UNIVERSIDAD DE LA RIOJA

ADVANCED TECHNIQUES IN STRUCTURAL DETERMINATION
2016-2017

Master Degree:	CHEMISTRY AND BIOTECHNOLOGY		760M
Course title:	ADVANCED TECHNIQUES IN STRUCTURAL DETERMINATION		5119
Year/Semester:	1st/2nd	ECTS Credits:	5

DEPARTMENT

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CONTENTS

The course is divided in three parts oriented to the structural determination of organic, inorganic and organometallic compounds using X-ray diffraction methods (solid state), nuclear magnetic resonance techniques (solution state) and computational methods, especially molecular dynamics. These complementary techniques help to the scientist to have a vision close to the reality about how the atoms of the molecules are arranged in 3D. This information is critical given the close relationship between structure and activity (SAR).

PART A. DIFFRACTION METHODS

UNIT1. Data collection: geometric data collection, data collection based on intensity (space group determination)

UNIT2. Data processing: Lorentz and polarization theories, data reduction and absorption correction, structure factor, Friedel's law, Fourier synthesis

UNIT3. Structural resolution: The phase problem, direct methods, Patterson methods, heavy atom methods

UNIT4. Refinement and results: Software (Wingx, SHELX, DIRDIF, SIR, PLATON, ORTEP, MERCURY, ENCIFER…), crystallographic files, data bases, validation of results

PART B. NUCLEAR MAGNETIC RESONANCE

UNIT1. Pulsed nuclear magnetic resonance

UNIT2. Chemical shift and coupling constants

UNIT3. Relaxation and NOE effect

UNIT4. Carbon-13 nuclear magnetic resonance and assignment techniques (Decoupling, attached proton test and DEPT)

UNIT5. 2D-Nuclear magnetic resonance: Proton-carbon heteronuclear correlations (HMBC, HSQC, HMQC), proton-proton homonuclear correlations (COSY, TOCSY, NOESY)

UNIT6. Dynamic nuclear magnetic resonance: concept, calculation of equilibrium constants, study of ligand-protein interactions using saturation transfer (STD-experiments, tr-NOESY experiments, Diffusion (DOSY).
PART C. COMPUTATIONAL CALCULATIONS. MOLECULAR DYNAMICS, QM/MM
UNIT1. General aspects of the molecular dynamic (MD) and molecular mechanical (MM)
UNIT2. MM and MD applications to the conformational analysis of interesting biological entities

REFERENCES

Title
Basic one- and two-dimensional NMR Spectroscopy
NMR spectroscopy and computer modeling of carbohydrates
The art of Molecular Dynamics Simulations
Introduction to Practice of Molecular Simulation
X-ray structure determination. A practical guide
Fundamentals of crystallography
Internet sources: NMR on-line: https://qshare.queensu.ca/Users01/sauriolf/www/webcourse/index.htm Molecular dynamics tutorials (AMBER): http://ambermd.org/tutorials/ GLYCAM web: http://glycam.org/ International Union of Crystallography: http://www.iucr.org/

EVALUATION SYSTEM

Tasks before final exam referred to resolution of structures from X-ray data (30%, unrecoverable)
Final exam (60%, recoverable)
Observation techniques (10%, unrecoverable)