ANALYTICAL CHEMISTRY

Training Plan

Class	Exams	Hours			Hours by years and semesters						
	Semesters	Total	Lectures	Laboratory classes	I	II	III	IV	V	VI	VII
Analytical Chemistry	II, III	150	60	90	-	2/3	2/3	-	-	-	-

Name of course
„Analytical Chemistry"

Type of course according to the Uniform State Requirements
Mandatory

Level of education
Master's degree **/MS/**

Forms of education:
Lecture course, laboratory classes, self-study

Duration of the course:
Two semesters

Auditorium classes:
Lectures 60 hrs, laboratory classes 75 hrs

Technical equipment applied in the training:
Multimedia presentations, classical and instrumental methods of analysis, other tools and technical devices for demonstration of the application of methods of quantitative chemical analysis in pharmacy, practical classes in analytical chemistry

Control and evaluation:
Ongoing evaluation –tests, oral examinations, writing papers based on the studied topics.
Final evaluation – examination

Formation of the final grade:
The current average grade for the semester is formed based on the tests and papers

Aspects in the formation of the final grade:
Participation in discussions, tests and evaluation of the written papers

Term exam:
Entrance test, written and oral examination

State final certification examination
No

Lecturer:
Professors and associated professors of the Department of "Chemical sciences".

Department: "Chemical sciences".

ANNOTATION

The course "Analytical Chemistry" is fundamental in the training of graduate pharmacists and sets theoretical basis of Pharmaceutical Chemistry and Analysis, Pharmacognosy. The "Analytical Chemistry" course allows students to get acquainted with the goals, objects and methods of analytical chemistry, to gain basic theoretical and practical knowledge of chemical and physical methods of qualitative and quantitative analysis. To gain knowledge and experience in solving important practical issues of chemical arts - calculating the pH of solutions, preparation of buffer solutions, calculation of solubility, stability properties of the constants and redox potential, under specified conditions. Analytical techniques are widely used in pharmaceutical product development and testing. Students will understand the chemical principle behind each analytical technique. This course is designed to be an integrative course on the basic principles of instrumental analysis with a focus on electrochemical and spectral methods. In laboratory classes, students will learn the methods of qualitative and quantitative analysis. Along with the analysis of inorganic materials, the curse covers the analysis of a number of organic substances used as medicaments. In parallel with the practical skills in qualitative and quantitative analysis, students will acquire knowledge of interpretation of physical parameters measured as well as quantitative treatment of obtained data.

COURSE OBJECTIVES

- Fundamentals of analytical chemistry;
- Qualitative chemical analysis - principles and methods;
- Principles and methods of quantitative chemical analysis (gravimetry and titrimetry);
- Understanding the theoretical basis and practical application of instrumental methods of analysis;
- Statistical analysis of experimental data.

MAJOR LEARNER OUTCOME

At the end of this course, the students should be able to:

- locate, select, organise and present information from a variety of sources;
- select and apply the most appropriate analytical technique/calibration strategy for the chosen analyte within a range of complex matrices;
- manipulate numerical and other data and translate information from one form to another;
- apply statistical analysis to experimental measurements.
- work with laboratory equipment– analytical balance, pH meter, spectrophotometer, etc.
- to use knowledge in future analyzes of pharmaceuticals and biologically active substances.

LECTURES – Theses

LECTURE № 1 - 2 hours

Introduction in Analytical Chemistry

1. The scope, function and role of analytical chemistry in modern science.
2. Key terms: sample, analyte, matrix, method, methodology of analysis, interfering components.
3. Classification of analytical techniques.
4. Steps in analytical process.

LECTURE № 2 - 2 hours

QUALITATIVE ANALYSIS – PRINCIPLES

1. Aims of qualitative analysis.
2. Analytical reagents used in qualitative analysis (specific, selective, group).
3. Characteristics of analytical reactions: sensitivity, identification limit, dilution limit, limiting concentration, pD value.
4. Application of qualitative analysis in pharmacy.

LECTURE № 3 - 2 hours

QUANTITATIVE ANALYSIS

1. Objectives and methods of quantitative analysis.
2. Errors in quantitative analysis.
3. Data analysis.
4. Solutions and their concentrations

LECTURE № 4 - 3 hours

CHEMICAL EQUILIBRIUM. BASIC TERMS. EQUILIBRIUM CONSTANTS IN ANALYTICAL CHEMISTRY

1. The law of mass actions. Equilibrium constants.
2. Real systems. Ideal systems. Activity coefficients. Ionic strength.
3. Equilibrium in heterogeneous systems. Equilibrium constants for a multistep reaction.
4. Equilibrium constants. Meaning of equilibrium constants for analytical chemistry.

LECTURE № 5 - 8 hours

ACID-BASE EQULIBRIA

1. General concepts of acids and bases.

1.1. Acid-base theories.

1.2. The Brønsted-Lowry theory.

1.3. Autoprotolysis. Dissociation of water.

2. Strength of acids and bases. Factors determining acids and bases strength.
3. Hydrogen ion exponent. pH scale in various solvents.

3.1. The hydrogen ion exponent.

3.2. pH scale of solutions.

3.3. Influence of solvent on proton exchange reactions.

4. Concentration of $[H_3O]^+$ in aqueous solutions.

4.1. pH and pOH calculations: solutions of a strong acid HA; solutions of a weak acid HA; solutions of a weak acid HA and its conjugate base A^-; solutions of polyprotic acids.

4.2. pH and pOH calculations: solution of a strong base; solution of a weak base; solution of a weak base and its conjugate acid.

4.3. Buffer solutions. Buffer capacity.

5. Graphical representations of equilibrium in proton exchange reactions: species distribution diagram and logarithmic charts.

LECTURE № 6 - 2 hours

TITRIMETRIC (VOLUMETRIC) ANALYSIS

1. Principles of titrimetric analysis.
2. Classification of volumetric methods for analysis.
3. Basic terms: titration, titrant, endpoint and equivalence point, titration curves.
4. Indicators.
5. Standard solutions. Methods of preparation.
6. Titration and titrimetric methods.
7. Calculations in volumetric analysis.
8. Errors.

LECTURE № 7 - 6 hours

VOLUMETRIC ANALYSIS: ACID BASE TITRATIONS

1. Principles of acid-base titrations.
2. Acid-base indicators – mechanism of action; indicator exponent and transition interval; mixed and universal indicators; mechanism of action.
3. The titration curve and their use at choosing suitable indicators: strong acid/strong base titrations; weak acid/strong base titrations; weak base / strong acid titrations; titrations of polyprotic acids.
4. Sources of errors in acid-base titrations.
5. Application of acid base titrations in pharmaceutical analysis.
6. Titration in non-aqueous solvents.

6.1. Principles of non-aqueous titrations.

6.2. Solvents, titrants and indicators in titration in nonaqueous medium.

6.3. Possibilities of the method. Application in pharmaceutical analysis.

LECTURE № 8 - 3 hours

SOLUBILITY AND PRECIPITATION. GRAVIMETRY

1. Conditions.

1.1. Solubility product.

1.2. Graphical representation of equilibrium.

1.3. Fractional precipitation.

2. Factors affecting solubility

2.1. Ionic strength.

2.2. Common ion effect.

2.3. Complex ions.

2.4. Conditional solubility product constant.

3. Gravimetry. Types of gravimetric methods.
4. Theory and practice of precipitation gravimetry.
5. Advantages and disadvantages of gravimetric analysis. Application of gravimetric analysis.

LECTURE № 8 - 2 hours

PRECIPITATION TITRATIONS

1. Principles of precipitation titrations. Classification.
2. Analytical reactions.
3. Titration curves.
4. Argentometry. Mohr's method, Volhard's method and Fajan's method**.**
5. Precipitation titration of mixtures.
6. Application in pharmaceutical analysis.

LECTURE № 12 - 2 hours

COMPLEXATION REACTIONS

1. Metal complexes. Composition of metal complexes. Ligands. Classification. Examples.
2. Stability of complexes. Stability constants.
3. Conditional metal–ligand formation constant.
4. Metal-EDTA equilibrium.

LECTURE № 13 - 2 hours

COMPLEXATION TITRATIONS

1. Principles of complex-formation titrations. Requirements.
2. Standard solutions.
3. Complexometric titration curves.
4. Metallochromic indicators. Mechanism of action.
5. Applications of complexation titrimetry.
6. Advantages and disadvantages. Application in pharmaceutical analysis.

LECTURE № 14 - 4 hours

OXIDATION–REDUCTION REACTIONS

1. Basic terms.

1.1. Oxidation and reduction.

1.2. Galvanic element.

1.3. Reversibility of electrode reactions.

2. Strength of oxidizing and reducing agents. Electrode potentials.

2.1. Standard hydrogen electrode.

2.2. Standard redox potential.

2.3. Nernst equation. Electrode potential.

3. Chemical equilibrium of oxidation-reduction reactions. Determination of the direction of the oxidation-reduction reactions.
4. Factors affecting electrode potential: ionic strength, precipitation, pH, complex formation. Conditional redox potential.

LECTURE № 15 - 4 hours

REDOX TITRATIONS

1. Principle of redox titrations.
2. Redox titration curves.

3. Redox indicators.
4. Types of redox titrations:

4.1. Permanganometry. Principle and application of permanganometry.
4.2. Chromatometry. Principle and application of chromatometry.
4.3. Iodometry/iodimetry. Principle and application of iodometry/iodimetry.
4.4. Bromatometry. Principle and application of bromatometry.
4.5. Nitritometry. Principle and application of nitritometry.

LECTURE № 16 - 2 hours

INSTRUMENTAL METHODS OF ANALYSIS. BASIC APPROACHES FOR OBTAINING AND INTERPRETING ANALYTICAL DATA

1. Classification of instrumental methods of analysis.
2. Calibration techniques: calibration curve method, standard additions method, internal standards.
3. Analytical characteristics of physical and physicochemical methods of analysis.

LECTURE № 17 - 6 hours

ELECTROCHEMICAL METHODS OF ANALYSIS

1. General characteristics.
2. Classification of electrochemical methods. Basic concepts: electrochemical cell; electrode; cell potential; electrode (electrochemical) reaction; polarization.
3. Conductimetry. Principles, classification, conductivity cell. Application.
4. Potentiometry.

4.1. Principles. Classification. Indicator and reference electrodes.
4.2. Direct potentiometry. Application.
4.3. Potentiometric titration: general discussion, reactions, titration curves, application.

5. Voltammetry: principles, electrodes, conditions. Polarographic analysis: half-wave potential, diffusion current, qualitative and quantitative polarographic analysis. Modern polarographic methods. Application.
6. Amperometric titration: general discussion, types of curves, application.
7. Coulometric methods of analysis Faraday's laws. Controlled-potential coulometry. Controlled-current coulometric methods (coulometric titrations). Application.

LECTURE № 18 - 5 hours

OPTICAL METHODS OF ANALYSIS

1. Classification. Electromagnetic radiation – basic concepts. Absorption and emission. Electronic, vibrational and rotational spectrums.
2. Regions of the electromagnetic spectrum. Methods.

3. Principles of quantitative and qualitative spectroscopic methods. Calibration.
4. Atomic spectroscopy: atomic absorption, emission and mass spectrometry. Principle of the methods, analytical characterization, application.
5. UV-VIS molecular absorption spectrometry. Quantitative and qualitative analysis.
6. Infrared spectroscopy. Principles. Application.

LECTURE № 18 - 5 hours
METHODS OF SEPARATION IN ANALYTICAL CHEMISTRY

1. Extraction. Types of extraction methods. Liquid-liquid extraction. Extraction constant, distribution coefficient. Application.
2. Chromatography. Principle. Classification. TLC. Application.
3. Ion exchange chromatography**.** Principles. Ion-exchange resins**.** Ion exchange equilibrium . Methods and application.
4. Liquid chromatography: high-performance liquid chromatography (HPLC). Theoretical principles. Application in pharmaceutical analysis.

LABORATORY CLASSES - THESES

LABORATORY EXERCISE № 1, 2, 3, 4, 5, 6 – 18 hours
QUALITATIVE ANALYSIS

1. Laboratory instruction, rules and safety guidelines for analytical chemistry laboratory. Qualitative chemistry analysis. Principle of the method. Classification. Guide in qualitative work. Laboratory glassware.
2. Quantitative analysis of cations of pharmaceutical interest.
3. Quantitative analysis of anions of pharmaceutical interest. Questions and calculations: limit of detection, limit of dilution, etc.

LABORATORY EXERCISE № 7 – 3 hours
TITRIMETRIC ANALYSIS

1. Volumetric (titrimetric) analysis: principles of the method, classification.
   Apparatus and basic techniques. Calculations, data acquisition and treatment. Errors.
2. Preparation of standard solutions.
   Calculations in analytical chemistry: concentration, dilutions, etc.

LABORATORY EXERCISE № 8, 9 – 6 hours
ACID-BASE TITRATIONS. ACIDIMETRY

1. Acid-base titration in aqueous solution. Principles of acid-base titrations, titration curves, standard solutions. Standardization of hydrochloric acid solution.
2. Determination of ammonia in solution. Problems.

LABORATORY EXERCISE № 10, 11 – 6 hours
ACID–BASE TITRATIONS. ALKALIMETRY

1. Standardization of sodium hydroxide solution. Primary and secondary standard solutions.
2. Determination of acetic acid in solution. Problems.

LABORATORY EXERCISE № 12, 13 – 6 hours
ACID–BASE TITRATIONS. APPLICATION

1. Determination of acetylsalicylic acid in aspirin tablets. Problems.
2. Determination of the acid neutralizing capacity of Antacid tablets.

LABORATORY EXERCISE № 14, 15 – 6 hours
ACID–BASE TITRATIONS IN NONAQUEOUS MEDIUM. APPLICATION

1. Acid-base titration in methanol. Preparation of hydrochloric acid in methanol. Exam.
2. Determination of sodium benzoate.
3. Acid-base titration in glacial acetic acid. Preparation of standard solution of perchloric acid.
4. Determination of papaverine hydrochloride.

LABORATORY EXERCISE № 16, 17 – 6 hours
PRECIPITATION TITRATIONS

1. Argentometry. Preparation of silver nitrate solution. Determination of chlorides. Problems.
2. Volhard's titration method. Determination of ranitidine hydrochloride in tablets. Problems.

LABORATORY EXERCISE № 18 – 3 hours
COMPLEXOMETRIC TITRATION

1. Preparation and standardization of complexone III solution. Determination of hardness of water. Determination of bismuth (III) and lead (II) in solution. Problems.

LABORATORY EXERCISE № 19, 20, 21, 22 – 12 hours
REDOX TITRATIONS

1. Permanganometry. Determination of hydrogen peroxide. Problems.
2. Iodometry. Preparation of standard solutions. Problems.
3. Iodometry. Determination of metamizole sodium in analgin tablets. Determination of glucose in aqueous solution.
4. Iodometry. Analysis of ascorbic acid in vitamin C tablets. Exam.

LABORATORY EXERCISE № 23, 24 – 6 hours
ELECTROCHEMICAL METHODS OF ANALYSIS

1. Direct potentiometry. Calibration of pH meter. Buffer solutions. Potentiometric determination of dissociation constant.
2. Potentiometric titration. Determination of phosphoric acid in solution. Problems.

LABORATORY EXERCISE № 25, 26, 27, 28 – 12 hours
SPECTROSCOPIC METHODS FOR ANALYSIS. UV-VIS SPECTROSCOPY

1. UV–VIS Spectroscopy. Absorption spectrum of vitamin B12. Quantitative determination of vitamin B12. Vitamin B12 UV-VIS purity test.
2. UV–VIS Spectroscopy. Determination of salicylic acid in acetylsalicylic acid.
3. UV–VIS Spectroscopy. Determination of active substances in binary mixture.
4. IR spectroscopy. Interpretation of IR spectrum.

LABORATORY EXERCISE № 29 – 3 hours
EXTRACTION

1. Ion-pair extraction. Determination of hydroxyzine dihydrochloride in tablets.

LABORATORY EXERCISE № 30 – 3 hours
CHROMATOGRAPHY

2. Chromatography. General concepts. Classification. Paper chromatography. Ion-exchange chromatography. Determination of distribution coefficient.

RECOMMENDED TEXTBOOKS AND INTERNET SITES

1. Fundamentals of analytical chemistry, Douglas A. Skoog; Donald M. West; F. James Holler and Stanley R. Crouch, Thomson-Brooks/Cole, 2004
2. Pharmaceutical Drug Analysys, Ashutosh Kar, New age international limited publichers, 2005.
3. http://freelecturesonline.blogspot.com/2011/11/analytical-chemistry-lecture-notes-pdf.html
4. http://ull.chemistry.uakron.edu/analytical/

ANALYTICAL CHEMISTRY
SYLLABUS

1. The scope of analytical chemistry. Objectives and tasks of analytical chemistry. Definitions and basic concepts: sample analyte, matrix method, a methodology for analyzing, interfering components. Classification of methods of analytical chemistry.
2. Chemical equilibrium. Law of mass action. Equilibrium constant. Real and ideal systems. Activity coefficient. Equilibrium in heterogeneous system.
3. Acid-base theories. The Brønsted-Lowry theory. Autoprotolysis, autoprotolysis constant. Factors affecting the strength of acids and bases.
4. The hydrogen ion exponent. pH scale in various solvents.
5. Acid-base equilibrium of strong monoprotic acids and bases. Calculations.
6. Acid-base equilibrium of monoprotic weak acids and bases. Calculations.
7. Graphical representations of acid-base equilibrium of monoprotic weak acids and bases.
8. Acid-base equilibrium of polyprotic acids and bases.
9. Buffer solutions. Buffer capacity.
10. Volumetric methods of analysis. Principles. Requirements to the analytical reaction. Classification. Basic terms: titration, titrant, equivalent and end pint, titration curves. Preparation of standard solutions. Titration and titrimetric methods.
11. Acid-base titration in aqueous solutions. Titration of monoprotic strong acids and bases. Titration curves and indicators.
12. Acid-base titration in aqueous solutions. Titration of monoprotic weak acids and bases. Titration curves and indicators.
13. Acid-base titration in aqueous solutions. Titration of polyprotic acid and strong base. Titration curves and indicators. Titrations of mixture.
14. Acid-base titrations in nonaqueous solvents. Solvents classification
15. Acid-base titrations in nonaqueous medium. Principles. Solvents, titrants and indicators in nonaqueous medium. Possibilities of the method. Application in pharmaceutical analysis.
16. Acid-base titration in nonaqueous medium. Advantages and disadvantages of methanol as a solvent.

17. Acid-base titrations in nonaqueous solvents. Titrations in glacial acetic acid. Advantages, disadvantages and considerations of the method.
18. Solubility equilibrium. Solubility and solubility product. Factors affecting solubility. Conditional solubility product constant.
19. Gravimetry. Basic concepts. Advantages and disadvantages of the gravimetric analysis.
20. Precipitation titrations. Principles of precipitation titrations. Requirements to the analytical reaction. Argentometry. Titration curves. Mohr's method, Volhard's method and Fajan's method of titration**.**
21. Metal complexes. Composition of metal complexes. Ligands. Classification. Examples.
22. Stability of complexes. Stability constants.
23. Conditional metal–ligand formation constant.
24. EDTA-metal ion complexation equilibria
25. Complexometric titration. Principles. requirements for complexometric titrations. Titration curves in complexometric titrations. Metallochromic indicators.
26. Application of complexometric titration.
27. Oxidation-reduction equilibrium. Oxidation-reduction reactions. Strength of oxidizing and reducing agents. Electrode potential. Conditional electrode potential.
28. Redox titrations. Principles. Redox titration curves. Redox indicators.
29. Classification of oxidation-reduction titrations. Principles and applications of the methods: permanganometry; chromatometry: iodometry/iodimetry; bromatometry; nitritometry.
30. Classification of instrumental analysis. Calibration techniques: calibration curve method, standard additions method.
31. Electrochemical methods of analysis. Classification of electrochemical methods. Basic concepts: electrochemical cell; electrode; cell potential; electrode (electrochemical) reaction; polarization.
32. Conductimetry. Principles. Classification. Conductivity cell. Application of conductimetry.
33. Potentiometry. General principles and classification. Indicator and reference electrodes
34. Potentiometry. Direct potentiometry. pH metry and ionometry. Potentiometric titration. Principle of potentiometric titration. Application of potentiometry.
35. Voltammetry. Principles of voltammetry. Polarographic analysis: half-wave potential; diffusion current, qualitative and quantitative polarographic analysis. Modern polarographic methods.
36. Amperometric titration. Principles of amperometric titration. Conditions. titration curves in amperometry.
37. Coulometry. Faraday’s Laws. Controlled potential coulometry. Coulometric titration. Application.
38. Classification of optical methods of analysis. Electromagnetic radiation - basic terms. Interaction of electromagnetic radiation with substance - absorption and emission. Electronic, vibrational and rotational spectrums.

39. Regions of the electromagnetic spectrum. Interaction of electromagnetic radiation with matter. Optical methods of analysis.
40. Principles of quantitative and qualitative optical methods. Calibration.
41. Atomic spectroscopy: atomic absorption, emission and mass spectrometry. Principles, analytical characterization and application of the methods.
42. UV-VIS molecular absorption spectrometry. Quantitative and qualitative analysis.
43. Infrared spectroscopy. Application.
44. Extraction. Classification. Distribution coefficient. Application.
45. Chromatography. Principle. Classification. Thin-layer chromatography (TLC). Application.
46. Liquid chromatography. Principle. Application.