

MINISTRY OF HEALTH OF THE REPUBLIC OF BELARUS
Educational Institution
BELARUSIAN STATE MEDICAL UNIVERSITY

Контрольный
экземпляр

APPROVED

by Rector of the Educational
Institution «Belarusian State
Medical University»



S.P. Rubnikovich

Reg. # UD-091-066/2425 /edu.

ANALYTICAL CHEMISTRY

**Curriculum of the educational institution
in the academic discipline for the specialty**

7-07-0912-01 «Pharmacy»

The curriculum is based on the educational program «Analytical Chemistry», approved 26.06.2024 registration # УД-091-066/2425/уч., on the education plan in the specialty 7-07-0912-01 «Pharmacy» approved 15.05.2024, registration # 7-07-0912-01/2425/mf.

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RECOMMENDED FOR APPROVAL:

The Pharmaceutical Chemistry Department of the educational institution «Belarusian State Medical University»
(protocol # 11 of 20.05.2024);

Scientific and Methodological Council of the educational institution «Belarusian State Medical University»
(protocol # 18 of 26.06.2024)

EXPLANATORY NOTE

«Analytical Chemistry» is an academic discipline of the Chemical Module containing systematized scientific knowledge about theoretical foundations, principles, methods and means of obtaining experimental information on the chemical composition and structure of a substance for identification and quantitative determination of pharmaceutical substances, medicines and other objects.

The aim of the academic discipline «Analytical Chemistry» is formation of basic professional competencies to gain a high level of knowledge, skills, and abilities to carry out identification and quantification of medicines and other objects.

The objectives of educational discipline «Analytical Chemistry» consists of developing students' scientific knowledge about theoretical foundations of chemical and instrumental methods of analysis to determine qualitative and quantitative composition of objects, sampling and sample preparation, methods of concentration and separation; methodology for conducting qualitative and quantitative analysis; statistical processing of chemical analysis results; skills and abilities necessary for identification and quantification of various objects including medicines quality monitoring.

Knowledge, abilities and skills acquired during study of the academic discipline «Analytical Chemistry» are necessary for the successful study of the following academic disciplines: «Pharmaceutical Chemistry», «Pharmacognosy», «Toxicological Chemistry», «Modern Methods of Analysis and Standardization of Drugs», «Biological Chemistry», «Pharmaceutical Ecology».

A student who has mastered educational material of the academic discipline «Analytical Chemistry» must have the following basic professional competencies:

apply knowledge of the basic physical, chemical and biological laws for quality control of medicines and medicinal plant raw materials.

As a result of studying the academic discipline «Analytical Chemistry» the student should

know:

basic concepts of analytical chemistry, role and significance of analytical chemistry methods in pharmacy;

basics of sampling, sample preparation and chemical analysis of samples;

basic methods of concentration and separation;

methods of preparation, standardization and storage of reagents including titrated solutions;

theoretical foundations of methods for qualitative and quantitative analysis;

arrangement of analytical equipment main types used in instrumental methods of analysis;

methods of chemical analysis results statistical processing and presentation;

be able to:

choose method and methodology for identification and quantification, carry out all the necessary calculations;

use techniques and methods of working with chemical reagents and laboratory glassware necessary for identification and quantification;

work with the main types of analytical instruments used in analysis;
 carry out identification and quantitative determination of substances using chemical and instrumental methods of analysis;

perform statistical processing of chemical analysis results;

master:

methods of sampling and sample preparation, separation and concentration of substances;

skills in working with chemical glassware, chemical reagents, analytical equipment and other instruments used in identification and quantification;

methods for identifying and quantifying of various objects;

skills in the preparation and standardization of chemical reagents including titrated solutions;

technique for analyzing substances using chemical and instrumental methods.

Total number of hours for the study of the discipline is 408 academic hours, of which 215 classroom hours and 193 hours of student independent work. Classroom hours according to the types of studies: lectures – 30 hours (including 9 hours of supervised student independent work (SSIW)), laboratory classes – 193 hours.

Intermediate assessment is carried out according to the syllabus of the specialty in the form of a credit (3 semester) and an exam (4 semester).

Form of higher education – full-time.

ALLOCATION OF ACADEMIC TIME ACCORDING TO SEMESTERS OF STUDY

Code, name speciality	Semester	Number of academic hours						Form of intermediate assessment
		total	in-class	including			out-of-class self-studies	
				lectures	SSIW	laboratory classes		
7-07-0912-01 «Pharmacy»	3	210	110	12	3	95	100	credit
	4	198	105	9	6	90	93	exam
		408	215	21	9	185	193	

THEMATIC PLAN

Section (topic) name	Number of class hours	
	lectures (including SSIW)	laboratory classes
1. Subject and tasks of analytical chemistry	1,5	5
2. Chemical methods for identifying inorganic substances		5
3. Separation and concentration methods		
4. Chemical equilibrium in analytical chemistry. Protolytic equilibria	3	20
4.1. Chemical equilibrium in analytical chemistry	1,5	5
4.2. Properties of a solvents that affect acid-base properties of substance		5
4.3. Calculation of protoliths aqueous solutions pH. Acid-base buffer solutions		10
5. Sampling and sample preparation in pharmaceutical analysis	1,5	10
6. Analytical chemistry and chemometrics. Use of mathematical statistics methods to process results of quantitative analysis		
7. Introduction into titrimetric method of analysis. Acid-base titration	3	15
7.1. General characteristics of titrimetric methods of analysis. Acid-base indicators	1,5	5
7.2. Types of acid-base titration curves. Titration errors	1,5	5
7.3. Acid-base titration in non-aqueous media. Application of acid-base titration in pharmaceutical analysis		5
8. Complexation equilibria. Organic reagents in chemical analysis	1,5	5
9. Complexometric titration	1,5	10
10. «Precipitate-solution» equilibria	1,5	5
11. Precipitation titration		5
12. Gravimetric method of analysis		5
13. RedOx equilibria	-	10
14. Methods of RedOx titration	3	20
14.1. General characteristics of RedOx titration methods. Iodometric titration. Chloriodometric titration	1,5	5
14.2. Iodatometric titration. Nitritometric titration. Dichromatometric titration		5
14.3. Permanganometric titration. Bromatometric titration. Cerimetric titration	1,5	10
15. General characteristics of instrumental methods of analysis. Law of electromagnetic radiation absorption.	1,5	5

Methods for calculating concentration of substance from the analytical signal value		
16. Absorption spectrometric methods of analysis	3	10
16.1. Atomic absorption spectrometry. Infrared spectrometry	1,5	5
16.2. Molecular absorption spectrometry in the ultraviolet-visible region	1,5	5
17. Emission spectrometric methods of analysis	1,5	5
17.1. Atomic emission spectrometry	1,5	5
17.2. Luminescence spectrometry		
18. Optical methods not related to absorption or emission of radiation	1,5	10
19. Chromatographic methods of analysis	-	10
19.1. General characteristics and theoretical foundations of chromatographic methods of analysis	-	5
19.2. Gas chromatography. Liquid chromatography	-	5
20. Electrochemical methods of analysis	3	20
20.1. General characteristics and classification of electrochemical methods of analysis. Conductometry. Coulometry	1,5	5
20.2. Potentiometric method of analysis	1,5	5
20.3. Voltammetry		5
21. Radiometric methods of analysis	1,5	15
Total hours	30	185

CONTENT OF THE EDUCATIONAL MATERIAL

1. Subject and tasks of analytical chemistry

Analytical chemistry as a science about methods of analyzing matter, its place in the system of sciences. Analytical service.

Basic concepts of analytical chemistry: principle, method and technique of analysis. Types of analysis. Main stages of analysis. Factors that determine formulation of analytical problem: level of component content, required accuracy, rapidity of analysis, cost.

Analytical chemistry methods: sampling methods, sample decomposition methods, separation and concentration methods, detection and quantification methods (chemical, instrumental, biological).

Chemical sensors, biosensors. Miniaturized analytical systems.

A brief historical sketch of analytical chemistry development. Application of analytical chemistry methods in pharmacy.

2. Chemical methods for identifying inorganic substances

Work and safety rules in a chemical laboratory. Basic principles of identification. Analytical effect and analytical reaction. Classification of analytical reactions and reagents used in qualitative analysis (group, selective, specific). The most important characteristics of analytical reaction (selectivity, detection limit, selectivity,

sensitivity). Fractional and systematic analysis. Analytical groups of cations and anions. Acid-base classification of cations. Classification of anions according to solubility of barium and silver salts and redox properties. General characteristics and analysis of anions and cations. Characteristic reactions for detection of groups I–VI cations of and groups I–III anions. Features of inorganic and organic substances identification. Preliminary tests for anion detection. Systematic analysis of halide anions and sulfur-containing anions mixtures. Ways to eliminate interfering influence of ions: separation, masking. Using qualitative analysis in pharmacy.

3. Separation and concentration methods

General characteristics and classification of separation and concentration methods. Quantitative characteristics of separation and concentration efficiency (separation coefficient, concentration coefficient). Basic concepts used in liquid-liquid extraction method (extractant, extract, reextraction, reextract, reextractant, extraction reagent, raffinate). Quantitative characteristics of extraction equilibrium (extraction constant, distribution constant, distribution coefficient, degree of single extraction, degree of multiple extraction). Extraction systems, extractants and extractable compounds. Influence of various factors on the extraction process. Extraction methods. Application of extraction in analytical chemistry.

4. Chemical equilibrium in analytical chemistry. Protolytic equilibria

4.1. Chemical equilibrium in analytical chemistry

General characteristics of chemical equilibrium. Chemical equilibrium constant. Types of chemical equilibrium constants used in analytical chemistry (thermodynamic, real and concentration, general, stepwise). Deviations from ideality in solutions of strong electrolytes. Activity and activity coefficient of ions (average, individual). Equations used to calculate activity coefficients. Ionic strength of solution. Dependence of activity of a strong electrolyte on ionic strength of solution. Reasons for changes in activity with changes in ionic strength. General principles for calculating composition of equilibrium systems. Total and equilibrium concentrations, mole fraction of a substance. Equations of material balance and electrical neutrality. Concept of methods for graphically describing equilibria.

4.2. Properties of a solvents that affect acid-base properties of substance

The most important theories of acids and bases: Arrhenius theory, Bronsted-Lowry protolytic theory, Lewis theory. Quantitative description of acids and bases strength (acidity constant, basicity constant, acidity constant of the conjugate acid, their indicators). Hydrogen indicator. Effect of solvent on the acid-base properties of solute. Protic and aprotic solvents. Classification of solvents according to acid-base properties and polarity. Solvent autoprotolysis. Autoprotolysis constant. Leveling and differentiating effect of solvent. Strong and weak acids and bases.

4.3. Calculation of protoliths aqueous solutions pH. Acid-base buffer solutions

pH calculation of aqueous solutions of strong and weak acids, mixtures of several acids, solutions of strong and weak bases, ampholytes and mixtures of several bases, multiproton protolytes. Calculation of equilibrium mixtures of protoliths composition at a given pH value.

Concept of buffer systems. Acid-base buffer solutions, general characteristics,

principle of operation. Calculation of buffer solution pH. Henderson-Hasselbach equation. Buffer capacity. Factors affecting buffer capacity. Working area of buffer solution. Criteria for choosing a buffer solution. Use of buffer systems in analysis.

5. Sampling and sample preparation in pharmaceutical analysis

Sample. Types of samples (general, average representative, laboratory, analyzed). Sampling of gases, liquids and solids, sample averaging. Causes of errors in sampling. Sample decomposition. «Dry» and «wet» methods of sample decomposition. Sample decomposition by dissolution (without a chemical reaction and with chemical reactions), fusion and thermal decomposition. Undesirable processes occurring during sample decomposition.

6. Analytical chemistry and chemometrics. Use of mathematical statistics methods to process results of quantitative analysis

Rules for working with rounded values. Concept of significant figures. Approximate calculations and significant figures. Concept of «measurement uncertainty» and «measurement error». Accuracy and reproducibility of analysis results. Types of errors: gross, random, systematic. Causes of systematic errors. Assessing correctness of analysis results (use of standard samples, analysis by other methods, additive method, introduced-found method).

Basic concepts of mathematical statistics used in analytical chemistry. Statistical processing and presentation of quantitative analysis results. Detection of gross errors (misses). The main characteristics of the analytical technique: detection limit, determination limit, limits of determined contents, sensitivity, reproducibility, accuracy. Reproducibility (convergence, repeatability) and correctness of analysis results. Comparison of reproducibility, average values of analysis results, verification of accuracy.

7. Introduction into titrimetric method of analysis. Acid-base titration

7.1. General characteristics of titrimetric methods of analysis. Acid-base indicators

Basic concepts associated with titrimetric methods of analysis: titration, titrant, equivalence point, titration end point, indicator, titration curve, degree of titration. Requirements for reactions in titrimetry. Classification of titrimetric methods of analysis and titration methods. Titrants and standard substances in titrimetric methods of analysis, requirements for them. Concept of indicators, their classification.

Calculations related to titration. Deriving calculation formulas used in titrimetry. Methods for describing quantitative composition of solutions in titrimetry. Calculations in titrimetric methods of analysis associated with preparation of titrant solutions and titration.

Characteristics of measuring utensils used for accurate and approximate measurement of liquid volume. Cleaning measuring utensils and preparing them for use. Rules for working with measuring utensils. Checking capacity of measuring containers.

Principles of acid-base titration. Acidimetry and alkalimetry. Preparation and standardization of acid-base titrant solutions. Primary and secondary standard solutions. Acid-base indicators, their classification. Color transition interval of acid-base indicators and its relationship with pK_a value of indicator. Reasons for changes

in color of acid-base indicators. Examples of acid-base indicators, proteolytic equilibria in their solutions (methyl orange, phenolphthalein, etc.).

7.2. Types of acid-base titration curves. Titration errors

Concept of titration curves. Degree of titration. Titration curves of a strong acid with a strong base and a strong base with a strong acid. Titration curves of a weak acid with a strong base and a weak base with a strong acid. Factors influencing magnitude of the acid-base titration jump. Titration errors. Systematic and random indicator errors in acid-base titration. Titration of polybasic acids and polyacid bases, mixtures of acids or bases. Analysis of alkali metal hydroxide and carbonate mixture, mixture of alkali metal carbonate and bicarbonate. Application of acid-base titration in pharmaceutical analysis.

7.3. Acid-base titration in non-aqueous media Application of acid-base titration in pharmaceutical analysis

Limitations of acid-base titration in aqueous media use. Essence of acid-base titration method in non-aqueous media. Criteria for choosing solvent for acid-base titration. Titration constant. Solvents, titrants, standards and indicators used in non-aqueous titrations. Solvent selection criteria. Practical application of non-aqueous acid-base titration in pharmaceutical analysis for determination of weak acids and bases.

Methods for titrimetric determination of ammonium salts. Determination of nitrogen in organic compounds by the Kjeldahl method. Determination of boric acid.

8. Complexation equilibria. Organic reagents in chemical analysis

Basic concepts related to complex compounds: complex compound, complexing agent, ligand, coordination number, ligand denticity. Classification of complex compounds. Chelates, intra-complex and outer-sphere compounds. Pearson's theory of soft and hard acids and bases. Equilibrium constants used to characterize complex compounds: formation constants, dissociation constants (general, stepwise, thermodynamic, real and conditional concentration). Kinetic stability of complex compounds.

Influence of various factors (nature of the complexing agent and ligands, temperature, ionic strength, side reactions) on the process of complex formation and stability of complex compounds. Irving-Williams series. Influence of reactants concentration on complex formation. Mole fractions calculation of free metal ions and complexes in an equilibrium mixture. Complexity function. Average ligand number.

Concept of organic reagents. Organic reagents as chelating ligands. Organic reagents that take part in processes not related to complex formation. Application of organic reagents in analytical chemistry. Functional-analytical grouping. Factors affecting activity of organic reagents. Classification of organic reagents by type of donor atoms. The most important organic reagents used in chemical analysis. Possibilities of using complex compounds in pharmaceutical analysis.

9. Complexometric titration

General characteristics and classification of complexometric titration methods. Requirements for reactions. Complexometric titration curve. Factors influencing magnitude of titration jump.

Mercurimetric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of

mercurimetric titration in pharmaceutical analysis.

General characteristics of complexometric titration. Characteristics of ethylenediaminetetraacetic acid (EDTA) properties and its interaction with metal cations. Equilibria in aqueous EDTA solutions. Composition and stability of metal complexonates. Methods for detecting titration end point in complexometry. Metal indicators: general characteristics, classification, interaction with metal ions, color transition interval of metallochromic indicators, indicator errors. Examples of metallochromic indicators. Complexometric titration curve. Factors influencing magnitude of titration jump. Titrants and methods of complexometric titration. Determined substances. Application of complexometric titration in pharmaceutical analysis.

10. «Precipitate-solution» equilibria

Equilibrium in «precipitate-solution» system. Solubility product constant (thermodynamic, real concentration, conditional concentration). Using solubility product constant to determine possibility of precipitation. Solubility. Relationship between the ionic and molecular solubility of a substance and solubility product constant. Calculation of molecular solubility of complex compounds and weak acids. Influence of various factors (nature of solute and solvent, temperature, ionic strength, presence of a common ion, side reactions-protolytic reactions and complexation reactions) on solubility of poorly soluble electrolytes. General principles for dissolving sediments of poorly soluble electrolytes.

11. Precipitation titration

General characteristics and classification of precipitation titration methods. Requirements for reactions. Precipitation titration curve. Factors influencing magnitude of titration jump.

Mercurimetric titration: principle of method, titration conditions, titrants, detection of titration end point, analytes. Practical application of mercurimetric titration in pharmaceutical analysis.

Argentometric titration. Essence of the method. Argentometric titration titrants, preparation methods, standardization. Detection of argentometric titration end point: methods of Mohr, Volhard and Fajans. Application conditions and selection of adsorption indicators. Determined substances. Practical application of argentometric titration in pharmaceutical analysis.

12. Gravimetric method of analysis

Essence of gravimetric analysis, advantages and disadvantages. Types of gravimetric determinations. Precipitable and gravimetric forms, requirements for them. The main stages of the gravimetric determination method by sedimentation. Concept of sediment formation mechanism. Formation of primary crystallization centers. Relative supersaturation and its influence on nature of the formed precipitate. Colloidal stage of slightly soluble compound formation. Amorphous and crystalline sediments, conditions of formation. Method of «emerging reagents». The main processes leading to sediment contamination, their causes and methods of elimination. Weighing technology using analytical and precision balances. Application of gravimetry in pharmaceutical analysis. Methods of direct and indirect distillation. Isolation methods in gravimetry.

13. RedOx equilibria

General characteristics of redox equilibria. Quantitative assessment of substances redox ability. Electrode potential. Nernst equation. Standard electrode half-reaction potential, formal potential. Electromotive force of reaction. Formal electrode potential. Equilibrium constant of redox reaction. Influence of various factors (temperature, foreign ions, pH, side reactions) on the course of redox reactions. Use of redox reactions in analytical chemistry and pharmaceutical analysis.

14. Methods of RedOx titration

14.1. General characteristics of RedOx titration methods. Iodometric titration. Chloriodometric titration

General characteristics and classification of redox titration methods. Requirements for reactions. Redox titration curve. Factors influencing magnitude of titration jump. Methods for detecting end point of redox titrations. Redox indicators, operating principle, indicator color transition interval. Systematic indicator errors.

Iodometric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of iodometric titration methods in pharmaceutical analysis. Determination of water by the Karl Fischer method. Determination of active chlorine.

Chloriodometric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of chloriodometric titration. Determination of iodine number.

14.2. Iodatometric titration. Nitritometric titration. Dichromatometric titration

Iodatometric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of iodate titration.

Nitritometric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of nitritometric titration. Determination of primary aromatic amines.

Dichromatometric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of dichromatometric titration. «Chemical oxygen demand».

14.3. Permanganometric titration. Bromatometric titration. Cerimetric titration

Permanganometric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of permanganometric titration.

Bromatometric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of bromatometric titration. Determination of phenols.

Cerimetric titration: principle of method, titration conditions, titrants, standard substances, detection of titration end point, analytes. Practical application of cerimetric titration.

Principle of choosing acids to create a medium in redox titration.

15. General characteristics of instrumental methods of analysis. Law of electromagnetic radiation absorption. Methods for calculating concentration of substance from analytical signal value

General characteristics and classification of instrumental methods of analysis. Concept of analytical signal. Reference and non-reference methods of quantitative analysis. Standard substances and reference materials. Methods for calculating concentration of a substance based on value of the analytical signal (calibration curve method, standard method, additive method).

Classification of spectrometric methods of analysis. Nature and properties of electromagnetic radiation. Use of electromagnetic radiation absorption law in analytical chemistry. Deviation from law of light absorption. Transmission and optical density. Molar and specific absorption coefficients. Law of optical densities additivity. Deviations from law of light absorption.

16. Absorption spectrometric methods of analysis

16.1. Atomic absorption spectrometry. Infrared spectrometry

Absorption spectrometric methods of analysis, concepts and classification. Principle of method. Atomic absorption spectrometry: processes leading to the appearance of an analytical signal, atomic absorption spectra. Design and principle of atomic absorption spectrometers operation. Possibilities, advantages and disadvantages of method. Factors affecting accuracy and reproducibility. Spectral and physico-chemical interference and methods for their elimination. Practical application in pharmaceutical analysis. Cold steam method, hydride generation method.

Infrared spectrometry: processes leading to the appearance of an analytical signal, general characteristics of IR spectra. Principle of method. Classification and design of IR spectrometers. IR spectrometry with Fourier transformation. Near-IR spectrometry. Practical application.

Concept of nuclear magnetic resonance, proton magnetic resonance, electron paramagnetic resonance.

16.2. Molecular absorption spectrometry in the ultraviolet-visible region

Molecular absorption spectrometry in the ultraviolet and visible region: processes leading to the appearance of an analytical signal, molecular absorption spectra. Principle of method.

Measurement of analytical signal, classification and design of instruments for measuring light absorption in the UV and visible regions of spectrum. Practical application of method. Methods for concentration determination.

The main techniques used in spectrophotometric analysis: direct, difference, derivative and multi-wavelength spectrophotometry, photometric reactions, extraction photometry, photometric titration. Examples of practical application. Colorimetry and photoelectrocolorimetry.

17. Emission spectrometric methods of analysis

17.1. Atomic emission spectrometry

Atomic emission spectrometry: processes leading to the appearance of an analytical signal, atomic emission spectra. Principle of method. Design of atomic emission spectrometers. Inductively coupled plasma atomic emission spectrometry. Possibilities, advantages and disadvantages of the method. Spectral and physico-

chemical interference and methods for their elimination. Practical application in pharmaceutical analysis.

17.2. Luminescence spectrometry

Luminescent methods of analysis. Classification, main characteristics and patterns of luminescence. Stokes-Lomel law. Vavilov's law. Kashi rule. Levshin's rule. Lomakin-Shaibe law. Fluorescence and phosphorescence. Jablonski diagram. Shpolsky effect. Stern-Volmer equation. Influence of various factors on fluorescence intensity of solutions. Design and principle of instruments used to measure fluorescence operation. Basic techniques used in luminescent methods of analysis. Fluorescence quenching. Luminescent indicators.

Concept of X-ray fluorescence spectrometry. X-ray diffraction.

18. Optical methods not related to absorption or emission of radiation

Determination of refractive index (refractometry), determination of optical activity (polarimetry), circular dichroism. Turbidimetry and nephelometry. Processes leading to analytical signal formation. Design and principle of corresponding devices operation. Factors affecting refractive index, optical rotation. Practical application in pharmaceutical analysis.

Raman spectrometry. Giant Raman scattering of light. Prospects for practical application.

19. Chromatographic methods of analysis

19.1. General characteristics and theoretical foundations of chromatographic methods of analysis

Principle underlying the method and classification of chromatographic methods of analysis. Retention and separation parameters in chromatography. Main characteristics of external chromatogram. Methods for obtaining chromatograms. Chromatographic peak parameters. Methods of quantitative determination in chromatography (methods of internal standard, external standard, internal normalization, calibration method). Theories of chromatographic separation (theory of theoretical plates and kinetic theory (Van Deemter equation)). Historical development of chromatographic methods of analysis.

19.2. Gas chromatography. Liquid chromatography

General characteristics and classification of gas chromatography method. Design and principle of gas chromatograph operation. Methods for quantitative processing of chromatograms. Practical application of gas chromatography in pharmacopoeial analysis.

Combined (hybrid) methods of analysis: gas chromatography-mass spectrometry. Basic concepts, mass spectrum of a substance.

Supercritical fluid chromatography (SFC).

General characteristics and classification of liquid chromatography methods. Planar (planar): paper and thin layer chromatography. Practical application of paper and thin layer chromatography.

General characteristics and classification of column liquid chromatography. Classical column chromatography. High performance (HPLC) and ultra-high performance liquid chromatography. Design and principle of liquid chromatograph operation. Practical application of HPLC in pharmaceutical analysis.

HPLC-mass spectrometry. Stages of analysis.

Size exclusion chromatography, its practical application.

Ion exchange chromatography. Ion chromatograph. Practical application. Concept of electrophoresis and capillary electrophoresis.

20. Electrochemical methods of analysis

20.1. General characteristics and classification of electrochemical methods of analysis. Conductometry. Coulometry

General characteristics and classification of electrochemical methods of analysis. Characteristics of electrodes and operating principle of an electrochemical cell.

Conductometric method of analysis: theoretical foundations and classification. Conductometric cell for measuring electrical conductivity. Analytical signal measurements. Factors affecting electrical conductivity. Kohlrausch's law. Practical application of direct conductometry. Conductometric titration: principle of method, types of conductometric titration curves, practical application. Concept of high-frequency conductometry.

Coulometric method of analysis: general characteristics and classification, types of coulometers, measurement of analytical signal in direct coulometry. Combined Faraday's law for electrolysis. Coulometric titration: principle of method, measurement of analytical signal, practical application.

20.2. Potentiometric method of analysis

Potentiometric method of analysis: theoretical foundations and classification, conditions for measuring analytical signal. Operating principle, classification and main characteristics of ion-selective electrodes. Methods for determining concentration of a substance in direct potentiometry. Potentiometric titration: principle of method, detection of titration end point using titration curve, its derivatives, as well as the Gran method, practical application.

20.3. Voltammetry

Voltammetric method of analysis, general characteristics and classification: polarography and voltammetry. Analytical signal measurement. Conditions required for voltammetric measurements. Polarographic curve. Polarographic wave. Half-wave potential. Diffusion current. Ilkovich equation. Practical application of voltammetry. Modern methods of voltammetry. Amperometric titration: principle of method, titration conditions, practical application. Amperometric titration with two indicator electrodes.

21. Radiometric methods of analysis

Basic concepts of radiometry. Methods for recording radioactive radiation. Measurement of natural or artificial elements radioactivity. Radiometric titration. Methods based on radiation absorption and reflection. Activation analysis.

EDUCATIONAL-METHODOLOGICAL MAP OF EDUCATIONAL DISCIPLINES «ANALYTICAL CHEMISTRY»

Section number, topic	Title of section, topic	Number of classroom hours		SSIW	Practical skill	Control form	
		lectures	laboratory classes			practical skill	current/ intermediate certification
3rd semester							
	Lectures	12	-	3			
1.	Subject and tasks of analytical chemistry. Chemical methods for identifying inorganic substances	1,5	-	-			
2.	Separation and concentration methods	-	-	1,5			electronic test
3.	Sampling and sample preparation in pharmaceutical analysis. Analytical chemistry and chemometrics. Using mathematical statistics methods to process results of quantitative analysis	1,5	-	-			
4.	Chemical equilibrium in analytical chemistry. Properties of a solvents that affect acid-base properties of substance	1,5	-	-			
5.	Calculation of protoliths aqueous solutions pH. Acid-base buffer solutions	1,5	-	-			
6.	General characteristics of titrimetric methods of analysis. Acid-base indicators	1,5	-	-			
7.	Types of acid-base titration curves. Titration errors	1,5	-	-			
8.	Complexation equilibria. Organic reagents in chemical analysis	-	-	1,5			electronic test
9.	Complexometric titration	1,5	-	-			
10.	Precipitate-solution equilibria. Precipitation titration. Gravimetric method of analysis	1,5	-	-			
	Laboratory lessons	-	95	-			
1.	Subject and tasks of analytical chemistry L.w. Reactions for detection of I–III analytical groups cations	-	5	-	Identification of 1-3 analytical groups	Laboratory report	Survey, electronic test

					cations using chemical reactions		
2. 3.	Chemical methods for identifying inorganic substances. Separation and concentration methods L.w. Extraction and re-extraction of bromine, extraction of perchromic acids. Reactions for detection of IV analytical groups cations	-	5	-	Identification of 4 analytical groups cations using chemical reactions. Extraction, bromine re-extraction, perchromic acid extraction	Laboratory report, solving of situational problems	Survey, electronic test
4.1.	Chemical equilibrium in analytical chemistry L.w. Reactions for detection of V-VI analytical groups cations	-	5	-	Identification of 4-5 analytical groups cations using chemical reactions. Calculation of theoretical chemical equilibrium values	Laboratory report, solving of situational problems	Survey, electronic test
4.2.	Properties of a solvents that affect acid-base properties of substance L.w. Analysis of unknown cations	-	5	-	Analysis of unknown cations. Calculation of theoretical values of chemical equilibrium	Laboratory report, solving of situational problems	Survey, electronic test
4.3.	Calculation of protoliths aqueous solutions pH L.w. Reactions for detection I analytical groups anions	-	5	-	Identification of 1 analytical groups anions using chemical reactions. Calculation of theoretical values of chemical equilibrium, incl. proteolytic	Laboratory report, solving of situational problems	Survey, electronic test
4.3.	Acid-base buffer solutions L.w. Reactions for detection of II-III analytical groups anions	-	5	-	Identification of 2-3 analytical groups anions using chemical reactions. Calculation of theoretical values of chemical equilibrium, incl. proteolytic	Laboratory report, solving of situational problems	Survey, electronic test
5.	Sampling and sample preparation in pharmaceutical analysis. Analytical chemistry and chemometrics. Using mathematical statistics methods to process results of quantitative analysis	-	5	-	Analysis of unknown anions. Statistical processing of chemical	Laboratory report, solving of situational	Survey, electronic test

	L.w. Identification of unknown anions				experiment results	problems	
6.	Final lesson on the topics «Chemical equilibrium. Protolytic equilibria» L.w. Identification of an unknown inorganic substance	-	5	-	Analysis of inorganic substances	Laboratory report	Colloquium*
7.1.	General characteristics of titrimetric methods of analysis. Acid-base indicators L.w. Checking capacity of measuring containers. Preparation and standardization of titrant solutions. Concentration determination of alkali solutions	-	5	-	Checking capacity of measuring containers. Preparation and standardization of hydrochloric acid solution. Determination of sodium hydroxide solution concentration. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test
7.2.	Types of acid-base titration curves. Titration errors L.w. Analysis of alkali metal carbonate and bicarbonate mixture. Determination of ammonium chloride by back titration	-	5	-	Analysis of sodium carbonate and sodium bicarbonate mixture. Determination of ammonium salts by back titration. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test
7.3.	Acid-base titration in non-aqueous media. Application of acid-base titration in pharmaceutical analysis L.w. Titrimetric determination of caffeine in non-aqueous medium	-	5	-	Titrimetric determination of caffeine in a non-aqueous medium. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test
8.	Complexation equilibria. Organic reagents in chemical analysis L.w. Complexometric determination of zinc sulfate (chloride)	-	5	-	Preparation and standardization of EDTA (sodium edetate) solution. Complexometric determination of zinc sulfate (chloride). Theoretical values	Laboratory report, solving of situational problems	Survey, electronic test

					calculation of chemical equilibrium, incl. complexation equilibria		
9.	Complexometric titration L.w. Determination of magnesium and calcium ions in water when they are present together	-	5	-	Determination of magnesium and calcium cations in their joint presence. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test
9.	Final lesson on the topics «Acid-base and complexometric titration. Complexation equilibria» L.w. Determination of boric acid in solution	-	5	-	Determination of boric acid	Laboratory report	Colloquium*
10.	Precipitate-solution equilibria L.w. Argentometric determination of chlorides	-	5	-	Argentometric determination of halides. Theoretical values calculation of chemical equilibrium, including sediment-solution equilibria	Laboratory report, solving of situational problems	Survey, electronic test
11.	Precipitation titration L.w. Argentometric determination of bromides and iodides	-	5	-	Argentometric determination of halides. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test
12.	Gravimetric method of analysis L.w. Determination of magnesium sulfate in hydrate	-	5	-	Determination of magnesium sulfate in hydrate	Laboratory report, solving of situational problems	Survey, electronic test
13.	RedOx equilibria	-	5	-	Theoretical values calculation of chemical equilibrium, incl. redox equilibria	Solving of situational problems	Survey, electronic test
13.	Final lesson on the topics «Precipitation and RedOx titration. Gravimetry» L.w. Presentation of chemical analysis results	-	5	-	Statistical processing of chemical experiment results	Solving of situational problems*	Control work* Credit

4th semester

Lectures		9	-	6			
1.	General characteristics of redox titration methods. Iodometric titration. Chloriodometric titration. Iodatometric titration. Nitritometric titration. Dichromatometric titration	1,5	-	-			
2.	Permanganometric titration. Bromatometric titration. Cerimetric titration	-	-	1,5			electronic test
3.	General characteristics of instrumental methods of analysis. Law of electromagnetic radiation absorption. Methods for calculating concentration of substance from the analytical signal value	1,5	-	-			
4.	Atomic absorption spectrometry. Infrared spectrometry	1,5	-	-			
5.	Molecular absorption spectrometry in ultraviolet-visible region	-	-	1,5			electronic test
6.	Atomic emission spectrometry. Luminescence spectrometry	1,5	-	-			
7.	Optical methods not involving absorption or emission of radiation	1,5	-	-			
8.	General characteristics and classification of electrochemical methods of analysis. Conductometry. Coulometry	1,5	-	-			
9.	Potentiometric method of analysis. Voltammetry	-	-	1,5			electronic test
10.	Radiometric methods of analysis	-	-	1,5			electronic test
Laboratory exercises		-	90	-			
14.1.	General characteristics of redox titration methods. Iodometric titration. Chloriodometric titration L.w. Iodometric determination of ascorbic acid and copper sulfate	-	5	-	Preparation and standardization of iodine solution. Preparation and standardization of sodium thiosulfate solution. Iodometric determination of ascorbic acid and copper sulfate. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test

14.2.	Iodatometric titration. Nitritometric titration. Dichromatometric titration L.w. Nitritometric determination of novocaine hydrochloride	-	5	-	Preparation and standardization of sodium nitrite solution. Nitritometric determination of novocaine (procaine) hydrochloride. Dichromatometric determination of iron salts. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test
14.3.	Permanganometric titration. Bromatometric titration. Cerimetric titration L.w. Permanganometric determination of hydrogen peroxide. Bromatometric determination of phenol	-	5	-	Standardization of potassium permanganate solution. Permanganometric determination of hydrogen peroxide. Bromatometric determination of phenol. Calculation of titrimetric results	Laboratory report, solving of situational problems	Survey, electronic test
14.3.	Final lesson on the topics «Redox equilibria and titrations» L.w. Bromatometric determination of phenol (resorcinol)	-	5	-	Bromatometric determination of resorcinol	Laboratory report	Colloquium*
15.	General characteristics of instrumental methods of analysis. Law of electromagnetic radiation absorption. Methods for calculating concentration of substance from the analytical signal value L.w. Photometric determination of iron (III)	-	5	-	Photometric determination of iron (III) salts. Calculation of spectrophotometric results	Laboratory report, solving of situational problems	Survey, electronic test
16.1.	Atomic absorption spectrometry. Infrared spectrometry L.w. Interpretation of IR spectra. Spectrophotometric determination of novocaine (procaine) hydrochloride	-	5	-	Spectrophotometric determination of novocaine (procaine) hydrochloride. Calculation of spectrophotometric results	Laboratory report, solving of situational problems	Survey, electronic test

16.2.	Molecular absorption spectrometry in the ultraviolet-visible region L.w. Spectrophotometric determination of cyanocobalamin and nitrofurantoin	-	5	-	Spectrophotometric determination of cyanocobalamin and nitrofurantoin. Calculation of spectrophotometric results	Laboratory report, solving of situational problems	Survey, electronic test
17.1. 17.2.	Atomic emission spectrometry. Luminescence spectrometry L.w. Fluorimetric determination of quinine and aluminum salts in complex with morine	-	5	-	Fluorimetric determination of quinine and aluminum salts in complex with morine. Calculation of spectrophotometric results	Laboratory report, solving of situational problems	Survey, electronic test
18.	Optical methods not involving absorption or emission of radiation L.w. Refractometric determination of substances concentration	-	5	-	Refractometric determination of magnesium sulfate, calcium chloride and glucose concentration. Calculation of polarimetric and refractometric results	Laboratory report, solving of situational problems	Survey, electronic test
18.	Final lesson on the topics «Spectrometric methods of analysis» L.w. Polarimetric study of sugar solutions	-	5	-	Polarimetric determination of sucrose and glucose solutions	Laboratory report	Colloquium*
19.1.	General characteristics and theoretical foundations of chromatographic methods of analysis L.w. Identification of metal cations using paper chromatography	-	5	-	Separation of iron and copper cations by paper chromatography. Calculation of chromatographic results	Laboratory report, solving of situational problems	Survey, electronic test
19.2.	Gas chromatography. Liquid chromatography L.w. Thin layer chromatography of biologically active substances	-	5	-	Thin layer chromatography of biologically active substances. Calculation of chromatographic results	Laboratory report, solving of situational problems	Survey, electronic test

20.1.	General characteristics and classification of electrochemical methods of analysis. Conductometry. Coulometry L.w. Conductometric determination of electrical conductivity	-	5	-	Determination of electrical conductivity of water and sucrose solution. Calculation of electrochemical results	Laboratory report, solving of situational problems	Survey, electronic test
20.2.	Potentiometric method of analysis L.w. Potentiometric pH determination	-	5	-	Potentiometric determination of pH. Calculation of electrochemical results	Laboratory report, solving of situational problems	Survey, electronic test
20.3.	Voltammetry L.w. Potentiometric titration of acid solutions	-	5	-	Potentiometric titration of hydrochloric and boric acids mixture. Calculation of electrochemical results	Laboratory report, solving of situational problems	Survey, electronic test
21.	Final lesson on the topics «Chromatographic and electrochemical methods of analysis»	-	5	-			Colloquium*, control work*
21.	Radiometric methods of analysis	-	5	-			Survey, electronic test
21.	Practical skills test	-	5	-		Laboratory report*, solving of situational problems*	
		21	185	9			Exam

*This is a mandatory form of current certification

INFORMATIONAL AND METHODOLOGICAL PART

LITERATURE

Main:

1. Kharitonov, Y. Y. Analytical chemistry. Analytics 1. General theoretical foundations. Qualitative analysis: textbook / Yu. Ya. Kharitonov; ed. V. Yu. Grigorieva. – Moscow: Geotar-Media, 2021. – 602 p.

2. Kharitonov, Y. Y. Analytical chemistry. Analytics 2. Quantitative analysis. Physical-chemical (instrumental). Analysis methods / Yu. Ya. Kharitonov; ed. V. Yu. Grigorieva. – Moscow: Geotar-Media, 2021. – 587 p.

Additional:

3. Analytical chemistry: textbook [the textbook for students of higher schools] / I. S. Grytsenko [и др.] ; National university of pharmacy ; ed. by I. S. Grytsenko. - Kharkiv : NUPh : Golden Pages, 2019. – 600 p.

4. Christian, G. D. Analytical chemistry. 7 ed. / G. D. Christian, P. K. Dasgupta, K. A. Schug. – Hoboken: Wiley, 2014. – 815 p.

METHODOLOGICAL RECOMMENDATIONS FOR ORGANIZING AND PERFORMING INDEPENDENT WORK OF STUDENTS IN AN ACADEMIC DISCIPLINE

Time allocated for independent work can be used by students to:

- preparation for lectures, laboratory classes;
- preparation for tests, credit and exam in the academic discipline;
- problem solving;
- performing research and creative tasks;
- preparation of thematic reports, abstracts, presentations;
- compiling a review of scientific literature on given topic;
- design of information and demonstration materials (stands, posters, graphs, tables, newspapers, etc.);
- compiling a thematic selection of literary sources and Internet sources;
- compiling tests for students to organize mutual control.

METHODOLOGICAL RECOMMENDATIONS FOR THE ORGANIZATION AND IMPLEMENTATION OF SUPERVISED INDEPENDENT WORK OF STUDENTS IN AN ACADEMIC DISCIPLINE

The main forms of organizing supervised independent work:

- writing and presentation of an abstract;
- giving a report;
- computer testing.

Control of supervised independent work is carried out in form of:
electronic test.

LIST OF AVAILABLE DIAGNOSTIC TOOLS

The following forms are used for competence assessment:

control work;
 electronic test;
 colloquium;
 survey;
 solving a situational problem;
 laboratory report.

LIST OF AVAILABLE TEACHING METHODS

Traditional method;

Active (interactive) methods:

Research-Based Learning (RBL).

Problem-Based Learning (PBL);

LIST OF PRACTICAL SKILLS

Name of practical skill	Practical skill control form
1. Identification of 1–6 analytical groups cations and 1–3 analytical groups anions using chemical reactions, analysis of unknown cations, anions, inorganic substances	laboratory report, solving of situational problems
2. Calculation of titrimetric, spectrophotometric, polarimetric, electrochemical, chromatographic or refractometric determination results; theoretical values calculation of chemical equilibrium, incl. proteolytic, redox equilibria, complexation equilibria and «precipitate-solution»	solving of situational problems
3. Preparation and standardization of hydrochloric acid solution	laboratory report
4. Determination of sodium hydroxide solution concentration	laboratory report
5. Analysis of sodium carbonate and sodium bicarbonate mixture	laboratory report
6. Preparation and standardization of EDTA (sodium edetate) solution	laboratory report
7. Complexometric determination of zinc sulfate (chloride)	laboratory report
8. Preparation and standardization of sodium thiosulfate solution	laboratory report
9. Preparation and standardization of iodine solution	laboratory report
10. Iodometric determination of ascorbic acid and copper sulfate	laboratory report
11. Standardization of potassium permanganate	laboratory report

solution	
12. Permanganometric determination of hydrogen peroxide	laboratory report
13. Preparation and standardization of sodium nitrite solution	laboratory report
14. Nitritometric determination of novocaine (procaine) hydrochloride	laboratory report
15. Spectrophotometric determination of cyanocobalamin and nitrofurantoin	laboratory report
16. Spectrophotometric determination of novocaine (procaine) hydrochloride	laboratory report
17. Refractometric determination of magnesium sulfate concentration	laboratory report
18. Refractometric determination of calcium chloride concentration	laboratory report
19. Refractometric determination of glucose concentration	laboratory report
20. Polarimetric determination of sucrose and glucose solutions	laboratory report
21. Determination of solution pH	laboratory report
22. Determination of electrical conductivity of water and sucrose solution	laboratory report
23. Potentiometric titration of hydrochloric and boric acids mixture	laboratory report
24. Statistical processing of chemical experiment results	solving of situational problem
25. Extraction, bromine re-extraction, perchromic acid extraction	laboratory report
26. Checking capacity of measuring containers	laboratory report
27. Determination of ammonium salts by back titration	laboratory report
28. Titrimetric determination of caffeine in non-aqueous medium	laboratory report
29. Determination of magnesium and calcium cations in their joint presence	laboratory report
30. Determination of boric acid	laboratory report
31. Argentometric determination of halides	laboratory report
32. Determination of magnesium sulfate in hydrate	laboratory report
33. Dichromatometric determination of iron salts	laboratory report
34. Bromatometric determination of phenol and resorcinol	laboratory report
35. Photometric determination of iron (III) salts	laboratory report
36. Fluorimetric determination of quinine and aluminum salts in complex with morine	laboratory report
37. Separation of iron and copper cations by paper	laboratory report

chromatography	
38. Thin layer chromatography of biologically active substances	laboratory report

LIST OF EQUIPMENT USED

1. Sets of chemical glassware for identification of substances.
2. Sets of chemical glassware for titration.
3. Sets of laboratory glassware.
4. Spectrophotometer.
5. Photoelectric colorimeter.
6. Fluorimeter.
7. Refractometer.
8. Polarimeter.
9. Set for performing thin layer chromatography.
10. Set for performing paper chromatography.
11. Set for column chromatography.
12. Gas chromatograph.
13. Liquid chromatograph.
14. Ion meter (pH meter).
15. Water and sand baths.
16. Drying cabinet.
17. Muffle furnace.
18. Centrifuge.
19. Filtration installation.
20. Carrier gas cylinder.
21. Aquadistiller.
22. Deionizer.
23. Conductivity meter.
24. Thermometer.
25. Analytical and precision balances.
26. Shaker mixers.
27. Multimedia projector, TV.
28. Computer.
29. Microscopes.
30. Set of dishes for extraction, extraction units.

PROTOCOL OF CURRICULUM APPROVAL

Title of the discipline requiring approval	Department	Amendments to the curriculum in the academic discipline	Title of the discipline requiring approval
1. Pharmacognosy	Pharmacy Organizations	no comments	dated May 22, 2024 No. 12

COMPILERS:

Head of the Pharmaceutical Chemistry
Department of the educational institution
«Belarusian State Medical University»,
Ph.D., Associate Professor



R.I. Lukashou

Associate Professor of the Pharmaceutical
Chemistry Department of the educational
institution «Belarusian State Medical
University», Ph.D., Associate Professor



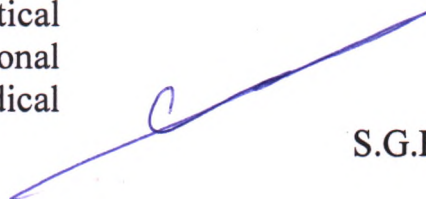
V.N. Belyatsky

Senior Lecturer of the Pharmaceutical
Chemistry Department of the educational
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N.M. Borabanova

Associate Professor of the Pharmaceutical
Chemistry Department of the educational
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University», Ph.D.



S.G. Khaminets

Curriculum content, composition and the accompanying documents comply with the established requirements.

Dean of the Medical Faculty for
International Students of the educational
institution «Belarusian State Medical
University»

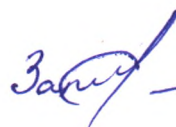
24. 06. 2024



O.S. Ishutin

Methodologist of the Educational and
Methodological Department of the Office
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Medical University»

24. 06. 2024



S.V. Zaturanova