



General information	
Academic subject	Laboratory of synthesis, extraction and drugs analysis
Degree course	CTF
Year of study	IV
European Credit Transfer and Accumulation System (ECTS)	9
Language	Italian
Academic Year	2022-23
Academic calendar (starting and ending date)	2° semester (February- June 2022)
Attendance	yes

Professor/ Lecturer	
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Department and address	Dept. of Farmacia – Scienze del Farmaco
Virtual headquarters	
Tutoring (time and day)	Agreed via email with the teacher

Syllabus	
Learning Objectives	Acquisition of the main techniques of extraction, purification and characterization of substances of pharmaceutical chemical interest.
Course prerequisites	Basic knowledge of General Chemistry, Organic Chemistry, Physical Methods in Organic Chemistry
Contents	Course program: Identification of molecules in the pure state and of organic substances in mixture. Purity Assessment Assays. Outward appearance of the substance. Determination of the solubility in water, ethyl acetate, acidic and basic aqueous solutions. Melting point and method of determination. Study of two-component solid mixtures. Sublimation, simple and fractional crystallization. Vapor pressure and evaporation, boiling, determination of the boiling point. Distillation of multi-component systems: simple, fractional and steam distillation. Continuous and discontinuous solvent extraction; extraction with supercritical fluids; solvent extraction accompanied by sonification and microwaves; solid phase extraction. Fractionation of organic mixtures and sorting of mixtures according to Staudinger. Identification reactions of substances listed in the Pharmacopoeia. Theoretical aspects of chromatography: separation mechanisms and chromatographic parameters (distribution constant, capacity factor, selectivity, efficiency, resolution, capacity. Van Deemter equation). Adsorption chromatography: stationary phases (types and characteristics), mobile phases (elutrope series), operating modes. Gel filtration chromatography: GFC parameters, calibration curve, gel structure, compatible mobile phases. Ion exchange chromatography: chemical-physical structure of the matrix, anion or cation exchangers, particle size, swelling capacity, exchange capacity, compatible mobile phases. Thin layer chromatography: operative technique and qualitative analysis (delay factor). Gas chromatography: theoretical principles, instrumentation, stationary phases. TCD, FID, ECD, MS detectors (sensitivity, selectivity, stability, response time, background noise, signal drift, detection limit, linearity range). Derivatization in gas chromatography. Combustion analysis. Qualitative-quantitative analysis in gas chromatography. High performance liquid chromatography (HPLC): theoretical



	<p>principles, instrumentation, stationary phases (normal phase, inverse phase), detectors (UV, refractive index, fluorescence, LC-MS interface). Analysis of chiral drugs: general aspects, polarimetry (polarized radiation, alpha-D, polarimeter, qualitative and quantitative uses), chiral stationary phases (Pirkle type; cellulose, cyclodextrins, corona ethers, ligand exchange, proteins, macrocyclic antibiotics). Synthesis and preparation of drugs: glassware and equipment. Synthetic procedures of reactions in anhydrous environment, at high pressure, at low temperature, with reflux heating. Examples: Grignard reaction, esterification, nucleophilic substitution in anhydrous and non-anhydrous environment, reduction with Nickel Raney, Friedel-Crafts acylation, Suzuki reaction. Microwave-assisted synthesis. Synthesis in unconventional solvents (DESS)</p> <p>Laboratory exercise program: Solubility; Determination of the melting point; Purification methods: crystallization, sublimation, distillation; Benzoic acid purification by crystallization; Recognition of some cations; Recognition of phenols, aromatic amines, carbonyl compounds; Recognition essays reported in F.U.; Unknown tests on organo-metallic substances and organic substances F.U.; TLC with Eluotropa series; Extraction and separation of a mixture, recognition of separated substances, through assays and TLC. Extraction of caffeine from tea leaves. Synthesis in the laboratory: ketone reduction reaction; esterification of carboxylic acids, microwave-assisted synthesis of aspirin. Synthesis in unconventional solvents (DESS)</p>
Books and bibliography	<ul style="list-style-type: none">- R. Cozzi, P. Protti, T. Ruaro. Elementi di Analisi Chimica Strumentale. Zanichelli Editore;- O. Bruno, F. Savelli. Analisi Chimico Farmaceutica. Piccin-Nuova Libreria;- V. Cavrini, V. Andrisano. Principi di Analisi Farmaceutica. Esculapio Editore;- G. Caliendo. Manuale di Analisi Quantitativa. EdiSES Università Editore;- Any text on organic chemistry.
Additional materials	

Work schedule			
Total	Lectures	Hands on (Laboratory, working groups, seminars, field trips)	Out-of-class study hours/ Self-study hours
Hours			
225	70	30	125
ECTS			
9	7	2	
Teaching strategy			
CLASSROOM LESSONS; LABORATORY TRAINING			
Expected learning outcomes			
Knowledge and understanding on:	<ul style="list-style-type: none">• Learning of traditional qualitative techniques in organic / inorganic chemistry; ability to understand the most recent instrumental techniques of organic qualitative analysis applied to substances belonging to the Official Pharmacopoeia		
Applying knowledge and understanding on:	<ul style="list-style-type: none">• Ability to apply instrumental and non-instrumental qualitative analytical techniques for the analysis of unknown compounds belonging to the Official Pharmacopoeia		
Soft skills	<ul style="list-style-type: none">• <i>Making informed judgments and choices:</i> Acquisition of judgment skills to undertake analysis of unknown substances (F.U.) autonomously and		



	<p>independently</p> <ul style="list-style-type: none">• <i>Communicating knowledge and understanding</i>: Development of communication skills suitable for the presentation of data collected during the analysis of unknown substances; ability to report results in an analytical and synthetic way• <i>Capacities to continue learning</i>: At the end of the course, the student must have developed a good autonomy both for carrying out qualitative analyzes of unknown organic / metallic organ substances and for updating their knowledge through the study of literature, databases, etc.
Assessment and feedback	
Methods of assessment	Oral test
Evaluation criteria	<ul style="list-style-type: none">• <i>Knowledge and understanding</i>: The oral test involves the assessment of knowledge of the program with particular reference to the understanding of qualitative analytical questions• <i>Applying knowledge and understanding</i>: During the laboratory exercises, the identification of unknown substances will take place, which involves the compilation of forms assessed by the teacher• <i>Autonomy of judgment</i>: It will evaluate the student's ability to independently apply the most appropriate analytical technique in solving analytical questions administered• <i>Communication skills</i>: The oral exam will take into account the student's communication skills in relation to the course contents and their critical application• <i>Capacities to continue learning</i>: The oral exam will measure the student's learning
Criteria for assessment and attribution of the final mark	
Additional information	