Experiment Time: 2019 / 06 / 11 14 : 00 ~ 16 : 30 Laboratory: DS2B402

Experimental Report

Experiment Course Title: Experimental of College Chemistry I

Experimental Project Name:

Preparation and Characterization of Anhydrous Copper Sulfate Crystal

Name: 易弘睿 Yi Hongrui Student No.: 20186103

College: JCI Specialty: Mechanical Engineering

Class: ME01 Mentor: Chen Gang

Score:_____

Teacher Comments:_____

_____

_____

_____

1. Purpose

(1) Master the preparation of compounds in the process of heating, filtration, recrystallization and other basic operations;

(2) Master the determination of copper content of indirect iodometry.

2. Principle and Method

（1）$CuSO_4 \cdot 5H_2O$ is a kind of blue translucent crystal. And it belongs to triclinic crystal system. Its luster is like glass, crispy and brittle . Molecular weight is 248.68 g/mol, the content of water is 36%. The informal name is blue-stone, which is dissolved in the water easily but difficultly in the ethanol. It will be weathered in the dry air, plus, when the temperature is 230℃, it will lose all of the hydrate to change to the white cupric sulfate.

There are various methods to produce the $CuSO_4 \cdot 5H_2O$, two common methods among them are 'Oxidation of heating the abandoned copper' and 'Using the oxidation of abandoned copper and HNO_3-H_2SO_4'. This experiment is applying the latter one to produce the CuSO4·5H2O crystal. The following is involved reaction:

$$Cu + 2HNO_3 + H_2SO_4 == CuSO_4 + 2NO_2 + 2H_2O$$

Filtering the solution after reaction to extract those impurities that cannot be dissolved. The impurities that could be dissolved in the solution are Fe2+ and Fe3+. For this, first, using H2O2 to oxidize the Fe2+ to Fe3+, second, adjusting the PH value to 3 approximately. Finally, heating the solution to boil, then the iron will be extracted as precipitation Fe(OH)3.

$$2Fe^{2+} + 2H^{+} + H_2O_2 == 2Fe^{3+} + 2H_2O$$

$$2Fe^{3+} + 6OH^{-} == 2Fe(OH)_3\downarrow$$

Due to the solubility of $CuSO_4 \cdot 5H_2O$ is increasing as the temperature increases in the water. Therefore, applying the 'evaporation and concentration' and 'cooling and filtering' to gain the respective pure blue $CuSO_4 \cdot 5H_2O$ crystal. Observing the appearance of the crystal by microscope.

（2） Analyze the components.

In the weak acid surroundings, Cu2+ reacts with excessive KI and produce the CuI and I2. Use the starch solution as indicator, what's more, using the $Na_2S_2O_3$ solution to titrate the produced I2.

$$2\ Cu2+ + 4I- == 2CuI\downarrow + I2$$

$$I2 + 2\ S2O32- == 2I- + S4O62-$$

According to the volume and concentration of the standard Na2S2O3 solution, calculate the amounts of Cu^{2+} to know the content of Cu in the $CuSO_4 \cdot 5H_2O$ clearly.

It is necessary to remember that the external CuI is easy to absorb the I_2 to reduce the accuracy and sensitivity of the end point of titration, which leads to the error. Therefore, usually, add fixed suitable amounts of KSN (or NH4SCN), It will transfer the CuI into CuSCN that is a precipitation which has smaller solubility.

$CuI + SCN^- == CuSCN\downarrow + I^-$

CuSCN is easy to absorb the SCN^- to leave the I2 which is stuck out to make the titration correctly. In addition, usually, using the NH4HF2 (or H3PO4 and NaF) to control the acid degree in the range of 3.5~4.0. If the solution contains As, Fe, As, Sb and so on, the F- that has been added could cover the Fe^{3+}, plus, when the PH>3.5, the pentavalent As. Sb could not oxidize I^-.

The hydrates of $CuSO_4 \cdot 5H_2O$ could be measured by the TGA (Thermogravimetric Analysis).

3. Materials and Instruments

Instruments: Analytical balance; Platform scale; Evaporating dish; Watch glass; Vacuum pump; Water bath pot; Suction flask; Volumetric flask; Iodine flask; Burette; Beaker.

Materials: Abandoned copper powder; H_2SO_4(3 mol/dm3); HNO_3(strong); H_3PO_4(strong); KI(1 mol/dm3); KSCN(10%); HCl(1:1); Starch solution(0.2%); Ammonia(1:1); HAc(1:1); H2O2(3%); $Na_2S_2O_3$ standard solution(0.1000 mol/dm3).

4. Experimental Records and Calculation

The mass of $CuSO_4 \cdot 5H_2O$ is 1.1999g

$C(Na_2S_2O_3)$ is 0.0508 mol/L

V1 (mL)	2.5	9.42	11.9
V2(mL)	9.42	11.9	15.3
V(Na2S2O3)	6.92	2.48	3.4

V1=6.92+2.48+3.4=12.8=12.8mL

V1 (mL)	13.6	20.2	23
V2(mL)	20.2	23	27.45
V(Na2S2O3)	6.6	2.8	4.45

V2=6.6+2.8+4.45=13.85mL

1^{st} $nCu^{2+}) = C(Na_2S_2O_3) \times \Delta V \times 10 = 6.5 \times 10\text{^-3}$ mol

$2^{nd}C(Cu^{2+}) = C(Na_2S_2O_3) \times \Delta V \times 10 = 7.0 \times 10\text{^-3}$mol

1^{st} $\omega Cu = 6.5 \times 10\text{^-3} \times 64/1.1999 \times 100\% = 34.67\%$

2^{nd} $\omega Cu = 7.0 \times 10\text{^-3} \times 64/1.1999 \times 100\% \times 5 = 22.41\%$

$\omega average = (34.67\% + 22.41\%)/2 = 28.54\%$

$\omega_{标准} = 64/250 \times 100\% = 25.6\%$

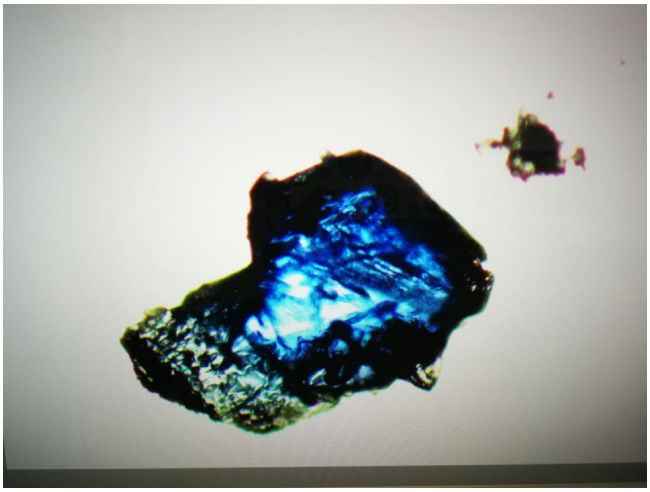
$Error = |\omega_{标准} - \omega average|/\omega_{标准} \times 100\% = 11.48\%$

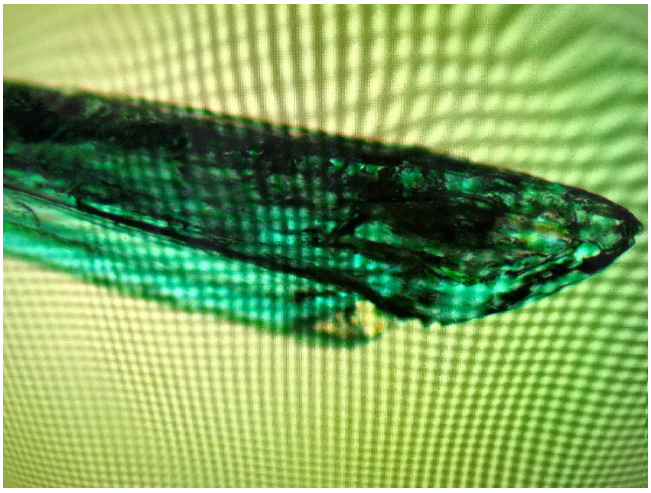
Phenomenon:

At first, the solution is brown yellow; after adding Na2S2O3, solution turns to grey dark blue; after adding 1mL starch, solution turns to grey dark blue (not transparent); after adding mediate Na2S2O3, solution turns to bright grey white; after adding 2mL KSCN, solution turn to dark grey blue (not transparent).

After washing: The copper sulfate crystal is a pile of blue rhombic crystal in different size.

In microscope: The copper sulfate crystal is blue-green and translucent. Some of the crystal are clumps, some are cone - shaped, and some are flaky.





5. Results and Discussion

①The function of calculating ωCu is $C(Na_2S_2O_3) \times \Delta V \times M_{Cu} / m(CuSO_4 \cdot 5H_2O) \times 100\% \times 5$

For the first time of the experiment we get

$1^{st}\ nCu^{2+}) = C(Na_2S_2O_3) \times \Delta V \times 10 = 6.5 \times 10\text{^-3 mol}$

For the second time of the experiment we get

$2^{nd} C(Cu^{2+}) = C(Na_2S_2O_3) \times \Delta V \times 10 = 7.0 \times 10\text{^-3mol}$

Then we calculate the average value of the result.

$\omega average = (34.67\% + 22.41\%)/2 = 28.54\%$

According to $\omega_{标准} = 64/250 \times 100\% = 25.6\%$, we can get the error.

$Error = |\omega_{标准} - \omega average| / \omega_{标准} \times 100\% = 11.48\%$

Error analysis:

The error is 11.48%. due to judging the change of color is hard, there is a great random error. The $Na_2S_2O_3$ added into the solution is superfluous so the ωCu we calculate is great.

②The observation of copper sulfate crystal

After wash : The copper sulfate crystal is a pile of blue rhombic crystal in different size.

In microscope: The copper sulfate crystal is blue-green and translucent. Some of the crystal are clumps,some are cone - shaped,and some are flaky.

③Question and answer

1) When using iodometric method to titrate the Cu^{2+}, what role do NaF and KSCN play? Why could not add KSCN prematurely?

NaF can control the PH between 3.5 and 4.0. NaF can also avoid the effect if there exists As,Sb, Fe. KSCN can transfer the CuI into CuSCN. KSCN can enable I_2 to be released from CuI, because KSCN can react with CuI which can adsorb I_2 easily.

If add the KSCN solution so early, it will react with Cu^{2+} , which will affect the result of the content of copper. It will effect the results of the experiment.

2) Why iodometric method can not only determine the reduction of substances, but also the determination of oxidizing substances?

Because the I2 has both oxidability and reducibility, it could be oxidizing agent and reducing agent.