

***Analytical Chemistry***

***CHEM234***

***Sec 1***

***Unknown: I***

***Exp7: Title***

***EDTA determination of water hardness using calmagite***

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**Abstract:**

Using complexes termed chelates, which are produced by binding a metal ion to a multidentate ligand like EDTA \* The EDTA is the most important chelating agent in analytical chemistry, its used for the determination of the metal ion contents in different samples. Complex metric titrations are especially effective for determining a mixture of various metal ions in solution and The “total hardness” of the water is defined as the concentration of Calcium plus Magnesium. While these metals are essential elements for human health, high concentrations of these cations (Ca2+ and Mg2+) in water can produce deposits in bathrooms and kitchens, resulting in "soap scum." The objective of this experiment is to use EDTA titration and a calmagite indicator to measure the overall hardness of water. To do this, EDTA solution is titrated against a main standard Calcium II solution using the calmagite indicator, which produces a colored complex when reacting with Ca2+, but this color disappears when an excess of EDTA is present, resulting in a colorless complex with Ca2+.

The color change from wine red to deep blue

**In our experiment, the major reactions that would occur are:**

**1). CaCO3(s) + 2H+ Ca2+ + H2O +CO2**

**2). 2H2Y2- + Ca2+ + Mg2+ CaY2- + MgY2- + 4H+**

**3). H2Y2- + MgIndic- MgY2- + HIndic2- + H+**

**The final result:**

The 95% confidence interval (µ) of EDTA concentration is 0.006614 ± 0.0000547 M and the total hardness of CaCO3 in unknown is 135.49 ± 23.79 ppm

**General observations:**

Color of the solution before adding Calmagite indicator: colorless

Color of the solution after adding Calmagite indicator: wine-red

* **Data and results:**

Mass of calcium carbonate: 0.4998 g

Molecular weight of calcium carbonate: 100.0869 ~ 100.09 g/mol

Table\_1: Standardization of EDTA for known sample

|  |  |  |  |
| --- | --- | --- | --- |
|  | Trial #1 | Trial #2 | Trial #3 |
| Initial volume of EDTA (mL) | 0.00 | 0.00 | 0.00 |
| Final volume of EDTA (mL) | 30.10 | 30.30 | 30.20 |
| Net volume of EDTA (mL) | 30.10 | 30.30 | 30.20 |
| Molar concentrations of EDTA (M) | 6.636 \* 10-3 | 6.592 \* 10-3 | 6.614 \* 10-3 |
| EDTA average volume (mL) | 30.20% |

* **For the known solutions (Trial 1):**
* Net volume of EDTA
 = Final volume of EDTA (mL) – Initial volume of EDTA (mL)
 = 30.10 – 0.00 = 30.10 mL
* EDTA average volume (mL)/3
= Trial1 +Trial2 + Trial3 / 3
= (30.10 + 30.30 + 30.20)/3 = 30.20 mL
* Moles of Ca (II) = mass of Ca(II)/ Molar mass of Ca(II)
= 0.4998/100.09 = 4.994 \*10-3 mole
* Molar concentration of Ca (II)

= moles of Ca (II) / total Volume of solution

 = (4.994 \*10-3/500.00) \*103 = 9.987 \* 10-3 M.

* Molar concentrations of EDTA
 =(Molar concentration of Ca(II) \* Volume of Ca(II))/ (Volume of EDTA))
 =((9.987 \* 10-3 \* 20)/30.10) = 6.636\* 10-3 M.
* **Average molar concentrations of EDTA :**
= Trial1 +Trial2 + Trial3 / 3
= (6.636 \* 10-3 + 6.592 \* 10-3 + 6.614 \* 10-3) / 3

**=** 6.614 \* 10-3 M

* **Standard deviation of sulfate ion in the samples (%):**
(s) =

2.2 \* 10-5

* **Grubbs test:**Gcalculated = ((questionable value – mean) / (s))
= ((6.636 \* 10-3 – 6.614 \* 10-3) / (2.2 \* 10-5))
= 1.000
- The suspension value isn’t outlier because the G table > G test
- **The G table** confidence level of 95% & n = 3 = **1.153
1.000 < 1.153**
* **RSD %:
Coefficient of variation = ((s\x) \* 100)**= (2.2\*10-5 / 6.614 \* 10-3) \* 100% = 0.3326 %
* **95 % confidence interval(**
= 0.006614 ± ((4.303 \* 2.2\*10-5) /
= 0.006614 ± 0.0000547
* Table\_2: Determination of Total Hardness of water for unknown sample

|  |  |  |  |
| --- | --- | --- | --- |
|  | Trial #1 | Trial #2 | Trial #3 |
| Initial volume of EDTA (mL) | 0.00 | 0.00 | 0.00 |
| Final volume of EDTA (mL) | 10.70 | 9.40 | 10.60 |
| Net volume of EDTA (mL) | 10.70 | 9.40 | 10.60 |
| Total hardness (ppm) CaCO3 | 141.67 ppm | 124.46 ppm | 140.34 ppm |

* **For trial 1 of the unknown solutions:**
* **Net volume of EDTA
 = final burette reading – initial burette reading
 =**10.70 – 0.00 = 10.70 ml**= 10.70 \* 10-3 L**
* **Total hardness (ppm) CaCO3**

= (Volume of EDTA (L) \* average concentration of EDTA \* molar mass of CaCO3\* 1000)/ Volume of the sample (L)

 = **(10.70\*10-3 \* 6.614 \* 10-3 \* 100.09\* 1000)/(0.05) = 141.67 ppm.**

* **Average hardness (ppm):**
= Trial1 +Trial2 + Trial3 / 3
= (141.67 + 124.46 + 140.34) / 3

**= 135.49** M

* **Standard deviation of average hardness (ppm):**(s) =

 9.575 ppm

* **Grubbs test:**Gcalculated = ((questionable value – mean) / (s))
= ((141.67-135.49) / (9.575))
= 0.6454
- The suspension value isn’t outlier because the G table > G test
- **The G table** confidence level of 95% & n = 3 = **1.153
0.6454 < 1.153**
* **RSD %:
Coefficient of variation = ((s\x) \* 100)**= (9.575 / 135.49) \* 100% = 7.067 %
* **95 % confidence interval(**
= 135.49 ± ((4.303 \* 9.575 ) /
= 135.49 ± 23.79 ppm
* **Discussion and conclusion:**

In the table\_1 the molar concentrations of EDTA 3 trial = 6.614 \* 10-3 M , and the Standard deviation of sulfate ion in the samples (%): = 2.2 \* 10-5

In table\_2 shows that no rejected samples were produced using the 95 percent G-Test that we used on our results in table\_2 of evaluating the unknown sample. As a result, the mean of the three trials' Total hardness (ppm) CaCO3 = ((141.67 + 124.46 + 140.34) / (3)) = 135.49 M, with a Standard deviation of average hardness (ppm) of 9.575 ppm.

The G test shows whether the values are far from each other or close \* and if the value is more than 1.153 (G table), this means that the value is outlier and should not be taken and if the value is less than 1.153 we take the value because it is true. however, my result was 1.000 in the known value while unknown my value was 0.6454. The both values are less than 1.153, so they are both true.

We added approximately 2.5 mL of HCl to enhance CaCO3 solubility since, as previously said, the complex is strong and stable, so we need to weaken it, and we heated the CaCO3 solutions to remove CO2 gas. The color of the Calmagite indication shifted from wine-red to dark blue.

The final result of 95% confidence interval for unknown = 135.49 ± 23.79 ppm

The final result of 95% confidence interval for known = 0.006614 ± 0.0000547, the average 7.161 \* 10-3 to 6.559 \* 10-3

There are many methodological errors that exist, and one of these common errors is that when the solution is poured into buret in the presence of a glass funnel, it isn’t removed during titration after the required solution has been poured causing an increase in the error rate, because it may be contaminated or otherwise, and it is possible that The buret is contaminated and reading through the buret may be inaccurate. One common error is bubbles in the buret. Finally, these errors should be avoided by paying attention that when filling the burette with the solution, we remove the glass funnel in order to take the reading correctly and that we wash the buret properly.