

PHARMACEUTICAL ANALYSIS II

Course type: ΝΠ18-29

	Background/General knowledge
X	Scientific area (pharmacy)

Credit Units (ECTS): 5.5

Lectures (hours/week): 3

Tutorial (hours/week): -

Laboratory work (hours/week): 1.5

Course coordinator: Markopoulou Catherine, associate professor,

Tutor (s):

- 1) Catherine K. Markopoulou Office 2nd floor, Pharmacy/Biology building ,
Cooperation with the students: Wednesday and Thursday 11:00 a.m.-1:00 p.m. e-mail:
amarkopo@pharm.auth.gr/ office telephone 2310 99 76 65/laboratory telephone 2310 99 76 67
- 2) Constantinos K. Zacharis, Assistant Professor, Office 2nd floor, Pharmacy/Biology building
, Cooperation with the students: e-mail: czacharis@pharm.auth.g/ office telephone 2310
99 76 63/laboratory telephone 2310 99 76 67

Aims of the course: The module enables students not only to identify medicines but also to quantify them in their pure form in pharmaceutical formulations and in biological fluids through various techniques such as **mass spectrometry** and chromatography (**thin layer, open column chromatography, gas chromatography and high-performance liquid chromatography**), high performance liquid chromatography, capillary electrophoresis etc . Particular emphasis is given to the various sample pretreatment techniques and methods used. This kind of knowledge is considered fundamental and essential for the possible future professional employment of a pharmacy graduate in a Pharmaceutical Analysis Laboratory.

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

Teaching methods: Lectures and laboratory practice. The laboratory practice has been enriched so that students can be acquainted with and become more specialized in understanding the spectra of different medicines` hence 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:

Chromatography: Introduction of chromatography, categorization of chromatographic techniques and nomenclature, chromatographic parameters, calculation of column yield, origin bandwidth in HPLC. Parameters that evaluate column performance, download / edit data.

Gas chromatography: Introduction, principle of the method, Organology, Selectivity of liquid static phases, Use of derivatization, Parameters affecting capillary performance, Detectors, Applications of GC in Quantitative Analysis, Determination of production residues and degradation by GC. Determination of solvent residues, solid phase microextraction, applications in bioanalysis.

High Performance Liquid Chromatography: Introduction. Organology. Stationary and mobile phases. Structural factors that determine the elution of the compounds from the columns. Advanced estimation of solvent selectivity in reverse phase chromatography. Effect of temperature on HPLC, Summary of static phases. Advanced assessment of reverse phase stationary phases. Detectors' summary. Applications of HPLC for quantitative drug analysis. Specific analytical techniques.

Thin layer chromatography: Introduction, Organology, TLC chromatogram. Static phases, Elution series and mobile phases, Modification of TLC adsorbents. Detection of compounds on plates after development. Applications. High performance thin layer chromatography (HPTLC).

Mass spectrometry: Introduction. Principle of the method. Ion production. Ionization methods. Ion separation techniques. Mass spectra. Molecular fragments. Gas chromatography-mass spectrophotometry. GC-MS applications with EI. Coupled tandem mass spectrometry. High

resolution mass spectrometry. Protein mass spectrometry. Mass spectrometry in the discovery of drugs.

Capillary electrophoresis: Introduction, organology, electro-osmotic flow, electrofusion, separation control, enantiomer separation. Use of additives (cyclodextrins, surfactants) in the carrier buffer, detection items, applications of capillary electrophoresis in pharmaceutical analysis.

Extraction methods in pharmaceutical analysis: Common excipients of pharmaceutical preparations, liquid-liquid extraction, microdialysis extraction, derivatization, solid phase extraction, adsorbents. Automated solid phase extraction, recent developments

Educational activities: Attendance of the lectures and the laboratories, handing in of the 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 has been enriched with electronic educational material, that is, special software in CD-ROM format which contains the spectra of the medicines as well as their solid chemical structure in space (bonds length, ring arrangement, various bonds angles) so that the relation of the medicine structure 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 course 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 is used to present the topics to be dealt with.

Lecture	Title	Tutor
1	Chromatography (Introduction, classification) Chromatographic parameters	C.K. Markopoulou C.K. Zacharis
2	Mobile / stationary phase, Solvents, Analytes, Development of chromatographic techniques	C.K. Markopoulou C.K. Zacharis
3	Thin layer chromatography	C.K. Markopoulou C.K. Zacharis
4	Gas chromatography	C.K. Markopoulou C.K. Zacharis
5	Gas chromatography	C.K. Markopoulou C.K. Zacharis
6	Liquid Chromatography/ properties of analytes	C.K. Markopoulou C.K. Zacharis
7	Liquid Chromatography	C.K. Markopoulou C.K. Zacharis
8	Mass Spectrometry	C.K. Markopoulou C.K. Zacharis
9	Mass Spectrometry	C.K. Makopoulou C.K. Zacharis
10	Mass Spectrometry	C.K. Markopoulou

		C.K. Zacharis
11	Capillary electrophoresis	C.K. Markopoulou C.K. Zacharis
12	Capillary electrophoresis	C.K. Markopoulou C.K. Zacharis
13	Extraction techniques in Pharmaceutical Analysis	C.K. Markopoulou C.K. Zacharis

B) Laboratory work

Lab	Title	Tutor
1	Determination (using the calibration curve) of furosemide in pharmaceutical formulations (tablets). Study with UV Spectrophotometry (Part A)	C.K. Markopoulou C.K. Zacharis
2	Determination (using the calibration curve) of furosemide in pharmaceutical formulations (tablets). Study with UV Spectrophotometry (Part B)	C.K. Markopoulou C.K. Zacharis
3	Study of UV spectrum of bromhexine hydrochloride and its calibration curve. Dilution factors, calculations. Overall Recovery	C.K. Markopoulou C.K. Zacharis
4	Study UV spectrum of Ifeprodil. Standard addition method. UV spectrum derivative techniques	C.K. Markopoulou C.K. Zacharis