Study program: Integrated Academic Studies in Pharmacy

Course title: Drug Analysis

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Course status: Compulsory

ECTS Credits: 4

Condition: Pharmaceutical chemistry III, Pharmacognosy II

Course aim

The main aim of the subject is introduction with analysis and control procedure of drug substance and final drug product, domestic and international regulatory rules and validation of analytical methods. Also, the aim is to gain knowledge about applications of analytical methods in pharmaceutical analysis, methods of analysis of various pharmaceutical forms and relationship between purpose of analysis and selection of analytical technique. Analysis of major active component(s), excipients and impurities. Student develops skills for practical laboratory work.

Expected outcome of the course:

Student learns about application and choice of analytical methods in analysis of actual samples. Knowledge about methods and phases of creation of specification of certain drug. Usage of Pharmacopoeia, specifications, law and regulatory rules. Approach to analysis as function of characteristics and quality of analyzed pharmaceutical form/drug substance. Methods of evaluation of analytical error and statistical data analysis. Real sample preparation and analysis of pharmaceutical forms.

The ability of choosing appropriate analytical method. Searching, interpretation and handling information necessary for appropriate analysis of samples. Preparation and defining procedure considering the aim and the purpose of the analysis. Practical performance of sample analysis. Data analysis, error evaluation and final results expression.

Course description

Theoretical education

Introduction in drug analysis. Law and regulatory rules. International Conference on Harmonization (ICH). Good laboratory practice (GLP). ISO 17025 standards. Pharmacopoeia-monographs. Physical and chemical properties of drug molecules, identification of drug substances, pH value, ionization of drug molecules and pKa value. Partition coefficient. Drug stereochemistry, polarimetry, refractometry. Physical constants- specific optical rotation, melting point, freezing point. Impurities in drug substances and products - organic and non-organic impurities. Residual solvents. Enantiomer impurities. Degradation products. Application of spectroscopy methods in pharmaceutical analysis - ultraviolet and visible spectroscopy (UV/Vis)-differential spectroscopy, multicomponent analysis. Interferences, correction techniques, derivative spectroscopy, determination of pKa value and solubility, dissolution test. Infrared spectroscopy (IR). Application of separation techniques in pharmaceutical analysis- review of separation methods. HPLC-special applications, chromatography with anionic/cationic ionic coupling agent, exclusion chromatography, ion-exchanging chromatography derivatization, separation of enantiomers with GC- derivatization chiral selectivity,. Determination of residual solvents-pharmacopoeia procedure, "head space" and "purge trap" analysis. Thin-layer chromatography-limit tests, determination of impurities in pharmaceutical products. Capillary electrophoresis. Extraction methods. Solid phase extraction. Drug analysis in pharmaceutical forms - sample preparation from different matrices. Characteristics of analysis of different pharmaceutical forms. Validation of analytical methods- Strategy for validation. Validation parameters - accuracy and precision, repeatability and reproducibility, range, linearity, limit of detection and quantification, robustness. Validation process. Providing drug quality - Drug quality. Drug specification, definition. Specification in different phases of drug development. Manufacturing and distribution of drugs. Quality control of drug products. Laboratory analysis. Releasing drug to the market, drug embargo, reclamation and drug withdrawal. Drug registration.

Practical education

Laboratory practice involves quality control of solvents, active drug substances and different pharmaceutical forms and evaluation of obtained results in accordance with appropriate regulation. Quality control of solvents - Characters, identification, physical and chemical properties, microbiological purity. Distilled water, 96% ethanol, European Pharmacopoeia regulation. Quality control of chemical substances, active pharmaceutical ingredient - appearance, identification, physical and chemical properties, impurities, assay, labelling. Sodium chloride, boric acid, European Pharmacopeia regulation. Quality control of liquid preparations for cutaneous application - appearance, identification, physical and chemical properties, impurities assay, microbiological purity. *Iodi solutio aquosa, Iodi solutio aethanolica, Acidi borici sol.,* European Pharmacopoeia regulation or manufacturer specification. Quality control of parenteral preparation - appearance, identification, physical and chemical properties, purity, assay, content uniformity, sterility,

bacterial endotoxins. Analysis, glucose infusion, according to manufacturer specification. Quality control of liquid preparations for oral use - appearance, identification, physical and chemical properties, purity, assay, content uniformity, preservative content, microbiological purity. Analysis of syrup/powder for syrup, according to manufacturer specification. Quality control of tablets and capsules - appearance, identification, physical and chemical properties, purity, assay, content uniformity, dissolution test, microbiological purity. Glibenclamide tablets, ampicillin capsules and folic acid tablets according to manufacturer specification. Validation of analytical methods. Analytical procedures that require validation. International regulations for validation of analytical methods, validation protocol. Validation of methods for quality control of pharmaceutical forms (HPLC). Validation documentation. Additional analysis methods. UV/Vis spectroscopy - content of ascorbic and acetylsalicylic acid in effervescent tablet, multicomponent analysis. Drug registration. Requirements, procedure and conditions for releasing drug on the market, required documentation.

Literature

Compulsory

1. Watson DG. Pharmaceutical analysis - A textbook for pharmacy students and pharmaceutical chemists. 4th ed. Elsevier; 2016.

Additional

1. Ahuja S, Scypinski S. Handbook of modern pharmaceutical analysis. 2nd ed. Amsterdam: Elsevier; 2011.
2. Cairns D. Essentials of pharmaceutical chemistry. 4th ed. UK: Pharmaceutical Press; 2012.
3. Snyder LR, Kirkland JJ, Glajch JL. Practical HPLC method development. 2nd ed. John Wiley & Sons; 1997.

Number of active classes	**Theoretical classes:** 30	**Practical classes:** 60

Teaching methods

Lectures. Laboratory work.

Student activity assessment (maximally 100 points)

Pre-exam activities	**points**	**Final exam**	**points**
Lectures	10	Written	60
Practices	10	Oral	
Colloquium	20	.....	