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Calcium Analysis in Ca-products

DESIGN REPORT

April 9, 2014

By Section 1, Group 1

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April 9, 2014

Dear Dr. Hamade:

It was the first time to learn chemistry laboratory in the university, I learned how to analyze general chemistry concepts in real laboratory as a future engineer. Due to your effort on devising your own straightforward method-"Dr.Hamade's way"-on understanding some complex concepts, we could learn better and clearly understood those concepts. Therefore, we decided to set our goal as giving clear and easy explanation so that every classmates or even those who are not so familiar with studying chemistry can explicitly understand. This design report introduces an experiment about the analysis of calcium in Ca-products. My efforts are about providing a way to measure the content of calcium in calcium tablet and use some equations to get the result. This experiment use the knowledge in the experiment 1 and experiment 3. Also I refer some information from books and websites. By this design report, I'm generally familiar with some concepts and know how to use them. So, Dr, Hamade, and thank you for letting me to have a chance to act in a way that professional engineers usually do. Thank you.

Yours Sincerely,
Peng Wei lun

Abstract

Calcium is essential for living organisms. As a major material used in mineralization of bone, teeth and shells, calcium is the most abundant metal by mass in many animals. Every day you eat enough calcium products, which could make you not only prevent osteoporosis, but also enhance weight loss.

In this experiment, I will design a method to determine the calcium content in calcium products. I will use Ammonium oxalate solution to separate calcium and provide a way called Potassium Permanganate Titration to measure the calcium content indirectly. In addition, I still need the knowledge that I learned in the experiment before like the filtration and pipet.

Potassium permanganate oxidation-reduction titration method is an important titration analysis method, which can be used to determine the content of oxidizing or reducing substances in water. Permanganate index of water quality monitoring is an important indicator which can reflect the degree of organic pollution in water

Introduction

Calcium is essential for living organisms, in particular in cell physiology, where movement of the calcium ion Ca^{2+} into and out of the cell functions as a signal for many cellular processes. As a major material used in mineralization of bone, teeth and shells, calcium is the most abundant metal by mass in many animals. Take a moment and look around your house. Many everyday chemicals used in your home contain calcium. Some of the most common things you may recognize and use include the milk and cheese. Every day we need to eat something to supply the content of calcium. As the most abundant mineral in your body, calcium is essential for our body's overall nutrition and health. As we age, we absorb less and less calcium from our diet, and less of calcium easily cause or contribute to osteoporosis.

Some food can supply enough the content of calcium each day like milk, cheese and yogurt, which are some of the top calcium-rich foods. Also some children eat some calcium tablet which fully contains calcium. The experiment will determine the calcium content in the calcium tablet and judge whether it satisfy the standard printed on the instruction. The experiment conduct the potassium permanganate titration method to determine the calcium content.

Potassium permanganate oxidation-reduction titration method is an important titration analysis method, which can be used to determine the content of oxidizing or reducing substances in water. The reason is that the potassium permanganate solution will change color from dark purple to colorless when it is used up, which can be as an important indicator.

Theory

a) The separation of Ca

In this experiment, we would take the calcium out by precipitating it. So by using $H_2C_2O_4$ to react with the Ca ion, then we could get the precipitation of CaC_2O_4. he main purpose of adding Ammonium oxalate solution in this experiment is to separate Ca^{2+} and avoid influence by other ions, such as Mg^{2+}, Ba^{2+}, Pb^{2+}, Cr^{2+}, Cu^{2+}, Zn^{2+}. By using Ammonium oxalate solution, only Ca^{2+} will precipitate with $C_2O_4^{2-}$. The ion reaction is as follow.

$$Ca^{2+}(aq) + C_2O_4^{2-}(aq) \longrightarrow CaC_2O_4(s)$$

By adding diluted H_2SO_4 solution, the CaC_2O_4 will dissolve. The reactant will be $H_2C_2O_4$ and Ca^{2+}. The ion reaction is as follow.

$$CaC_2O_4(s) + 2H^+(aq) \longrightarrow Ca^{2+}(aq) + H_2C_2O_4(aq)$$

The mole ratio of the reaction is actually 1:1, and thus Ca^{2+} can be determined indirectly by testing the moles of $H_2C_2O_4$. The method to test the moles of $H_2C_2O_4$ is potassium permanganate titration, which will be introduced below.

b) Potassium Permanganate Titration

The main theory in this experiment is potassium permanganate titration. Potassium permanganate, $KMnO_4$, is a strong oxidizing agent. Permanganate, MnO_4^-, is an intense dark purple color. However Mn^{2+} ion is colorless. The standardized solution is then added slowly to the flask until the end point is reached. The solution will turn from dark purple to colorless at the equivalence point. The ion reaction is as follow.

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O$$

In this experiment, potassium permanganate will be as oxidation by $C_2O_4^{2-}$ in acidic conditions. The ion reaction is as follow.

$$2MnO_4^-(aq) + 5H_2C_2O_4(aq) + 6H^+(aq) \longrightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

Experiment setup

Chemicals used	Materials used
1. Standardization of 0.1 *mol / L* KMn O_4 solution	250mL volumetric flask
2. Concentrated H_2SO_4 solution	Erlenmeyer flask
3. 30% H_2O_2 solution	250mL beaker(3)
4. 6 M Ammonia	Glass rod
5. Ammonium oxalate solution(5%)	Funnel
6. Ammonium oxalate solution(0.01%)	Pipet
7. 10% $BaCl_2$	Dropper
8. 10% H_2SO_4	Buret
9. Methyl orange indicator	
10. Calcium tablet sample	

Procedure

a) Take 2g Calcium tablet sample accurately and slowly put it into the bottom of Erlenmeyer flask. Then add concentrated H_2SO_4 into the Erlenmeyer flask and wet the sample evenly. It begin to come up a lot of white smoke. Start heat the solution to boiling for nearly 5 minutes. Then take it down and cool it for nearly half a minute. After that add several drops of 30% H_2O_2 to the solution. Repeat from heating steps to adding several drops of H_2O_2 until the solution are transparent. In the end, boil the solution for nearly 5 minutes to take the H_2O_2 out from solution. Then cool down it.

Caution: If any caustic material is spilled on your skin, immediately rinse with running water and inform your laboratory instructor. In addition, we need to wear our googles at all times when working in the laboratory. Also we need to pay attention to dispose of peroxide waste according to instructor's directions. In general, peroxide waste is not compatible with other chemical waste.

b) Transfer the solution to 250mL volumetric flask. Use the de-ionized water to rinse the Erlenmeyer flask for several times and put it all into the volumetric

flask. Add the de-ionized water to volumetric flask to make a sample solution.

c) Pipet 25mL sample solution from the volumetric flask and add it into a 250mL beaker. Then add 25mL de-ionized water to dilute the sample solution. Along the glass rod adding 25mL 5% Ammonium oxalate solution to the beaker and heating it at 70℃～80℃.

d) Then add 2 or 3 drops of Methyl orange indicator solution to the beaker. With stirring the solution, add 6 M Ammonia until the solution changes color from red to yellow and over add it to make sure the precipitation come out completely. Once the precipitation is not complete, continue to add the 5% Ammonium oxalate solution until the precipitation totally get out. Then heat the solution to make a CaC2O4 crystal precipitation.

e) Rinse the CaC_2O_4 crystal precipitation with 0.1% Ammonium oxalate for three times. Every time use the glass rod to stir the solution in the beaker and put the beaker aside. When the solution is clear, then filtrate it. Then use 40℃ de-ionized water to rinse the CaC_2O_4 crystal precipitation again until the $C_2O_4^{2-}$ ion does not exist. We could use the 10% $BaCl_2$ solution to check it whether the precipitation is rinsed clean.

f) Put the precipitation into a 250mL beaker and add 10% H_2SO_4 to make the precipitation dissolve. Then dilute the solution to nearly 100mL and heat it to 70℃～80℃.

g) Titrate this solution with standardization of 0.1 mol/L $KMnO_4$ solution until the solution changes color to red and it does not change in 30s. Record the volume of standardization of 0.1 mol/L $KMnO_4$ solution used V_1mL.

h) Repeat this experiment for 2 times and record the volume of standardization of 0.1 mol/L $KMnO_4$ solution used V_2mL and V_3mL.

Data and Calculation

a) Data Table

Trail / Vol. (mL)	#1	#2	#3
V_{KMnO_4}			
Average V			

b) Formula and Calculation

$$W_{Ca} = \frac{c_{KMnO_4}(mol/L) \cdot V_{KMnO_4}(mL) \cdot M_{Ca^{2+}}(g/mol) \cdot \frac{n_{Ca^{2+}}}{n_{KMnO_4}} \cdot \frac{1L}{1000mL} \cdot \frac{1000mg}{1g}}{m_{tablet}(mg)} \times 100\%$$

W_{Ca} is the calcium content of the calcium tablet sample;

V_{KMnO_4} is the average V in the data table, and it is the only variable in this equation;

All the followings are constant in this experiment:

$c_{KMnO_4} = 0.1mol/L$; $M_{Ca^{2+}} = 40.08g/mol$; $\frac{n_{Ca^{2+}}}{n_{KMnO_4}} = \frac{5}{2}$; $m_{tablet} = 1250mg$.

Compare the calculation result with the value printed on the instruction of calcium tablet. The standard of calcium content is nearly 19.5%.

The absolute error = $\left|W_{Ca(std)} - W_{Ca(test)}\right|$.

The relative error = $\frac{\left|W_{Ca(std)} - W_{Ca(test)}\right|}{W_{Ca(std)}} \times 100\%$.

Discussion

a) Determination of the Solution Acidity

CaC_2O_4 is weak acid precipitate, the solubility will increase with increasing acidity. When pH is equal 4, almost all of the CaC_2O_4 precipitate will be transfer to $C_2O_4^{2-}$ and Ca^{2+}, and the loss of CaC_2O_4 in dissolution is negligible. So we need to add the acid to make the reaction in a acidic environment.

b) Rinsing the Precipitation of Cac2o4

If the excess 0.25 *mol / L* $(NH_4)_2C_2O_4$ solution cannot be wash clean, the result will be unexpected high. So, I wash the CaC_2O_4 with 0.1% Ammonium oxalate for three times firstly. Then I also use the d-ionized water to rinse it again. The function of 10% BaCl2 solution is to examine whether the $C_2O_4^{2-}$ has been rinsed off.

c) Temperature Control in Titration

When titration is processing, we need to keep the temperature between 70℃ and 80℃. This is because if the reaction temperature lower than 70℃, the reaction rate will be low. If the reaction temperature is higher than 80℃, oxalate would decompose. The reaction formula is as follow.

$$H_2C_2O_4(aq) \xrightarrow{>85℃} CO(g) + CO_2(g) + H_2O(l)$$

d) The security problem

In this experiment, we will use the concentrated H_2SO_4 solution. It is a caustic acid which could easily hurt the skin. So we had better wear gloves. If any is spilled on your skin, immediately rinse with running water and inform your laboratory instructor. In addition, we need to wear our googles at all times when working in the laboratory. Also we need to pay attention to dispose of peroxide waste according to instructor's directions. In general, peroxide waste is not compatible with other chemical waste.

Question

a) Pre-lab question

i. What is method of the Potassium Permanganate Titration

Answer: The potassium permanganate solution will change color from dark purple to colorless when it is used up.

ii. **Write down the ion reaction equation about the Potassium Permanganate Titration in this experiment**

Answer:

$$2MnO_4^-(aq) + 5H_2C_2O_4(aq) + 6H^+(aq) \longrightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

b) **Post-lab question**

i. **Why should we rinse the crystal precipitation so complexly?**

Answer: Because the $(NH_4)_2C_2O_4$ will stick on the surface of precipitation. If the excess $0.25\ mol/L$ $(NH_4)_2C_2O_4$ solution cannot be wash clean, the result will be unexpected high.

ii. **When using $KMnO_4$ to determine the calcium content, could we use HNO_3 or concentrated H_2SO_4 to control the acidity?**

Answer: Nitric acid has oxidation, and hydrochloric acid has reducibility. Using these two chemicals will easily occur side reactions.

Conclusion

Through this design experiment, I learn how to design an experiment by my own. I use the knowledge I got in the experiments before like the titration, filtration and some other reaction methods to figure out a way to determine the calcium content in Cal-products. I also learn how to search the reference to supply my design experiment. Although the experiment I do not really conduct, I at least provide a way to solve calcium content measurement problems. So it really helps me a lot and it will exert an significant effect on my further study.

Reference

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Excellent!