GHENT UNIVERSITY

Course Specifications

Valid in the academic year 2020-2021

Molecular Structure Analysis (C004127)

Due to Covid 19, the education and assessment methods may vary from the information displayed in the schedules and course details. Any changes will be communicated on Ufora.

Course size *(nominal values; actual values may depend on programme)*

Credits 6.0 **Study time 150 h** **Contact hrs** 67.5h

Course offerings and teaching methods in academic year 2020-2021

A (semester 1)	English	Gent	lecture	22.5h
			group work	30.0h
			seminar: coached exercises	25.0h
			online seminar: coached exercises	0.0h
			online lecture	0.0h

Lecturers in academic year 2020-2021

Martins, José	WE07	lecturer-in-charge
Lynen, Frederic	WE07	co-lecturer
Van Hecke, Kristof	WE06	co-lecturer

Offered in the following programmes in 2020-2021	**crdts**	**offering**
Master of Science in Teaching in Science and Technology(main subject Chemistry)	6	A
Master of Science in Chemistry(main subject (Bio)Organic and Polymer Chemistry)	6	A
Master of Science in Chemistry(main subject Analytical and Environmental Chemistry)	6	A
Exchange Programme in Chemistry (master's level)	6	A

Teaching languages

English

Keywords

Nuclear magnetic resonance spectroscopy, X-ray diffraction, Organic mass spectrometry, Structural analysis.

Position of the course

The student gathers both theoretical and interpretative skills allowing him/her to elucidate structures of molecules based on the interpretation of data obtained from advanced 1D and 2D nuclear magnetic resonance (NMR) spectroscopy, mass spectrometric fragmentography (MS) and X-ray diffraction (XRD) analysis. The student develops skills in the use of spectral information obtained from the most common methods of NMR, MS and XRD and learns to establish integretated analysis strategies allowing to elucidate the structure of simple as well as relatively complex molecules. Regarding NMR, after a theoretical introduction, a selection of important NMR techniques is discussed, most frequently incountered in chemical literature of organic and organometallic compounds, and hence regarded within the structural analysis community as the most informative and hence find very broad applications in the elucidation of molecular structures. The focus will be on 1D and 2D NMR techniques. The modules (1) NMR spectroscopy and XRD and (2) mass spectrometry can be followed as Advanced Topics in Chemistry.

Contents

NMR spectroscopy

- Ground zero: nuclear spin, energy levels and resonance
- Towards a realistic measurement:: a collection of nuclear spins, boltzman statistics and the magnetic field together provide the bulk magnetization
- Relaxation-Pulse-Measurement-Repeat: description of the simplest NMR experiment
- Pulse and waiting times: manipulate bulk magnetization
- At home in all markets: the spin echo

- Living apart together: spin-decoupling
- Relaxation and its practical importance
- Relative and absolute quantification with NMR
- Sensitivity or lack of it and what you can do about it
- From 1 to 2 to N dimensions in NMR
- General principle, correlations throughout the chemical bond, through space and through chemical exchange.
- The diffusion dimension in NMR

XRD

- X-ray diffraction: principles
- Recording techniques
- Scattering of X-rays
- Fourier Synthesis: structure factors vs electron density
- Patterson function
- The phase problem
- Friedel's law
- Absolute configuration
- Crystal structure determination
- Crystal structures: examples

MS

- Principles of structural elucidation based on electron ionization EI-MS spectra.
- Relevance of the unit and high resolution MS and of isotopic distributions in the deterimation of the molecular ion
- Interpretation of direct fragmentation processes (sigma electron ionization, charge and radical site initiation)
- Fragmentations at the benzylic and allylic bond, rearrangements (McLafferty, retro-Diels-Alders, neutral losses)
- Origin and use of characteristic ions for structural elucidation
- Soft ionization techniques I: Chemical ionization and field desorption ionization as alteratives for the determination of the molecular ion.
- Soft ionization techniques II: Electrospray Ionisation (ESI), Atmospheric Pressure Chemical Ionization (APCI) and Matrix assisted laser desorption/ionization (MALDI) of non-volatile molecules, charge deconvolution approaches.
- Introduction to MS/MS of biomolecules.

Initial competences

Fundamental aspects of structural analysis, symmetry, nuclear resonance, X-ray diffraction and mass spectrometry and fundamental knowledge of organic chemistry, both in agreement with the bachelor program.

Final competences

Being able to select a suitable spectroscopic technique allowing structural elucidation of given molecular (sub)structures.

- Being able to combine given spectroscopic data obtained from MS, NMR and XRD, individually or together in a strategy for the determination of organic structures.
- Being able to differentiate relevant spectroscopic signals in complex spectra.
- To have insight in the most important parameters in the practical assessment of the different spectroscopic and diffraction techniques.

Conditions for credit contract

Access to this course unit via a credit contract is determined after successful competences assessment

Conditions for exam contract

This course unit cannot be taken via an exam contract

Teaching methods

Practicum, Demonstration, Online lecture, Group work, Lecture, Online seminar: coached exercises, Seminar: coached exercises

Extra information on the teaching methods

Due to the COVID19 crisis modification in the teaching methods are possible if this would prove necessary.

Learning materials and price

- PowerPoint presentations (Ufora)
- Articles and book chapters from recent literature

References

- The following handbooks are relevant with regard to the NMR spectroscopy section: High-Resolution NMR techniques in Organic Chemistry 2nd Edition. Timothy DW Claridge, Elsevier, ISBN-13: 978-0-08-054628-5. This book is electronically available via Ufora.
- The following handbooks are relevant with regard to the mass spectrometry section. They do not have to be purchased. Students can always receive a copy upon request. Mass Spectrometry (3rd edition), J.H. Gross, Springer, ISBN 978-3-319-54397-0.
- The following handbook is relevant with regard to integrated structural analysis strategies Organic Structure Analysis (2nd edition), P Crews, J. Rodriguez, M. Jaspars, Oxford university press, students can always receive a copy upon request.
- The following handbooks are relevant with regard to X-ray analysis and crystal structure determinations: The Basics of Crystallography and Diffraction (IUCr, 4th Edition), Christopher Hammond, Oxford University Press; Crystal Structure Analysis - Principles and Practice (IUCr, 2nd Edition), Alexander J. Blake, William Clegg, Jacqueline M. Cole, John S. O. Evans, Peter Main, Simon Parsons, David J. Watkin
- Analytical chemistry (ACS)
- Rapid communications in Mass Spectrometry

Course content-related study coaching

Interactive support via Ufora, or through interaction with the docent(s) and assistants during coached excercises and practica. Individual support after evaluation of the request by the docent(s), by appointment. Additional information on the group work NMR by the responsible docent and assistants.

Assessment moments

end-of-term and continuous assessment

Examination methods in case of periodic assessment during the first examination period

Oral examination, Open book examination

Examination methods in case of periodic assessment during the second examination period

Skills test, Report, Oral examination, Open book examination, Job performance assessment

Examination methods in case of permanent assessment

Skills test, Report, Job performance assessment

Possibilities of retake in case of permanent assessment

not applicable

Extra information on the examination methods

- Periodic evaluation methods: 10 out of 20 points
- MS: Clarification of structures based on MS spectra via an open book exercise exam (6/10)
- XRD: Oral exam with written preparation, with focus on integrated applications (4/10)
- Non-periodical evaluation methods: 10 out of 20 points.

At the start of the semester, students are presented in small groups with a structural analysis problem with corresponding NMR spectra that they must tackle as a team. The work activities are recorded in a logbook that is scored (2/10). The result of the analysis is the subject of a written report that is submitted no later than the end of week 11 and is also scored (3/10). Finally, each student gives an individual presentation in which he / she answers a specific question that is assigned to them personally and is situated within the context of the group work (5/10). The presentation is also made available at the end of week 11.
Please note: If a student does not submit the written paper or oral presentation in time, the student is considered not to take part in the periodic evaluation, and the student is automatically referred to the second examination opportunity by being given a non-passing grade.

Calculation of the examination mark

- 50% NMR
- 30% MS
- 20% XRD