MARITIME UNIVERSITY OF SZCZECIN

Institute of Mathematics, Physics and Chemistry
Department of Chemistry

EXERCISE INSTRUCTION

Laboratory Exercise 5

Reduction-oxidation reactions (Redox reactions)

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EXERCISE SHEET

<table>
<tr><td rowspan="5">1</td><td colspan="3">Relation to subjects: ESO/25, 27 DiRMiUO/25, 27 EOUNiE/25, 27</td></tr>
<tr><td>Specialty/Subject</td><td>Learning outcomes for the subject</td><td>Detailed learning outcomes for the subject</td></tr>
<tr><td>ESO/26 Chemistry of water, fuels and lubricants</td><td>EKP3
K_U014, K_U015, K_U016.</td><td>SEKP3 – Water quality indicators;
SEKP6 – Performing determinations of selected indicators of technical water quality;</td></tr>
<tr><td>DiRMiUO/26 Chemistry of water, fuels and lubricants</td><td>EKP3
K_U014, K_U015, K_U016.</td><td>SEKP3 – Water quality indicators;
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SEKP6 – Performing determinations of selected indicators of technical water quality;</td></tr>
<tr><td>2</td><td colspan="3">Purpose of the exercise:
deepening and expanding the chemical knowledge of the oxidation and reduction processes as well as the ability to balance redox reactions and identify the oxidant and reducer</td></tr>
<tr><td>3</td><td colspan="3">Prerequisites:
general knowledge, obtained from high school, on the principles of determining the oxidation states of elements, balancing redox reactions and recording half reactions, knowledge of the principles of work in a chemical laboratory</td></tr>
<tr><td>4</td><td colspan="3">Description of the laboratory workplace:
a set of laboratory glassware, a set of reagents for carrying out redox reactions, indicators</td></tr>
<tr><td>5</td><td colspan="3">Risk assessment:
the likelihood of chemical burns from exposure to 0,2 M sulfuric acid is very small, and the effects are minor,
Final assessment – VERY SMALL THREAT
Security measures required:
1. Lab coats, gloves and safety glasses
2. Health and safety cleaning products, paper towels</td></tr>
<tr><td>6</td><td colspan="3">The course of the exercise:
1. Getting to know the workplace manual (appendix 1)
2. Performing a redox reaction in solution</td></tr>
<tr><td>7</td><td colspan="3">Exercise report:
1. Prepare a report in accordance with the instructions contained in the workplace manual.
2. Solve the given task and/or answer the questions included in the set of tasks and questions to be completed by the student.</td></tr>
<tr><td>8</td><td colspan="3">Archiving of research results:
report on exercises - prepared in accordance with the rules applicable in the laboratory - should be submitted in writing to the academic teacher during the next classes.</td></tr>
<tr><td>9</td><td colspan="3">Assessment method and criteria:
a) EKP1, EKP2 – checking the knowledge of basic chemical concepts related to oxidation and reduction processes during classes,</td></tr>
</table>

	b) SEKP4 - the detailed learning outcome for an individual student will be assessed on the basis of the solutions to tasks and problems presented in the report, given for independent solution/development: – mark 2,0 – the student has too little knowledge about the redox reaction in the solution, or is unable to use it in practice – mark 3,0 – has basic chemical knowledge of redox reaction in solution and is able to use it to a small extent – mark 3,5 – 4,0 – has an extensive knowledge of chemical and redox reactions in solution and is able to use it to solve basic potential technical problems arising in his specialty, – mark 4,5 – 5,0 – has the ability to apply complex chemical knowledge of oxidation and reduction processes and is able to use it to solve complex technical problems
10	**References:** 1. https://assets.openstax.org/oscms-prodcms/media/documents/Chemistry2e-WEB.pdf (accessed 15.07.22). 2. A. Kozłowski, A. Kalbarczyk-Jedynak, M. Ślączka-Wilk, K. Ćwirko, C. Wiznerowicz, G. Gorzycka, Instrukcje stanowiskowe do ćwiczeń laboratoryjnych: Reakcje utleniania i redukcji w roztworach, AM Szczecin 2022 (in Polish). 3. J. E. McMurry, R. C. Fay, J. K. Robinson, Chemistry, 7th edition, global edition, publisher: Pearson, 2016. 4. A. Blackman, S. Bottle, S. Schmid, M. Mocerino, U. Wille, Chemistry, 2nd edition, publisher: John Wiley&Sons, 2012. 5. G. Curran, Chemistry, publisher: The Career Press, 2011. 6. J. T. Moore, Chemistry for Dummies, publisher: Wiley Publishing, 2015. 7. D. Kealy, P.J. Haines, Analytical Chemistry, publisher: BIOS Scientific Publishers Limited, 2002. 8. Sparkcharts Chemistry, 2002 Spark Publishing, A Division of Barnes & Noble, Canada 2014. 9. M. D. Jackson, Chemistry, 2015 BarCharts, Inc. (Quickstudy.com). 10. M. Charmas, English for Students of Chemistry, Maria Curie-Skłodowska University Press, Lublin 2012. 11. Stundis H., Trześniowski W., Żmijewska S.: Ćwiczenia laboratoryjne z chemii nieorganicznej. WSM, Szczecin 1995 (in Polish). 12. M. Wesołowski, K. Szefer, D. Zimna, Zbiór zadań z analizy chemicznej, Wydawnictwa Naukowo – Techniczne, Warszawa 1997 (in Polish).
11	Notes

1. THEORY

KEYWORDS:

- redox reactions,
- oxidation and reduction,
- oxidation number,
- rules for assigning oxidation numbers,
- oxidizing agent and reducing agent,
- half – reactions,
- examples of redox reactions.

Reduction – oxidation reactions (Redox reactions) – reactions in which electrons are exchanged.
Oxidation – electron loss – the oxidation number increases.
Reduction – electron gain – the oxidation number decreases.

OIL RIG
OXIDATION IS LOSS (OIL)
REDUCTION IS GAIN (RIG)
OF ELECTRONS

Oxidation numbers – numbers that allow chemists to for example balance redox reactions.

Rules for assigning oxidation numbers:

- free element (e.g., Ca, O_2, Al, Br_2) – the oxidation number is zero;
- elements in group I have an oxidation number of +1 in compounds;
- elements in group II have an oxidation number of +2 in compounds;
- ionic substance (e.g., Mg^{2+}, Fe^{3+}, Cl^{-}) – the oxidation number = the charge of the ion;
- hydrogen – the oxidation number is +1 in all compounds except for hydrides (e.g., KH, CaH_2), where it has an oxidation number of –1;
- oxygen – the oxidation number is –2 in all compounds except for the peroxides, where it is of –1;
- in all neutral compounds, the sum of the oxidation numbers equals zero;
- in polyatomic ions, the sum of the oxidation numbers equals the charge of the polyatomic ion.

Example 1 – Identify the oxidation numbers for each substance involved:

$$Al_2O_3,\ H_2SO_4,\ Cl^{-},\ Mg^{2+},\ MnO_4^{-},\ Cr_2O_7^{2-}$$

Al_2O_3 the oxidation number of Al is? The oxidation number of oxygen is –II

$$2x + 3 \cdot (-2) = 0$$ – the oxidation number of Al in Al_2O_3 is III

H_2SO_4 the oxidation number of hydrogen is I, oxygen –II, sulfur?

$$2 \cdot 1 + x + 4 \cdot (-2) = 0$$ – the oxidation number of S in H_2SO_4 is VI

Cl^{-} the oxidation number of Cl in Cl^{-} is –I
Mg^{2+} the oxidation number of Mg in Mg^{2+} is II
MnO_4^{-} the oxidation number of oxygen is –II, the oxidation number of Mn is?

$$x + 4 \cdot (-2) = -1$$ – the oxidation number of Mn in MnO_4^- is VII

$Cr_2O_7^{2-}$ the oxidation number of oxygen is –II, the oxidation number of Cr is?

$$2x + 7 \cdot (-2) = -2$$ – the oxidation number of Cr in $Cr_2O_7^{2-}$ is VI

Oxidizing agent – the reactant in a oxidation – reduction reaction that gains electrons.
Reducing agent – the reactant in a oxidation – reduction reaction that donates electrons.

Examples of common oxidizing agents	Examples of common reducing agents
MnO_4^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, HNO_3, $HClO_4$, H_2SO_4, F_2, Cl_2, O_2, O_3	active metals like Na, Mg, Zn, Al, metal hydrides like NaH, CaH_2

Half – reactions – consist of chemical equations that show oxidation and reduction separately and can be combined to give the overall equation for an oxidation – reduction reaction.

Example 2 – Balancing a redox reaction:

e.g., $$SnCl_2 + 2FeCl_3 \longrightarrow 2\,FeCl_2 + SnCl_4$$

- Assign oxidation numbers for each substance involved: chlorine shows an oxidation number –I in all 4 compounds; the tin (Sn) on the reactant side has an oxidation number II; the tin (Sn) on the product side has an oxidation number IV; the iron (Fe) on the reactant side has an oxidation number equals III, on the product side II. Iron (Fe^{3+}) is the oxidizing agent, tin (Sn^{2+}) is the reducing agent.
- Separate the half – reactions:

$$Fe^{3+} + e^- \longrightarrow Fe^{2+}$$
$$Sn^{2+} \longrightarrow Sn^{4+} + 2e^-$$

- Balance the electrons in the equations; in this case the electrons are balanced by multiplying the entire first half – reaction by 2 and leaving the second half – reaction as it is:

$$Fe^{3+} + e^- \longrightarrow Fe^{2+} \,/x2$$
$$Sn^{2+} \longrightarrow Sn^{4+} + 2e^-$$

$$2Fe^{3+} + 2e^- \longrightarrow 2Fe^{2+}$$
$$Sn^{2+} \longrightarrow Sn^{4+} + 2e^-$$

- Add two equations and cancel out the electrons:

$$2Fe^{3+} + \cancel{2e^-} + Sn^{2+} \longrightarrow 2Fe^{2+} + Sn^{4+} + \cancel{2e^-}$$
$$SnCl_2 + 2FeCl_3 \longrightarrow 2\,FeCl_2 + SnCl_4$$ – balanced redox reaction

Whole numbers in front of the compounds are called coefficients (coefficients must be added to the chemical reaction in order to balance it = the left side of the reaction = the right side of the reaction).

Example 3 – Balancing redox reactions in acidic solutions, step by step:

e.g., $MnO_4^- + Fe^{2+} + H^+ \rightarrow Mn^{2+} + Fe^{3+} + H_2O$

a) identify a reactant and a product of each of the oxidation and reduction reaction:

$MnO_4^- \rightarrow Mn^{2+}$ reduction

$Fe^{2+} \rightarrow Fe^{3+}$ oxidation

b) balance atoms other than H, O

$$MnO_4^- \rightarrow Mn^{2+}$$
$$Fe^{2+} \rightarrow Fe^{3+}$$

c) balance oxygen by adding H_2O

$$MnO_4^- \rightarrow Mn^{2+} + 4H_2O$$
$$Fe^{2+} \rightarrow Fe^{3+}$$

d) balance hydrogen by adding H+

$$8H^+ + MnO_4^- \rightarrow Mn^{2+} + 4H_2O$$
$$Fe^{2+} \rightarrow Fe^{3+}$$

e) balance the charge by adding electrons

$$5e^- + 8H^+ + MnO_4^- \rightarrow Mn^{2+} + 4H_2O$$

$Fe^{2+} \rightarrow Fe^{3+} + e^-$ / * 5 multiply the second reaction by 5 because the number of electrons must be the same

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

f) cancel 5 electrons from both sides to obtain the final balanced chemical equation (chemical reaction)

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

Example 4 – Balancing redox reactions in basic solutions: **first follow the first steps: from a) to e) just like in acidic solutions** **than perform additional steps typical for basic solutions**: check the number of H^+ in the balanced chemical reaction and add the same number of OH^- ions to each side of a reaction, combine each pair of H^+ and OH^- ions to form H_2O than cancel any H_2O molecules that occur on both sides of a chemical reaction and get a fully balanced chemical equation (reaction).

e.g., $\mathbf{2}MnO_4^- + SO_3^{2-} + \mathbf{2}OH^- \rightarrow \mathbf{2}MnO_4^{2-} + SO_4^{2-} + H_2O$

Redox potential (reduction potential; reduction/oxidation potential) is a measure of the tendency of a chemical species to gain (acquire) electrons in order to be reduced. It is measured in volts [V] or in millivolts [mV]. The more positive redox potential the greater tendency to be reduced. In other words a more positive electrode potential will oxidize a more negative potential [4].

Redox potential (E): Nernst equation:

$$E = E^\circ + \frac{RT}{n\mathrm{F}} \cdot \ln c$$

where:

E	–	redox potential,
E^o	–	standard electrode potential,
R	–	gas constant (= 8.314 J/mol · K),
T	–	temperature [^{o}K],
n	–	number of electrons in the reaction (cation + $ne^- \rightleftarrows$ atom),
F	–	Faraday constant (96 500 C/mol),
c	–	molar concentration [mol/dm^3; mol/l].

Selected applications of redox reactions: electrochemical cells or batteries, corrosion, combustion, electrolysis, photosynthesis.

Additional tasks and questions to be performed by the student:

1. Assign an oxidation number to each element in the following ions:

   a) SO_4^{2-}, MnO_4^-, PO_4^{3-}, ClO_4^-, CO_3^{2-}, NO_2^-, Br^-;
   b) AsO_2^-, AsO_3^{3-}, AsO_4^{3-}, MnO_3^{2-}, $H_2PO_4^-$, Ca^{2+};
   c) ClO_3^-, HPO_4^{2-}, $Cr_2O_7^{2-}$, SO_3^{2-}, ClO^-, IO_3^-, Na^+.

2. Assign an oxidation number to each element in the following neutral compounds as well as free elements:

   a) H_2SO_4, H_2SO_3, H_3PO_4, H_2S, HCl, HClO, $HClO_3$, O_3;
   b) Na_2SO_4, K_2SO_3, P_2O_5, Na_2S, KCl, $HClO_4$, $NaClO_3$, F_2;
   c) NH_3, N_2O, NO, SO_2, SO_3, P_2O_3, NH_4Cl, Al;
   d) $NaAlO_2$, Al_2O_3, NaOH, H_2O, HIO_3, Na_2HPO_4, S;
   e) MnO_2, $KMnO_4$, K_2CrO_4, $K_2Cr_2O_7$, K_2MnO_4, Cr_2O_3, O_2;
   f) NaH, MgH_2, AlH_3, CH_4, NH_3, H_2S, H_2O_2, K.

3. Balance each of the following chemical equations:

   a) $S^{2-} + I_2 \longrightarrow S + I^-$
   b) $Na + Cl_2 \longrightarrow 2NaCl$
   c) $Br^- + SO_4^{2-} + H^+ \longrightarrow Br_2 + SO_3^{2-} + H_2O$
   d) $MnO_4^- + I^- + H^+ \longrightarrow Mn^{2+} + I_2 + H_2O$
   e) $HI + H_2SO_4 \longrightarrow I_2 + H_2S + H_2O$
   f) $MnO_4^- + SO_3^{2-} + OH^- \longrightarrow MnO_4^{2-} + SO_4^{2-} + H_2O$

2. INSTRUCTION 5 – LABORATORY EXERCISE 5

Materials and reagents (Experiments: 1 – 4):

Glass test tube set, micro spatula, polyethylene plastic pipettes, measuring cylinder, solutions of: potassium hexacyanoferrate (III) (0.1M $K_3[Fe(CN)_6]$), iron (II) sulfate (5% $FeSO_4$), hydrogen peroxide (3% H_2O_2), potassium permanganate (0,1M $KMnO_4$), potassium bromide (5% KBr), potassium iodide (5% KI), potassium chloride (5% KCl), sodium sulfide (2M Na_2S), iron (III) sulfate (0,1M $Fe_2(SO_4)_3$), iodine solution in potassium iodide (I_2 in KI), sulfuric acid (H_2SO_4 1 : 3), sodium hydroxide (1M NaOH), chloroform (trichloromethane) ($CHCl_3$), solid sodium sulfite (Na_2SO_3).

Experiment 1 – Determining of oxidizing and reducing agent based on products of selected oxidation-reduction reactions

Experimental procedure:

To each of three test tubes pour:

1. First test tube – pour 4 cm^3 of potassium permanganate (0.1M $KMnO_4$) and add just a little bit of sulfuric acid solution (H_2SO_4 1 : 3) in order to acidify the solution.
2. Second test tube – pour 4 cm^3 of iodine solution in potassium iodide (I_2 in KI).
3. Third test tube – pour 4 cm^3 of potassium iodide (5% KI) and add just a little bit of sulfuric acid solution (H_2SO_4 1 : 3) in order to acidify the solution.
   First test tube – divide the solution by pouring equal parts into two test tubes. One test tube treat as a control sample. Add a few drops of iron (II) sulfate (5% $FeSO_4$) solution into the second test tube.
   Second test tube – divide the solution by pouring equal parts into two test tubes. One test tube treat as a control sample. Add a few drops of sodium sulfide (2M Na_2S) solution into the second test tube.
   Third test tube – divide the solution by pouring equal parts into two test tubes. One test tube treat as a control sample. Add just one drop!!! Of hydrogen peroxide (3% H_2O_2) into the second test tube.

Data analysis (after the experiment):

1. Write balanced chemical equations for oxidation-reduction reactions (reactions from the experiment) – ionic form.
2. Identify the oxidizing and reducing agent as well as fill in the table below.

Table with the results of experiment 1

	Test tube 1	Test tube 2	Test tube 3
Reagent I	$KMnO_4$	I_2 w KI	KI
Reagent II	$FeSO_4$	Na_2S	H_2O_2
Nature of the solution (acidic/basic/neutral)			
Initial colour/ colour source			
Final colour/ colour source			
Oxidizing agent			
Reducing agent			

Experiment 2 – Oxidation-reduction reactions in acidic/basic/neutral solution

Experimental procedure:

Pour 4 cm^3 of potassium permanganate (0.1M $KMnO_4$) to each of four test tubes. Add 1cm^3 of sodium hydroxide (1M NaOH) into the first test tube, 1 cm^3 of distilled water into the second test tube and 1 – 3 drops of sulfuric acid solution (H_2SO_4 1 : 3) into the third test tube. The fourth test tube treat as a control sample. Add a pinch of solid sodium sulfite (Na_2SO_3) to each of three test tubes (first, second, third). Record the color of the solution in test tubes (after the reaction). Compare the color with the control sample.

Data analysis (after the experiment):

1. Write balanced chemical equations for oxidation-reduction reactions (reactions from the experiment) – ionic form.
2. Identify the oxidizing and reducing agent.

Experiment 3 – The oxidation-reduction potential

Experimental procedure:

Pour 4 cm^3 of potassium permanganate (0.1M $KMnO_4$) into the first test tube. Pour 4 cm^3 of iron (III) sulfate (0.1M $Fe_2(SO_4)_3$) into the second test tube. Add a few drops of sulfuric acid solution (H_2SO_4 1 : 3) to each of the two test tubes (first and second test tube). Divide the obtained solutions (test tube 1 and test tube 2) by pouring equal parts into three test tubes. Test tubes with potassium permanganate (0.1M $KMnO_4$) should be placed behind the test tubes with iron (III) sulfate (0.1M $Fe_2(SO_4)_3$).

Add into the first pair of test tubes solution of potassium iodide (5% KI), into the second pair of test tubes solution of potassium bromide (5% KBr) and into the third pair of test tubes potassium chloride (5% KCl).

Data analysis (after the experiment):

1. Write balanced chemical equations for oxidation-reduction reactions (reactions from the experiment) – ionic form.
2. Identify the oxidizing and reducing agent.
3. Fill in the table below: insert „+” where the reaction occurs and „–“ where it does not occur:

Reagents	KI	KBr	KCl
$KMnO_4$			
$Fe_2(SO_4)_3$			

$$E_{MnO_4^-/Mn^{2+}} = 1.52\ V \quad E_{Cl_2/2Cl^-} = 1{,}40\ V$$
$$E_{Br_2/2Br^-} = 1.08\ V \quad E_{Fe^{3+}/Fe^{2+}} = 0{,}75\ V$$
$$E_{I_2/2I^-} = 0.58\ V$$

Experiment 4 – The effect of the oxidizing agent on the oxidation-reduction reaction

Experimental procedure:

Pour 4 cm^3 of potassium iodide (5% KI) into the test tube, add a few drops of sulfuric acid solution (H_2SO_4 1 : 3), just one drop of hydrogen peroxide (3% H_2O_2) and 3 cm^3 of chloroform ($CHCl_3$). Shake it for a minute or two.

Data analysis (after the experiment):

1. Write balanced chemical equations for oxidation-reduction reaction (reaction from the experiment) – ionic form.
2. Identify the oxidizing and reducing agent.
3. What type of extraction is it?

3. Guidelines for writing the final laboratory report

1. First page of the report – The Laboratory Report Cover Sheet found on our website: https://www.am.szczecin.pl/en/facilities/institute-of-mathematics-physics-and-chemistry/department-of-chemistry/chemistry-lab-manuals/
2. Second page of the report – „The Theoretical Part” – on a maximum of one page including brief description of keywords.
3. Third page of the report – „The Experimental Part” – including all performed experiments with titles, raw data, reactions, calculations, tables, graphs, etc. It should be written in accordance with „Data analysis (after the experiment)”.
4. Additional task/tasks given by the academic teacher.
5. References.

4. In order to pass the laboratory exercise students must pass „the entry test” and submit the final laboratory report at the next laboratory meeting. The lab report must be accepted by the academic teacher.