THE MINISTRY OF EDUCATION AND SCIENCE OF UKRAINE
NATIONAL TECHNICAL UNIVERSITY
"DNIPROVSKA POLYTECHNIC"

FACULTY OF GEOLOGICAL PROSPECTING
Departament of Chemestry

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CHEMISTRY

METHODICAL RECOMMENDATIONS FOR THE IMPLEMENTATION OF LABORATORY WORK ON DISCIPLINE

for students of all specialties

Dnipro
NTU “DP”
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The theoretical positions are considered and methods of carrying out of laboratory works on a course "Chemistry" are given. Each work presents control questions and tasks for independent work.

Розглянуто теоретичні положення та методики виконання лабораторних робіт з курсу «Хімія». До кожної роботи подано контрольні питання й завдання для самостійної роботи.

Відповідальна за випуск завідувач кафедри хімії, д-р техн. наук, проф. О.Ю. Свєткіна.

PREFACE

Laboratory work on chemistry is a form of a training session in which a student, under the direction of a teacher, personally conducts chemical experiments with the aim of practical confirmation of certain theoretical provisions. At the same time, a student acquires skills of working with laboratory equipment and reagents mastering the methodology of experimental studies, learns independently to solve research and design problems.

Didactic aims of conducting laboratory works on the discipline of "Chemistry":

– mastering of methods of experimental research (preparation and conducting of chemical experiments, analysis and processing of their results);

– formation of skills of using special equipment and mastering the specifics of practical work;

– assimilation of safe methods of conducting a chemical experiment;

– deeper assimilation of the theoretical material due to its practical application.

Skills acquired on the conceptual and analytical level are an essential component of the competence of a specialist.

A teacher is responsible for the organization of laboratory work, it is he who is obliged to create appropriate conditions for this, to provide necessary material, technical and informational support.

During the laboratory work a student must:

− be sure and careful to follow the rules of safety and health protection;

− get acquainted with methodical recommendations for conducting laboratory works;

− perform laboratory work according to the appropriate methodology;

− make a report on the implementation of the laboratory work;

− present the results of the laboratory work;

− get an assessment for the laboratory module through the appropriate form of modular control (presentation of reports on laboratory work).

A teacher has to:

− organize proper methodological, informational and logistical support of carrying out laboratory classes according to the requirements of the rules of safety and health protection;

− instruct students about the rules for safe laboratory work;

− conduct laboratory work;

− carry out current control of the students' mastering of conducting chemical experiments;

− ensure compliance with the rules of safety and health protection during the performance of each laboratory study;

− draw up a schedule of consultations and strictly follow it;

− assess the student's educational activity in mastering the laboratory module.

Requirements for registration of educational documents:

During the laboratory work, students must keep records in a laboratory journal and make a report.

Report structure:

It is recommended to make a report on laboratory work in the following sequence:

− theme;

− the purpose of laboratory work;

− equipment and reagents;

− methodology of research (conditions under which the research is carried out, schemes of devices);

− protocol of research (observation, reaction equation, table);

− processing the results of the experiment (calculations by the formulas);

− conclusions.

ORDER OF WORK AND RULES OF SAFETY IN CHEMICAL LABORATORIES

1. During the experiments, carefully observe all changes that occur with the substances in the process of reaction.

2. Required chemical reagents are placed on the tables, and general reagents are in the draught cupboard.

3. After using the solution, the glass with its residue is closed and put into the place.

4. It is forbidden to pour unused reagent in the same glass.

5. It is prohibited to put out common-purpose reagents from the draught cupboard.

6. Experiments with poisonous substances and those with sharp smell are carried out in the draught cupboard.

7. When heating the liquid, the tube is held a hole away from yourself and from those who are present.

8. When performing experiments, ensure that chemical reagents do not fall onto the face, hands, clothing.

9. When determining the type of gas by smell, its stream is directed by moving the hand from the test tube to yourself.

10. Sulfate acid is diluted, adding it in small portions to the water (and not vice versa!) and stirring constantly.

11. You cannot lean over the liquid during its heating.

Laboratory work № 1

DETERMINATION OF THE MOLECULAR MASS OF METAL EQUIVALENT

Theoretical provisions

Equivalent of a substance is its amount that is coupled with one mole of hydrogen atoms or replaces the same amount of hydrogen atoms in chemical reactions. Example:

$CuO + H_2 = Cu + H_2O$; E (CuO) = 1/2 mol Cooper (II) oxide

The molar mass of the equivalent is the mass of one equivalent of the substance. The molar mass of the equivalent of hydrogen is 1 g / mol, and oxygen is 8 g / mol. The molar amount of hydrogen equivalent at standard conditions: T = 273°K, P = 101.3 kPa (s.t.p.) is equal to11.2 l / mol, and oxygen – 5.6 l / mol. The molar mass of the equivalent of a simple substance can be calculated by dividing its molar mass by valency, that is:

$$M_{eq} = M/B.$$

There are various ways of determining the molar masses of equivalents: by joining, decomposition, substitution, exchange and electrochemical methods.

In this paper, we use the method of determining the molar mass of the equivalent of a metal by volume of hydrogen, which is released when the metal is dissolved in acid.

Purpose: to master the method of determining the equivalent mass of a metal using the law of equivalents; to acquire skills of safe conducting of chemical research and to master the methods of calculating the obtained data.

Setting objectives

Given: heavyweight of metal zinc.

Determine: molar mass equivalent of zinc per volume of hydrogen released during the reaction.

Equipment and reagents: a device for determining the molar mass of the equivalent of a metal, a thermometer, a barometer, a 10 % solution of chloride acid and distilled water.

Research methodology

To determine the molar mass of the equivalent of a metal, it is used a device consisting of a test tube 1, a funnel 2 and a burette 3, connected by rubber tubes (Fig. 1). Burette and funnel are filled with water.

First check the leak tightness of the appliance. For this, the funnel is lowered by 10 – 15 cm, observing the level of water in the burette. If the appliance is sealed, the water level in the burette will slightly decrease, and then remain unchanged. Having checked the leak proofness of the device, open the cork in the test tube, install the funnel in such a position that the water in it occupied about 1/3 of the volume, and the water level in the burette would be 1 ml below zero. Figures are determined by fixing the lower edge of the meniscus (level) of water in the burette.

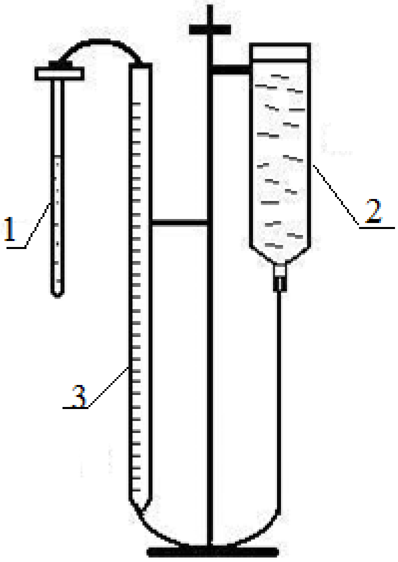

Fig. 1 Scheme of the device for determining the molar mass of the equivalent of the metal

The test tube is filled with 1/3 volume of acid. Close the test tube with a stopper and record the water level indicator V1 in the burette in the observation table (Table 1). Again open the test tube, tilt it and place the prepared zinc in the edge of the opening. Close the tube with a stopper, shake it, plunging the metal into acid.

Isolated hydrogen displaces water from the burette into the funnel. When the metal completely reacts with acid, the vials are allowed to cool to room temperature, and then balance the water levels in the burette and funnel. Take and record water level impressions in burette V_2. The difference between V_1 and V_2 is equal to the amount of hydrogen discharged.

In table 1 there are introduced figures of a thermometer , barometer and a pressure of water vapor according to the experimental temperature (Table 2).

Table 1

Research results

m,g	V_1 , ml	V_2, ml	$V(H_2)$, l	P_{atm}, kPa	P_{vapour}, kPa	$P(H_2)$, kPa	T, (K) (273 + t °C)

Processing the results of the experiment

Using the obtained experimental data, calculations are performed using the formulas given below:

1. The volume of hydrogen isolated

$$V(H_2) = V_2 - V_1.$$

2. Absolute temperature

$$T = 273 + t\ {}^{o}\mathrm{C}.$$

3. Pressure of hydrogen

$$P(H_2) = P_{atm} - P_{\text{saturation of water vapour}},\ \text{kPa}.$$

4. Bring the volume of hydrogen to normal conditions (bearing in mind that P_0= 101,3 kPa or 760 mmHg and T_0 = 273 K) according to the combined gas law, that is:

$$\frac{PV}{T} = \frac{P_0 V_0}{T_0}.$$

So,

$$V_0(H_2) = \frac{P(H_2) \cdot V(H_2) \cdot T_0}{P_0 \cdot T}.$$

5. The experiment value of the molar mass of zinc equivalent is calculated using the law of equivalents, namely:

$$\frac{m(\mathrm{Zn})}{M_{eq\ of\ \exp}(\mathrm{Zn})} = \frac{V_0(\mathrm{H_2})}{11{,}2}.$$

6. Relative error of determination of molar mass of zinc equivalent is calculated by the following formula:

$$\Delta = \pm\frac{M_{eq\ of\ theory}(Zn) - M_{eq\ of exp}(Zn)}{M_{eq\ of\ theory}(Zn)} \cdot 100,\%$$

here $M_{eq\ of\ theory}(Zn) = \frac{M(\mathrm{Zn})}{\mathrm{B}} = \frac{65{,}39}{2} = 32{,}695$ g/mol.

Table 2

Dependence of pressure of saturated water vapor on temperature

T, k	P, kPA	T, K	P, kPA	T, K	P, kPa
283	1,22	290	1,93	297	2,97
284	1,31	291	2,03	298	3,16
285	1,40	292	2,19	299	3.34
286	1,49	293	2,34	300	3,58
287	1,59	294	2,48	301	3,89
288	1,70	295	2,64	302	4,00
289	1,82	296	2,80	303	4,24

Test questions and tasks for self-study work

1. Give the definition of the molar mass of the equivalent. In what units is it measured?
2. How is the molar mass of the metal oxide equivalent calculated, when the molar mass equivalent of an element, that is coupled with oxygen is known?
3. Give the mathematical expression of the equivalence law for the gas system.
4. What is the equivalent of a substance?
5. Formulate the law of equivalents.
6. Determine the molar mass of the equivalent of aluminum oxide.
7. Formulate the law of Avogadro.

Laboratory work № 2

CHEMICAL KINETICS

Theoretical provisions

Chemical kinetics is the doctrine of the rate of chemical reactions. The rate of chemical reactions is measured by changing the concentration of each of the reactants per unit time, provided that the volume of the reacting system remains constant. In this case, molar concentration is mostly used. Units of time are chosen as seconds, minutes, hours depending on the speed of the reaction. It is advisable to correlate the change in concentration with infinitely small intervals of time and to determine the true reaction rate as a derivative of concentration over time, namely:

$$\upsilon = dC/dt.$$

There is an inverse proportional relationship between the rate and the time of the reaction. The rate of chemical reactions is usually uneven and conditioned by the nature of the reactants, their concentration, temperature and the presence of a catalyst.

The dependence of the reaction rate on the concentration of the reactants is expressed by ***the law of mass action***, according to which the reaction rate is directly proportional to the product of the concentrations of the reacting substances in degrees that are numerically equal to their stoichiometric coefficients, that is,

$$\upsilon = k\,[\mathrm{A}]^{\mathrm{m}}[\mathrm{B}]^{\mathrm{n}},$$

where [A] and [B] are the concentration of reactants; m, n are stoichiometric coefficients; *k* is the rate constant depending on the nature of the reactants.

Quantitatively, *k* is equal to the reaction rate, provided that the product of the concentrations of the reactants is equal to one.

Concentrations of solids in the expression to determine the reaction rate are not taken into account.

The influence of temperature on the reaction rate is ***determined by the Vant-Hoff*** rule: with the temperature increase for every 10 degrees, the reaction rate increases by 2 − 4 times, namely:

$$\upsilon_2 = \upsilon_1 \cdot \gamma^{\frac{T_2 - T_1}{10}},$$

where υ_2 is the reaction rate at T_2; υ_1 is reaction rate at temperature T_1; γ is temperature coefficient of Van-Hoff (varies from 2 to 4, which is due to the nature of the reactants).

Pressure affects only the reaction rate between the gaseous substances. With increasing pressure the speed of such reactions increases.

Purpose: to master the method of studying the influence of some factors on the speed of chemical reactions.

Setting objectives

To investigate: the effect of changes in the concentration of sodium thiosulfate and system temperature on the reaction rate.

Equipment and reagents: test tubes, stopwatch, thermometer, a glass of water, a 10 % solution of sodium thiosulfate and a dilute solution of sulfate acid.

Methodology of research

1. The influence of the concentration of substances on the rate of reactions

The interaction of sodium thiosulfate with sulfate acid occurs according to the following scheme:

$$Na_2S_2O_3 + H_2SO_4 = Na_2SO_4 + H_2SO_3 + S.$$

As you can see, due to the appearance of free sulfur, opalescent solution is formed.

It is respectively poured, 9, 6 and 3 ml of $Na_2S_2O_3$ in three test tubes. To the second and third samples it is added 3 and 6 ml of water. 3 ml of $H_2SO_4$are added in the first tube. The stopwatch counts the time from the moment of the merging of the solutions to the appearance of turbidity. Consistently, in the second and third test tubes, 3 ml of H_2SO_4 are added. The tubes are shaken and the time of the appearance of turbidity is fixed in all three cases with the same degree of turbidity. Data is entered in the table 3.

Table 3

Research Results

№ of research	Volume, ml			τ, c	$\upsilon = 1/\tau$	$C_{relative}$
	$Na_2S_2O_3$	H_2O	H_2SO_4			
1	9	0	3			1
2	6	3	3			2/3
3	3	6	3			1/3

Taking the concentration of the first test tube per unit, in the second and third test tubes the relative concentrations of substances are 2/3 and 1/3. According to these data, the dependency graph between the relative reaction rate ($\upsilon = 1/\tau$) and relative concentration of the solution $C_{relative}$ are drawn up and concluded accordingly.

2. Influence of temperature on the reaction rate

It is poured 5 ml of $Na_2S_2O_3$ solution in three test tubes, and in the other three – 5 ml of H_2SO_4 solution. Depending on the contents of the test tube, they are grouped in pairs (acid - thiosulfate). The thermometer and the first pair of test tubes are placed in a glass of water at room temperature. After 3 – 5 minutes, the content of one test tube is poured into the second one, they are shaken and the time from the beginning of mixing to the appearance of white haze is set.

The second pair of test tubes is placed in water, the temperature of which is 10 ºC higher than in the previous experiment, and the third in the aquatic environment, where the temperature is still 10 ºC higher.

The last two experiments are conducted similarly to the first one. All data is entered in the table 4

Table 4

Research Results

№ of research	T, ºC	τ, c	$\upsilon = 1/\tau$	γ

According to the results of the received data make the appropriate conclusion.

Test questions and tasks for self-study work

1. How will the reaction rate change with a temperature increase from 50 to 100 °C, if the temperature coefficient is equal to 2?

2. How will the reaction rate $2NO + O_2 = 2NO_2$ change, when increasing the concentration of NO and O_2 3 times?

3. The temperature coefficient of the reaction rate is 2.4. At what temperature should the reaction be performed to reduce its rate by 10 times (initial reaction temperature of 60 ºC)?

4. What is the concept of chemical kinetics?

5. Give the definition of the rate of chemical reactions.

6. Give a mathematical expression to determine the rate of chemical reactions.

7. What factors does the speed of chemical reactions depend on?

8. What law expresses the dependence of the reaction rate on the concentration of reactants? Give its mathematical expression.

9. What rule determines the effect of temperature on the reaction rate? What mathematical expression is it related to?

Laboratory work № 3

ION REACTIONS

Theoretical provisions

In solutions of electrolytes, chemical reactions pass through the interaction of ions only in those cases when due to reaction ***hardly soluble substances*** are formed which precipitate, volatile (gaseous) compounds or ***weakly dissociates***. Under these conditions, the equations are written in molecular and ionic forms.

In ionic equations, strong electrolytes should be written in the form of ions, and weak, hardly soluble and gaseous compounds are in the form of molecules.

For example:

$BaCl_2 + Na_2SO_4 = BaSO_4\downarrow + 2NaCl$ (molecular equation);

$Ba^{2+} + 2Cl^{-} + 2Na^{+} + SO_4^{2-} = BaSO_4\downarrow + 2Na^{+} + 2Cl^{-}$ (complete ion equation).

Ions of sodium and chlorine do not take part in this process. Let's rewrite the last equation without these ions, then

$Ba^{2+} + SO_4^{2-} = BaSO_4\downarrow$ (reduced ion equation).

Purpose: To master the method of performing a chemical experiment of interaction of electrolytes and compiling reaction equations in molecular and ionic forms.

Setting objectives

Conduct possible ionic reactions between the following electrolytes and formulate their equations.

Equipment and reagents: test tubes, burner, 10% solution of electrolytes $NaOH$, CH_3COONa, H_2SO_4, $BaCl_2$, Na_2SO_4, $Al_2(SO_4)_3$, $FeSO_4$, $CuSO_4$, Na_2S. Ammonium chloride NH_4Cl must be in crystalline form.

Methodology of research

1. Ionic reactions with the formation of weak electrolyte

a) Inject a few crystals of ammonium chloride into a test tube and add 2 – 3 ml of sodium hydroxide solution. Slightly heat the test tube. By smell, determine which gas is allocated. Formulate reaction equations.

b) In a test tube with sodium acetate solution it is added sulfate acid. The smell is determined by the presence of acetic acid. Formulate reaction equations.

2. Ionic reactions with the formation of sediments

a) Pour a few drops of chloride barium solution into three test tubes and add in one of them a solution of sodium sulfate, in the second one – a solution of sulfate acid, in the third one – a solution of aluminum sulfate. Observe the appearance of identical sediments. Formulate reaction equations.

b) In the first test tube it is added 2 – 3 drops of iron (II) sulfate solution, and in the second – the same amount of copper (II) sulfate solution. In both test tubes, add sodium sulfide solution. Formulate reaction equations.

Test questions and tasks for independent work

1. In which cases there is a reaction between solutions of electrolytes?
2. Make the molecular and ion equations for the following reactions:

a)$Al_2(SO_4)_3 + Pb(NO_3)_2 \rightarrow$ b) $ZnS + HCl \rightarrow$

c) $Zn(OH)_2 + NaOH \rightarrow$ d) $HCN + NaOH \rightarrow$

e) $Fe(OH)_3 + HNO_3 \rightarrow$

Laboratory work №. 4

IONIC PRODUCT OF WATER. WATERPROOF INDICATOR. SOLID HYDROLYSIS

Theoretical provisions

Water is a very weak electrolyte. To a small extent, it dissociates the following ions:

$$H_2O \Leftrightarrow H^+ + OH^-.$$

Applying the law of mass action to this turnaround process, we obtain the expression of the dissociation constant of H_2O

$$K_d = \frac{[H^+]\cdot[OH^-]}{[H_2O]}.$$

At a temperature of 22 °C $K_d(H_2O) = 1.8\ 10^{-16}$. From the expression K_d we find the product of equilibrium concentrations of H^+ and OH^- ions, that is,

$$[H^+][OH^-] = 1{,}8\ 10^{-16}\cdot[H_2O].$$

Since the ions of hydrogen and hydroxide in water is insignificant, it will practically exist in the form of molecules, and therefore the number of moles of water in 1 liter is equal to its mass, divided by the molar mass (water density is 1 g / cm^3), that is

$$[H_2O] = 1000/18 = 55{,}56 \text{ mol};$$

$$[H^+][OH^-] = 1{,}8\ 10^{-16}\cdot 55{,}56 = 10^{-14};$$

$$[H^+]\cdot[OH^-] = 10^{-14}.$$

The obtained value of the ***ionic product of water*** is a constant value for this temperature.

The amount of ions of hydrogen hydroxide in a neutral solution is the same, namely:

$$[H^+] = [OH^-] = 10^{-7} \text{ mol/l}.$$

The concentration of H^+ ions is calculated by the following formula:

$$[\,H^+] \;=\; C_м \cdot \alpha \cdot k\,,$$

where $C_м$ is molar concentration, mol / l; α – the degree of dissociation; k – number of H^+ ions formed during the dissociation of one molecule of acid.

The nature of the medium is judged by concentration of hydrogen ions in the solution, in particular

$[H^+] = 10^{-7}$ – neutral;
$[H^+] > 10^{-7}$ – acid;
$[H^+] < 10^{-7}$ – alkaline.

But it is more convenient to characterize the environment not by the concentration of hydrogen ions, but by its decimal logarithm with the opposite sign. This quantity is called the ***hydrogen value***, namely:

$$\mathrm{pH} = -\lg[\mathrm{H}^+].$$

If the pH of the solution is 7, then this medium is neutral, pH > 7 is alkaline, pH < 7 is acid. Practically, the pH varies from 0 to 14. Sometimes, by the analogy with the hydrogen index, the ***hydroxyl value*** is determined, that is,

$$\mathrm{pOH} = -\,\mathrm{l}g\left[\mathrm{OH}^-\right].$$

For any aqueous solution at 22 °C

$$\mathrm{pH} + \mathrm{pOH} = 14.$$

The environment can also be characterized by acid-base indicators. The most common among them are litmus, phenolphthalein and methylorange.

Hydrolysis of salts is the interaction of salt ions with water. The following variants of hydrolysis are possible:

a) salt formed by a strong base and a weak acid:

$KCN + H_2O \Leftrightarrow HCN + KOH$;

$CN^- + H_2O \Leftrightarrow HCN + OH^-$ –alkaline medium, pH > 7;

b) salt formed by a weak base and a strong acid:

$NH_4Cl + H_2O \Leftrightarrow NH_4OH + HCl$;

$NH_4^+ + H_2O \Leftrightarrow NH_4OH + H^+$ –acid medium, $pH < 7$;

c) salt formed by weak acid and weak base:

$CH_3COONH_4 + H_2O \Leftrightarrow CH_3COOH + NH_4OH$.

The acidity of the environment is determined by the relative strength of the weak acid or weak base formed as a result of hydrolysis.

Salts, formed by strong acids and strong bases, practically do not undergo hydrolysis.

In the hydrolysis of salts of weakly monosaccharides, the reaction proceeds in such a way that acidic salts are formed on the first stage, and on the second one there are weak acids, that is,

I stage

$Na_2CO_3 + H_2O \Leftrightarrow NaHCO_3 + NaOH$; $CO_3^{2-} + H_2O \Leftrightarrow HCO_3^- + OH^-$;

II stage

$NaHCO_3 + H_2O \Leftrightarrow NaOH + H_2CO_3$; $HCO_3^- + H_2O \Leftrightarrow H_2CO_3 + OH^-$.

If the salt is formed by a weak, multi-acid base, then the hydrolysis also occurs step by step, in particular for $ZnCL_2$:

I stage

$ZnCL_2 + H_2O \Leftrightarrow ZnOHCl + HCl$; $Zn^{2+} + H_2O \Leftrightarrow ZnOH^+ + H^+$;

II stage

$ZnOHCl + H_2O \Leftrightarrow Zn(OH)_2 + HCl$; $ZnOH^+ + H_2O \Leftrightarrow Zn(OH)_2 + H^+$.

Purpose: to investigate processes of hydrolysis of salts in solutions.

Setting objectives

Determine: the hydrogen index of the medium of the solutions listed below and compile the molecular and ionic equations of salt hydrolysis.

Equipment and reagents: test tubes, burner, indicator paper,

10 % solution of salts of zinc sulfate, sodium carbonate, sodium acetate; 0,1N solution of chloride and acetic acid; 0,1N solution of ammonium hydroxide and

1 % solution of litmus, phenolphthalein, methylorange, distilled water.

Research methodology

1. Color of indicators in different environments

Place 10 drops of distilled water in six tubes and add 1 – 2 drops: in the first two - a solution of litmus, the next two - a solution of methylorange, and in the rest - a solution of phenolphthalein. Tick in table 5 color of these solutions in a neutral environment.

Add in the first of three pairs of tubes a few drops of a solution of alkali and fill their coloring in the table.

In the remaining samples add a few drops of acid and also fix the color of the solutions in the table 5.

Table 5

Research results

Name of indicators	Coloring of solutions		
	acid	neutral	alkaline
Litmus			
Phenolphthalein			
Methylorang			

2. Determination of pH using all-purpose indicator

Pour a small amount of solutions in four test tubes: in the first one – chloride acid, in the second one – vinegar, in the third one – ammonium hydroxide, in the fourth one- distilled water. Measure the pH of these solutions using an all-purpose indicator paper.

3. Hydrolysis of salts

Pour a few drops in three test tubes: in the first one - distilled water, in the second one - a solution of zinc sulfate, in the third one- sodium carbonate. Add 1 - 2 drops of litmus in each of the samples. Compare the colors of the solutions and determine what the environment is. Make molecular and ionic equations of salt hydrolysis.

4. Influence of temperature on hydrolysis of salts

Add 1 – 2 drops of phenolphthalein to the sodium acetate solution and determine the intensity of its color. Heat the test tube to boil and again evaluate the color of the solution.

Test questions and tasks for self-study work

1. Determine the pH of the solution and the nature of the environment in which the molar concentration of H^+ ions is $2{,}8\ ^{10-3}$ mol / l.

2. pH = 9,05. Calculate $[OH^-]$ and $[H^+]$.

3. What medium will solutions of salts $AlCl_3$, Na_3PO_4, $NaHCO_3$ have? Write the molecular and ionic equations of hydrolysis.

4. Give the definition of the ionic product of water.

5. What are the hydrogen and hydroxyl indicators?

7. Record the possible variants of salt hydrolysis with the corresponding ionic equations.

8. What salts are practically not hydrolyzed?

Laboratory work № 5

OXIDATION-REDUCTION REACTIONS

Theoretical provisions

Oxidizing-reducing reactions are called reactions in which there is a change in the oxidation steps of the elements that are a part of the reactants.

The degree of oxidation is called a conditional charge that would have received this element if all the separated electron pairs in it were fully extended in the direction of a more electronegative element.

The degree of oxidation may be positive, negative or zero. The zero degree of oxidation has atoms and molecules of elementary substances. The algebraic sum of all degrees of oxidation in any compound must be zero.

Oxidation is the process of electron returns by atoms or ions, and the reduction is the process of adding them. **An oxidizer** is a substance that includes an element that can connect electrons, thereby reducing its degree of oxidation. **A reducer** is a substance that includes an element that can lose electrons, thereby increasing its degree of oxidation.

When compiling the equations of oxidation-reduction reactions it is advisable to observe the following order:

1. Write a reaction scheme.
2. Set the values of the oxidation steps over the elements and emphasize those that change the degree of oxidation.
3. Make the electronic equations and calculate the coefficients using the electronic balance method.
4. Transfer the calculated values of the coefficients into the reaction scheme, then equalize the metals that have not changed the degree of oxidation, then equate the acid residues and the H + ions.
5. To check the found coefficients, use the oxygen balance.

For example:

$$K^{+1}Mn^{+7}O_4^{-2} + 16H^{+1}Cl^{-1} = 2K^{+1}Cl^{-1} + 2Mn^{+2}Cl_2^{-1} + 5Cl_2^0 + 8H_2^{+1}O^{-2}$$

$$\begin{array}{r|l} 2 & Mn^{+7} + 5\bar{e} = Mn^{+2} \text{ – reduction, } \quad KMnO_4 \text{ – oxidizer;} \\ 5 & 2Cl^{-1} - 2\bar{e} = Cl_2^0 \text{ – oxidation, } \quad HCI \text{ – reducer.} \end{array}$$

The purpose of the work: to investigate the oxidation-reduction processes and to master the technique of compiling the equations of oxidative-reducing reactions.

Setting objectives

Determine: oxidation, reduction, oxidant and reductant processes, to determine the coefficients in the equations of oxidation-reduction reactions, to analyze the oxidizing properties of $K_2Cr_2O_7$ in the acidic medium.

Equipment and reagents: test tubes, iron nail, iodine solution,

10 percent solutions of sulfur dioxide sulfate, sodium sulfite, sulfate acid, potassium dichromate and potassium iodide; 0.1 % solution of starch and distilled water.

Methodology of research

1. Oxidation of iron with Cu^{2+} ions

Pour a solution of $CuSO_4$ salt in a test tube to one third of its volume and immerse a carefully cleaned iron nail in it. The appearance on the surface of iron of a red plaque of metallic copper indicates the reduction of Cu^{2+} ions.

Make electronic equations of oxidation and reduction processes. Identify an oxidizer and a reducer. Write the equation of the total reaction of these processes.

2. Interaction of iodine with sodium sulfite

Put a few drops of iodine solutions in a test tube and add a solution of sodium sulfite (Na_2SO_3). The reaction follows the following pattern:

$$I_2 + Na_2SO_3 + H_2O \rightarrow Na_2SO_4 + HI.$$

Make an equation of oxidation and reduction processes. Identify an oxidizer and a reducing agent.

3. Interaction of potassium dichromate with potassium iodide in acid solution

Mix a few drops of potassium dichromate solution ($K_2Cr_2O_7$) in a test tube, and the same amount of 10 % solution of sulfate acid, then add 2 – 3 drops of starch as an indicator. Add a solution of potassium iodide to the obtained solution by dropping, shaking to change the color to the blue one. The reaction proceeds according to the following scheme:

$$K_2Cr_2O_7 + H_2SO_4 + KI \rightarrow Cr_2(SO_4)_3 + K_2SO_4 + I_2 + H_2O.$$

4. Interaction of potassium dichromate with potassium sulphite in acid solution

Add the same amount of 10% solution of sulfate acid in a tube with several drops of potassium dichromate. Then add to the contents of the test tube a drop of sodium sulfite solution until the color changes to green. The reaction follows the following pattern:

$$K_2Cr_2O_7 + H_2SO_4 + Na_2SO_3 \rightarrow Cr_2(SO_4)_3 + Na_2SO_4 + K_2SO_4 + H_2O.$$

Make an equation of oxidation and reduction processes. Identify an oxidizer and a reducing agent. Write the equation of the total reaction of these processes.

Test questions and tasks for self-study work

1. What reactions are called oxide-reducing?
2. Define the degree of oxidation.
3. Describe the oxidation and reduction processes.

4. What substances can be only oxidants, which are only reductants, and which one are both oxidants and reducing agents?

5. What order should be followed when writing oxidation-reduction reactions?

6. Write the equation of oxidation-reduction reactions which take place under the following schemes:

1. $K_2Cr_2O_7 + H_2SO_4 + C_2H_5OH \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + CH_3COH + H_2O$;

2. $Mn(NO_3)_2 + PbO_2 + HNO_3 \rightarrow HMnO_4 + Pb(NO_3)_2 + H_2O$;

3. $NaCrO_2 + H_2O_2 + NaOH \rightarrow Na_2CrO_4 + H_2O$.

Laboratory work № 6

ELECTROLYSIS

Theoretical provisions

Electrolysis is an oxidation-reduction process that occurs when a constant electric current passes through a solution or melt of an electrolyte.

Under the action of a direct current, the ions of the electrolyte acquire a directed motion: the negative ions move to the **anode** (positively charged electrode) and therefore they are called **anions**, and positive ions - to the **cathode** (negatively charged electrode) and they are called **cations**. The oxidation process takes place at anode, and reduction occurs on the cathode.

The peculiarities of the chemical reactions that accompany the electrolysis process are due to the nature of the electrolyte and the material of the electrodes. The electrolysis of molten salts is most easily carried out, ions that are a part of the electrolyte are discharged on the electrodes. For example:

$$CaCl_2 \Leftrightarrow Ca^{2+} + 2Cl^-$$

Melt

(–) cathode $Ca^{2+} + 2\bar{e} = Ca^0$ – reduction;

(+) anode $2Cl^- - 2\bar{e} = Cl_2^0$ – oxidation.

Significantly more complicated are the processes of electrolysis of aqueous solutions of electrolytes, where, in addition to the electrolyte ions, water is present. In the process of electrolysis it can be used **insoluble** and **soluble** anodes. Insoluble (inert) anodes are often made of platinum and graphite.

At electrolysis of aqueous solutions of electrolytes with using insoluble anodes there is an order of discharge of ions on electrodes:

- the ions of active metals that are located in the range of the metal volumes from the beginning including the cation of aluminum are not reduced on the cathode, but the N + from the water is reduced by the following reaction:

$$2H_2O + 2\bar{e} = H_2 + 2OH^-;$$

On the anode there is no oxidation of anions of oxygen-containing acids (CO_3^{2-}, SO_4^{2-}, NO_3^-, etc.), and O^{2-} oxidizes from water the reaction:

$$2H_2O - 4\bar{e} = O_2 + 4H^+.$$

Consider an example of electrolysis of an aqueous solution of potassium nitrate with an insoluble anode:

$$KNO_3 \Leftrightarrow K^+ + NO_3^-;$$

$$(-)\ \text{cathode: } K^+, H_2O; \quad 2H_2O + 2\bar{e} = H_2 + 2OH^-;$$
$$(+)\ \text{anode: } NO_3^-, H_2O; \quad 2H_2O - 4\bar{e} = O_2 + 4H^+.$$

As the written equation shows, at electrolysis of this salt, none of the ions of the electrolyte is discharged, but electrolysis of water occurs.

In electrolysis process with the use of a soluble anode, there is oxidation of neither electrolyte ions nor water, but the metal dissolves from which the anode is made of.

Consider electrolysis of nickel sulfate solution with nickel anode:

$$NiSO_4 \Leftrightarrow Ni^{2+} + SO_4^{2-};$$

$$(-)\ \text{cathode: } Ni^{2+}, H_2O; \quad Ni^{2+} + 2\bar{e} = Ni^0;$$
$$(+)\ \text{anode: } SO_4^{2-}, H_2O; \quad Ni^0 - 2\bar{e} = Ni^{2+}.$$

The number of substances isolated on the electrodes is determined according to the Faraday law, using the following formula:

$$m = \frac{M_{\text{eq}} \cdot I \cdot t}{F},$$

where M_{eq} is the molar mass of the equivalent of a substance isolated on the electrode, g / mol; And I is current strength, A; t – hour, c; Faraday number F = 96500 Cl / moles.

Purpose: to study the processes of electrolysis of solutions of electrolytes.

Setting objectives

Master the method of electrolysis of salt solutions and compile schemes of electrolysis processes.

Accessories and reagents: a device for studying the electrolysis process (U-shaped tube with graphite electrodes), a DC source, 0,5 M solution of salts of potassium (II) chloride, potassium iodide and sodium sulfate; 1 % solution of phenolphthalein, starch and litmus.

Methodology of research

1. Electrolysis of the solution of copper (II) chloride

Pour a copper (II) chloride solution into a U-shaped tube. Insert graphite electrodes into both ends of the tube and connect them to a DC source. What happens on electrodes? Make a diagram of electrolysis.

2. Electrolysis of potassium iodide solution

Pour into a U-shaped tube a solution of potassium iodide, to which 3 to 4 drops of phenolphthalein are added. Insert graphite electrodes in both joints and turn on the current. What is observed near the electrodes? Add 1 to 2 drops of starch solution into anode space. What's happening? Make a diagram of electrolysis.

3. Electrolysis of sodium sulfate solution

Pour into a U-shaped tube a solution of sodium sulfate, to which add 3 to 4 drops of litmus solution. Immerse the graphite electrodes in the tube and turn on the current. Observe the allocation of gas bubbles on the electrodes and the change of color of the solution. Make a diagram of electrolysis.

Test questions and tasks for self-study work

1. What oxidative-reducing process is called electrolysis?
2. What is the name of the electrolyte ions moving to the anode or cathode?

3. Describe the soluble or insoluble anodes. What processes occur on soluble anodes?

4. What process occurs on the cathode during electrolysis? Draw a scheme of electrolysis of a solution of nickel (II) chloride using graphite and nickel anodes.

5. What process is observed during electrolysis of aqueous solutions of $CdSO_4$, $NaNO_3$, KCl, if the anode is graphite?

6. Make diagram of electrolysis of solution of nickel(II) chloride using graphite and nickel anodes.

7. What processes occur during electrolysis of aqueous solutions of $CdSO_4$, $NaNO_3$, KCI, if the anode is graphite?

8. During what time it is necessary to pass a current of 3 A through a solution of nitrogen argentum so that 1 g of silver is isolated on the cathode?

Laboratory work № 7

CORROSION OF METALS AND PROTECTION AGAINST IT

Theoretical provisions

Corrosion is chemical or electrochemical destruction of metals, which occurs under the influence of the environment. There are two types of corrosion: **chemical** and **electrochemical**. Chemical corrosion occurs when metals contacted with dry gases, solutions of non-electrolytes (gas, gasoline, technical lubricants).

Electrochemical corrosion is observed when metals contacted with water, electrolyte solutions, and with damp air. When electrochemical corrosion is formed, a lot of micro-agglomeration elements which are called corrosion. As an example of electrochemical corrosion, let’s consider the effect of sulfate acid on technical zinc containing iron admixtures. On the surface of such zinc there is a large number of micro-galvanic elements in which zinc serves as a negatively charged electrode, and iron is positively, that is,

$$(-)\ Zn\ |\ H_2SO_4\ |\ Fe\ (+).$$

$$(-)\ Zn^0 - 2\,\bar{e} = Zn^{2+};$$
$$(+)\ 2H^+ + 2\,\bar{e} = H_2^0.$$

Thus, the impurities contained in the metal are one of the causes of electrochemical corrosion.

Electrochemical corrosion can also occur when two metals are contacted.

The corrosion process is largely influenced by the polarization of electrodes, the formation of films on metals, and others. There are various ways to fight corrosion, the main of which is the application of protective coatings (lamination, galvanizing, nickel, chromium, etc.), electrochemical protection, adding corrosive substances (inhibitors) into corrosive media.

Coatings can be cathodic (lignified iron) and anodic (galvanized iron). For **cathode coating** it is used less active metal than protected, for **anode** - more active.

In **electrochemical protection**, metal is connected to a negative electrode of an external current source (**cathodic protection**) or to a more electronegative metal (**ground protection**). Electrochemical protection is appropriate to be used in solutions of electrolytes, in soil, in atmospheric conditions.

The effectiveness of the protective substances used in the industry (inhibitors) is high. Thus, adding of small amounts (0.1 – 0.5 % by weight) of acid inhibitors of PB and FM marks, reduces the rate of corrosion of iron in tens and hundreds of times.

The purpose of the work: the study of corrosion processes and the explanation of oxidation-reduction reactions that take place on the anode and cathode areas.

Setting objectives

Simulate corrosion processes and draw up schemes of formed corrosion elements.

Equipment and reagents: chemical glass, porcelain cup, glass pipette, U-shaped tube, coal and iron electrodes, DC source galvanized and laminated iron plates, zinc granulate, copper wire, 0.01 N solution of sulfate acid, 10 % solution of cooper (II) sulfate and potassium hexacyanoferrate (III) and 3 % solution of sodium chloride.

Research methodology

1. Corrosion on the contact of two metals

a) Pour a solution of sulfate acid in a glass. Immerse a zinc pellet in it and observe the slow release of hydrogen.

b) In the same glass, put the copper wire without touching zinc. Does hydrogen deposit on a copper wire?

c) Engage zinc granule and copper wire. Draw a scheme of action of the formed galvanic couple. Explain the increase in zinc corrosion rate.

2. Formation of micro-galvanic couples

a) Place a zinc pellet in a test tube and add a small amount of dilute sulfate acid. Pay attention to the slow release of hydrogen.

b) Add a few drops of copper (II) sulfate solution to the same test tube. What is observed? Write the equation of the corresponding reactions.

3. Properties of metal coatings

Apply one scratch with a knife or a nail on galvanized and laminated iron plates. Using a glass pipette, place a solution of dilute sulfate acid containing $K_3[Fe(CN)_6]$ in place of scratches.

In what specimen will there be corrosion of iron? Explain the results of the experiment, draw up a scheme of corrosion galvanic couples.

4. Electrochemical protection

Add a NaCl solution to the U-shaped tube and a few drops of $K_3[Fe(CN)_6]$ solution. Insert into the tube two electrodes – carbon (anode) and iron (cathode). Impose current for 5 – 10 minutes through them. For comparison, another sample of iron is immersed in a glass of solution of the same electrolyte.

In which case will the corrosion process occur faster? Explain the results of the experiment.

Test questions and tasks for self-study work

1. What process is corrosion of metals?
2. Name the types of corrosion and characterize each of them.
3. What are the methods of protecting metals from corrosion?
4. What differs iron corrosion in acidic environment from corrosion in neutral?
5. For which metals can the alkaline environment be harmful?
6. Give examples of using electrochemical protection of metals from corrosion in the industry.
7. What are the features of corrosion transformation of nickel-plated iron in neutral and acidic environments at perishing of its protective coating?

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Хімія
МЕТОДИЧНІ РЕКОМЕНДАЦІЇ ДО ВИКОНАННЯ ЛАБОРАТОРНИХ РОБІТ З ДИСЦИПЛІНИ
(Англійською мовою)

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