



**Institute of Mathematics,
Physics and Chemistry
Department of Chemistry**

Technical chemistry laboratory

Laboratory exercise

Electrolysis

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EXERCISE SHEET

1	Relation to subjects: ESO/26, DiRMiUO/26, EOUNIE/26		
	Specialty/Subject	Learning outcomes for the subject	Detailed learning outcomes for the subject
	ESO/25 Technical Chemistry	EKP1 K_W01, K_W02, K_U05 EKP2 K_U08, K_U09	SEKP7 – Mastering the experimental knowledge in studying the phenomenon of electrolysis and drawing conclusions from the conducted experiments. Expanding knowledge on the applications of the electrolysis process in technology and industry.
	ESO/26 Chemistry of water, fuels and lubricants	EKP3 K_U014, K_U015, K_U016	SEKP6 – Determination of oxygen and ammoniacal nitrogen content in technical water. SEKP6 – Determination of corrosion inhibitors in technical water.
	DiRMiUO/26 Chemistry of water, fuels and lubricants	EKP3 K_U014, K_U015, K_U016	SEKP6 – Determination of oxygen and ammoniacal nitrogen content in technical water. SEKP6 – Determination of corrosion inhibitors in technical water.
	EOUNIE/26 Chemistry of water, fuels and lubricants	EKP3 K_U014, K_U015, K_U016	SEKP6 – Determination of oxygen and ammoniacal nitrogen content in technical water. SEKP6 – Determination of corrosion inhibitors in technical water.
2	Purpose of the exercise: 1. Learning and consolidation of the basic concepts related to the phenomenon of electrolysis and the principles of operation of the electrolyser. 2. Understanding and analyzing the reactions taking place in the electrolyser, knowledge of the laws relating to the phenomenon of electrolysis. 3. Getting to know practical examples of the use of the phenomenon of electrolysis and the use of basic calculations relating to the studied phenomenon.		
3	Prerequisites: general chemical knowledge of the phenomenon of electrolysis		
4	Description of the laboratory workplace: Electrolyser, set of electrodes, wash bottle, glass rod, desiccator, beaker with a capacity of 100 cm ³ , solutions: concentrated sulphuric acid(VI) H ₂ SO ₄ , (1 : 1), nitric acid (V)		

	HNO ₃ , 1% sodium sulphide solution Na ₂ S, ethyl alcohol C ₂ H ₅ OH, (1%), copper(II) sulphate(VI) solution CuSO ₄ , solids: ammonium nitrate(V) (NH ₄ NO ₃)
5	<p>Risk assessment: the likelihood of chemical burns from contact with sulphuric acid is very small, and the effects are minimal. Final assessment – VERY SMALL THREAT</p> <p>Safety measures required:</p> <ol style="list-style-type: none"> 1. Lab coats, gloves and safety glasses. 2. Health and safety cleaning products, paper towels.
6	<p>The course of the exercise:</p> <ol style="list-style-type: none"> 1. Getting to know the workplace instructions for the exercise (appendix 1), 2. Performing individual exercises according to the instructions.
7	<p>Exercise report:</p> <ol style="list-style-type: none"> 1. Develop an exercise in accordance with the instructions contained in the workplace manual. 2. Solve the given tasks and/or answer the questions included in the set of tasks and questions to be completed by the student.
8	Archiving of research results: Submit the report on the exercise in the applicable form at the beginning of the next laboratory exercises.
9	<p>Assessment method and criteria:</p> <ol style="list-style-type: none"> a. EKP1, EKP2 – checking the knowledge of basic chemical concepts related to the phenomenon of electrolysis during the classes, b. SEKP7 – the detailed effect of the student's education will be assessed on the basis of the observations, conclusions and solved tasks and problems presented in the report, recommended for independent solution/development: <ul style="list-style-type: none"> – mark 2,0 – the student has too little knowledge of the electrolysis process, electrode reactions and the laws governing this phenomenon, or is unable to solve simple tasks regarding the above-mentioned concepts; – mark 3,0 – has basic chemical knowledge of the electrolysis process, electrode reactions and the laws governing this phenomenon and is able to solve simple problems in his profession regarding the above-mentioned concepts; – mark 3,5 – 4,0 – has extensive knowledge of chemistry and in the field of the electrolysis process, electrode reactions and the laws governing this phenomenon, and has the ability to solve complex tasks in his specialty regarding the above-mentioned concepts; – mark 4,5 – 5,0 – has the ability to apply complex knowledge of the electrolysis process, electrode reactions and the laws governing this phenomenon, and is able to solve problem tasks in his specialty regarding the above-mentioned concepts.
10	<ol style="list-style-type: none"> 1. Stundis H., Trzeźniowski W., Żmijewska S., Ćwiczenia laboratoryjne z chemii nieorganicznej, WSM, Szczecin 1995. 2. Pazdro K., Rola-Noworyta A. Chemia Repetytorium dla przyszłych maturzystów i studentów, Oficyna Edukacyjna. Krzysztof Pazdro, Warszawa 2015 r. 3. Jones L., Atkins P. Chemia Ogólna, Wydawnictwo Naukowe PWN, Warszawa 2014. 4. Praca zbiorowa pod redakcją K. Pazdro: Chemia nieorganiczna. WSiP, Warszawa 1992. 5. Molenda J. Technologia chemiczna. WSiP, Warszawa 1996. 6. Bortel E., Koneczny H. Zarys technologii chemicznej. PWN, Warszawa 1992.

	<p>7. Barbir F. PEM electrolysis for production of hydrogen from renewable energy sources. Solar Energy 2005, Vol. 78.</p> <p>8. Kotowski W. Gdy zabraknie ropy i gazu. Wodór staje się paliwem przyszłości. Nafta & Gaz Biznes, 2004, nr 2.</p> <p>9. Educational Web www.szkolnictwo.pl</p> <p>10. https://www.greatmining.com/magnesium.html – from 1.03.2021</p> <p>11. https://zasoby1.open.agh.edu.pl/dydaktyka/chemia/a_e_chemia/7_elektrochemia/07_03_01.htm – z dnia 1.03.2021 r.</p> <p>12. https://fineartamerica.com/featured/classic-chrome-grill-bill-schaudt.html – z dnia 1.03.2021 r.</p>
11	Notes

APPENDIX 1 – MANUAL

1. SCOPE OF THE EXERCISE:

Issues and keywords:

- The phenomenon of electrolysis,
- Principles of operation of the electrolyser,
- Reactions taking place in the electrolyser,
- Faraday's laws,
- Practical use of the phenomenon of electrolysis.

2. THEORETICAL INTRODUCTION TO THE EXERCISE

2.1. Definition of electrolysis and the principle of operation of the electrolyser

Electrolysis is a method of carrying out a non-spontaneous reaction with the help of electric current. It is a set of changes at the interface between a metallic conductor and an ionic conductor, taking place under the influence of a sufficiently high electric voltage applied to the metallic conductors of the electrolyser.

The **electrolyser** is a system in which the electrolysis process is carried out. It consists of two electrodes (metallic phases, plates) immersed in an electrolyte solution. The electrolysis process begins when the electrodes of the electrolyser are connected to a DC source (for example, it may be a set of cells with an appropriately selected voltage or an AC rectifier). A simplified diagram of the electrolyser is shown in Fig. 1.

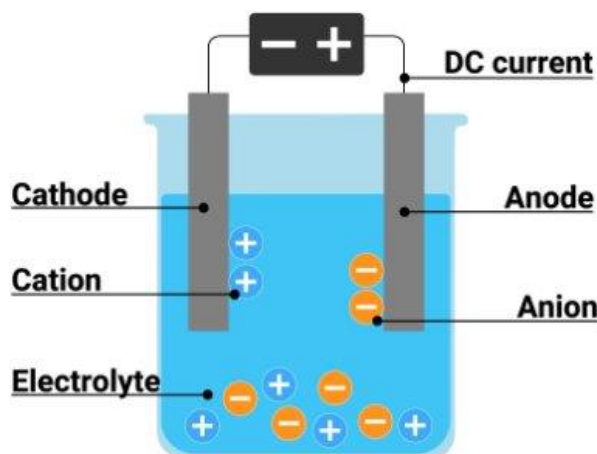
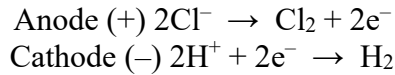


Fig. 1. Schematic diagram of the electrolyser

source: <https://www.proprofs.com/quiz-school/story.php?title=electrolysis-quiz>
(accessed October, 5, 2021)

In an electrolyser, the electrode connected to the positive pole of the current source is called the **anode**, and the electrode connected to the negative pole of the current source is called the **cathode**. The **oxidation** process takes place at the anode, the **reduction** process takes place at the cathode. In the case of, for example, the electrolysis of an aqueous HCl solution, reactions take place:



Galvanic cells enable the transfer of energy from the cell to the environment, while in the electrolyser, an external source of current transfers energy to the reactants of the electrolyser.

2.2. Faraday's laws

An important issue in the electrolysis process is to calculate the amount of product formed by a certain amount of electricity. This is possible thanks to the calculations made by Michael Faraday, the English physicist and chemist who created the laws of Faraday electrolysis. Two Faraday's laws describe the electrolysis process quantitatively.

The first Faraday's law says that the mass of the substance (m) separated on one of the electrodes is directly proportional to the intensity of the current I and the duration of electrolysis t :

$$m_e = k \cdot I \cdot t$$

where:

- m_e – mass of substance released on one of the electrodes [g];
- k – electrochemical equivalent (this is the mass of the substance that is released on the electrode when a unit charge passes through the electrolyte, for example 1 C) [g/C];
- I – the intensity of the electric current flowing through the electrode [A];
- t – duration of electrolysis [s].

Faraday's second law says that the ratio of the molar mass M of the substance that is released at the electrode to the product of its electrochemical equivalent (k) and the charge number (z) of the electrode reaction, which is recorded for 1 mole of a substance with a molar mass M , is a constant value for all electrode processes and amounts to **96 500 C/mol** (it is the Faraday constant, symbol **F**):

$$\frac{M}{kz} = F$$

where:

- M – molar mass of substance evolving at the electrode [g/mol];
- z – the charge number of the reaction (positive, dimensionless quantity, equal to the stoichiometric electron coefficient in the electrode reaction equation);
- k – electrochemical equivalent [g/C];
- F – Faraday constant of approximately 96 500 [C/mol].

Faraday's second law also says that the same amount of electricity flowing through different electrolytes will release on the electrodes masses of substances proportional to their electrochemical equivalents according to the relationship:

$$\frac{m_1}{m_2} = \frac{k_1}{k_2}$$

After transforming the two Faraday laws, an equation is obtained, which is the basis for quantitative calculations of the electrolysis process:

$$m_e = \frac{M}{zF} \cdot I \cdot t$$

where:

- m_e – mass of substance evolving on one of the electrodes [g];
- M – mass of mole of substance evolving on the electrode [g/mol];
- z – the charge number of the reaction (positive, dimensionless quantity, equal to the stoichiometric electron coefficient in the electrode reaction equation);
- F – Faraday constant of approximately 96 500 [C/mol];
- I – the intensity of the electric current flowing through the electrode [A];
- t – duration of electrolysis [s].

1.1. The use of electrolysis in practice

Electrolysis of molten binary salts

Electrolytic methods are widely used in the preparation of base metals such as alkali metals, beryllium metals and aluminum. Elements with the most negative standard potentials are obtained only by electrolytic means (lithium, sodium, potassium, magnesium, aluminum). Figure 2 shows a schematic of the electrolyser used in the *Dow* process to separate magnesium from molten magnesium chloride (it is the electrolyte in the process).

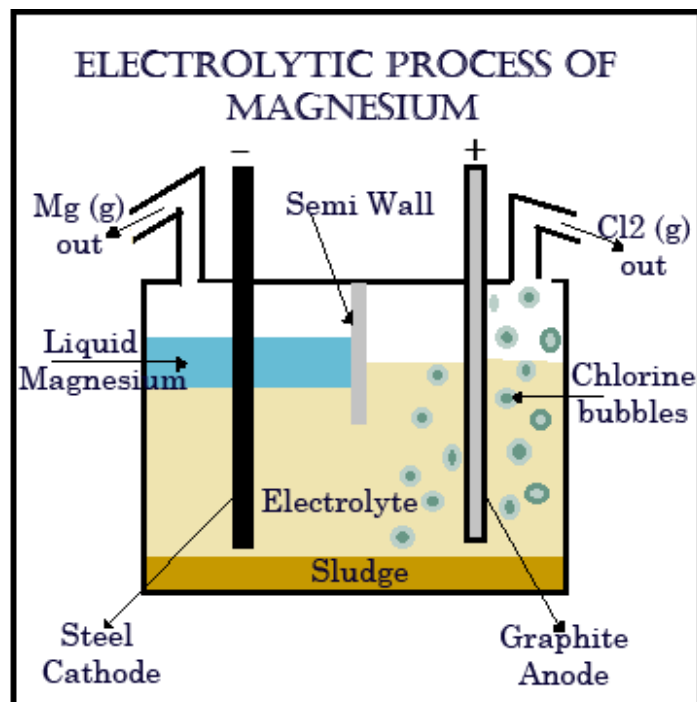


Fig. 2. The electrolyser used to separate magnesium
 source: <https://www.greatmining.com/magnesium.html>
 (accessed October, 5, 2021)

As a result of the high temperature in this process, the crystal lattice of the fused magnesium chloride is destroyed, thanks to which the free movement of the ions towards the electrodes becomes possible. When electricity from an external source flows through the electrolyser, metallic magnesium (Mg) is formed at the cathode and chlorine gas (Cl₂) at the anode.

Another practical example of an oxygen-free molten salt electrolysis process is the Downs process for the production of metallic sodium. In this process, the electrolysis of molten rock salt (sodium chloride) is carried out. At the graphite anode, Cl⁻ ions oxidize to chlorine, while at the steel cathode, Na⁺ ions are reduced to metallic sodium (Fig. 3).

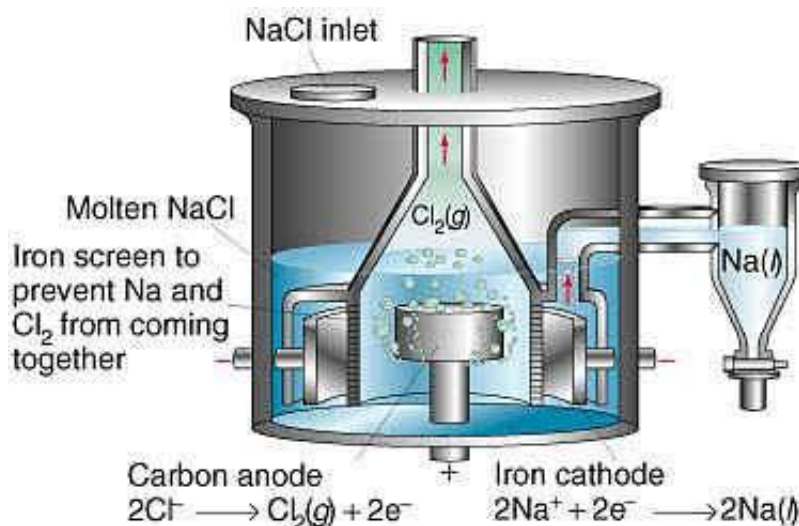


Fig. 3. Electrolyser used in the Downs process to obtain metallic sodium and chlorine
 source: <https://corrosion-doctors.org/Electrowinning/Sodium.htm>
 (accessed October, 5, 2021)

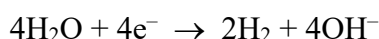
Electrolysis of oxygen salts

If electrolysis is carried out in a solution of active metals, e.g. sodium sulphate(VI), Na₂SO₄, then Na⁺ and SO₄²⁻ ions serve as charge transporters that must be delivered to the cell, and water is electrolysed.

At the anode, oxygen is released according to the equation:



At the cathode, hydrogen is released:



According to the mechanism of the electrolysis reaction, in this case the salts of oxygen acids and the active metal on both electrodes give way to the processes of water electrolysis. It is worth noting that the volume of generated hydrogen is twice as large as that of oxygen released at the anode. It is of great practical importance in various industrial processes (obtaining metallic sodium, producing gases, etc.).

An example comparison of the reactions for the electrolysis of molten binary salts on the example of NaCl (Fig. 4a) and salts of oxoacids on the example of AgNO₃ (Fig. 4b), is given below. In both cases, we assume that the electrolysis is carried out with the use of neutral electrodes, made, for example, of platinum or graphite. In the diagram, the symbol K (-) denotes the cathode, A (+) – the anode.

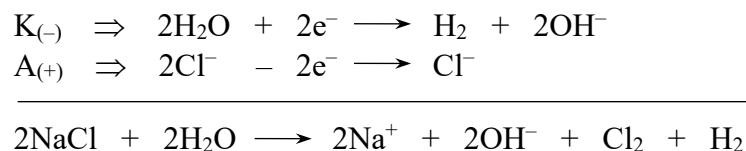


Fig. 4a. An example of an electrolysis reaction of molten binary salts

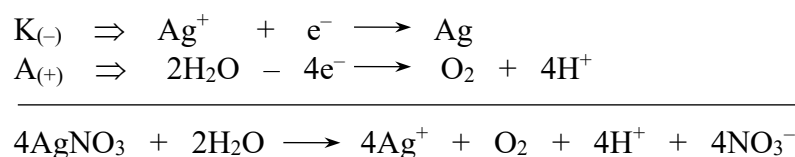
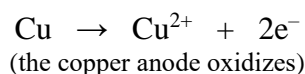


Fig.4b. An example of an electrolysis reaction of molten salts of oxoacids

If the anode is made e.g. of copper (noble metal), the process of its anodic dissolution should be additionally taken into account, e.g. during electrolysis of CuSO₄ water solution on copper electrodes:



If the cathode is made, for example, of mercury, then under these conditions all metals are reduced at the cathode.

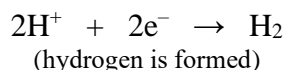
If molten electrolytes such as oxides or hydroxides were subjected to electrolysis, then under these conditions all metals are reduced at the cathode.

Electrolysis of an aqueous acid solution

During the electrolysis of binary acids, as a rule, hydrogen is released at the cathode, and oxygen or other products are released at the anode (e.g. Cl₂ with HCl).

Oxoacids solutions (H₂SO₄, H₃PO₄, HNO₃, H₂CO₃) always give oxygen and hydrogen.

If we are dealing with solutions of dilute acids, the reactions taking place in the solution are the same as for dilute salt solutions. In the case of higher concentrations on the cathode, we deal with the following reaction:



On the other hand, at the anode, the product of anodic oxidation in the case of anions of oxoacids oxygen is supplied by OH⁻ ions or water molecules, and in the case of binary acids, the anions of acidic residues are oxidized with the release of the product, e.g. chlorine:



A schematic comparison of the electrolysis processes for oxoacids and binary acids (using neutral electrodes) is given in Fig. 5. (a, b). In the diagram, the symbol K (-) denotes the cathode, A (+) – the anode.

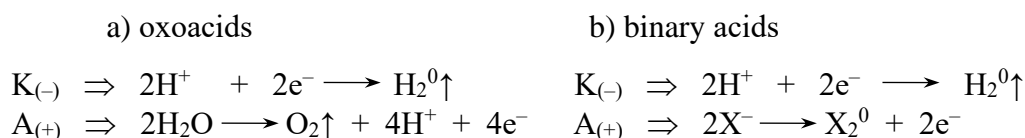
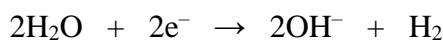


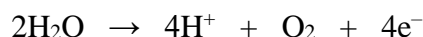
Fig. 5. (a, b) Comparison of electrolysis processes for oxoacids and binary acids

Alkaline electrolysis

If a very dilute base solution is electrolysed, and no excessive voltage is applied to the electrodes from an external power source, then mainly water is decomposed. The cathode reaction produces hydrogen as shown in the equation:

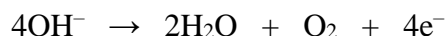


As a result of the anode reaction, oxygen is released:



It is also worth noting that the volume of formed hydrogen is twice as large as the volume of formed oxygen (under the same conditions). As can be seen from the reactions written above, the solution around the anode becomes acidic due to the formation of hydrogen ions and the solution around the cathode becomes alkaline due to the formation of hydroxyl ions.

If concentrated alkaline solutions are electrolysed, the anode reaction is slightly different:



In summary, almost all electrode reactions that occur when electric current flows through very dilute aqueous solutions lead to the evolution of hydrogen and oxygen.

Taking into account the position of metals in the voltage series, we can conclude that:

- Light metal, i.e. located in the electrochemical-series at the beginning – including aluminium ($E^0 \leq -1.66\text{V}$) does not reduce at the cathode;
- At the cathode, water is reduced, hydrogen gas is released, and the pH of the solution increases;
- Oxidation of OH^- ions takes place at the anode, oxygen gas is released, the pH of the solution decreases;
- The metal behind the aluminium in the electrochemical series ($E^0 > -1.66\text{V}$) is reduced at the cathode.

Electroplating

This process involves the electrolytic deposition of a very thin layer of metal on an object. The electroplated object made of metal or graphite-coated plastic forms the cathode and

the electrolyte is an aqueous salt solution of the deposited metal. Thus, the metal is deposited on the cathode as a result of the reduction of the metal's cations, which may come from the added salt or the oxidation of the anode made of the deposited metal. For the chrome plating process, the electrolyte is prepared by dissolving CrO_3 in dilute sulphuric acid. In the process of electrolysis, chromium(VI) is reduced to chromium(III) and then to chromium(0) according to the equation:



Chromium is deposited on the cathode as a compact protective layer. The anode, i.e. the positive pole of a direct current source, in galvanizing processes most often consists of metal plates from which the coating is made (e.g. chromium, nickel, copper, etc.) and which dissolve in the electrolyte during electrolysis. However, in the process of chrome plating, the positive pole is made of plates made of a material insoluble during electrolysis, and the metal for the coating comes from the electrolyte.

The chrome plating process (shown in Fig. 6) uses a lot of electricity, because six electrons are needed to form one deposited chromium atom. In addition, it can also be burdensome due to the generated waste posing a threat to the environment.



Fig. 6. Chrome-plating of steel parts of classic cars used to give a decorative and protective effect
source: <https://rhkhydraulics.com/blog/hard-chrome-plating-extending-the-life-of-your-equipment>
(accessed January, 14, 2022)

Summarizing the above considerations, it can be stated that electrolysis is a process used on an industrial scale, inter alia, for:

- metal production: aluminum, copper, lithium, sodium, potassium;
- production of various chemical compounds, including aspirin, trifluoroacetic acid, sodium hydroxide, potassium, sodium chlorate and potassium chlorate – the use of various redox reactions – oxidation and reduction that occur during electrolysis;
- production of gases: hydrogen, chlorine and oxygen – as a result of electrolysis of appropriate salts, where the desired gases are released;
- electroplating – covering with a thin layer of metal another metal.

3. PERFORMING THE EXERCISE

Experiment 1 – Analysis of the phenomenon of electrolysis

Instruments and reagents:

Electrolyser, set of electrodes, wash bottle, glass rod, desiccator, beaker with capacity 100 cm³, solutions: concentrated sulphuric acid(VI) H₂SO₄, (1 : 1), nitric acid(V) HNO₃, 1% sodium sulphide solution Na₂S, ethyl alcohol C₂H₅OH, (1%), copper(II) sulphate(VI) solution CuSO₄, solids: ammonium nitrate(V) (NH₄NO₃).

Performance:

The person conducting the exercises according to the scheme connects the prepared electrodes to the electrolyser (clean and dried to a solid mass). Then, he adds about 2 cm³ of concentrated sulphuric acid and 2.5 g of ammonium nitrate (NH₄NO₃) to the test solution. Platinum electrodes connected to the electrolyser are immersed in the heated to 353 K solution and the electrolysis is carried out at a voltage of 3 – 4 V using a magnetic stirrer. When the solution becomes discoloured (after 30 – 60 minutes), which indicates that almost all of the copper has been released from the solution, it increases the voltage to 4.2 V and the electrolysis process continues for another 30 minutes. You can tell if the copper has released completely by adding a little water or by immersing the electrodes deeper and observing whether a new layer of copper is depositing on the freshly submerged platinum electrode surface. The presence or absence of copper in the electrolyte can be checked more accurately by means of a drop reaction on a paper with sodium sulphide, Na₂S. For this purpose, the instructor takes 2 drops of the electrolyte (test solution) onto a filter paper and adds 1 drop of sodium sulphide (Na₂S). If copper is present in the test sample, the spot will turn light brown. After electrolysis is completed, the current flow should not be interrupted until the cathode with the separated copper is removed from the test solution and rinsed with distilled water. Premature power failure can dissolve some of the copper. After rinsing the electrode (cathode) with distilled water, the instructor immerses the cathode in alcohol and dries it for about 3 minutes at 373 K. The cathode cooled in the desiccator is weighed on an analytical balance. It then removes the copper from the electrode by immersing it in a nitric acid solution (HNO₃) (1 : 1) under the hood.

Elaboration of the results:

1. Knowing the intensity of the current and duration of electrolysis (given by the academic teacher) and the electrochemical equivalent, calculate the amount of released copper.
2. Describe and present the scheme of the electrolysis of copper(II) sulphate(VI) solution (CuSO₄).

4. DEVELOPMENT OF THE EXERCISES

1. Prepare a report according to the guidelines in the experimental section.
2. Place the cover sheet as the first page of the report.
3. After the theoretical part has been concisely developed, include in the report the study of individual experiments and the solved task/additional tasks given by the academic teacher.

5. THE FORM AND CONDITIONS FOR PASSING THE LABORATORY EXERCISE

1. Passing the so-called „entry test” before starting the exercise.
2. Submission of a correct written laboratory report on the performed exercise in accordance with the guidelines for the preparation of the laboratory report, please see the link below:
<https://www.am.szczecin.pl/en/facilities/institute-of-mathematics-physics-and-chemistry/department-of-chemistry/technical-chemistry/tech-chemistry-lab-manuals/>

I. Examples of tasks with a solution

Example 1

How many grams of copper are released on the platinum cathode during the electrolysis of an aqueous solution of $\text{Cu}(\text{NO}_3)_2$ with a current of 1 A during 9650 seconds?

Solution:

First we write down the cathode reaction:



The mass of the substance released on the electrodes during electrolysis is proportional to the current intensity and duration of the electrolysis and is expressed by the formula:

$$m = k \cdot I \cdot t$$

The proportionality coefficient k , called the electrochemical equivalent, is determined from the dependence:

$$k = \frac{M}{z \cdot F}$$

The molar mass of copper is 63.5 [g/mol], while the formal charge of the z ion is 2, F – Faraday constant 96500 [C/mol].

By combining the above relationships, we determine the mass of copper m , separated at the cathode:

$$m = \frac{M}{z \cdot F} \cdot I \cdot t = \frac{63.5 \left[\frac{\text{g}}{\text{mol}} \right]}{2 \cdot 96500 \left[\frac{\text{C}}{\text{mol}} \right]} \cdot 1 \left[\frac{\text{C}}{\text{s}} \right] \cdot 9650 [\text{s}] = 3.17 [\text{g}]$$

Answer: 3.17 g of copper will be released on the cathode.

Example 2

The electrolysis of the aqueous CuSO_4 solution was carried out for 1.5 hours with a current of 5 A. What is the value of the charge that passed through the solution?

Solution:

The value of the charge that has flowed through the solution during the electrolysis process is calculated from the dependence:

$$Q = I \cdot t = 5 \left[\frac{\text{C}}{\text{s}} \right] \cdot 5400 [\text{s}] = 27000 [\text{C}]$$

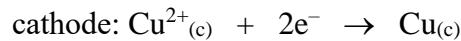
Answer: A charge of 27000 C passed through the solution.

Example 3

Calculate how much copper is released on the electrolyser electrode, if a current of 0.5 A flows through the electrolyser within 1 hour. 4 min. 20 sec.

Solution:

First, we write the half-equation for obtaining copper and determine the molar ratio of electrons and product:



Then we calculate the delivered charge Q based on the dependency:

$$Q = I \cdot t = 0.5 \left[\frac{\text{C}}{\text{s}} \right] \cdot 3860[\text{s}] = 1930[\text{C}]$$

We convert a charge into a number of moles of electrons:

$$\text{number of moles } e = \frac{Q}{1F} = \frac{1930[\text{C}]}{193000 \left[\frac{\text{C}}{\text{mol}} \right]} = 0.01[\text{mol}]$$

We calculate the mass of copper released on the electrode based on the data from the half equation. According to these data, 2 moles of electrons will allow us to obtain 1 mole of copper, i.e. 63.55 g of Cu, respectively 0.01 mole of electrons will allow us to obtain – 0.6355 g of Cu.

Answer: 0.6355 g of copper will be released on the electrode of the electrolyser.

II. Tasks and questions to be completed by the student

1. Calculate how many grams of magnesium can be obtained by electrolysis of 1500 g of magnesium chloride.
2. Write the equations of the reactions taking place during the electrolysis of the aqueous solution of copper(II) sulphate(VI), occurring at the cathode and at the anode, and write down the sum equation for this reaction.
3. The electrolysis reaction of molten lead(II) chloride was performed. Write down the equations of reactions at the cathode and at the anode. Calculate the volume of chlorine released during total electrolysis of 50 g of molten lead(II) chloride
4. How many copper atoms are released on the platinum cathode during the electrolysis of the aqueous CuSO_4 solution with the current of 1A, within 30 minutes?
5. 50 g of water was decomposed during the electrolysis. Calculate the volume of oxygen obtained (normal conditions) and the mass of hydrogen released.
6. What is electrolysis and what is it used for?
7. What current is needed to produce hydrogen by electrolysis and why?
8. Discuss the practical applications of the electrolysis process.
9. What reactions take place on the electrodes in the case of electrolysis of aqueous NaOH solution?
10. What is the electrochemical equivalent of the substance?