2024-2025学年春夏学期《无机及分析化学实验》实验报告

实验名称：Preparation and Characterization of Mohr's Salt

姓名/同组同学：_____ 实验时间：2025 年 3 月 25 日

指导老师/助教：_____/_____ 第 1 页

Ⅰ. Objectives

(1). Understand the concept and characteristics of double salt.

(2). Prepare and characterize Mohr's salt.

(3). Understand the principles and procedures of permanganate titration.

Ⅱ. Principles

· Mohr's salt: $[(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O]$, light green or blue-green crystalline compound. It is soluble in water but insoluble in several organic solvents.

It does not cake and it is less prone to oxidization, which making it a primary standard.

· Double salt: a crystalline compound that forms from a mixture of two simple salts with different cations but the same anion, and it has a distinct crystal structure compared to either of the simple salt. Solubility is lower than 2. Terefore, Mohr's salt can be obtained through evaporation (蒸发) and cooling of its solution.

· prepare Mohr's salt:

$$Fe + H_2SO_4 = FeSO_4 + H_2\uparrow$$

$$FeSO_4 + (NH_4)_2SO_4 + 6H_2O = (NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$$

Determine Fe: use $KMnO_4$ (self-catalyst and self-indicator)

When using $(Na_2C_2O_4)$ to standardize the $KMnO_4$, it's important to contol the temperature, acidity and titration speed.

$$5Fe^{2+} + MnO_4^- + 8H^+ = 5Fe^{3+} + Mn^{2+} + 4H_2O$$

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ = 2Mn^{2+} + 10CO_2\uparrow + 8H_2O$$

Ⅲ.Pre-lab Questions

(1). How do you prepare a standard $KMnO_4$ solution? Provide a brief description.

First, weight the $KMnO_4$ and dissolve it with water. Then, use the standard $Na_2C_2O_4$ solution to titrate the $KMnO_4$ solution we've prepared. Finally, calculate its solution.

(2). For the standarlization of a $KMnO_4$ solution, what are the effects when the following conditions are higher or lower: temperature, acidity, and titration speed? Give a brief explanation.

- Temperatue: i). higher: lower concentration of $KMnO_4$
  ii). lower: higher concentration of $KMnO_4$.
- Acidity: i). higher: lower concentration
  ii). lower: higher concentration.
- Titration speed: i). higher: lower concentration
  ii) lower: ~~per~~ precise concentration.

(3). When using permanganate titration to determine the iron content in the product, why should the product solution not be heated before titration?

To prevent Fe^{2+} react with O_2 in the air and turns into Fe^{3+}. Also can prevent the Mohr's salt react with $KMnO_4$ vice reaction.

Ⅳ.Procedures

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1. Preparation of $FeSO_4$.

100 mL beaker → iron powder 1.0 g → 3 mol/L H_2SO_4 8mL → stir gently → hot-water bath → heat and stir for about 10 min → add water to 30 mL → stir completely → suction filtration → pale green and clear $FeSO_4$ solution.

ps. (1). If a significant amount of white or pale green solids form in the beaker before suction filtration, add a little more water to dissolve them.

(2). Ensure the total volume is controlled to 30mL, as exceeding this volume will prolong the evaporation time.

2. Preparation of $(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$.

100mL evaporating dish → above solution → $(NH_4)_2SO_4$ 2.3g → boiling water heating → stir to dissolve completely → keep concentrating → form a thick crystal membrane → solve cooling to room temperature → suction filtration rinse twice with ethanol → transfer on a watch glass → dry at 50°C for 5~10min → weight the product and record.

ps: We can gradually see the crystal come out. (Refer to figures in the appendix part.)

Ps. (1). Pour about 180mL of deionized water into a 250 mL beaker, and heat it on a hot plate to prepare a boiling water bath.

(2). Avoid stirring the solution while concentrating to prevent the formation of small, sandy crystals.

(3). Rinse the product with ethanol twice, using 5 mL each time. (Decrease polarity of the solution).

3. Standardization of 0.02 mol/L $KMnO_4$ Solution.

i). Preparation $KMnO_4$ Solution. (0.02 mol/L).

1L beaker → $KMnO_4$ 3.2g → H_2O 1000mL → boil for 15 min → cool to RT → 1L brown reagent bottle

ii). Standardization. 加热一份做一份

150mL Erlenmeyer flask $\xrightarrow[0.12\sim0.15g]{Na_2C_2O_4}$ $\xrightarrow[20\sim30mL]{H_2O}$ $\xrightarrow[10mL]{3mol/L\ H_2SO_4}$ heat to 75~85°C

→ titrate with $KMnO_4$ → from colorless to light pink (no change within 30s) → read the final volume → repeat 2 more times.

4. Determination of the Iron Content in the Product

100mL beaker $\xrightarrow[3.5g]{product}$ $\xrightarrow[6\sim8mL]{3mol/L\ H_2SO_4}$ $\xrightarrow[30mL]{water}$ dissolve completely $\xrightarrow{transfer}$ 100mL volumetric flask $\xrightarrow[mark]{dilute\ to\ the}$ sample solution

100mL Erlenmeyer flask $\xrightarrow[20.00mL]{sample\ solution}$ $\xrightarrow[5mL]{3mol/L\ H_2SO_4}$ titrate with 0.02mol/L standard $KMnO_4$ solution → till light red solution.

Ⅴ.Data Record Tables

Table 1 Preparation of Mohr's salt

Fe/g	$(NH_4)_2SO_4$/g	yield/g	Outside observation of the product
1.01	2.21	5.09	浅绿色晶体

Table 2 Standardization of 0.02 mol·L^{-1} $KMnO_4$ Solution

	1	2	3
$m(Na_2C_2O_4)$ /g	0.1227	0.1281	0.1213
$V_1(KMnO_4)$/mL	2.35	0.64	0.80
$V_1(KMnO_4)$/mL	20.68	19.76	18.80
$\Delta V(KMnO_4)$/mL	18.33	19.12	18.00

Ⅵ. Key Steps and Precautions

1. Avoid stirring the solution while concentrating to prevent the formation of small, sandy crystals.
2. Notice the overall volume of the solution before concentrating the products. Don't keep a large amount of volume.
3. Before the titration step, don't use tap water to wash the burette. Rinse it with deionized water.
4. When titrating with the $KMnO_4$, read the point where the liquid surface meets the scale on the burette.
5. When concentrating the solution, be cautious to identify the crystal coating which determines the time to stop heating.

Ⅶ.Results and Data Processing

According to the recorded data, we can get the following tables.

Table 1 Preparation of Mohr's salt

Fe/g	$(NH_4)_2SO_4$/g	yield/g	Outside observation of the product	Theoretical yield/g	Percent yield/%
1.01	2.21	5.09	浅绿色晶体	7.07	72.0

Table 2 Standardization of 0.02 mol·L^{-1} $KMnO_4$ Solution

	1	2	3
$m(Na_2C_2O_4)$ /g	0.1227	0.1281	0.1213
$V_1(KMnO_4)$/mL	2.35	0.64	0.80
$V_1(KMnO_4)$/mL	20.68	19.76	18.80
$\Delta V(KMnO_4)$/mL	18.33	19.12	18.00
$c(KMnO_4)$/mol·L^{-1}	0.01998	0.02000	0.02012
$\overline{c}(KMnO_4)$/mol·L^{-1}	0.02003		
$\overline{d_r}$ /%	0.29		

Data Treatment: In the standardiztion of 0.02 mol·L^{-1} $KMnO_4$ Solution experiment, calculate the following treatments:

1：$c(KMnO_4)=\frac{m(Na_2C_2O_4)}{M(Na_2C_2O_4)\times\Delta V(KMnO_4)\times10^{-3}}\times\frac{2}{5}=0.01998\ mol\cdot L^{-1}$

2：$c(KMnO_4)=\frac{m(Na_2C_2O_4)}{M(Na_2C_2O_4)\times\Delta V(KMnO_4)\times10^{-3}}\times\frac{2}{5}=0.02000 mol\cdot L^{-1}$

3：$c(KMnO_4)=\frac{m(Na_2C_2O_4)}{M(Na_2C_2O_4)\times\Delta V(KMnO_4)\times10^{-3}}\times\frac{2}{5}=0.02012\ mol\cdot L^{-1}$

$\overline{d_r} = \frac{1}{3} \times \frac{\sum|d_i|}{\overline{c}} \times 100\% = \frac{1}{3} \times \frac{\sum|c_i - \overline{c}|}{\overline{c}} \times 100\% = 0.29\%$

Relative errors: $E_{r_1} = \frac{x_1 - x_T}{x_T} \times 100\% = -0.1\%$, $E_{r_2} = \frac{x_2 - x_T}{x_T} \times 100\% = 0$, $E_{r_3} = \frac{x_3 - x_T}{x_T} \times 100\% = 0.6\%$

Standard deviation: $s = \sqrt{\frac{\sum_{i=1}^{3}(x_i - \bar{x})^2}{2}} = 7.583 \times 10^{-5}$, Class A uncertainty: $u_A = \sqrt{\frac{\sum_{i=1}^{3}(x_i - \bar{x})^2}{3 \times 2}} = 0.00004$

Therefore, the concentration of the $KMnO_4$ solution is $(0.02003 \pm 0.00004)\ mol/L$. This indicates that the solution is very close to the $0.02\ mol/L$. And within a certain margin of error, we can consider its concentration is $0.02\ mol/L$.

Ⅷ.Analysis, Discussion and Summary

Error analysis:

In the experiments, the yield of Mohr's salt is a little bit low and the concentration of the KMnO4 Solution is somehow higher.Here are the possible reasons of the errors:

1. The Mohr's salt crystal is made by heating and cooling, and the precondition is the saturation solution. Therefore, its reasonable that the yield is not 100%.
2. The cooling time maybe not so long so that the crystal's incomplete precipitation.
3. Probably the drying time is a little long, which causes the lose of water in the Mohr's salt.
4. There is some solution remaining in the containers during the pouring process.
5. In the titration process, the temperature of heating $Na_2C_2O_4$ is so high that causes it decompose.
6. While titrating, the temperature is gradually decreasing. This can also influence the usage of $KMnO_4$.
7. Because the color of $KMnO_4$ is purplish, it's difficult to read the burette correctly.

Summary:

1. When seperating the double salt, using its physical characteristics can help a lot.
2. High yield and high quality can not be gotten at the same time.
3. When analyzing the results, I tried to use more analysis methods to process my data, such as relative errors and uncertainty. I'm sure these can verify the results more persuasive.
4. To simplify the total procedures of the experiments, constrain the solution's volume.

Ⅸ. Post-lab Questions

How can all the ions in the product be identified using qualitative analysis methods? Write out all the related chemical equations.

(1) NH_4^+: NaOH solution and heating, use wet phenolphthalein test paper it turns to red.

$$NH_4^+ + OH^- \xrightarrow{\Delta} NH_3\uparrow + H_2O$$

(2) Fe^{3+}: Add KSCN solution and then it turns biood red.

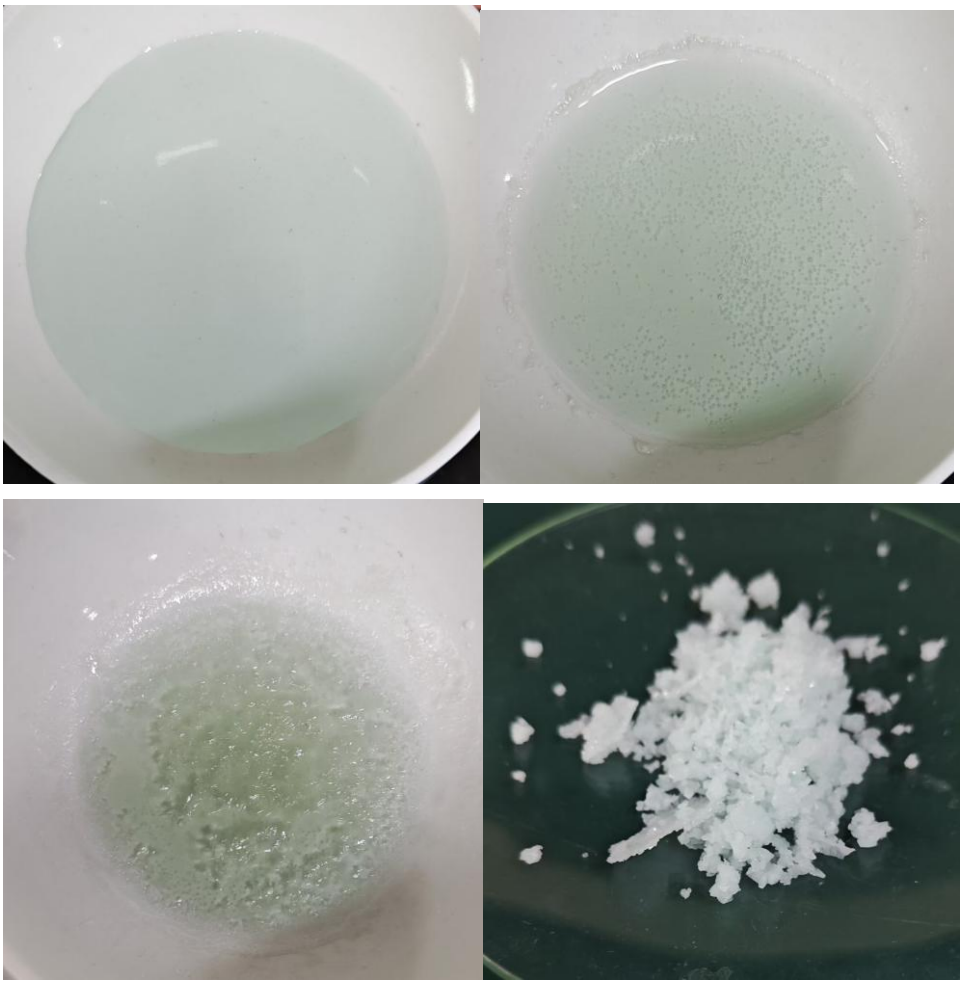
$$F_e^{3+} + 3SCN^- = F_e(SCN)_3$$

(3) SO_4^{2-}: Add $BaCl_2$ solution and it generate white precipitate. After adding HCl solution, it doesn't disappear.

$$Ba^{2+} + SO_4^{2-} = BaSO_4\downarrow$$

Ⅹ. Appendix

Here are some pictures which show the experimental phenomena during the process.



The figures shows the crystal flower, crystal film and the Mohr's salt crystal during the process of concentrating accordingly.

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评分项目	学术规范	书写工整	写作表达	数据结果和分析讨论	课前和课后思考题	总分
分值	30 分	10 分	10 分	30 分	20 分	100 分
得分/分						
评语						