Study program: Integrated academic studies of Pharmacy

Type and level of the study program: integrated academic studies

Course title: INSTRUMENTAL PHARMACEUTICAL ANALYSIS (PhII-IPHA)

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Course status: compulsory

ECTS Credits: 8

Condition: Physical chemistry

Course aim

The main goal of Instrumental Pharmaceutical Analysis course is introduction of students with principals 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- spectroscopy, electromagnetic rays, absorption and emission of radiation, spectra, terms, types of instruments. **3.** Atomic Spectrometry- Atomic absorption spectrometry (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 spactra 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 (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 elution 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. Capillary electrophoresis (CE) - principle of electrophoretic separation. Instruments. Applications of CE in pharmaceutical analysis. High performance capillary electrophoresis (HPCE). **13.** Preparation of the samples. Liquid-liquid extraction, principle, applications. Solid phase extraction (SPE), types of adsorbents, methodology, application. **14.** Electroanalytical chemistry- 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: exercises, other forms of education, research related activities

1. Introduction to organization and laboratory practice in instrumental pharmaceutical analysis- Rules and handlings in laboratory practice, organization of laboratory work, potential risks, safety. Regularly performance of basic operations or practical laboratory practice. **2.** Statistical processing of data- evaluation of analytical parameters, standard deviation, coefficient of variation, recovery, relative fault. Calculation of the parameters of calibration curve. Parameters of statistical processing. Methods of quantification (external standard, internal standard, standard addition). **3.** Ultraviolet and visual spectroscopy (UV/VIS)- Instrument parts, preparation of samples, measurement techniques, Qualitative analysis, interpretation of UV/VIS spectra. Influence of the polarity, types of solvent and pH values on spectra. Quantitative analysis, Lambert-Beer formula. Calculation of unknown concentration from the data obtained by UV- spectroscopy, calculation of molar and specific absorption coefficient. Assignment of concentration of acetylsalicylic and ascorbic acid. Managing of the data and presentation of the results. **4.** Infrared spectroscopy (IR)- Instrument parts, preparation of solid and liquid samples, techniques of measurements. Qualitative analysis. Interpretation of IR spectra, identification of characteristic stripes. Recording, interpretation and identification of spectra of pharmaceuticals. **5.** Atomic spectroscopy- theoretical basis, instrument parts. Techniques of measurements. **6.** Thin layer chromatography (TLC)- Equipment for thin layer chromatography performance, preparation of layers. Infliction of the samples. Chromatogram development, detection of the analyte on plates. Qualitative test, retention time. Determination of the optimal mobile phase system for efficient separation of mixture components. **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 life pigments. Eluate analysis. **8.** High performance liquid chromatography (HPLC)- Instrument parts, 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. Assignment of vitamin C in tablets, lemon and multivitamin preparation. Preparation of samples- solid phase extraction (SPE), principles, type of stationary phases, performance. **9.** Preparation of samples- Liquid-liquid extraction, principle, usage. Solid phase extraction (SPE), principles, type of stationary phases, performance. Separation of colored analytes from mixture. **10.** Electroanalytical chemistry- calculation of pH value, degree of ionization, acidoalcalimetry. Potentiometry. Potentiometric titration of hydrochloric acid. Graphic management of data. Ion selective measurements. Measurements of fluorides in toothpaste using ion selective electrodes. Graphical presentation of obtained data.

Literature

Compulsory

1. Skoog DA, West DM, Holler FJ, Crouch SR. Fundamentals of Analytical Chemistry (9th Edition). Brooks/Cole Thomson Learning-USA, 2013.
2. Cvejić J, Dimitrovska A, Atanacković M. Instrumental pharmaceutical analysis-practicum, Ortomedics, Novi Sad, 2010. (translated from Serbian version into English).

Additional

1. Skoog DA, West DM, Holler FJ, Crouch SR. Analytical Chemistry An Introduction (7th Edition). Brooks/Cole Thomson Learning-USA, 2000.

Number of active classes				Other:
Lectures: 60	Practice: 60	Other types of teaching:	Research related activities:	

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		