

<b>2023/2024. ACADEMIC YEAR</b>	
<b>PROGRAM OF STUDY (FOR STUDENTS OF 4TH YEAR)</b>	
<b>Full (Hun) name of the subject: MŰSZERES GYÓGYSZERANALÍZIS</b>	
<b>Program: Undivided program (pharmaceutical)</b>	
<b>Schedule: full-time</b>	
<b>Short name of the subject: MGYA</b>	
<b>English name of the subject: Instrumental Pharmaceutical Analysis</b>	
<b>German name of the subject: Instrumentelle Arzneimittelanalytik</b>	
<b>Type of registration: obligatory/obligatory elective/elective/criteria requirement</b>	
<b>Neptun code of the subject: GYKGYK285E1A</b>	
<b>Responsible Department: Department Of Pharmaceutical Chemistry</b>	
<b>Responsible tutor</b> <b>Dr. György Tibor Balogh</b>  <b>Contact information: Semmelweis University, Department of Pharmaceutical Chemistry</b> 1092 Budapest, Hőgyes Endre u. 9.  - <b>phone:</b> 06-1-217-0891 - <b>email:</b> <a href="mailto:balogh.gyorgy.tibor@semmelweis.hu">balogh.gyorgy.tibor@semmelweis.hu</a>	<b>Title, academic degree:</b>  <b>Head of department, DSc, PhD, dr. habil</b>
<b>Name of the persons responsible for the teaching of the subject:</b> <b>Dr. György Tibor Balogh</b> <b>Dr. Péter Horváth</b> <b>Dr. István Antal</b> <b>Dr. László Órfi</b> <b>Dr. Károly Mazák</b> <b>Dr. Márta Kraszni</b> <b>Dr. Gergő Tóth</b> <b>Dr. Arash Mirzahosseini</b> <b>Dr. Tamás Páll</b> <b>Dr. Marcell Krekó</b> <b>Dr. Vivien Bárdos</b> <b>Szabina Kádár</b> <b>Dr. Balázs Simon</b> <b>Dr. Rita Szolláth</b>	<b>Title, academic degree:</b>  <b>Head, full professor, PhD</b> <b>Associate professor, PhD</b> <b>Dean, full professor, PhD</b> <b>Full professor, PhD</b> <b>Associate professor, PhD</b> <b>Associate professor, PhD</b> <b>Adjunct, PhD</b> <b>Adjunct, PhD</b> <b>Assistant professor, PhD</b> <b>Assistant professor,</b> <b>PhD student</b> <b>PhD student</b> <b>PhD student</b> <b>PhD student</b>
<b>Class per week: 2 hours lectures</b> <b>5 hours practices</b>	<b>Credit point(s): 2 credits lectures</b> <b>4 credits practices</b>
<b>Professional content, intent of acquirement and it's function in order to implement the goals of the program:</b>  The theoretical presentation and practical application of modern routine and large-scale analytical techniques used in the pharmaceutical industry, as found in the monograph of the pharmacopoeia, aiming to provide a comprehensive understanding of the testing of pharmaceutical raw materials, intermediates, and products produced through small, medium, and large-scale processes. Additionally, this encompasses the interpretation of the advanced instrumental methods employed in various sections of the Pharmacopoeia. Another objective is to equip our students for easier integration into the pharmaceutical industry.	

<b>Short description of the subject:</b> The fundamental objective of the course is to acquaint students with the essential routine and large-scale analytical measurement methods necessary for determining the quality of pharmaceutical preparations and raw materials. Students will become familiar with the construction and operation of various instruments. They will gain insight into the potential applications of each instrument and, through hands-on experience, acquire knowledge of the key instrumental techniques in pharmaceutical analysis.							
<b>Course data</b>							
Recommended term	Contact hours (lecture)	Contact hours (practice)	Contact hours (seminar)	Individual lectures	Total number of contact hours/semester	Normal course offer	Consultations
7th semester	28	70	-	-	98	Autumn semester* Spring semester Both semesters (* Please underline)	-
<b>Program of semester**</b>							
<b>Topics of theoretical classes (pro week):</b>							
1 UV/VIS spectroscopy							
2 Electroanalytical methods (potentiometry, amperometry)							
3 IR, Raman spectroscopy							
4 HPLC I.							
5 HPLC II.							
6 ORD, CD spectroscopy							
7 Capillary electrophoresis							
8. Mass spectrometry, LC-MS							
9 HOLIDAY							
10 NMR spectroscopy I							
11 NMR spectroscopy II.							
12 HTS methods							
13 Analytical techniques in solid state characterization. Nondestructive and Process Analytical Technology (PAT) approaches							
14 Consultation							
<b>Topics of practical classes (pro week):</b>							
1 UV spectroscopy , optical rotation							
2 UV spectroscopy II, amperometry							
3 IR, RAMAN							
4 Potenciometry							
5 HPLC I							
6 ORD, CD							
7 HPLC II							
8 Capillary electrophoresis							
9 HPLC III							
10 MS							
11 NMR							
12 NMR II							
13 Plate-based methods, permeability measurements							
14 Consultation							
<b>Other subjects (both compulsory and optional) relating to the transversal issues of the subject. Possible overlaps between subjects:</b>							
Biophysics, analytical chemistry							
<b>Schedule of consultations: based on requirements</b>							
<b>Course requirements</b>							

<p><b>Prerequisites:</b> Pharmaceutical Chemistry and Analysis II. Pharmaceutical Technology II.</p>
<p><b>Conditions of attending the classes, amount of acceptable absents, way of presentation of leave, opportunity for makeup:</b> Based on study and examination regulations</p>
<p><b>The grading method; the conditions for getting the signature; the number, topic(s) and date(s) of the mid-term assessments, (reports, term tests), and the process in which they contribute to the final grade; and the possibility of their retake or their upgrading retake (as provided in §§ 25-28 of the STUDY AND EXAMINATION REGULATIONS):</b> Throughout the semester, there will be 3 midterms (in the 5th week, 9th week, and 13th week), the results of which must achieve an average of at least 2.0 without rounding. The topics will be detailed in the topic list provided at the beginning of the semester (via Moodle).</p>
<p><b>Requirements of signature</b>(as provided for in STUDY AND EXAMINATION REGULATIONS § 29):</p> <ol style="list-style-type: none"> <li>1) It is compulsory to be present on the practices.</li> <li>2) Maximum 3 absences are acceptable. In case of a further absence the semester has to be repeated.</li> <li>3) The practices begin and finish punctually. Latecomers can be sent home by the instructor.</li> <li>4) You have to be prepared for the practices. Your preparation can be checked by the instructor at any time, without former notification, both in written and oral form. Unprepared students can be sent home by the instructor.</li> <li>5) Scheduled project reports must be written on the designated practice. Students absent during scheduled project reports must retake them within a week.</li> <li>6) A practical course grade can only be given to a student if       <ol style="list-style-type: none"> <li>a) the average of the scheduled project reports is at least 2.00 and at least 2 of them must be passed;</li> <li>b) he/she accumulates the required minimum amount of points (min 50%) during practices;</li> <li>c) the number of the absences does not exceed the acceptable limit.</li> </ol> </li> <li>7) After the end of the study programme the practical course grade can only be improved in the first week of the exam period.</li> <li>8) A laboratory report must be kept about your work in the lab. All the examination results of the unknowns must be written into this and handed in by the end of the practice or until a deadline agreed with the instructor. Otherwise, no points will be rewarded for the practice.</li> </ol>
<p><b>Number and type of projects students have to perform independently during the semester and their deadlines:</b> Completing the tasks defined in the practical curriculum, creating reports on the measurements conducted on a weekly basis. The reports must be submitted by the end of the lab session or within the timeframe agreed upon by the lab instructor and the students.</p>
<p><b>Type of the semester-end examination:</b> signature*/<u>practical grade*</u>/ comprehensive examination*/final/end-term examination*</p> <p><b>Examination requirements:</b> as published by the education-research department on the MOODLE interface by the start of the academic term.</p>

Form of the semester-end examination: written\*/oral\*/combined examination/practical examination/the assessment of completing project work (according to STUDY AND EXAMINATION REGULATIONS 30.§)\* (*Please underline*)

#### REQUIREMENTS FOR EXAM

UV/VIS Spectroscopy (Electronic excitation, HOMO-LUMO orbitals, chromophore, auxochrome, spectral shifts, solvent cut-off values, steps of spectrophotometer qualification (wavelength, intensity, linearity; scattered light); Transmittance-absorbance; Lambert-Beer law, specific and molar absorptivity (+calculation), quantitative analytical possibilities (overlapping/non-overlapping/partially overlapping spectra, difference spectroscopy +calculation examples), concept of derivative spectroscopy, its application)

ORD, CD Spectroscopy (Optical (physical) background of ORD spectrum formation, Cotton effect curve, specific rotation and calculation; Optical (physical) conditions for CD spectrum formation, chiral and chiral perturbed chromophore, rotator strength concept, TEM, TMM, and relationship to rotator strength. Comparison of CD and ORD spectroscopy, range of applicability. Induced CD, its uses, protein structural analysis, absolute configuration determination, concept of dissymmetry factor ( $g$ ) and its application)

Electroanalytical Methods (Potentiometry (description of electrode potential using Nernst and Nikolsky-Eisenmann equations, classification of indicator electrodes with examples, pH measurement with combination glass electrode (examples from the pharmacopoeia)); Voltammetry (cell setup, types of electrodes, polarographic curve and segments, cyclic voltammetry), Amperometry (titration curve, examples from the pharmacopoeia); Coulometry; Biocatalytic electrodes (three generations of amperometric glucose electrodes in detail)

Vibrational Spectroscopy IR and RAMAN (Infrared spectroscopy ranges and characteristics, normal modes of vibration, selection rules, basics of quantitative and qualitative analysis, IR spectrum, ATR measurement setup, FT-IR, sample preparation and measurement setups, theory of Raman spectroscopy, Raman scattering, Raman spectrum, Raman spectrometer setup, comparison of NIR and Raman spectroscopy, chemical mapping, applications)

HPLC (classification of chromatographic methods, separation mechanisms, HPLC instrument structure, concepts, detection methods, comparison of UPLC and HPLC, causes and consequences of peak broadening in HPLC, reversed-phase chromatography, C18 column classification, normal-phase chromatography, normal-phase fillers, HILIC, polar organic mode, chiral chromatography, main chiral selectors)

MS, LC-MS (definitions: mass spectrometry, monoisotopic mass, mass spectrum, molecular ion, base peak, fragment ion, mass spectrometer structure, ion sources (electron impact, chemical, ESI, MALDI), comparison of ionization types, analyzers (TOF, Q), comparison of analyzers, tandem mass spectrometry, uses of mass spectrometry, qualitative LC-MS)

Capillary Electrophoresis (apparatus structure, separation principles, electroosmotic flow, factors influencing electroosmotic flow, injection, detectors, UV detection in capillary electrophoresis, CZE, MEKC, chiral capillary electrophoresis, capillary electrochromatography)

NMR (spin, spin quantum number, and magnetic quantum number relationship, NMR-active/inactive nuclei, precession, magnetic energy levels, Larmor frequency (formula), NMR experiment, FID definition and characteristics, NMR spectrum, instrument structure, calculation of chemical shift,  $^1\text{H}$  and  $^{13}\text{C}$  chemical shift scales, spin-spin coupling, Karplus equation, multiplicity of NMR signals, magnetic equivalence, splitting caused by equivalent and non-equivalent nuclei, intensity/integrated area of NMR signals, relaxation definition and types, inversion recovery pulse sequence and T1 relaxation time measurement, effect of field strength on spectrum, ways to increase signal-to-noise ratio,  $^{13}\text{C}$  acquisition modes and their comparison in terms of sensitivity and quantitiveness, nuclear Overhauser effect, basis of quantitative NMR measurement, advantages over other analytical methods, parameters for quantitative NMR acquisition and processing,  $^{13}\text{C}$  satellite signal, inverse gated decoupling mode, actual concentration determination, criteria for quantitative NMR standard materials and 2 examples, determination of hydroxypropylbetadex molar substitution by NMR)

Solid-Phase Analysis (DSC, XRD, thermoanalytical methods, distinction between amorphous and crystalline forms)

Plate-Based Methods (HTS method principles, practical applications, possibilities of permeability determination)

**The possibility and conditions for offering grades:**

There are no possibilities for offering grades.

**A list of the basic notes, textbooks, resources and literature that can be used to acquire the knowledge necessary to master the curriculum and to complete the assessments, ~~\*\*\*\*~~with exact description about which of them is required to acquire which part of the syllabus (e.g. description based on topics)), as well as the main technical and other aids and study aids that can be used:**

All the lectures will be available in the Moodle system with the syllabus and main questions regarding each topic.

**In the case of a subject lasting more than one semester, the position of the teaching/research department on the possibility of parallel enrolment and the conditions for admission\*\*\*\*:**

yes\*/no\*/on and individual assesment basis\* (*Please underline*)

**The course description was prepared by::  
Dr. Gergő Tóth**