

The National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute"



Ecology and Plant Polymers Technology

# Instrumental Methods of Chemical Analysis Work program of the discipline (Syllabus)

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

The program of the discipline

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

"Instrumental Methods of Chemical Analysis" – the science of the principles and methods of determining the qualitative and quantitative composition of a substance through the use of various devices and chemical equipment.

**The subject of study of the discipline "Instrumental Methods of Chemical 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 "Instrumental Methods of Chemical Analysis" requires the formation of students' competencies:

• the ability to use methods of observation, description, identification, classification of objects of chemical technology and industrial products;

- the ability to choose and use appropriate equipment, tools and methods for control and management of technological processes of chemical production;
- the ability to apply up-to-date experimental methods of working with technological objects in industrial and laboratory conditions.

According to the requirements of the program of the discipline "Instrumental Methods of Chemical 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;
- 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.

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

Microsoft\* Translator×

Ориглал Дисципліна «Інструментальні методи хімічного аналізу» забезпечує дипломне проєктування.

#### 3. The content of the discipline

Section 1. Introduction to instrumental methods of analysis. Spectroscopic methods of analysis.

Section 2. Electrochemical methods of analysis. Section 3. Chromatographic methods of analysis. Section 4. Kinetic methods of analysis.

#### 4. Learning materials and resources

#### **Basic literature**

- 1. 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, "Polytechnic", 2017. 152 p.
- 2. Slobodnyuk R.E., Goralchuk A.B. Analytical chemistry and analysis of food products. K.: Publishing House "Condor", 2018. – 336 p.
- 3. Slobodnyuk R.E. Course of analytical chemistry. Kherson: Oldie Plus, 2020. 256 p.

#### Supporting literature

4. 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

р.

- 5. Bolotov V.V., Svechnikova O.M., Golik M.Yu. and others. Analytical chemistry. Vinnytsia: New Book, 2011. – 424 p.
- 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.
- 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.
- Toporov S.V. Physico-chemical methods of research of substances and materials: method. instructions for students of the faculty of chemistry and pharmacy / S.V. Toporov, R.E. Khoma, O.M. Chebotaryov. – Odessa: Odessa National University named after I.I. Mechnikov, 2021. – 74 p.
- 9. Moroz A.S. Medical Chemistry / Moroz A.S., Lutsevych D.D., Yavorska L.P. Vinnytsia: New Book, 2008. 776 p.
- 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.

## Information resources on the Internet

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

5. Methods of mastering the discipline (educational component)

## Lectures

Lectures are aimed at:

- providing modern, holistic, interdependent knowledge of the discipline "Instrumental Methods of Chemical 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.

Nº	Title of the lecture topic and list of main questions (list of didactic means, references to literature and tasks for IWS)	Hours
1	Section 1. Introduction to instrumental methods of analysis. Spectroscopic methods of analysis. Tothe lassification of instrumental measurements of analysis. Use and importance of instrumental methods for the development of science, technology, control of production and economics. Content and classification of spectral methods of analysis. Electromagnetic spectrum. Literature: [2] pp. 197-198; [3] pp. 206-207. Tasks on the IWS. Comparison of the possibilities of chemical and instrumental methods of analysis.	
2	<ul> <li>Molecular absorption spectroscopy. The nature of the absorption of light by matter. Chemistry of the occurrence of color. Methods for obtaining colored compounds. Basic law of photometry, causes of deviation.</li> <li>Literature: [2] pp. 205-213.</li> <li>Tasks on the IWS. Nefelometric and turbidimetric analysis methods. Use of fluorescent analysis methods. Advantages compared to molecular absorption photometry methods.</li> </ul>	3

3	General positions of atomic spectroscopy. Methods of atomization of matter. Atomic-	4
	emission spectral analysis. Atomic absorption spectral analysis: basics of the method,	
	instruments, quantitative measurements. X-ray methods of analysis: basics of the	
	method, X-ray spectra. X-ray spectral and rentgenofluorescent analysis, devices and	
	their application in quality and quantitative analysis.	
	Literature: [3] pp. 214-223; [4] pp. 264-270.	
	Tasks on the IWS. Quenching of luminescence. Luminescence of crystal phosphors.	
	Using methods of atomic emission and atomic absorption spectroscopy.	
4	Section 2. Electrochemical methods of analysis.	1
,	Voltamperometric analysis: qualitative and quantitative analysis, instruments.	
	Literature: [3] pp. 228-233; [5] pp. 329-331.	
	Tasks on the IWS. Conductometry: direct conductometry and conductometric	
	titration. Electrogravimetric, coulometric analysis. Faraday's Law. Types of	
	coulometers. Coulometric titration. Methods for generating titrant.	
5	Section 3. Chromatographic methods of analysis.	3
	Theoretical bases and classification of chromatographic methods of analysis.	
	Chromatographic parameters. Theories of chromatographic separation. Gas-solid-	
	phase and gas-liquid chromatography: general characteristics of the method,	
	schematic diagram of the installation, qualitative and quantitative analysis.	
	Literature: [2] pp. 222-231; [5] pp. 395-401.	
	Tasks on the IWS. Areas of use of different types of chromatography. Types of	
	detectors in gas chromatography. Chromato-mass spectrometry.	
6	Ion exchange chromatography: general characteristics of the method, classification	4
	of ionites. Planar chromatography: qualitative and quantitative analysis, methods of	
	development of chromatograms. Hemiochromatography.	
	Literature: [1] pp. 41-46; [3] pp. 233-247; [5] pp.402-405.	
	<b>Tasks on the IWS.</b> Factors that affect ionic metabolism. Regeneration of ionites. Use	
	of ion exchange chromatography.	1
7	Section 4. Kinetic methods of analysis.	1
	General characteristics of the method. Reaction speed. Catalytic and non-catalytic	
	reactions. Indicator substance and indicator reaction. Methods for determining the	
	concentrations of indicator substances.	
	Literature: [9] pp. 420-454.	
	<b>Tasks on the IWS.</b> Catalytic reactions and their advantages in relation to non-	
	catalytic. Practical application of kinetic methods of analysis. Just	18
	<i>Just</i>	10

# Laboratory classes

The main objectives of the laboratory cycle are:

• help students systematize, consolidate and deepen theoretical knowledge of qualitative and quantitative analysis of instrumental 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 <sup>0</sup>	The name of the topic of the laboratory lesson and the list of main questions (list of didactic support, references to literature)	Hours
1	Determination of titanium (IV) in the form of a peroxide complex. Definition of	4
	copper (II) in the form of complex ammonia	
	Literature: [7] pp. 4-6.	
2	Determination of iron (III) in aluminum in the form of a monosalicylate complex.	4
	Literature: [7] pp. 3-4.	
3	Analysis of the mixture of cobalt and nickel and their quantitative determination.	4
	Literature: [6] pp. 45-47.	
4	Determination of Cu <sup>2+</sup> micronumbers .	4
	Literature: [7] pp. 6-7.	
	Test paper number 1.	
5	Potentiometric determination of chromic acid.	4
	Potentiometric determination of ferrum (III).	
	Literature: [7] pp. 10-12.	
6	Determination of KNO <sub>3</sub> by the method of ion exchange.	4
	Literature: [7] pp. 15-17.	
7	Chromatographic separation and determination of cadmium and zinc.	6
	Literature: [6] pp. 23-25.	
	Test paper number 2.	
8	Determination of molybdenum (VI) by fixed time method.	4
	Literature: [8] pp. 35-37.	
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 <sup>0</sup>	The name of the topic submitted for independent study	Number of hours of IWS		
Sect	Section 1. Introduction to instrumental methods of analysis. Spectroscopic methods of analysis.			

1	Comparison of the possibilities of chemical and instrumental methods of analysis.	3
	Literature: [2] pp.197-198 ; [5] pp.309-310.	
2	<i>Nef</i> elometric and turbidimetric analysis methods. Use of fluorescent analysis methods. Advantages compared to molecular absorption photometry methods. <i>Literature:</i> [5] pp. 361-368.	11
2		
3	Quenching luminescence. Luminescence of crystal phosphors. Use of atomic emission and atomic absorption spectroscopy methods. Literature: [4] pp. 254-258.	9
	Section 2. Electrochemical methods of analysis.	
4	Conductometry: direct conductometry and conductometric titration.	
	Electrogravimetric, coulometric analysis. Faraday's law. Types of coulometers.	10
	Coulometric titration. Methods for generating titrant.	10
	Literature: [3] pp. 223-228; [4] pp. 277-281.	
	Section 3. Chromatographic methods of analysis.	
5	Areas of use of different types of chromatography. Types of detectors in gas chromatography. Chromato-mass spectrometry. Literature: [4] pp. 575-597.	
	Factors that affect ionic exchange. Regeneration of ionites. Use of ion exchange chromatography.	11
	Literature: [2] pp. 222-230; [4] pp. 336-345.	
6	Section 4. Kinetic methods of analysis. Catalytic reactions and their benefits in relation to non-catalytic ones. Practical	
0	application of kinetic methods of analysis.	8
	Literature: [9] pp. 420-454.	0
7	Writing a settlement work	8
8	Preparation for the test	6
	Total hours	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 <u>https://kpi.ua/code</u>".

## 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 <u>https://kpi.ua/code</u>".

#### 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:

	Educational time		Distribution of study hours				Control measures		
Semester	Credits	Acad. H.	Lecture	Practical	Lab. Work	IWS	МСТ	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 2 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:

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

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

## 2. Writing a modular test:

1 modular test is divided into 2 tests, 30 points each.

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

## 30 points • 2 = 60 points.

Evaluation criteria	Points
"excellent", complete answer (at least 95% of the necessary	30-28
information)	
"good", incomplete disclosure of one of the issues (at least	27-22
75% of the required information)	
"satisfactory", incomplete answer (at least 65% of the	21-18
necessary information)	
unsatisfactory work (does not meet the requirements for	17-0
"satisfactory").	

## 3. Writing a calculation work:

## The maximum number of points for writing a settlement 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

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

According to the results of educational work for the first 7 weeks, the "ideal studentnt" 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.

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

Criteria for evaluating theoretical questions and questions from laboratory works

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

#### Criteria for evaluating the solution of the problem

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
10095	Perfectly
9485	very good
8475	well
7465	Satisfactory
6460	enough
R<60 points	Disappointing
Conditions of admission have not been met	not allowed

#### 9. Additional information on the discipline (educational component)

## An approximate list of questions for credit in the discipline "Instrumental methods of chemical analysis"

- 1. Present the advantages of instrumental methods in comparison with chemical ones.
- 2. Present a classification of spectral methods of analysis.
- 3. Explain the nature of the absorption of light by matter and the chemistry of the occurrence of color.
- 4. Reveal the meaning of the concept of electromagnetic spectrum.
- 5. To reveal the meaning of the concept of chromophore, auxochromes, bathochromic and gypsochromic displacements.
- 6. Give the conditions for using the basic law of photometry and the reasons for the deviation.
- **7.** Present methods for determining the concentration of colored compounds.

- 8. To reveal the physical meaning of the concept of the molar coefficient of light absorption.
- 9. Formulate the II law of light absorption (the law of additiveness of optical densities).
- 10. Formulate the main provisions of atomic emission spectral analysis.
- 11. To present the main nodes of spectral devices and their characteristics.
- 12. Reveal the content of atomic absorption spectral analysis.
- 13. To present the main nodes of instruments for atomic absorption spectral analysis.
- 14. To present a comparative description of the methods of atomic emission and atomic absorption spectroscopy.
- 15. Reveal the content of X-ray spectral analysis.
- 16. Present the main units of devices for X-ray spectral analysis and their characteristics.
- 17. Explain the qualitative and quantitative X-ray spectral analysis.
- 18. Reveal the essence of X-ray fluorescent analysis.
- 19. Reveal the content of voltamperometric analysis.
- 20. Explain the qualitative and quantitative voltamperometric analysis, give the calculated formulas.
- 21. Reveal the content of the chromatographic method of analysis.
- 22. Present a chromatographic peak.
- 23. Present qualitative and quantitative chromatographic analysis.
- 24. Reveal the content of ion exchange chromatography.
- 25. Present the classification of ion exchange sorbents, give examples.
- 26. To reveal the content of gas-solid-phase chromatography, to present the schematic diagram of the installation .
- 27. To reveal the content of gas-liquid chromatography, to present the schematic diagram of the installation.
- 28. Present planar chromatography, its qualitative and quantitative analysis.
- 29. Reveal the content of chemiochromatography.
- *30. Present a general characteristic of kinetic methods of analysis.*
- 31. To reveal the concepts of catalytic and non-catalytic reactions, indicator substance and indicator reaction.

#### The work program of the discipline (syllabus):

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

**Approved** by the department <u>E and PPT</u> (protocol  $N^0$  <u>14</u> from <u>18.05.2023</u>)

Approved by the Methodical Commission

Faculty of Chemical Engineering (protocol N<sup>0</sup> <u>10</u> from <u>26.05.2023</u>)

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11. Представити основні вузли спектральних приладів та їх характеристики.