Complexiметric Analysis of River Water for Magnesium and Calcium Ions

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October 22nd, 2019 Partner: Linh Nguyen

Summary Report

The objective of this experiment was to determine the concentration of magnesium and calcium ions in water sample. In order to do that, the water samples were titrated with ethylenedinitrilotetraacetic acid (EDTA). At first, the titration was done at pH 10, where the total concentration of magnesium (Mg^{2+}) and calcium (Ca^{2+}) ions in the water were determined. Then, at pH 12, only the concentration of calcium ions in the water could be determined, in which consequently, the concentration of magnesium ions was calculated by finding the difference. To prepare the EDTA solution, sodium EDTA solid was weighed and dissolved with distilled water (DI), before being diluted into a 250 mL volumetric flask up to its mark. About 0.9 g of EDTA solid was used to prepare the solution. The final concentration of EDTA solution was as shown in **Table 1** below. At pH 10, a reference sample containing 75 mL of DI water was made, and 2 mL of pH 10 buffer solution and 4 drops of Eriochrome Blank T indicator were added. The Eriochrome Black T indicator will form a red complex with either Ca^{2+} or Mg^{2+} ions in the water sample. This sample was used as reference for the next three titrations, to ensure that the titrations had reached their endpoints, from red to blue color. For each replicate of titration, 75 mL of water sample was pipetted into 250 mL Erlenmeyer flask and 2 mL of pH buffer and 4 drops of Eriochrome Black T indicator were added. A 50-mL buret was filled with the EDTA solution and the initial buret reading was recorded. The solution was titrated into each of the flask until it reached its endpoint. The final buret reading was recorded and the titrated volume of EDTA solution was calculated, as tabulated in **Table 2** below. By using the same EDTA solution, it was used to titrate the next three replicate solutions at pH of 12. Like the solution at pH 10, a reference sample was made containing 75 mL of distilled water, 10 mL of 0.1 M sodium hydroxide (NaOH) solution and 0.1 g of calcon (the indicator). The NaOH was added to react with Mg^{2+} ions to form a solid $Mg(OH)_2$, in order to only isolate and determine the concentration of Ca^{2+} ions in water sample. This sample was being used as the reference to determine the end-point color of the titrated sample, which turned from purple to blue. For each replicate titration, 75 mL of water sample was pipetted into the 250 mL Erlenmeyer flask and 10 mL of NaOH solution was added. About 0.1 g of calcon indicator was added into one of the flasks at a time, right before it was titrated. The buret was refilled with the EDTA solutions and the initial buret reading was recorded. The sample was titrated until the endpoint had reached. After 5 minutes of permanent blue color, the final buret reading was recorded and the titrated volume was calculated and tabulated as shown in **Table 3.** Titration of EDTA was continued if the sample changed color back from blue to purple.

Mass of empty beaker (g)	119.8945
Mass of beaker + EDTA (g)	120.7598
Mass of EDTA solid (g)	0.8653
Total volume (mL)	250.00
Concentration of EDTA solution (g/L)	3.4612
Molecular weight of disodium EDTA (g/mol)	372.24
Molarity of EDTA solution (M)	0.009298

Table 1: Molarity of EDTA solution used in both pH 10 and pH 12 solution

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

Table 2: The total number of moles of Mg^{2+} and Ca^{2+} ions at pH 10 solution

Based on **Table 2**, the number of moles of EDTA solution titrated was equivalent to the total number of moles of Mg^{2+} and Ca^{2+} ions in the water sample. This was because the reaction between the ions in the water sample and the EDTA solution was 1:1 ratio stoichiometrically.

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		

Table 3: The number of moles of Mg^{2+} and Ca^{2+} ions in the water sample

By referring to **Table 3**, the number of moles of Ca^{2+} ions were equivalent to the number of moles of EDTA solution titrated. This was again because the reaction between EDTA solution and the Ca^{2+} ions was in 1:1 ratio stoichiometrically. The number of moles of Mg^{2+} ions were then calculated by subtracting the total number of moles of Mg^{2+} and Ca^{2+} ions in **Table 2** with the number of moles of Ca^{2+} ions in **Table 3**. The concentration of $CaCl_2$ and $MgSO_4$ in water in mg/L were then determined by using the molar mass as provided in **Table 4**. The formula used to calculate data values tabulated in **Table 1 - 3** were as listed in **Table 5**.

Molar mass (g/mol)	
$MgSO_4$ molar mass	120.366
$CaCl_2$ molar mass	110.98

Table 4: Molar mass used to determine each concentration of ions in the water

Formula	
Concentration of EDTA solution (g/L)	Mass of EDTA solid (g) / Total volume of solution (L)
Molarity of EDTA solution (M)	Concentration of EDTA solution (g/L) / Molar mass of EDTA (g/mol)
Mole of EDTA titrated (mol)	Molarity of EDTA solution (mol/L) * Titrated volume (L)
Mole of Mg^{2+} and Ca^{2+} ions (mol)	Same as mol of EDTA, since it's 1:1 ratio of reaction
Mole of Ca^{2+} ions (mol)	Same as mol of EDTA, since it is a 1:1 ratio reaction
Mole of Mg^{2+} ions (mol)	Average mol of Mg2+ and Ca2+ ions in the black indicator solution - mol of Ca2+ ions in the calcon indicator solution
Mass of $CaCl_2$ (mg)	Mole of Ca2+ (mol) * Molar mass CaCl2 (g/mol) * 1000 (mg/g)
Mass of $MgSO_4$ (mg)	Mole of Mg2+ (mol) * Molar mass MgSO4 (g/mol) * 1000 (mg/g)
Concentration Ca^{2+} in water (mg/L)	Mass of CaCL2 (mg) / Volume of sample water (L)
Concentration Mg^{2+} in water (mg/L)	Mass of MgSO4 (mg) / Volume of sample water (L)
Average concentration Ca^{2+} (mg/L)	AVERAGE(three sample's concentration) function in excel
Average concentration Mg^{2+} (mg/L)	AVERAGE(three sample's concentration) function in excel
Standard deviation (mg/L)	STDEV(three sample's concentration) function in excel
RSD (%)	Standard deviation / Average * 100 %

Table 5: Formula used to evaluate data values in Table 1 – 3