Study program: Integrated Academic Studies in Pharmacy

Course title: Instrumental Pharmaceutical Analysis

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Course status: Compulsory

ECTS Credits: 8

Condition: Physical chemistry, Organic chemistry I

Course aim

The main goal of Instrumental Pharmaceutical Analysis course is introduction of students with principles of instrumental analysis and assumption of knowledge about theory and principles on which techniques of analytical measurements are established. Understanding of the analytical instruments, their parts and the way measurements are performed. It is necessary for students to learn the skills for practical applying of knowledge learned in theory. In the laboratory, students practically use learnt measurement techniques and become experienced in modern analytical techniques.

Expected outcome of the course:

It is necessary that student comprehend theory and practice of instrumental measurements, as well as consecutive steps in pharmaceutical analysis. Instruments – parts and operation principle, amenities and limitations of different measurement techniques. Estimation of analytical errors and statistical analysis.

Appliance of knowledge into the practice. Preparation of analytical procedure and definition of consecutive steps of analysis. Qualitative and quantitative analysis of the samples. Use of techniques for the increase of the accuracy and precision of measurements. Data analysis, estimation of the errors and presentation of the results.

Course description

Theoretical education

1. Introduction to instrumental pharmaceutical analysis- Classification of analytical methods. Characteristics of analytical methods, idioms in analytical process. Quality control of analytical methods, control of faults.
2. Introduction of spectroscopy methods - electromagnetic rays, absorption and emission of radiation, spectra, terms, types of instruments.
3. Atomic Spectroscopy - Atomic absorption spectroscopy (AAS)- principle, appliance in pharmaceutical analysis, interference in AAS. Atomic emission spectroscopy (AES) - principle, instruments, applications, interference in AES analysis. Inductively coupled plasma (ICP)- principle, application, in pharmaceutical analysis, characteristics.
4. Luminescent spectroscopy – Theory of phosphorescence and fluoroscence. Excited states. Emission and excitation spectra. Instruments. Application.
5. Ultraviolet and visible spectroscopy (UV/VIS) - UV/VIS radiation, absorption. Lambert-Beer formula and its limitations, UV spectra, chromophores, instruments, qualitative and quantitative analysis.
6. Infrared spectroscopy (IR)- principle, molecule vibrations, spectra, interpretation, absorption- factors which influence absorption, instruments, preparation of the samples. Applications, examples of molecule spectra of pharmaceuticals.
7. Mass spectroscopy- theory of molecule mass spectroscopy, molecule fragmentation, homolytical and heterolytical α-cleavage. Instruments, ionization techniques, EI, PICI, NICI. Ions in spectra, isotopic peaks, transfer of protons. Application, mass spectra of some pharmaceuticals.
8. Nuclear magnetic resonance spectroscopy (NMR) - Theory of nuclear magnetic resonance. Types of NMR spectra. NMR instruments. Applications.
9. Chromatography- introduction to chromatography techniques, classification of chromatography methods. Theory of chromatography, parameters, (indexing ratio, selectivity factor, resolution factor, number of theoretical plates). Principle of the separation, Rf value, applications.
10. Thin layer chromatography (TLC) - purpose of TLC, stationary and mobile phases, types of detections. Applications, examples. High performance thin layer chromatography (HPTLC) - principle, applications.
11. High performance liquid chromatography (HPLC) - purpose of HPLC, instruments, principle. Partition, adsorption, ion-exchange and size exclusion chromatography. Stationary phase, types of interaction, normal and reversed phased chromatography systems. Mobile phase, isocratic and gradient elution. Influence of polarity, lipophility and pH values on elution. Qualitative and quantitative analysis, applications.
12. Gas chromatography (GC) - principle of gas chromatography, instruments. Types of columns and stationary phases. Selectivity of liquid stationary phase, retention index, system. Kovats index. Joining of the gas chromatography with spectroscopy methods.

13. Capillary electrophoresis (CE) - principle of electrophoretic separation. Instruments. Applications of CE in pharmaceutical analysis. High performance capillary electrophoresis (HPCE).
14. Preparation of the samples. Liquid-liquid extraction, principle, applications. Solid phase extraction (SPE), types of adsorbents, methodology, application.
15. Electrochemistry - Introduction to electroanalytical chemistry, electrochemical cell, potential in electrochemical cell, electrode potential, current in electrochemical cell. Types of electrodes. Potentiometry. Direct potentiometric measurements, potentiometric titrations. Introduction to voltammetry.

Practical education

1. Introduction to organization and laboratory practice in instrumental pharmaceutical analysis - Rules and handlings in laboratory practice, organization of laboratory work, potential risks, safety. Performance of basic operations in laboratory practice.
2. Statistical analysis of data- evaluation of analytical parameters, standard deviation, coefficient of variation, recovery, error. Calculation of the parameters of calibration curve. Parameters of statistical analysis. Methods of quantification (external standard, internal standard, standard addition).
3. Ultraviolet and visual spectroscopy (UV/VIS) - Instrument, preparation of samples, measurement techniques. Qualitative analysis, interpretation of UV/VIS spectra. Chromophores. Influence of the polarity, types of solvent and pH values on spectra. Quantitative analysis, Lambert-Beer law. Calculation of molar and specific absorption coefficient. Determination of concentration of acetylsalicylic and ascorbic acid. Data processing and discussion of results.
4. Infrared spectroscopy (IR) - Instrument, preparation of solid and liquid samples, techniques of measurements. Qualitative analysis. Interpretation of IR spectra, identification of characteristic bands. Measurement, interpretation and identification of spectra of pharmaceuticals.
5. Atomic spectroscopy - theoretical basis, instrument. Measurement techniques.
6. Thin layer chromatography (TLC) – TLC apparatus, plates for TLC. Application of the samples. Development of TLC plate, detection of the analyte. Qualitative test, retention factor. Determination of the optimal mobile phase system for efficient separation of mixtures.
7. Elution column chromatography - separation of mixture components by column chromatography. Stationary and mobile phase, preparation of chromatography column. Characteristics of separation process. Separation of leaf pigments. Eluate analysis.
8. High performance liquid chromatography (HPLC) - Instrument, preparation of samples, measurement techniques. Calculation of column parameters - capacity factors, resolution. Adjustment of analysis parameters, computer control, management and saving of the data. Qualitative and quantitative analysis. Application of liquid chromatography. Determination of vitamin C in tablets and natural products. Preparation of samples.
9. Sample preparation - Liquid-liquid extraction, principle, application. Solid phase extraction (SPE), principles, type of stationary phases, performance. Separation of colored analytes from mixture.
10. Gas chromatography – Instrument. Sample preparation – internal standard. Determination of fatty acids in dietary supplements.
11. Electrochemical methods - Potentiometry. Potentiometric titration of hydrochloric acid. Graphic management of data. Ion selective measurements. Measurements of fluorides in toothpaste and mouthwash using ion selective electrodes.

Literature

Compulsory

1. Skoog DA, West DM, Holler FJ, Crouch SR. Fundamentals of Analytical Chemistry. 9th ed. Cengage Learning; 2013.
2. Cvejić J, Dimitrovska A, Atanacković M. Instrumental pharmaceutical analysis – practicum (translated to English). Department of Pharmacy, Faculty of Medicine, Novi Sad.

Additional

1. Skoog DA, West DM, Holler FJ, Crouch SR. Analytical Chemistry An Introduction. 7th ed. Brooks Cole Thomson Learning; 2000.
2. Cairns D. Essentials of pharmaceutical chemistry. 2nd ed. UK: Pharmaceutical Press; 2003.
3. Pungor E. A practical guide to instrumental analysis. CRC press; 1995.

Number of active classes	**Theory:** 60	**Practice:** 60

Teaching methods

Lectures. Laboratory work.

Student activity assessment (maximally 100 points)

Pre-exam activities	**points**	**Final exam**	**points**
Lectures	10	Oral	40
Practices	10		
Colloquium	20	.....	
Test	20		