

NP18-22-Pharmaceutical Analysis I

Aims of the course: The course enables students not only to identify medicines (active ingredients) but also to quantify them in their pure form, in pharmaceutical formulations and in biological liquids (active ingredients and metabolites). Ultraviolet spectrophotometry is considered a very useful technique in Pharmaceutical Analysis because it is highly sensitive (due to electronic excitations-orbitals are involved in electronic transitions). Furthermore, identification of medicines is carried out on the basis of their various chromophore groups (the chromophore concept) because they provide (give) specific spectrum which is characterized from its fine structure: maxima, minima, diversions, shoulders, width of electronic excitations, that is, width in the spectrum where the electronic promotions occur, values of specific absorption coefficient, $A_{1\%, 1\text{cm}}$, ratios of absorption intensities in specific maxima, minima or combination of them, pH solution etc). The method, because of the above mentioned advantages, can be combined with High-Performance Liquid Chromatography in order to detect, identify and quantify the medicines in the presence of the various impurities and metabolites. Ultraviolet spectrophotometry is described extensively in various Pharmacopoeias (Greek, European etc) as a useful means for the identification of drugs, their purity control and the quantitative determinations of various medicines in pharmaceutical formulations. Also, instrumentation is described. This kind of knowledge is considered fundamental and essential for a professional employment of a Pharmacy graduate in a Pharmaceutical Analysis Laboratory.

Skills: To become acquainted with basic concepts of Pharmaceutical Analysis.

Teaching methods: Lectures and Laboratory practice. The laboratory practice has been enhanced so that students can be acquainted with and become more familiar with the spectra of the medicines and with the additional use of computers to make possible the supervised self-study, which is very useful for the training of the students.

Contents of the course:

Principles of instrumental methods. Classification of instrumental analytical methods. General characteristics of instrumental methods that are used in Pharmaceutical Analysis. Energy and the electromagnetic spectrum. The nature of the radiated energy (ultraviolet/visible is a form of energy, which can be described by two complementary theories: the wave theory and the corpuscular theory. Classification of spectroscopic analytical techniques which can be applied in Pharmaceutical Analysis. Generally, about spectroscopic methods.

Control of the quality of analytical methods: Introduction, errors, Accuracy and precision, Validation of an analytical procedure, Standard operating procedure (SOP), basic calculations

Ultraviolet spectrophotometry: Absorption and emission spectra. Principles, applications and methods. Study of ultraviolet spectrum of various representative organic molecules (called chromophores) which are of particular interest in Pharmaceutical Analysis (spectra of benzen, aniline, pyridine, phenol and diphenols, barbituric acid and its derivatives, xanthenes, hormones, corticosteroids (steroid enones), anti-inflammatory, anti-histamine).

Quantifications through ultraviolet spectrophotometry: The Beer-Lambert Law. The importance of specific absorption coefficient in quantitative analysis. Spectrophotometric quantifications of active ingredients in different formulations. Polar and non-polar solvents. Stock solutions and dilutions. Methods of extraction in Pharmaceutical Analysis (liquid-liquid and solid-phase extraction). Construction of calibration graph-Standard Addition Method. Instrumentation: the light sources, the monochromator, the optics, diode array instruments, instrument calibration, calibration of absorbance and wavelength scale. Determination of instrumental resolution and stray light. Difference spectrophotometry and derivative spectra

Flame spectrophotometry and spectrophotometry of individual absorption: flames, burners nebulisers. Transmission Flame spectrophotometry. Individual absorption spectrophotometry. Instrumentation, sensitivity and detection limit. Applications

Infrared spectrophotometry, key points, instrumentation, factors determining intensity and energy, application. Near Infrared analysis, key points, instrumentation, additional problems, examples.

Fluorescence spectroscopy, key points, instrumentation, molecules which exhibit Fluorescence, applications.

Raman spectroscopy, theory, key points, instrumentation, applications.

Nuclear Magnetic Resonance Spectroscopy (NMR), theory, key points, instrumentation, proton NMR, carbon NMR, two dimensional NMR spectra, applications.

Proposed literature:

Educational activities: Attendance of the module and the laboratories, handing in of laboratory notebook.

Evaluation process and methods: Once the laboratory course is successfully completed with a pass mark(passing grade) and the laboratory notebook is handed in corrected, each student with his/her team should hand in a project on an earlier assigned topic (the project is prepared individually with the responsibility of the student) -the project should be corrected-. At the end of the academic year, students have to undergo oral exams. After having completed the above mentioned obligations, students can participate in the written examinations of the pharmaceutical analysis modules. Cooperativeness, initiative and interpersonal relationship with the instructor plays an important role in the student assessment.

Use of ICTs (Information and Communication Technologies) / Electronic distribution of the lectures: The laboratory content of the module has been enriched with electronic educational material that is special software in CD-ROM format which contains the spectra of some representative medicines as well as their spatial configurations, that is, the chemical structure in space (bonds length, ring arrangement, various bonds angles) so that the relation of the structure of the medicine with the relevant spectrum to be obvious. Moreover, a large part of the laboratory practice is done by the students with the application of a special UVPC programme.

The lectures of the modules are enriched by multimedia use (power point presentations, videos etc).

Teaching:

The module is taught both through lectures and laboratory practice.

A) **Lectures.**

The lectures, which last for two hours, take place twice a week in Classroom D12 and multimedia are used to present the topics to be dealt with.

Lecture	Title	Tutor
1	Principles of Instrumental Pharmaceutical Analysis, Electromagnetic Radiation	Catherine K. Markopoulou
2	Control of the quality of analytical methods	Catherine K. Markopoulou
3	Control of the quality of analytical methods	Catherine K. Markopoulou
4	Ultraviolet spectrophotometry	Catherine K. Markopoulou
5	Study of UV spectra (theory needed to interpret Spectra)	Catherine K. Markopoulou
6	<i>Infrared spectrophotometry</i>	Catherine K. Markopoulou
7	<i>Infrared spectrophotometry</i>	Catherine K. Markopoulou

8	<i>Fluorescence spectroscopy</i>	Catherine K. Markopoulou
9	<i>Nuclear Magnetic Resonance Spectroscopy (NMR)</i>	Catherine K. Markopoulou
10	<i>Nuclear Magnetic Resonance Spectroscopy (NMR)</i>	Catherine K. Markopoulou
11	Flame Photometry and atomic absorption spectrophotometry	Catherine K. Markopoulou
12	Flame Photometry and atomic absorption spectrophotometry	Catherine K. Markopoulou
13	Raman spectroscopy	Catherine K. Markopoulou

B) Laboratory work

La b	Title: Pharmaceutical Analysis	Tutor
1	Ultraviolet Spectrophotometry, Indroduction, Instrumentation	John E. Koundourellis Catherine K. Markopoulou
2	a) Study of Benzene UV spectra with changes in the monochromator slit width. b) Study of UV spectra of acetylsalicylic acid, phenol, naphthalene, naphazoline ,etc	John E. Koundourellis Catherine K. Markopoulou
3	Changes in UV absorption spectra due to methyl substitution on the aromatic ring of xylene, use of UVPC software	John E. Koundourellis Catherine K. Markopoulou
4	Application of (Standard addition method) to perphenazine, amitriptyline, cyproterone acetate using the UVPC software	John E. Koundourellis Catherine K. Markopoulou