OFFICE FOR INTERNATIONAL STUDY PROGRAM

FACULTY OF CHEMICAL ENGINEERING

VIETNAM NATIONAL UNIVERSITY – HO CHI MINH CITY

HO CHI MINH CITY UNIVERSITY OF TECHNOLOGY

EXPERIMENTAL REPORT

GENERAL CHEMISTRY LABORATORY

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Class CC02 - Group 4

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TABLE OF CONTENTS:

Date: 23/10/2023

Class CC02 - Group 4

<u>**Unit 1**</u>: **EXPERIMENTAL TECHNIQUES**

1.Abstraction

In this experiment, we are learning experimental techniques as well as how to use the apparatus correctly. We learned how to determine exactly the volume of liquid by using apparatus and learned how to have distilled liquid.

2. Introduction

2.1. Glassware

Glassware is the apparatus which is used in many experiments. Glassware can be classified into three main categories:

- Glassware is used for temporary storing chemicals and conducting reactions:

> + A test tube.
>
> + A beaker.
>
> + An Erlenmeyer flask.
>
> + A round-bottom flask.

- Glassware is used for measuring chemicals (volumetric types):

> + A volumetric pipette.
>
> + A volumetric flask.

-Glassware is used for measuring chemicals (Graduated types):

> + A burette.
>
> + A graduated pipette.
>
> + A graduated cylinder.

2.2. Other apparatus:

A separatory funnel and a condenser.

2.3. Filtration:

A filtration is used to separate the solid from a fluid through the use of a porous medium as a filter. Paper, charcoal or sand can be a filter. These materials allow the fluid to go through them but the solid is not allowed:

> + Gravity filtration.
>
> + Vacuum filtration.

2.4. Some common devices:

To have a nearly exact measurements during laboratory practices, several measuring devices are used:

+ Top-loading balances.

+ Analytical balances.

+ Furnaces, drying ovens and desiccator

2.5. Warewashing:

Every laboratory ware needs to be washed before using them. First, rinse them with tap water. Whenever the dirt is not removed, we need to use a special test-tube brush. There are several chemicals that can be used to clean glassware such as a warm 10% detergent solution, a sulfochromic mixture, concentrated acid, and alcohol solution of sodium hydroxide.

When needed, dry wares in a drying oven (except for measuring glassware, which are never dried by heating). Another drying method is by rinsing with pure acetone and blowing to dry.

2.6. Theory:

Law of equivalent proportion: Substances react or combine with each other in quantities proportional to their equivalent weights. In a reaction, the number of equivalents of all substances should be equal.

2.7. Rinsing of labware and handling chemicals:

Ordinary glassware can be rinsed with distilled water after cleaning and then used if they do not have to be dried. Graduated cylinders and funnels can be rinsed with the liquid to be measured. Two or three small volumes of solution must be used for rinsing pipettes and burettes before filling inside. Rinsing the apparatus can be done by rotating them in a horizontal position so that all the surface can be rinsed wetly.

Nevertheless, reagent bottles and volumetric flasks are never manipulated directly, in this case, a beaker should be used. Subsequently, never pour the unconsumed reagents back into their storage bottles. Thus, merely take the fixed amount of chemicals.

2.8. Deviation and Error:

2.8.1. Deviation:

From the series of measurement values X_1, X_2, . . . , X_n, we get the average:

$$\overline{X} = \frac{X_1 + X_2 + \dots + X_n}{1 + 2 + \dots + n}$$

For each value, the deviation is:

$$\Delta X_i = X_i - \overline{X}$$

Standard deviation:

$$s = \sqrt{\frac{(\Delta X_1)^2 + (\Delta X_2)^2 + \dots + (\Delta X_n)^2}{n - 1}}$$

Deviation:

$$\Delta X = \frac{|X_1| + |X_2| + \dots + |X_n|}{n}$$

2.8.2. Error:

Error can be different between measured value and actual value. This can be estimated as a standard deviation or deviation.

3. Materials and Methods

3.1. Using pipette:

Using a pipette and a bulb rubber to measure 10 mL of water. Do this several times to practice.

3.2. Using burette:

● Use a beaker and pour water into a burette.

● Wait until all bubbles disappear.

● Open the tap to fill the jet, release bubbles inside.

● Close the tap and fill the burette with water.

● Adjust the level of fluid to 0.

● Transfer exactly 10 mL of water from the burette into the beaker.

● Do and repeat some times to practice.

3.3. Oxidation-reduction titration:

● Measure 0.6g of oxalic acid. Record the exact mass to the nearest 0.01g.

● Transfer measured acid into a 100-mL volumetric flask.

● Add a small amount of distilled water and swirl to dissolve the solid.

● Filled the distilled water until reaching the mark.

● Finally, cap, mix, swirl and invert to completely mix the solution.

● Pour this solution into a beaker. Clean the volumetric flask.

- Rinse a burette by distilled water, rinsed again with $KMnO_4$ 0.1 N solution.
- Pour 0.1 N $KMnO_4$ solution into the burette, adjust the liquid level to the zero mark and no bubbles of air are trapped in the jet.
- Transfer 10mL of prepared oxalic acid by a pipette into an Erlenmeyer flask. Add 2mL of concentrated H_2SO_4 solution.
- Titrate the acid solution by 0.1N $KMnO_4$.
- Stop the titration when the solution in conical flask changes to faint-pink color.
- Read the consumed volume of $KMnO_4$ and calculate the concentration of oxalic acid.
- Repeat three times and get the average.

3.4. Dilution of solution:

Measure exactly 10 mL of HCl 1 M by pipette, diluted exactly to 100 mL of the final solution by volumetric flask.

3.5. Checking the concentration of the diluted solution:

- Prepare burette containing 0.1 M NaOH solution.
- Transfer 10 mL of diluted HCl to the Erlenmeyer flask which was rinsed by distilled water.
- Add two drops of phenolphthalein indicator. Titrate the solution. The endpoint is the appearance of faint-pink that does not disappear in 30 seconds.
- Read the consumed volume of NaOH. Compute the concentration of dilute acid solution.
- Repeat three times and get the average.

4. Result and Discussion

Each experiment is conducted for 3 times:

4.1. *Experiment 1*: Oxidation-reduction titration:

	1st time	2nd time	3rd time
Mass of Oxalic Acid (g)	0,61	0,59	0,60
Volume of $KMnO_4$ (ml)	11,4	10,5	10,3

4.2. __Experiment 2__: Checking the concentration of the diluted solution:

	1st time	2nd time	3rd time
Volume of $KMnO_4$ (ml)	10,3	10,4	10,7

5. Conclusion

To conclude, this experiment help us to know the right method to use these apparatus and the significance of the experimental safety. Thanks to these experiments, we canknow how to have an exact data in laboratory.

6. References

[1] Nguyen Tuan Anh, Huynh Ky Phuong Ha, Le Minh Vien (2020), *Laboratory Experiments in General Chemistry.*

Date: 30/10/2023

Class CC02 - Group 4

<u>**Unit 2**: **HEAT OF REACTION**</u>

1. Abstraction

In this experiment, we are learning about the heat of reaction. We determine the heat of reaction in 4 experiments. The result shows that the energy can transfer in every change. These findings have significant implications for our understanding in the heat of the reactions.

2. Introduction

All chemical and physical changes involve the transfer of heat (energy); heat may be either released (exothermic) or absorbed (endothermic). A calorimeter is the laboratory apparatus that is used to measure the quantity and direction of heat flow accompanying a chemical or physical change. The heat change in chemical reactions is quantitatively expressed as the enthalpy (or heat) of reaction, ΔH, at constant pressure. ΔH values are negative for exothermic reactions and positive for endothermic reactions.

Hess' law states that the change of enthalpy in a chemical reaction (i.e the heat of reaction at constant pressure) is independent of the pathway between the initial and final states.

3. Materials and Methods

    1) <u>Determination of calorimeter constant</u>

- Place 50 ml H_2O at room temperature to beaker, measure temperature t_1.

- Place 50 ml H_2O at about 60 Celsius degrees to the calorimeter, wait for the temperature stable, read temperature t_2. Keep the thermometer inside the calorimeter.

- Using a funnel, quickly transfer the water from beaker to calorimeter. Cap, wait for the temperature stable, read the temperature t_3.

- Determine m_0c_0 of the calorimeter from the heat balance equation.

$$m_0c_0 = mc\frac{(t_3-t_1) - (t_2-t_3)}{t_2 - t_3}$$

m: mass of water = 50g. c: heat capacity of water = 1 cal/(g.K).

    2) <u>Neutralization reaction between HCl and NaOH</u>

- Prepare burette containing NaOH 1M. In the preparation step, measure the temperature t_1 of NaOH. After measurement, rinse the thermometer.

- Prepare burette containing HCl 1M and transfer 25 ml HCl 1 M to the calorimeter,

measure the temperature t_2. Keep the thermometer inside the calorimeter.

- From the burette containing NaOH 1M, transfer 25 ml to the calorimeter. Cap and mix the solution, measure the temperature t_3.

- Calculate the heat quantity Q and ΔHr Assume heat capacity of diluted solution is 1 cal/g.K. Specific weight of salt solution is 1.02g/ml

$$Q = (m_{sol} \cdot C_{sol} + m_0 c_0) \Delta t$$

$$\Delta H = -\frac{Q}{n}$$

3) Heat of dissolution of anhydrous CuSO4 (copper(II) sulfate)

- Place into a calorimeter 50 ml of water. Measure the temperature t_1

- Weight exactly about 4g anhydrous $CuSO_4$.

- Quickly transfer $CuSO_4$ to the calorimeter, stir to dissolve completely, measure the temperature t_2 - Determine the heat Q and ΔHsolution. Confirm Hess's law.

$$Q = (m_{sol} \cdot C_{sol} + m_0 c_0) \Delta t$$

$$\Delta H = -\frac{Q}{n}$$

4) Heat of dissolution of NH$_4$Cl

- Repeat the procedure in section 3, using NH_4Cl (solid) in the place of $CuSO_4$ (solid). The specific heat capacity of the solution is approximately 1 cal/g.K

  Note: How to determine the temperature after the reaction.

- Measure the temperature before the reaction.

- Place reactants into the calorimeter, measure the temperature every 30 seconds.

- Draw the graph of temperature versus time.

- The maximum temperature is the intersection point of 2 lines: (1) the best line drawn through the data points on the cooling portion of the curve and (2) a line drawn perpendicular to the time axis at the starting time.

- The maximum temperature should not be recorded because of some, though very small, heat loss to the calorimeter wall.
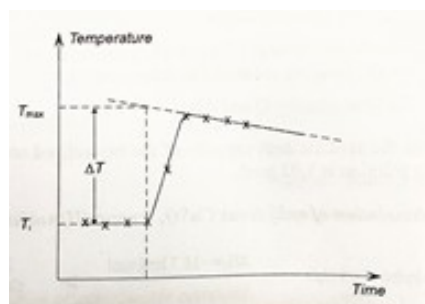
Figure 1: Heat of dissolution of NH$_4$Cl

4. Result and Discussion

Experiment 1: Determine the calorimeter constant

Temperature °C	First time	Second time	Third time
t_1	25	25	25
t_2	70	71	69
t_3	49	49	48
$m_0 c_0$	7.1	4.5	4.8
$m_0 c_0$ (Average)	5.47		
Deviation	1.63	0.97	0.67
Deviation (Average)	1.09		

$m_0 c_0 = 5.47 \pm 1.09$ (cal/K)

Experiment 2: Neutralization reaction between HCl and NaOH:

$$Q = (m_{sol} \cdot C_{sol} + m_0 c_0) \Delta t$$

$$\Delta H = -\frac{Q}{n}$$

$$\Delta t = t3 - \frac{t1 + t2}{2}$$

Temperature (°C)	First time	Second time	Third time
t_1	26	26	26
t_2	26	26	26
t_3	33	33	34
Q	395.29	395.29	451.76
Q_{ave}	414.113		
ΔH (cal/mol)	-16564.52		

$c_{NaCl} = 1 \ cal/gK; \ p_{NaCl} = 1.02 \ g/ml$

$m_{ddNaCl} = V_{dd} \cdot D_{NaCl} = (25 + 25) \times 1.02 = 51 \ (g)$

$$Q = (m_{sol} \cdot C_{sol} + m_0 c_0)\Delta t = (m_{sol} \cdot C_{sol} + m_0 c_0)(t3 - \frac{t1+t2}{2}) = (5.47 + 51 * 1)(t3 - \frac{t1+t2}{2})$$

$$n_{NaCl} = 0.025 \ (mol)$$

$$\Delta H = -\frac{Q}{n} = -\frac{414.113}{0.025} = -16564.52 \ (cal/mol)$$

Experiment 3: Heat of dissolution of anhydrous $CuSO_4$

$m_1 = 4,00g; m_2 = 4.01g; m_3 = 3.99g$

Temperature (°C)	First time	Second time	Third time
t_1	26	26	26
t_2	31	32	32
Q (cal)	297.35	356.88	356.76
ΔH (cal/mol)	-11894.0	-14239.6	-14306.2
ΔH_{ave} (cal/mol)	-13479.93		

$$Q = (m_{sol} \cdot C_{sol} + m_0 c_0) \Delta t = (m_0 c_0 + m_{H_2O} c_{H_2O} + m_{CuSO_4} c_{CuSO_4})(t_2 - t_1)$$

$$= (5.47 + m_{CuSO_4} \cdot 1 + 50.1).(t_2 - t_1)$$

$$n_{CuSO_4} = \frac{m_{CuSO_4}}{M_{CuSO_4}}$$

$\Rightarrow n_1 = 0.0250 \ (mol); n_2 = 0.0251 \ (mol); n_3 = 0.0249 \ (mol)$

$$\Delta H = -\frac{Q}{n} \Rightarrow \text{As } \Delta H_{ave} < 0 \Rightarrow \text{Exothermic process.}$$

Experiment 4: Heat of dissolution of NH_4Cl

Similar to previous section 3, replace $CuSO_4$ by NH_4Cl

$c_{NH_4Cl} = 1 \ (cal/g.K); m_{H_2O} = 50 \ (g)$

$m_1 = 4,01g; m_2 = 3.99g; m_3 = 3.99g$

Temperature °C	First time	Second time	Third time
t_1	26	26	26
t_2	22	22	21
Q (cal)	-237.92	-237.84	-297.30
ΔH (cal/mol)	3174.24	3189.08	3986.35
ΔH_{ava} (cal/mol)	3449.890		

$$Q = (m_{sol} \cdot C_{sol} + m_0 c_0) \, \Delta t = (m_0 c_0 + m_{H_2O} c_{H_2O} + m_{NH_4Cl} c_{NH_4Cl})(t_2 - t_1)$$

$$= (5.47 + m_{NH_4Cl} \cdot 1 + 50.1) \cdot (t_2 - t_1)$$

$$n_{NH_4Cl} = \frac{m_{NH_4Cl}}{M_{NH_4Cl}} \approx 0.075 \text{ (mol)}$$

$$\Delta H = -\frac{Q}{n} \Rightarrow \text{As } \Delta H_{ave} > 0 \Rightarrow \text{Endothermic process.}$$

5. Answer the questions:

5.1. ΔH of the reaction HCl + NaOH → NaCl + H2O is calculated based on the molar of HCl or NaOH when 25 ml of HCl 2M solution reacts with 25 ml of NaOH 1M solution? Explain.

We have:

- $n_{NaOH} = 0.025.1 = 0.025$
- $n_{HCl} = 0.025.2 = 0.05$
- HCl + NaOH → NaCl + H2O

Initial: 0.05   0.025

React: 0.025   0.025

Remain: 0.025   0

As can be seen from the equation above, NaOH is the limiting reagent and oxygen is the excess reagent so the equation is calculated based on the molar of NaOH.

5.2. If we replace HCl 1M with HNO$_3$ 1M, will the result of experiment 2 change or not?

The result of experiment 2 will not change because HCl and HNO$_3$ are both strong acids, completely dissociated. The reaction with NaOH if we use HNO$_3$ is still a neutralization reaction:

HNO$_3$ + NaOH → NaNO$_3$ + H$_2$O, with the ion equation: H$^+$ + OH$^-$ → H$_2$O.

5.3. Calculate ΔH$_3$ based on Hess's law. Compare to experimental results. Considering 6 reasons that might cause the error

- **Heat loss due to the calorimeter**

- **Thermometer**

- **Volumetric glassware**

- **Balance**

- **Copper (II) sulfate absorbs water**

- **Assume specific heat of copper (II) sulfate is 1 cal/mol.K**

In your opinion, which one is the most significant? Explain? Is there any other reason?

- Base on Hess's law:

ΔH$_3$ = ΔH$_1$ + ΔH$_2$ = -18.7 + 2.8 = -15.9 kcal/mol = -15900 cal/mol.

⇨ There is a great difference between ΔH_3 calculated by Hess's law (-15900 cal/mol) and the experimental result (-15978.01 cal/mol).

- Heat loss due to the calorimeter is the most significant reason that causes the error. Since carelessness or incorrectness happens while conducting the experiment will quickly lead to loss of heat to the environment.

- Another reason is copper (II) sulfate absorbs water $CuSO_4 + 5H_2O \rightarrow CuSO_4.5H_2O$ (caused by the speed used when operating with copper (II) sulfate is not quick enough) which results in a thermal effect of $CuSO_4.5H_2O$.

6. Conclusion

In conclusion, this experiments help us to find out the heat energy transfer in everyreaction. Thanks to these experiments, we can determine the heat transfer in everyreaction.

7. References

[1] Nguyen Tuan Anh, Huynh Ky Phuong Ha, Le Minh Vien (2020), *Laboratory Experiments in General Chemistry.*

Date: 30/10/2023

Class CC02 - Group 4

<u>**Unit 4**: REACTION RATES</u>

<u>**1. Abstraction**</u>

In this experiment, we are learning the reaction rate. We determine the rate of reaction in 2 experiments. The result shows that in distinct reactions, there is different reaction rate, it can be fast or slow. These findings have significant implications for our understanding of reaction rates.

<u>**2. Introduction**</u>

Chemical kinetic is the study of reaction rates and reaction mechanisms. Some reactions, such as acid-base or precipitation reactions, occur instantaneously. Other reactions, such as the rusting of iron, occur very slowly. Understanding the factors that affect reaction rates has important biological, industrial, and environmental significance.

The rate of a chemical reaction may be expressed as a change in the amount of a reactant (or product) per unit volume in a unit of time. When the volume is constant, the rate is the change in concentration per unit time.

$$r = \pm \frac{\Delta n}{V \Delta t} = \pm \frac{\Delta C}{\Delta t} \text{ (average rate)}$$

$$= \pm \frac{dC}{dt} \text{ (instantaneous rate)}$$

The rate of chemical reactions depends on a number of factors, including the nature of the reactants, reactant concentrations, reaction temperature, and the presence of catalysts.

3. Materials and Methods

   1) **Determining reaction order with respect to $Na_2S_2O_3$**

Prepare 3 test tubes containing H_2SO_4 and 3 conical flasks containing $Na_2S_2O_3$ and H_2O as the following table:

No.	Test tube V (ml) H_2SO_4 0.4M	Erlenmeyer Flask	
		V (ml) $Na_2S_2O_3$	V (ml) H_2O
1	8	4	28
2	8	8	24
3	8	16	16

- Use graduated pipette to place acid solution into test tubes.
- Use a burette to add water to three conical flasks. After that, rinse the burette with $Na_2S_2O_3$ and use the burette to deliver $Na_2S_2O_3$ to the flask.
- Repare the stopwatch.
- Alternately conduct the reactions in the pair of test tube and conical flask as follows:
  - Rapidly pour acid in the test tube to conical flask.
  - Start the stopwatch when two solutions are mixed.
  - Gently shake the conical flask and rest. Observe until the solution becomes slightly turbid, stop the watch.
  - Record Δt.
- Repeat each experiment at least one more time to get the average.

2) Determining reaction order with respect to $Na_2S_2O_3$

Conduct similarly to part 1 with the amount of acid and $Na_2S_2O_3$ as the following table

No.	Test tube V (ml) H_2SO_4 0.4M	Erlenmeyer Flask	
		V (ml) $Na_2S_2O_3$	V (ml) H_2O
1	4	8	28
2	8	8	24
3	16	8	16

4. Result and Discussion

Experiment 1: Reaction order with respect to $Na_2S_2O_3$:

a) Procedure:

Determining reaction order with respect to $Na_2S_2O_3$

$$r = \pm \frac{\Delta n}{V \Delta t} = \pm \frac{\Delta C}{\Delta t} = kC_A{}^m C_B{}^n$$

$$Na_2S_2O_3 + H_2SO_4 \rightarrow Na_2SO_4 + SO_2 + S\downarrow + H_2O$$
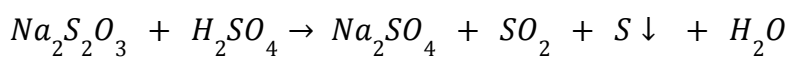
b) Result

No.	Initial Concentration (M)		Δt_1	Δt_2	Δt_{ave}
	$Na_2S_2O_3$	H_2SO_4			
1	0.1	0.4	151	150	150.5
2	0.1	0.4	72	73	72.5
3	0.1	0.4	35	37	36.0

Reaction order with respect to $Na_2S_2O_3$

- From Δt_{ave} of experiment 1 and 2, determine m_1 (sample calculation)

$$n_1 = log_2(\frac{\Delta t_1}{\Delta t_2}) = log_2(\frac{150.5}{72.5}) = 1.05$$

- From Δt_{ave} of experiment 2 and 3, determine m_2

$$n_2 = log_2(\frac{\Delta t_2}{\Delta t_3}) = log_2(\frac{72.5}{36.0}) = 1.01$$

$\rightarrow$ Reaction order with respect to $Na_2S_2O_3$ = $(n_1 + n_2) / 2 = 1.030$

Experiment 2: Reaction order with respect to H_2SO_4:

a) Procedure

Conduct similarly to part 1 with the amount of acid and $Na_2S_2O_3$ as the following table

No.	Test tube V (ml) H_2SO_4 0.4M	Erlenmeyer flask	
		V (ml) $Na_2S_2O_3$	V (ml) H_2O
1	4	8	28
2	8	8	24
3	16	8	16

b) Result

No.	Initial Concentration (M)		Δt_1	Δt_2	Δt_{ave}
	$Na_2S_2O_3$	H_2SO_4			
1	0.1	0.4	72	70	71.0
2	0.1	0.4	57	59	58.0
3	0.1	0.4	52	54	53.0

Reaction order with respect to H_2SO_4:

- From Δt_{ave} of experiment 1 and 2, determine n_1 (sample calculation)

$$n_1 = log_2(\frac{\Delta t_1}{\Delta t_2}) = log_2(\frac{71.0}{58.0}) = 0.29$$

- From Δt_{ave} of experiment 2 and 3, determine n_2

$$n_2 = log_2(\frac{\Delta t_2}{\Delta t_3}) = log_2(\frac{58.0}{53.0}) = 0.13$$

$\rightarrow$ Reaction order with respect to H_2SO_4= $(n_1 + n_2) / 2 = 0.210$

5. Answer the questions:

5.1. In the experiment above, what is the effect of the concentrations of $Na_2S_2O_3$ and H_2SO_4 on the reaction rate? Rewrite the reaction rate expression. Determine the order of the reaction.

- The concentration of $Na_2S_2O_3$ is directly proportional to the reaction rate.
- The concentration of H_2SO_4 almost has no effect on the reaction rate.
- Reaction rate expression: $v = k. [Na_2S_2O_3]^m. [H_2SO_4]^n$ in which m is the reaction order with respect to $Na_2S_2O_3$ and n is the reaction order with respect to H_2SO_4 (m and n are determined through experiments).

$\Rightarrow$ Reaction rate expression: $v = k. [Na_2S_2O_3]^{1.030}. [H_2SO_4]^{0.210}$.

$\Rightarrow$ The order of the reaction: $m + n = 1.030 + 0.210 = 1.240$.

5.2. Mechanism of the reaction can be written as

$$H_2SO_4 + Na_2S_2O_3 \rightarrow Na_2SO_4 + H_2S_2O_3$$

$$H_2S_2O_3 \rightarrow H_2SO_3 + S \downarrow$$
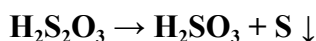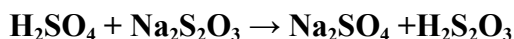
Based on the experimental results, may we conclude that the reaction (1) or (2) is the rate determining step, which is the slowest step of the reaction? Recall that in the experiments, the amount of the acid H_2SO_4 is always used in excess.

- Reaction (1) is an ion-exchange reaction so the reaction quickly happens.
- Reaction (2) is a self-redox reaction so the reaction slowly happens.

$\Rightarrow$ Reaction (2) is the rate determining-step, which is the slowest step of the reaction, because the order of the reaction is the order of reaction (2).

5.3. Based on the principle of the experimental method, the reaction rate is considered as an instantaneous rate or average rate.

- Base on the principle of the experimental method, the reaction rate is considered as instantaneous rate because the reaction rate is determined using the ratio $\Delta C/\Delta t$ in which $\Delta C \approx 0$ ($\Delta C \approx dC$) since the changes in the amount of sulfur is not noticeable).

5.4. Reverse the order of adding H$_2$SO$_4$ and Na$_2$S$_2$O$_3$, does the reaction order change? Explain?

- The reaction order does not change when we reverse the order of adding H$_2$SO$_4$ and Na$_2$S$_2$O$_3$.

- Explain: At a specific temperature, the order of the reaction depends on the nature of the reaction (concentration, temperature, surface area, pressure) but not the order of the reactant added.

6. Conclusion

In conclusion, this experiments help us to figure out the heat rate in every reaction. Thanks to these experiments, we can determine the heat rates in every reaction.

7. References

[1] Nguyen Tuan Anh, Huynh Ky Phuong Ha, Le Minh Vien (2020), *Laboratory Experiments in General Chemistry.*

<u>**Unit 8**</u>: **VOLUMETRIC ANALYSIS**

<u>**1. Abstraction**</u>

In this experiment, we are learning volumetric analysis. We determine the concentration based on measurement of volumes of reaction in solutions in 3 experiments. These findings have significant implications for our understanding in volumetric analysis.

<u>**2. Introduction**</u>

Volumetric analysis is the method of quantitative determination of species X based on the measurement of the volume. Volumetric analysis is often referred to as titration, a technique in which typically the exact volume of unknown concentration of component X (measured by pipette and placed into conical flask) is reacted with the standard solution. the standard solution whose concentration is accurately known is usually added gradually to the flask from a burette.

The standard solution is called the titrant; the solution being analyzed is called the analyte.

Delivery of the titrant until the process is judged to be complete is called a titration.

The reaction $C + X \rightarrow A + B$ is called titration reaction.

The principle of calculation: law of equivalent proportion $C_C V_C = C_X V_X$

The equivalence point is the point at which a complete chemical reaction takes place. At this point, equivalent quantities of the reagents are used.

The equivalence point can only be estimated by observing a physical change such as color change, clouding. The point in a titration when this physical change occurs is called the end point. Indicators are usually added to the analyte to give a signal of the end point.

A titration curve is a plot typically showing the changes of logarithm of the concentration of the titrated solution versus the volume of the added standard solution (titrant). The shape of a titration curve depends on the concentration, equilibrium constant of titration reaction.

The reflection point, the steepest part of the titration curve, is the equivalence point.

Instant change in curve when a small amount of titrant is added is called titration jump.

The requirement for the indicator is the end point within the titration jump.

Note that the equivalence point in acid-base titration will have the pH corresponding to the pH of the salt solution. When a strong acid is neutralized by a strong base (for example HCl and NaOH), the salt is neutral and the pH at the equivalence point is 7. When a weak acid is

neutralized by a strong base or a weak base is neutralized by a strong acid, the pH at the equivalence point is differ from 7.

The following table shows the transition range of some common indicators.

Indicator	Color		
	acidic form	pH range of transition	basic form
Thymol blue	red	1.2 - 2.8	yellow
Methyl orange	red	3.1 - 4.4	yellow
Phenolphthalein	colorless	8.0 - 10	purple - pink
Indigo camine	blue	11.4 - 13	yellow
Alizarine yellow	yellow	10 - 12	red

3. Practice

1) Experiment 1: Titration curve of HCl by NaOH

Construct the titration curve of the titration of a strong acid by a strong base

V_{NaOH}	0	2	4	6	8	9	9.2	9.4	9.6	9.8	10	11	12	13
pH	0.96	1.14	1.33	1.59	1.98	2.36	2.56	2.73	3.36	7.26	10.56	11.70	11.97	12.01

Base on the titration curve, determine the pH jump, equivalence point and appropriate indicators.

2) Experiment 2:

- Rinse the burette with NaOH 0.1N, and then pour NaOH to the burette. Adjust the liquid level to zero.

- Use pipette to measure 10 ml HCl solution of which the concentration is unknown and transfer to the erlenmeyer flask. Add 10 ml distilled water and two drops of phenolphthalein.

- Open the stopcock to slowly drop the NaOH solution to the Erlenmeyer flask. Swirl the flask while dropping. Until the solution in the flask changes to faith-pink, close the stopcock. Read the consumed volume of NaOH. Repeat the experiment one more time.

3) Experiment 3:

Repeat the procedure in experiment 2, using methyl orange indicator in the place of phenolphthalein. Stop the titration when the color of the solution changes from red to orange.

4) Experiment 4:

Do similar to experiment 2, but replace the HCl solution by acid acetic solution. Repeat two times, first time using phenolphthalein, the later using methyl orange.

4. Result and Discussion

Experiment 1: Titration curve of HCl by NaOH

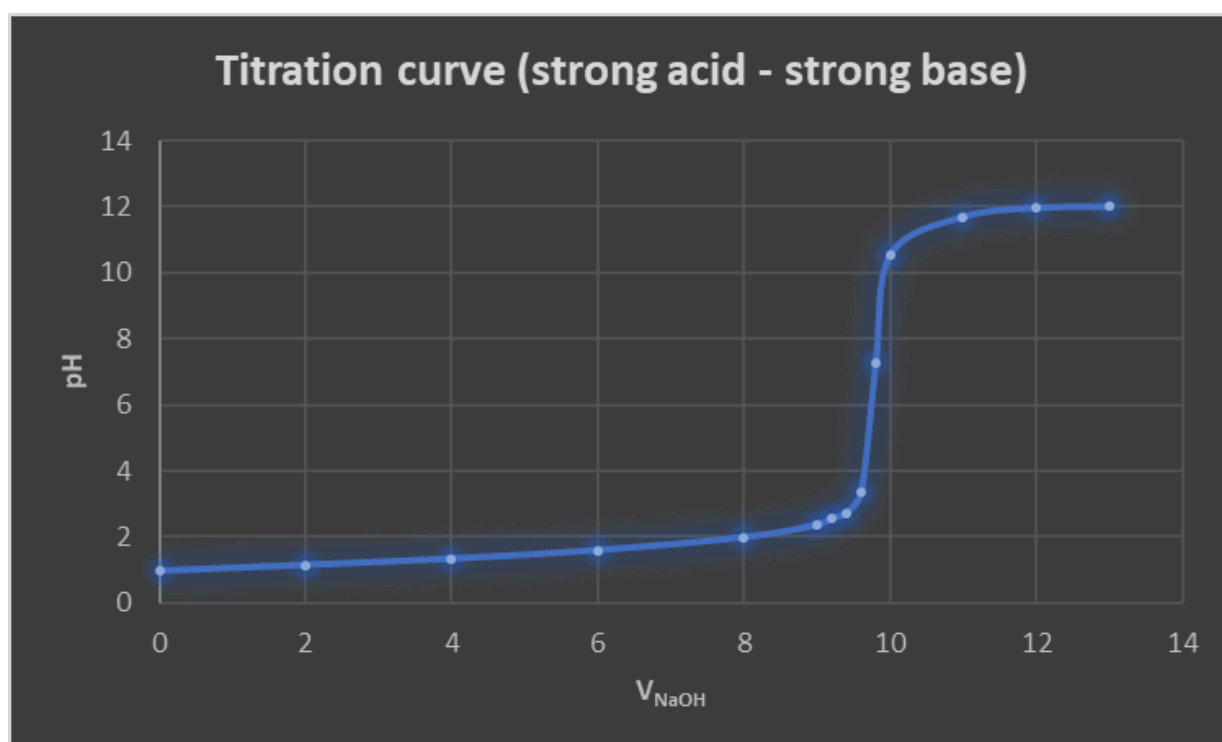| V_{NaOH}	0	2	4	6	8	9	9.2	9.4	9.6	9.8	10	11	12	13
pH	0.96	1.14	1.33	1.59	1.98	2.36	2.56	2.73	3.36	7.26	10.56	11.70	11.97	12.01

Figure 1: The titration curve of the titration of a strong acid by a strong base

Result:

- pH at equivalence point is 7.26
- pH jump: from pH 3.36 to 10.56
- Appropriate indicator: phenolphthalein, since it changes its color in the pH range between 8 and 10.

Experiment 2:

- Rinse the burette with NaOH 0.1N, and then pour NaOH to the burette. Adjust the liquid level to zero.

- Use pipette to measure 10 ml HCl solution of which the concentration is unknown and transfer to the erlenmeyer flask. Add 10 ml distilled water and two drops of phenolphthalein.

- Open the stopcock to slowly drop the NaOH solution to the Erlenmeyer flask. Swirl the flask while dropping. Until the solution in the flask changes to faith-pink, close the stopcock. Read the consumed volume of NaOH. Repeat the experiment one more time.

No.	V_{HCl} (ml)	V_{NaOH} (ml)	C_{NaOH} (N)	C_{HCl} (N)	Deviation
1	10	12.7	0.1	0.127	0.0023
2	10	12.4	0.1	0.124	0.0007
3	10	12.3	0.1	0.123	0.0017
Average	x	x	x	0.1247	0.0016

$C_{HCl} = 0.1247 \pm 0.0016 \ (N)$

Experiment 3:

Follow the procedure of the experiment 1. Replace the indicator phenolphthalein by methyl orange.

No.	V_{HCl} (ml)	V_{NaOH} (ml)	C_{NaOH} (N)	C_{HCl} (N)	Deviation
1	10	12.5	0.1	0.125	0.0017
2	10	12.3	0.1	0.123	0.0003
3	10	12.2	0.1	0.122	0.0013
Average	x	x	x	0.1233	0.0011

$C_{HCl} = 0.1233 \pm 0.0011 \ (N)$

Experiment 4:

- Follow the procedure of the experiment 1.

- Replace the HCl solution with an acid acetic solution.

- Repeat two times, first time using phenolphthalein, the later using methyl orange.

No.	Indicator	$V_{CH_3COOH}(ml)$	$V_{NaOH}(ml)$	$C_{NaOH}(N)$	$C_{CH_3COOH}(N)$
1	Phenolphthalein	10	11.6	0.1	0.116
2	Methyl orange	10	3.4	0.1	0.034

5. Answer the question:

5.1. When changing the concentration of HCl or NaOH, does the titration curve change? Explain.

- When changing the concentration of HCl or NaOH, the titration curve does not change because the HCl titration method using NaOH is determined based on the equation:

$$HCl + NaOH \rightarrow NaCl + H_2O$$
$$C_{HCl}.V_{HCl} = C_{NaOH}.V_{NaOH}$$

- With V_{HCl} and C_{NaOH} remain unchanged, when C_{HCl} increases or decreases, V_{NaOH} also increases or decreases. In conclusion, even if we expand or narrow down, the titration curve does not change. The same for the case when we change the concentration of NaOH.

5.2. The determination of the concentration of HCl in experiment 2 and 3, which one is more precise?

The determination of the concentration of HCl in experiment 2 is more precise. Because phenolphthalein helps us determine the color more accurately, since its color changes from colorless to light pink, which is easier to notice than from red to orange.

5.3. From the result of experiment 4, for the determining concentration of acid acetic solution, which indicator is more precise?

Indicator choice is based on the pH of the equivalence point for the acid-base titration. A suitable indicator should change color at or near the equivalence point.
Phenolphthalein changes color at a pH above 7. So it is quite good as an indicator for titrations of strong acids with strong bases.
$\Rightarrow$ Phenolphthalein is more precise than methyl orange since acetic acid is a weak acid so if the norm point is greater than 7, we should use phenolphthalein (because the jump from 3.0 to 4.4 is too far).

5.4. In volumetric titration, if NaOH and HCl are interchanged, does the result change? Explain?

In volumetric titration, if NaOH and HCl are interchanged, the result does not change since this is an equilibrium reaction.

6. Conclusion

In conclusion, this experiments help us to find out the applications of volumetric analysis. Thanks to these experiments, we can determine the deviation in every reaction.

7. References

[1] Nguyen Tuan Anh, Huynh Ky Phuong Ha, Le Minh Vien (2020), *Laboratory Experiments in General Chemistry.*

RECORDED DATA IN LAB

DATA OF LAB 1

Nhóm 4 : Nguyễn Tiến Hưng 2252280
           Nguyễn Ngọc Khôi   2252378
           Nguyễn Quang Phú   2252621

③ — Oxalic Acid 1) m = 0,61g → 11,4 ml V_{KMnO_4}
                   2) m = 0,59g → 10,5 ml V_{KMnO_4}
                   3) m = 0,60g → 10,3 ml V_{KMnO_4}

⑤   1) V_{NaOH} = 10,3 ml
    2) V_{NaOH} = 10,4 ml
    3) V_{NaOH} = 10,7 ml

23/10/23

DATA OF LAB 2

Unit 2 : Heat of Reaction

Experiment 1 : Determine the calorimeter constant

T°C	First time	Second Time	Third time
t_1	25	25	25
t_2	70	71	69
t_3	49	49	48
$m_0 c_0$	7,1	4,5	4,8
$m_0 c_0$ Ave	5,47		

Họ và Tên : Nguyễn Quang Phú - 2252621   Nhóm : 4
Nguyễn Ngọc Khôi - 2252378   Lớp : CC02
Nguyễn Tiến Hưng - 2252280

$$m_0 c_0 = mc \frac{(t_3 - t_1) - (t_2 - t_3)}{t_2 - t_3} \quad (m = 50g, c = 1 cal/gk)$$

30/10/2023

Experiment 2 : Neutralization reaction between HCl and NaOH

T°C	First time	Second Time	Third Time
t_1	26	26	26
t_2	26	26	26
t_3	33	33	34
Q			
Q_{Ave}			
ΔH (cal/mol)			

Experiment 3 : Heat of dissolution of anhydrous CuSO4

T°C	First time	Second Time	Third Time
t_1	26	26	26
t_2	31	32	32
Q (cal)			
ΔH (cal/mol.k)			
ΔH_{Ave}			

$m_1 = 4,00g$
$m_2 = 4,01g$
$m_3 = 3,99g$

Experiment 4 : Heat of dissolution of NH4Cl

T°C	First time	Second Time	Third Time
t_1	26	26	26
t_2	22	22	21
Q (cal)			
ΔH (cal/mol.K)			
ΔH_{ave} (cal/mol.K)			

$m_1 = 4,01g$
$m_2 = 3,99g$
$m_3 = 3,99g$

DATA OF LAB 4

Unit 4: Reaction Rates

Experiment 1: Reaction order with respect to $Na_2S_2O_3$

30/10/2023

No.	Initial concentration (M)		Δt_1 (s)	Δt_2	Δt_3	Δt_{ave}
	$Na_2S_2O_3$	H_2SO_4				
1	0,1	0,4	-151	150		
2	0,1	0,4	72	.73		
3	0,1	0,4	35	37		

Experiment 2: Reaction order with respect to H_2SO_4

No	Initial concentration (M)		Δt_1	Δt_2	Δt_3	Δt_{ave}
	$Na_2S_2O_3$	H_2SO_4				
1	0,1	0,4	72	70		
2	0,1	0,4	57	59		
3	0,1	0,4	52	54		

DATA OF LAB 8

6/11/23

Nguyễn Ngọc Khôi – 2252378
Nguyễn Tiến Hưng – 2252280
Nguyễn Quang Phú – 2252621

Experimental report of unit 8

Date: 6/11/2023

Group: 4 .... Class: CC04 CC02

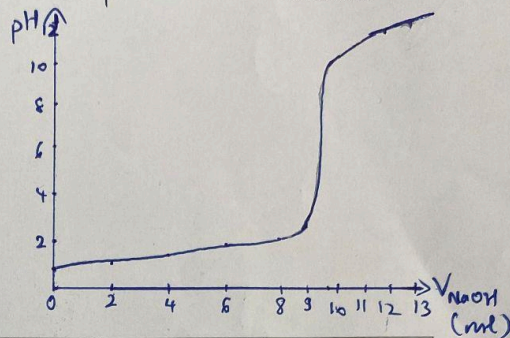### I. EXPERIMENT RESULTS

1) Titration curve of HCl by NaOH

From the graph determine:

- pH at equivalence point

- Titration jump

2) Experiment 2

No.	V_{HCl} (mL)	V_{NaOH} (ml)	C_{NaOH} (N)	C_{HCl} (N)	Devitation
1	10	12,7			
2	10	12,4			
3	10	12,3			

$C_{HCl}^3 = \quad$ N

3) Experiment 3

No.	V_{HCl} (mL)	V_{NaOH} (ml)	C_{NaOH} (N)	C_{HCl} (N)	Devitation
1	10	12,5			
2	10	12,3			
3	10	12,2			

4) Experiment 4

No.	Indicator	V_{CH_3COOH} (ml)	V_{NaOH} (ml)	C_{NaOH} (N)	C_{CH_3COOH} (N)
1	Phenolphthalein	10	11,6		
2	Metyl orange	10	3,4		