Experiment 3: EDTA Determination of Total Calcium and Determination of Individual Calcium and Magnesium

Quantitative Analysis

Section: 001

Date lab completed: February 22, 2012

Unknown #E

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Purpose:

The purpose of this experiment is to determine the amount of total calcium and the individual values of calcium and magnesium. In addition, this lab also concerns the determination of water hardness. Hardness is defined as the presence in water of salts of calcium and/or magnesium. The notion of hardness is caused by either bicarbonates, such as Ca(HCO3)2 and Mg(HCO3)2, and sulfates.

Ca(HCO3)2 🡸🡺 CaCO3 + H2O + CO2 (1)

Mg(HCO3)2 🡸🡺 MgCO3 + H2O + CO2 (2)

The total hardness of water varies and can be determined through calculation for ppm. In this lab, total hardness is determined through titration using ethylenediaminetetraacetic disodium salt, EDTA. The complete dissociation of the disodium dihydrogen salt is shown in the subsequent reaction.

Na2H2Y 🡺 H2Y2- + 2Na+ (3)

The reactions shown below are used to determine the amount of calcium and magnesium in relation to EDTA.

Ca2+ + H2Y2- 🡺 CaY2- + 2H+ (4)

Mg2+ + H2Y2- 🡺 MgY2- + 2H+ (5)

Materials:

|  |  |
| --- | --- |
| Equipment used: | Chemicals used: |
| Analytical scale #4  25 mL volumetric flask  50 mL Burette  Glass stopper  1 L storage bottle  400 mL beaker  100 mL beaker  10 mL graduated cylinder  100 mL graduated cylinder  Pipette  Stirring rod  25 mL volumetric pipette and bulb  Parafilm  Hot plate  1L volumetric flask  Scoopula  250 mL Erlenmeyer flask  1000 µL micropipette with tips  Ion Chromatography  Drying oven  Desiccator | Nanopure water  EDTA  MgCl2\*6H2O  CaCO3  12 M HCl  NH4/NH4OH buffer A  Absorbic Acid  Calmagite indicator  Unknown E |

Procedure:

In a beaker, transfer 3.75-4.00g EDTA and 0.1g MgCl2\*6H2O. Add 400 mL of nanopure water to the same beaker. Using a stirring rod, make sure the solution is homogenous. Transfer the solution to a 1L volumetric flask. Dilute the solution to the meniscus with nanopure water. Transfer the solution to a 1L glass stock bottle. In a separate 1L volumetric flask, prepare the calcium standard. Weigh 1.00g dried primary standard CaCO3 to the nearest 0.1mg. Add 400mL of nanopure water and then 6mL concentrated HCl using a graduated cylinder. Dilute the solution using nanopure water to the meniscus. Using parafilm, cover the top of the volumetric flask. Invert and mix the solution so that the solution becomes homogenous. Calculate the molarity of the solution prepared using the molecular weight for CaCO3.

Obtain a 25mL volumetric pipet and transfer 25mL of the calcium carbonate standard solution previous prepared into a 250mL Erlenmeyer flask. Add 10 mL NH4/NH4OH buffer and 10mg absorbic acid. Add 4-5 drops of the calmagite indicator, which will turn the solution to a deep red color. Fill the 50mL buret with the prepared EDTA solution. Titrate to endpoint. The endpoint is achieved once the solution turns from a deep red to a deep blue with no violet present. Repeat this titration four more times. Calculate the molarity of the EDTA solution.

Obtain a 25mL volumetric pipet and transfer 25mL of the unknown solution into a 250mL Erlenmeyer flask. Add 10 mL NH4/NH4OH buffer and 10mg absorbic acid. Add 4-5 drops of the calmagite indicator, which will turn the solution to a deep red color. Fill the 50mL buret with the prepared EDTA solution. Titrate to endpoint. The endpoint is achieved once the solution turns from a deep red to a deep blue with no violet present. Repeat this titration four more times. Calculate the total hardness in ppm.

Prepare an analysis of the unknown for ion chromatography. Make a 10x dilution of the unknown by transferring 2.5 mL of the unknown into a 25 mL volumetric flask using a micropipette. Dilute the volumetric flask to volume using nanopure water. Cover with parafilm and mix by inverting flask. Run unknown in the ion chromatography and record results.

Results:

Molecular Weight CaCO3= 100.09g

Molarity of CaCO3 standard solution =

(Amount of CaCO3) (mol CaCO3/Molecular Weight of CaCO3)

(Total volume of the standard solution)

M CaCO3= (1.0003 g)(1 mol CaCO3/100.09g CaCO3) = 0.009994 M CaCO3

(1.0 L)

MEDTAVEDTA = MCa solutionVCa solution

Trial 1: (MEDTA)(25.40 mL) = (0. 009994 M)(25.00 mL) MEDTA=0.009837 M EDTA

Trial 2: (MEDTA)(25.50 mL) = (0. 009994 M)(25.00 mL) MEDTA=0.009798 M EDTA

Trial 3: (MEDTA)(25.60 mL) = (0. 009994 M)(25.00 mL) MEDTA=0.009760 M EDTA

Trial 4: (MEDTA)(25.70 mL) = (0. 009994 M)(25.00 mL) MEDTA=0.009722 M EDTA

**Mean= ∑ X / n where n is the number of trials**

**SD = √ [ ∑(Xi – Mean)2/ (n-1) ]**

**ppt = 1000 \* SD / Mean**

Mean: (0. 009837 M + 0. 009798 M + 0. 009760 M + 0. 009722 M)/4 = 0.009779 M EDTA

SD= √ [ (0.009837 -0.009779)2 + (0.009798 -0.009779)2 + (0.009760 -0.009779)2 + (0.009722 -0.009779)2/ (4-1) ] = 0.000049

PPT= 1000\*(0.000049)/0.009779 = 5.05

**Table 1: Preparation of Standard Solutions**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Trials | Volume of EDTA solution (mL) | Volume of CaCO3 solution (mL) | Amount of Absorbic Acid (g) | Volume of NH4/NH4OH buffer (mL) | Calculated Molarity of EDTA solution (M) | Total hardness (ppm) |
| 1 | 25.45 | 25.00 | 0.0106 | 10 | 0.009837 | 398.20 |
| 2 | 25.50 | 25.00 | 0.0124 | 10 | 0.009798 | 399.76 |
| 3 | 25.60 | 25.00 | 0.0108 | 10 | 0.009760 | 401.33 |
| 4 | 25.70 | 25.00 | 0.0114 | 10 | 0.009722 | 402.90 |
|  |  |  |  | Mean= | 0.009779 | 400.55 |
|  |  |  |  | SD= | 0.000049 | 2.02 |
|  |  |  |  | PPT= | 5.05 | 5.05 |

*Determination of Total Hardness of unknown E:*

(#M unknown) x (25.0mL sample) = MEDTAVEDTA

Trial 1: (# M unknown)(25.0 mL unknown solution) = (0.009779M)(24.65 mL) = 0.009642 M unknown

0.009642M x 40.078g Ca/mol Ca x 1000 mg/1 g = 386.44 ppm

Trial 2: (# M unknown)(25.0 mL unknown solution) = (0.009779M)(24.50 mL) = 0.009583 M unknown

0.009583M x 40.078g Ca/mol Ca x 1000 mg/1 g = 384.09 ppm

Trial 3: (# M unknown)(25.0 mL unknown solution) = (0.009779M)(24.70 mL) = 0.009662 M unknown

0.009662M x 40.078g Ca/mol Ca x 1000 mg/1 g = 387.22 ppm

Trial 4: (# M unknown)(25.0 mL unknown solution) = (0.009779M)(24.85 mL) = 0.009720 M unknown

0.009720M x 40.078g Ca/mol Ca x 1000 mg/1 g = 389.57 ppm

Mean: (386.44 + 384.09 + 387.22 + 389.57)/4 = 386.83ppm

SD= √ [ (386.44-386.83)2 + (384.09-386.83)2 + (387.22-386.83)2 + (389.57-386.83)2/ (4-1) ] = 2.26

PPT= 1000\*(2.26)/386.83= 5.85

**Table 2: unknown**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Trials | Volume of EDTA solution (mL) | Volume of unknown solution (mL) | Amount of Absorbic Acid (g) | Volume of NH4/NH4OH buffer (mL) | x M Ca (M) | Total Hardness (ppm) |
| 1 | 24.65 | 25.00 | 0.0111 | 10 | 0.009642 | 386.44 |
| 2 | 24.50 | 25.00 | 0.0105 | 10 | 0.009583 | 384.09 |
| 3 | 24.70 | 25.00 | 0.0115 | 10 | 0.009662 | 387.22 |
| 4 | 24.85 | 25.00 | 0.0113 | 10 | 0.009720 | 389.57 |
|  |  |  |  | Mean= |  | 386.83 |
|  |  |  |  | SD= |  | 2.26 |
|  |  |  |  | PPT= |  | 5.85 |

*Determination of Ion Chromatography Calculations:*



Figure 1: Chromatogram: Conductivity vs Time (minutes). This graph illustrates the relationship of conductivity and time. The area under the curve represents proportionality to the concentration of calcium and magnesium.

Run #1: Unknown E

Values displayed on chromatogram, in order: Ion name, retention time (minutes), concentration in ppm

Calcium, 10.06 minutes, 14.751 ppm

Magnesium, 12.30 minutes, 14.814 ppm

Since there was a 10x dilution:

14.751 x (10)= 147.51 ppm Ca2+

14.814 x (10)= 148.14 ppm Mg2+

Convert ppm Mg2+ to ppm Ca2+

148.14 ppm Mg2+ x 40.078g/mol Ca = 244.28 ppm as Ca2+

24.3050 g/mol Mg

Total ppm Ca2+ = 147.51 ppm Ca2+ + 244.28 ppm as Ca2+ = 391.79 ppm total Ca2+



Figure 2: Chromatogram: Conductivity vs Time (minutes). This graph illustrates the relationship of conductivity and time. The area under the curve represents proportionality to the concentration of calcium and magnesium.

Run #2: Unknown E

Values displayed on chromatogram, in order: Ion name, retention time (minutes), concentration in ppm

Calcium, 10.06 minutes, 14.740 ppm

Magnesium, 12.30 minutes, 14.825 ppm

Since there was a 10x dilution:

14.740 x (10)= 147.40 ppm Ca2+

14.825 x (10)= 148.25 ppm Mg2+

Convert ppm Mg2+ to ppm Ca2+

148.25 ppm Mg2+ x 40.078g/mol Ca = 244.46 ppm as Ca2+

24.3050 g/mol Mg

Total ppm Ca2+ = 147.40 ppm Ca2+ + 244.46 ppm as Ca2+ = 391.86 ppm total Ca2+

Average total hardness in ppm from runs #1 and #2: (391.79 + 391.86)/2 = 391.82 ppm

Compare to total hardness calculated from titration: 386.83 ppm Ca2+

The purpose of this experiment as shown from the results is to determine the amount of total calcium and the individual values of calcium and magnesium. In addition, this lab also concerns the determination of water hardness. Additionally, the interpretation of the ion chromatogram results illustrates how the area under the curve is integrated to reveal the relative concentrations of calcium and magnesium. In this experiment, the result of the ion chromatogram is compared to the results of the titration using an unknown E solution.

Conclusion:

The results of this experiment in regards to the Unknown E solution indicated that the unknown solution had a total hardness of 391.82 ppm. Through this experiment, there was the possibility of errors within the technique or data collection. A methodological error could have been possible if the experimenter used an inadequate indicator for the titration. This could have affected the detection of the endpoint for a certain indicator will change abruptly when the titration is complete; the color change is caused by the appearance of excess titrant. In addition, a personal error could have arisen by titrating the solution past the endpoint and misidentifying the endpoint. This systematic error in regards to the determination of the pH levels could have been due to instrumentation that was not properly calibrated. By overshooting past the endpoint, this could increase our ppt values indicating that the values obtained were not acceptable for the precision was compromised. The errors in this experiment are propagated by the standard deviations and the ppt calculated throughout the experiment. The formation of errors could have subsequently increased the standard deviation and ppt as a result of over-titrating the solution. To improve the experimenter’s technique for future experiments, the experimenter should proceed cautiously by doing the following: awareness of contaminants, proper calibration of instrumentation, and observant of the correct stage in the chemical reaction in which the endpoint takes place. In addition, if time permits, repeat procedure for multiple trials. All of the previous tips listed are to ensure precision and accuracy of the data for future experiments. To increase precision and accuracy, performing the experiment again with multiple trials would ensure reliable data.