



Physical and Chemical Methods of Analysis

Work program of the discipline (Syllabus)

Details of the discipline

Level of higher education	<i>first (bachelor's)</i>
Field of knowledge	<i>16 Chemical and Bioengineering</i>
Speciality	<i>161 Chemical Technologies and Engineering</i>
Educational program	<i>Industrial Ecology and Resource Efficient Clean Technologies</i>
Discipline status	<i>Optional</i>
Form of study	<i>full-time (day)/remote/mixed</i>
Year of preparation, semester	<i>3rd year, autumn semester</i>
Scope of discipline	<i>4 credits (120 hours)</i>
Semester control/ control measures	<i>Test</i>
Schedule of classes	<i>3 hours a week (1 hour of lectures and 2 hours of laboratory classes)</i>
Language of instruction	<i>Ukrainian</i>
Information about to the eminent course / teachers	Lecturer: https://eco-paper.kpi.ua/pro-kafedru/vykladachi/vizytyky/krisenko-tamara-volodimirivna-2.html Laboratory: https://eco-paper.kpi.ua/pro-kafedru/vykladachi/vizytyky/krisenko-tamara-volodimirivna-2.html
Course placement	https://do.ipk.kpi.ua/

The program of the discipline

1. Description of the discipline, its purpose, subject of study and learning outcomes

"Physical and Chemical Methods of Analysis" – the science of the principles and methods for determining the qualitative and quantitative composition of a substance using various devices.

The subject of study of the discipline "Physical and Chemical Methods of Analysis" is the study of the theoretical foundations of physical and physico-chemical methods, the improvement of existing and the development of new methods of analysis, their practical use.

The purpose of studying this discipline is to form in students a set of knowledge of the theoretical foundations of methods for identifying and quantifying the studied substances, a set of skills and abilities necessary to perform basic chemical operations and take measurements on devices.

In accordance with the goal, the study of the discipline "Physical and Chemical Methods of Analysis" requires the formation of students' competencies:

- to carry out qualitative and quantitative analysis of substances of inorganic and organic origin, using appropriate methods of general and inorganic, organic, analytical, physical and colloidal chemistry;
- to select and use appropriate equipment, tools and methods to solve complex problems of chemical engineering, control and management of technological processes of chemical production.

According to the requirements of the program of the discipline **“Physical and Chemical Methods of Analysis”**, students after mastering the discipline, students must demonstrate the following **learning outcomes**:

- to carry out qualitative and quantitative analysis of substances of inorganic and organic origin, using appropriate methods of general and inorganic, organic, analytical, physical and colloidal chemistry;
- choose and use appropriate equipment, tools and methods to solve complex problems of chemical engineering, control and control of technological processes of chemical production.

2. Pre-requisitions and post-requisitions of disciplines (place in the structural and logical scheme of education according to the relevant educational program)

The study of the discipline **“Physical and Chemical Methods of Analysis”** is based on the principles of integration of knowledge gained by students during the first and second year in the study of disciplines "General and Inorganic Chemistry", "Organic Chemistry", "Physics", "Higher Mathematics", "Analytical Chemistry". Discipline **“Physical and Chemical Methods of Analysis”** provides diploma design.

3. The content of the discipline

Topic 1. Photometric method of analysis.

Topic 2. IR spectroscopy.

Topic 3. Fluorescent analysis.

Topic 4. Potentiometric method of analysis.

Topic 5. Polarographic method of analysis.

Topic 6. Amperometric titration.

Topic 7. Chromatographic methods of analysis.

4. Learning materials and resources

Basic literature

1. Slobodnyuk R.E. *Course of analytical chemistry*. – Kherson: Oldie plus, 2020. – 256 p.
2. Butchenko L.I., Khokhotva O.P., Tereshchenko O.M., Glushko O.V., Krysenko T.V. *Analytical chemistry. Methods of qualitative chemical analysis (theoretical aspects and laboratory workshop): Textbook*. – Kyiv: Igor Sikorsky Kyiv Polytechnic Institute, Publishing house "Polytechnic", 2017. – 152 p.
3. Slobodnyuk R.E., Goralchuk A.B. *Analytical chemistry and analysis of food products*. – K.: Publishing House "Condor", 2018. – 336 p.

Supporting literature

4. Fedushchak N.K., Bidnychenko Yu.I., Kramarenko S.Yu., Kalibabchuk V.O. *Analytical Chemistry*. – Vinnytsia: Nova Kniga, 2012. – 640 p.
5. Shevryakov M.V., Povstyanyi M.V., Yakovenko B.V., Popovych T.A. *Analytical chemistry. Theoretical foundations of qualitative and quantitative analysis*. – Kherson: Ailant, 2011. – 404 p.
6. Bolotov V.V., Svechnikova O.M., Golik M.Yu. and others. *Analytical chemistry*. – Vinnytsia: New Book, 2011. – 424 p.
7. Chebotaryov O. M. *Analytical chemistry. Quantitative analysis: workshop for students of the faculty of chemistry and pharmacy / O. M. Chebotaryov, S. V. Toporov, O. M. Guzenko, R. E. Khoma, D. V. Snihur*. – Odessa: Odessa National University named after I. I. Mechnikov, 2019. – 80 p.
8. Segeda A.S. *Analytical Chemistry. Quantitative analysis*. – K.: Phytosociocenter, 2006. – 544 p.
9. Studenyak Ya.I., Voronych O.G., Sukharev O.Yu., Fershal M.V., Basel Y.R. *Workshop on Analytical Chemistry. Instrumental methods of analysis*. - Uzhgorod, 2014.- 129 p.
10. Butchenko L.I., Kutsyi V.G. *Instrumental methods of analysis / Methodical instructions for laboratory work on the course "Analytical Chemistry", for students of all forms of education*. – K.: NTUU "KPI", 2003. – 20 p.
11. Butchenko L.I., Khokhotva O.P., Tereshchenko O.M. *Methods of analytical chemistry in environmental research. Part II. Physical and physico-chemical (instrumental) methods of analysis / Methodical instructions for studying the discipline for students of the direction of training 6.040106 "Ecology, environmental protection and balanced environmental management"* – K: NTUU "KPI", 2011. – 68 p.
12. Butchenko L.I., Khokhotva O.P., Tereshchenko O.M. *Methods of analytical chemistry in environmental research. Ch.III. Physical, physico-chemical and biochemical methods of analysis / Methodical instructions for studying the discipline for students of the direction of training 6.040106 "Ecology, environmental protection and balanced environmental management"* – K: NTUU "KPI", 2011. –56 p.
13. Butchenko L.I., Khokhotva O.P., Tereshchenko O.M., Glushko O.V. *Methodical instructions for solving typical problems from the course "Instrumental methods of analysis" for students of the direction of training 6.040106 "Ecology, environmental protection and balanced use of nature"* – K: NTUU "KPI", 2012. – 56 p.
14. Dorokhova E.M., Prokhorova G.V. *Tasks and questions on analytical chemistry*.— K.: Kyiv. univer., 2001.—282 p.
15. Lysenko O.M., Nabyvanets B.Y. *Introduction to chromatographic methods*. – K.: Corvin-press, 2005. – 187 p.

Information resources on the Internet

16. Union of Chemists of Ukraine <http://www.chemunion.org.ua/uk/>

Educational content

5. Methods of mastering the discipline (educational component)

Lectures

Lectures are aimed at:

- providing modern, holistic, interdependent knowledge of the discipline "**Physical and Chemical Methods of Analysis**", the level of which is determined by the target setting for each specific topic;
- ensuring in the process of the lecture the creative work of students together with the teacher;
- education of students' professional and business qualities and the development of their independent creative thinking;
- formation of students' necessary interest and providing direction for independent work;
- determination at the modern level of development of science and technology in the field of chemical technology, forecasting their development for the coming years;
- reflection of the methodological processing of the material (selection of the main thoughts and provisions, underlining the conclusions, repeating them in different formulations);
- acquisition of clarity, combination, if possible, with the demonstration of audiovisual materials, layouts, models and samples;
- teaching in a clear and clear language, explaining all newly introduced terms and concepts;
- accessibility for perception by this audience.

N ^o	<i>Title of the lecture topic and list of main questions (list of didactic means, references to literature and tasks for IWS)</i>	<i>Hours</i>
1	<p>Topic 1. Photometric method of analysis. <i>Features and classification of physicochemical methods of analysis. Photometric method of analysis. Booger-Lambert-Baer law and conditions for its implementation. Methods for determining the concentration of colored compounds.</i> Literature: [6] pp. 368-376. Tasks on the IWS. <i>Advantages of physicochemical methods in comparison with chemical. Refractometry. Polarimetry.</i></p>	2
2	<p>Topic 2. IR spectroscopy. Theoretical bases of IR spectroscopy, preparation of samples for analysis, quality and quantitative analysis, devices. Literature: [3] pp. 208-213; [4] pp. 466-474. Tasks on the IWS. <i>Types of oscillations in IR spectroscopy. Detectors in IR spectroscopy, their types. Use of IR spectroscopy.</i></p>	1
3	<p>Topic 3. Fluorescent analysis. General characteristics of fluorescent analysis. Methods of excitation of luminescence, the main characteristics and rules of luminescence. Qualitative and quantitative fluorescent analysis. General <i>positions of atomic spectroscopy, classification.</i> Literature: [6] pp. 357-366, 387-389.</p>	4

	Tasks on the IWS. Fluorescent poisons, the use of fluorescent analysis. Bioluminescence. Advantages of fluorescent methods of analysis.	
4	Topic 4. Potentiometric method of analysis. <i>Potentiometric analysis: absolute potentiometry and potentiometric titration, electrodes in the potentiometric method of analysis.</i> Literature: [1] pp. 218-222; [6] pp.319-327. Tasks on the IWS. Standard electrodes their structure. Advantages of potentiometric titration over normal. Use of potentiometric titration.	1
5	Topic 5. Polarographic method of analysis. <i>Polarographic curve and its characteristics. Qualitative and quantitative analysis.</i> Topic 6. Amperometric titration. <i>Fromcities of amperometric titration, titration curves.</i> Literature: [4] pp. 526-532. Tasks on the IWS. Migration current, causes, methods of elimination. Characteristics of the capillary electrode. Polarogram distortion: causes and remedies. Application of polarography and amperometric titration.	2
6	Topic 7. Chromatographic methods of analysis. <i>Theoretical bases and classification of chromatographic methods of analysis. Chromatographic parameters. Theory of chromatographic separation. Adsorption chromatography, requirements for sorbents. Gas-solid-phase and gas-liquid chromatography, schematic diagram of the installation, qualitative and quantitative analysis.</i> Literature: [1] pp. 211-221; [6] pp. 395-415. Tasks on the IWS. Sensors: structure, types, use.	6
7	<i>Thin-layer and paper chromatography, qualitative and quantitative analysis. Advantages of thin-layer chromatography over paper. Methods of development of chromatograms. Molecular sieve chromatography.</i> Literature: [4] pp. 551-564. Tasks on the IWS. Methods of solvent supply in thin-layer chromatography. Selection of sorbent and solvent. Affine chromatography.	2
	Just	18

Laboratory classes

The main objectives of the laboratory cycle are:

- ◆ *help students systematize, consolidate and deepen theoretical knowledge of qualitative and quantitative analysis of physicochemical methods;*
- ◆ *teach students the basic skills of working with devices and contribute to the mastery of skills and abilities to perform analysis with their help;*
- ◆ *teach students techniques for solving practical problems, promote mastery of skills and abilities to perform calculations and other types of tasks.*

N^o	<i>The name of the topic of the laboratory lesson and the list of main questions (list of didactic support, references to literature)</i>	<i>Hours</i>
1	Photometric determination of Ti ⁴⁺ . Photometric determination of Cu ²⁺ . Literature: [7] pp. 34-37.	4
2	Photometric determination of Fe ³⁺ . Literature: [7] pp. 37-41.	4
3	Extraction-photometric determination of copper. Literature: [7] pp. 27-29.	4
4	Separation of inorganic ions by extraction. Literature: [7] pp. 25-26. Test paper number 1.	4
5	Potentiometric determination of H ₂ CrO ₄ . Amperometric definition of Hg (II). Literature: [9] pp. 37-39. Test paper number 2.	4
6	Pseparation and detection of Fe ³⁺ , Co ²⁺ and Cu ²⁺ by chromatography. Literature: [10] pp. 14-15. Test paper number 3.	5
7	Determination of the concentration of potassium nitrate by the method of column chromatography. Literature: [9] pp. 97-99.	4
8	Distribution chromatography on paper. Literature: [10] pp. 18-19. Test paper number 4.	5
9	Test	2
	Just	36

6. Independent work of the student

Independent work takes 55% of the time to study the credit module. The main task of independent work of students is to master knowledge in areas that are not included in the list of lecture questions through personal search for information, the formation of an active interest in a creative approach in educational work. In the process of independent work within the framework of the educational component, the student must learn to analyze the information received and use it to solve the tasks.

N^o	<i>The name of the topic submitted for independent study</i>	<i>Number of hours of IWS</i>
Topic 1. Photometric method of analysis.		
1	<i>The advantages of physicochemical methods in comparison with chemical. Refractometry. Polarimetry. Literature: [6] pp.350-361.</i>	3

Topic 2. IR spectroscopy.		
2	<i>Types of oscillations in IR spectroscopy. Detectors in IR spectroscopy, their types. Use of IR spectroscopy. Literature: [4] pp. 465-474.</i>	7
Topic 3. Fluorescent analysis.		
3	<i>Fluorescent poisons, the use of fluorescent analysis. Bioluminescence. Advantages of fluorescent methods of analysis. Literature: [11] pp. 17-23.</i>	8
Topic 4. Potentiometric method of analysis.		
4	<i>Standard electrodes their structure. Advantages of potentiometric titration over normal. Use of potentiometric titration. Literature: [6] pp. 309-327.</i>	10
Topic 5. Polarographic method of analysis. Topic 6. Amperometric titration.		
5	<i>Migration current, causes, methods of elimination. Characteristics of the capillary electrode. Polarogram distortion: causes and remedies. Application of polarography and amperometric titration. Literature: [6] pp. 327-344.</i>	10
Topic 7. Chromatographic methods of analysis.		
6	<i>Sensors: structure, types, use. Literature: [12] pp. 39-54.</i>	8
7	<i>Methods of solvent supply in thin-layer chromatography. Selection of sorbent and solvent. Affine chromatography. Literature: [6] pp. 400-416; [11] pp. 61-65.</i>	6
8	<i>Writing a settlement work</i>	8
9	<i>Preparation for the test</i>	6
	<i>Total hours</i>	66

Policy and control

7. Policy of the discipline (educational component)

Rules for attending classes and behavior in the classroom

Attendance is a mandatory component of the assessment. Students are obliged to take an active part in the educational process, not to be late for classes and not to miss them without a good reason, not to interfere with the teacher to conduct classes, not to be distracted by actions that are not related to the educational process.

Rules for assigning incentive and penalty points

- Incentive points can be awarded by the teacher solely for performing creative work in the discipline or additional completion of online specialized courses with the receipt of the appropriate certificate:
<https://www.coursera.org/learn/spectroscopy>*

But their amount cannot exceed 10% of the rating scale.

- penalty points are not provided.*

Deadlines and Rescheduling Policy

In case of debts in the discipline or any force majeure circumstances, students should contact the teacher through the available (provided by the teacher) communication channels to solve problematic issues and agree on an algorithm of actions for working out.

Academic Integrity Policy

Plagiarism and other forms of dishonest work are unacceptable. Plagiarism includes the lack of links when using printed and electronic materials, quotes, opinions of other authors. Unacceptable hints and write-offs when writing tests, conducting classes; passing the exam for another student; copying materials protected by the copyright system without the permission of the author of the work.

The policy and principles of academic integrity are defined in Chapter 3 of the Code of Honor of the National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute <https://kpi.ua/code>".

Academic Conduct and Ethics Policy

Students should be tolerant, respect the opinions of others, formulate objections in the correct form, constructively maintain feedback in the classroom.

The norms of ethical behavior of students and employees are defined in Chapter 2 of the Code of Honor of the National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute <https://kpi.ua/code>".

8. Types of control and rating system for evaluating learning outcomes (RS)

Distribution of study time by types of classes and tasks in the discipline in accordance with the working curriculum:

Semester	Educational time		Distribution of study hours				Control measures		
	Loans	Acad. H.	Lecture	Practical	Lab. Work	IWS	MCT	SW	Semester control
5	4	120	18	-	36	66	1	1	Test

The student's rating on the discipline consists of points that he receives for:

- performance of laboratory works (10 works);
- writing a modular test (1 modular test is divided into 4 tests);
- writing a settlement work.

Semester control is test.

The system of rating (weight) points and evaluation criteria

1. Performance of laboratory works:

The necessary conditions **for admitting** a student to laboratory work are:

- availability of a protocol for appropriate laboratory work;
- a positive answer to the question of incoming express quality control of the theoretical preparation of the student for laboratory work (express control is carried out in the form of an

oral survey conducted at the beginning of the lesson). In case of non-compliance with these conditions, the student is not allowed to perform laboratory work.

Weight score – 3. The maximum number of points for the performance of all laboratory work is equal to:

$$3 \text{ points} \cdot 10 = 30 \text{ points.}$$

Criteria for evaluating the performance of laboratory work

Completeness and signs of completing the task	Points
The work was performed in full (relative error in determining the $\delta \leq 3\%$, appropriate design (protocol and calculations are presented on time)).	3
Minor shortcomings in execution (relative error of determination of $3 < \delta \leq 5\%$, there are certain shortcomings in the design (the protocol is presented on time and calculations are some late)).	2
Gross shortcomings in execution (work performed with gross errors, relative error of determination of $5 < \delta \leq 8\%$, inappropriate design (protocol and calculations are not presented on time)).	1
Failure to complete the task (Work not completed (relative error of the experiment $\delta > 8\%$)).	0

2. Writing a modular test:

1 modular test is divided into 4 tests, 15 points each.

The maximum number of points for writing all tests is equal to:

$$15 \text{ points} \cdot 4 = 60 \text{ points.}$$

Evaluation criteria	Points
"excellent", complete answer (at least 95% of the necessary information)	15-14
"good", incomplete disclosure of one of the issues (at least 75% of the required information)	13-11
"satisfactory", incomplete answer (at least 65% of the necessary information)	10-9
unsatisfactory work (does not meet the requirements for "satisfactory").	8-0

3. Writing a calculation work:

The maximum number of points for writing a roaring work is 10 points.

Points	Evaluation criteria
10	"excellent" - the work is done carefully, in full, the answers are justified, examples and equations of chemical reactions are given, which are discussed in the question
9-8	"good" - theoretical questions and calculations contain minor flaws (or the work is completed with some delay from the deadline)
7-6	"satisfactory" - there are no answers to 1-2 questions or theoretical questions

	<i>and calculations are presented with certain errors (or the work is completed with a significant delay from the deadline)</i>
5-0	<i>"unsatisfactory" - the task is not completed or there are gross errors, the work is not credited</i>

According to the results of educational work for the first 7 weeks, the "ideal student" must perform all laboratory work (at the time of certification) and score 27 points. **At the first certification** (8th week), the student receives "enrolled" if he has completed all the laboratory work (at the time of certification) and his current rating is at least 15 points.

According to the results of educational work for 13 weeks of study, the "ideal student" must complete all the laboratory work (at the time of certification) and score 72 points. **At the second certification** (14th week), the student receives "credited" if he has completed all the laboratory work (at the time of certification), 3 tests are written and his current rating is at least 40 points.

Thus, the sum of rating points is:

$$R_s = 30 + 60 + 10 = 100.$$

The condition for admission to the test is the enrollment of all laboratory works, all tests, and calculation work.

To receive credit from the credit module "**automatic**" you need to have a rating of at least 60 points.

Students who score less than 0,6 R_s during the semester, as well as those who want to increase the overall rating, perform a test paper. At the same time, all points received by them during the semester are canceled, except for points for settlement work.

4. The test paper is estimated from 90 points. The control task of this work consists of four tasks (questions): two theoretical, one task (question) from laboratory work and a problem. Each theoretical task (question) and task (question) from the laboratory course is estimated at 20 points, and the task is estimated at 30 points.

Criteria for evaluating theoretical questions and questions from laboratory works

Mark	Completeness of the answer
20-19	<i>"excellent", complete answer (at least 95% of the necessary information)</i>
18-17	<i>"very good", complete answer with minor inaccuracies (at least 85% of the necessary information)</i>
16 – 15	<i>"good", a sufficiently complete answer (at least 75% of the necessary information)</i>
14-13	<i>"satisfactory", incomplete answer (at least 65% of the necessary information)</i>
12-11	<i>"enough", incomplete answer with certain errors (at least 60% of the required information)</i>
10-0	<i>unsatisfactory work (does not meet the requirements for "enough").</i>

Criteria for evaluating the solution of the problem

<i>Mark</i>	<i>Completeness of the answer</i>
<i>30-29</i>	<i>"excellent", complete error-free solution of the problem (at least 95% of the necessary information)</i>
<i>28-26</i>	<i>"very good", complete answer with minor inaccuracies (at least 85% of the necessary information)</i>
<i>25 – 23</i>	<i>"good", complete solution of the problem with insignificant inaccuracies (at least 75% of the necessary information)</i>
<i>22-20</i>	<i>"satisfactory", ncomplete answer (at least 65% of the necessary information)</i>
<i>19-18</i>	<i>"enough", incomplete answer with certain shortcomings (at least 60% of the necessary information)</i>
<i>17-0</i>	<i>unsatisfactory work (does not meet the requirements for "enough").</i>

To obtain a test score, the sum of all received rating points R_s is translated according to the table:

Points: automaton (laboratory classes + MCT + SW) or test: test work	Score
<i>100...95</i>	<i>Perfectly</i>
<i>94...85</i>	<i>very good</i>
<i>84...75</i>	<i>well</i>
<i>74...65</i>	<i>Satisfactory</i>
<i>64...60</i>	<i>enough</i>
<i>R<60 points</i>	<i>Disappointing</i>
<i>Conditions of admission have not been met</i>	<i>not allowed</i>

9. Additional information on the discipline (educational component)

***An approximate list of questions for credit in the discipline
"Physical and Chemical Methods of Analysis"***

- 1. Reveal the content of photolorimetric analysis.*
- 2. Explain the chemistry of the occurrence of color.*
- 3. Derive the Booger-Lambert-Baer law.*
- 4. Present the structure of devices for photometric analysis.*
- 5. Present a graphic representation of the Booger-Lambert-Baer law. Formulate the reasons that cause deviations from the linearity of the graph.*
- 6. Present the content of the main methods of photometry and the calculated formulas of these methods.*
- 7. Present absorption spectra.*
- 8. Cite the Planck equation.*
- 9. Formulate the main provisions of atomic emission spectral analysis.*

10. *Reveal the essence of the method of infrared spectroscopy.*
11. *Present the blocks of the infrared spectroscopy device and their characteristics.*
12. *Explain the features of sample preparation for analysis in infrared spectroscopy.*
13. *Reveal the content of fluorescent analysis.*
14. *Present the main characteristics and rules of luminescence.*
15. *Reveal the theoretical foundations of potentiometry.*
16. *Present the galvanic element.*
17. *Cite the Nernst equation.*
18. *Explain the essence of indicator electrodes. Present their classification, give examples and describe the structure.*
19. *Reveal the essence of standard electrodes, give examples and describe their structure.*
20. *Explain the content of potentiometric titration, its types.*
21. *Reveal the content of the method of classical polarography.*
22. *Present a polarographic installation scheme.*
23. *Explain the qualitative and quantitative polarographic analysis, give the calculated formulas.*
24. *Reveal the content of amperometric titration.*
25. *To present the types of amperometric titration curves, to give examples.*
26. *Reveal the content of the chromatographic method of analysis.*
27. *Present chromatographic parameters.*
28. *Explain the theories of chromatography.*
29. *Reveal the content of liquid-adsorption and gas-adsorption chromatography.*
30. *Reveal the content of gas-liquid chromatography.*
31. *Reveal the content of thin-layer chromatography.*
32. *Reveal the content of molecular sieve chromatography.*
33. *Reveal the content of paper chromatography.*

The work program of the discipline (syllabus):

Compiled by Ph.D., assoc. prof. Krysenko T.V.

Approved by the department E and PPT (protocol N^o 14 from 08.06.2022)

Approved by the Methodical Commission

Faculty of Chemical Engineering (protocol N^o 10 from 24.06.2022)