL)		0.31	0.69	13.35
Final buret reading (mL)		12.97	13.35	26.02
Titrated volume (mL)		12.66	12.66	12.67

Eriochrome Blank T indicator			
Sample number	1	2	3
Volume of sample water (mL)	75.0	75.0	75.0
Initial buret reading (mL)	0.31	0.69	13.35
Final buret reading (mL)	12.97	13.35	26.02
Titrated volume (mL)	12.66	12.66	12.67
Mol EDTA titrated (mol)	1.177E-04	1.177E-04	1.178E-04
Total mol of Mg^{2+} and Ca^{2+} (mol)	1.177E-04	1.177E-04	1.178E-04

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4. After the EDTA dissolves, transfer the solution quantitatively to a 250mL volumetric flask. Fill the flask to the mark with water, insert the stopper & shake to mix the solution thoroughly.

5. Make a reference sample containing 75.0mL of DI water, the pH 10 buffer (2mL), & the indicator (2-4 drops of Eriochrome Black T). Use this sample to find out the end point of titration.

6. Use a pipet (3 x 25.0mL) to deliver 75.0mL water sample into each of 3, 250mL Erlenmeyer flasks.

7. Add 2mL of pH 10 buffer & 2-4 drops of Eriochrome Black T indicator solution to each flask (solutions should be red)

8. Fill a 50mL buret with the EDTA solution & record the initial volume to the nearest 0.01mL. Titrate each water sample to the end point where the titrant solution changes abruptly from red to blue. Record each end-point volume to the nearest 0.01mL).

9. Make a reference sample containing 75.0mL of DI water, the NaOH solution (10mL of 0.1M), & the indicator (0.1g of calcon). Use this sample to find out the end point of titration.

10. Again pipet 75.0mL of water into each of 3 250mL Erlenmeyer flasks. Add 10.0mL of 0.1M NaOH into each flask using a graduated cylinder, and swirl to mix well. A white precipitate of $Mg(OH)_2$ may be visible at this point.

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Calcon as indicator

Sample	Reference	1	2	3
Initial buret reading (mL)	∅	0.68	11.4	22.19
Final buret reading (mL)		11.4	22.19	32.95
Titrated volume (mL)		10.72	10.79	10.76

Calcon Indicator			
Sample number	1	2	3
Volume of sample water (mL)	75.0	75.0	75.0
Initial buret reading (mL)	0.68	11.4	22.19
Final buret reading (mL)	11.4	22.19	32.95
Titrated volume (mL)	10.72	10.79	10.76
Mol EDTA titrated (mol)	9.968E-05	1.003E-04	1.000E-04
Mol of Ca^{2+} (mol)	9.968E-05	1.003E-04	1.000E-04
Mol of Mg^{2+} (mol)	1.807E-05	1.742E-05	1.770E-05
Mass of $CaCl_2$ (mg)	1.106E+01	1.113E+01	1.110E+01
Mass of $MgSO_4$ (mg)	2.1750	2.0966	2.1302
Concentration $CaCl_2$ in water (mg/L)	147.5	148.5	148.0
Average concentration $CaCl_2$ (mg/L)	148.0		
Standard deviation of $CaCl_2$ (mg/L)	0.5		
RSD of $CaCl_2$ (%)	0.33		
Concentration $MgSO_4$ in water (mg/L)	29.0	28.0	28.4
Average concentration $MgSO_4$ (mg/L)	28.5		
Standard deviation of $MgSO_4$ (mg/L)	0.5		
RSD of $MgSO_4$ (%)	1.8		

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11. Add about 0.1 g of calcon indicator to 1 of the flasks. Record the initial volume to the nearest 0.01mL, then titrate to a blue end point. (not to overshoot and compare the colors with reference sample ~~to~~, which has calcon in excess EDTA, to check for the end point).

12. Let the flask stand for 5 minutes after reaching the end point to ensure any $Ca(OH)_2$ that precipitates can dissolve and react with EDTA.

13. Continue the titration to a blue, permanent end point (5-10 minutes), if the color of the titrant solution has changed back from blue to purple.

14. Repeat the steps with the remaining samples.

15. Dispose of the EDTA standard solution, the titration solutions & other solution as indicated.

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Analysis & Results:

The analysis and calculations were made in the Excel file included together with this lab report. The concentration of each ions in the water sample are as affected in this book.

Conclusion

Two different pH (10 & 12) solutions were prepared to determine the concentration of Ca^{2+} & Mg^{2+} ions in the water sample. Eriochrome Black T indicator is used to form a red complex in the water sample with either Ca^{2+} & Mg^{2+} ions. In this pH 10 solutions, the total mol of both ions are determined. At pH 12 meanwhile, ~~a~~ calcon was used as indicator, and NaOH was added to isolate the Mg^{2+} ions and formed $Mg(OH)_2$ solid, in order to determine the concentration of Ca^{2+} ions only. From here, the concentration of Mg^{2+} ions are determined by finding the different between the average total moles of Ca^{2+} and Mg^{2+} in the pH 10 solution and each beaker's corresponding mol of Ca^{2+}. Then, the concentration of $MgSO_4$ and $CaCl_2$ in the water sample in mg/L can be determined.