GHENT UNIVERSITY

Course Specifications

Valid in the academic year 2024-2025

Molecular Structure Analysis (C004127)

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

Credits 6.0 **Study time 150 h**

Course offerings and teaching methods in academic year 2024-2025

A (semester 1)	English	Gent	lecture
			seminar
			group work

Lecturers in academic year 2024-2025

Lynen, Frederic	WE07	lecturer-in-charge
Van Hecke, Kristof	WE06	co-lecturer
Vianney, Lie	WE07	co-lecturer

Offered in the following programmes in 2024-2025	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 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 and learns to establish integrated analysis strategies allowing to elucidate the structure of simple as well as relatively complex molecules. The most frequently used NMR techniques for structure analysis are covered with attention for important concepts required for their successful analysis. The modules (1) NMR spectroscopy and XRD and (2) mass spectrometry can be followed as Advanced Topics in Chemistry.

Contents

NMR spectroscopy
- Useful concepts underlying NMR spectroscopy
- How the signal is generated: description of a simple NMR experiment
- From signal to spectrum: processing
- Relaxation and its impact on spectral appearance, relative and absolute quantification
- Sensitivity, resolution and how to improve these
- From 1 to 2 to N dimensions in NMR
- Correlations through bonds and through space
- Understanding chemical exchange phenomena

XRD
- (Advanced) crystallization techniques
- The phase problem

- Direct methods
- Patterson
- Friedel's law - absolute configuration
- Molecular replacement
- Anomalous scattering
- Structure refinement
- Quality assessment crystal structures
- Cambridge Structural Database
- (Advanced) crystal structure determinations

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 determination 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 alternatives 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

1 Being able to select a suitable spectroscopic technique allowing structural elucidation of given molecular (sub)structures.
2 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.
3 Being able to differentiate relevant spectroscopic signals in complex spectra.
4 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

Group work, Seminar, Lecture

Study material

Type: Slides

Name: Molecular Structure Analysis - X-ray diffraction
Indicative price: Free or paid by faculty
Optional: no
Available on Ufora : Yes

Type: Software

Name: Cambridge Structural Database - Enterprise suite
Indicative price: Free or paid by faculty
Optional: no
Online Available : Yes

Additional information: Betaald door Facultaire Bibliotheek

Type: Software

Name: CrysAlisPro
Indicative price: Free or paid by faculty
Optional: no
Online Available : Yes

Type: Software

Name: SHELX
Indicative price: Free or paid by faculty
Optional: no
Online Available : Yes

References

The following handbook isrelevant 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 978-3-31954397-0.

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)

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.

Course content-related study coaching

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

Assessment moments

end-of-term and continuous assessment

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

Written assessment

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

Written assessment

Examination methods in case of permanent assessment

Participation, Presentation, Assignment

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• (5/10)
- XRD: Written assessment, with focus on integrated applications (5/10)

Non-periodical evaluation methods: 10 out of 20 points

- At the start of the semester, students are assigned to small groups with a structural analysis problem and corresponding NMR spectra that they must tackle as a team. The work activities are recorded in a logbook that is scored for participation and content (2/10). The end 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 made available at the end of week 11 and evaluated in week 12.
- 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
- 25% MS
- 25% XRD