Advanced NMR Spectroscopy: Application to Structure Analysis
(C002551)

Course Specifications
Valid as from the academic year 2016-2017

Course size
Credits 6.0 Study time 160 h Contact hrs 77.5 h

Course offerings and teaching methods in academic year 2017-2018
A (semester 1) lecture 25.0 h
demonstration 2.5 h
seminar: coached exercises 22.5 h
self-reliant study activities 27.5 h

Lecturers in academic year 2017-2018
Martins, José WE07 lecturer-in-charge
Sinnaeve, Davy WE07 co-lecturer

Offered in the following programmes in 2017-2018
Master of Science in Chemistry 6 A
Exchange Programme in Chemistry (master's level) 6 A

Teaching languages
English

Keywords
Structure Analysis, Nuclear Magnetic Resonance Spectroscopy, 2D-NMR, NOE, Relaxation, Conformation

Position of the course
This course is the logical continuation of the course on Structure Analysis (2nd Bachelor in Chemistry). The student acquires a theoretical understanding of the principles and practical application of structure analyses using advanced NMR spectroscopic techniques. An in depth theoretical insight is provided. The student learns to draft a structure analysis methodology, using the knowledge of various NMR techniques and information on the compound at hand. The student learns to understand the necessary measurements and the analysis of the spectra obtained, to report on the results obtained, and to formulate recommendations for further analysis. Finally, the student applies the theory and practice through an individual molecular assignment task using a series of spectra on a given compound or a compound of interest.
The knowledge and skills acquired are relevant and can directly be applied to structure analysis issues in chemical synthesis, for instance in the context of master thesis research.
This course contributes to the following education skills ('opleidingscompetenties'): M.2.3, M.2.4, M.2.5., M.3.4., M.4.1., M.4.3., M.6.2., M.O.2

Contents
1. Introduction:
   • The nuclear spin as the basis for a spectroscopic technique
   • Precession
   • Magnetization
   • The vector model
   • Pulse Fourier Transform NMR : the technique in terms of the vector model
   • The Spin Echo: a 'jack of all trades'
Introduction of concepts that allow to introduce the important theoretical and practical aspects of NMR spectroscopic techniques used for structural analysis.
2. Relaxation

(Approved)
• Relaxation processes: a phenomenological view
• T1 en T2 processes and their impact on the measurement process
• Measuring T1 and T2
• The nuclear Overhauser effect (nOE) als the source of conformational information.

3. Advanced NMR techniques for structure analysis
• Practical aspects of measuring and processing NMR spectra.
• 1D techniques: APT, Polarization transfer : INEPT
• 2D techniques: principles (presented phenomenologically using the vector model)
• 2D J-correlation techniques: COSY, TOCSY, HSQC, HMBC
• 2D nOE-correlation techniques: NOESY, ROESY

An overview is presented of the most used techniques and practical aspects related to advanced structural analysis is presented. The emphasis is put on the spectra that result from these techniques, rather than on the pulse sequences themselves.

4. Structure Analysis with NMR: integrated approach
• Integration of 1D and 2D NMR techniques into a protocol for advanced structure analysis
• Molecular Modelling based on NMR data
• Illustration with selected examples (during tutorials)
• Recent examples of structure analysis with NMR based on literature examples

5. Beyond Structure Analysis
• Dynamic Phenomena : Chemical Exchange
• NMR and intermolecular interactions : from host-guest chemistry to biomolecular screening
• The diffusion dimension in NMR

Initial competences
The student must have successfully completed and obtained a credit for the complete mathematics (15 studypoints) and physics (15 studypoints) curriculum, or have obtained credits for a similar program, as well as the structure analysis course (5 studypoints).

Final competences
1. An understanding of the fundamental aspects of NMR spectroscopy in one and multiple dimensions.
2. An understanding of the ways in which covalent structure and conformation can be visualized through NMR spectroscopy.
3. A rational assessment of the strengths and weaknesses of NMR for structure analysis.
4. Assess whether a structure analysis problem is amenable to investigation by NMR.
5. Independently devise, plan and execute a procedure for structure analysis using NMR spectroscopy.
6. Process multidimensional and multinuclear NMR spectra.
7. Interpret spectra of molecules with (partially) known structure.
8. Communicate the result of the analysis by means of a written report and be able to discuss orally on the issue.

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
Demonstration, lecture, self-reliant study activities, seminar: coached exercises

Extra information on the teaching methods
The 'Zelfstandig werk' or individual assignment consists in further developing structure analysis skills by performing the complete assignment of a specific molecule of interest using advanced 1D and 2D spectra. Each student is assigned a different molecule. For 2nd Master students, a molecule issued from organic synthesis work in the context of the master thesis may be selected in agreement with the lecturer in charge and the master thesis promotor.

Learning materials and price
Cost: 20.0 EUR
English lecture notes available electronically on Minerva

References

(Approved)
Course content-related study coaching
Interactive support via Minerva (forum and e-mail)
Personal support: following an appointment with the lecturer

Evaluation methods
end-of-term evaluation and continuous assessment

Examination methods in case of periodic evaluation during the first examination period
Oral examination

Examination methods in case of periodic evaluation during the second examination period
Oral examination

Examination methods in case of permanent evaluation
Assignment

Possibilities of retake in case of permanent evaluation
examination during the second examination period is possible

Extra information on the examination methods
- Examination on the course material: partly oral, with written preparation, partly written
- The students receive a list of ca 30 questions in week 10. All examination questions will be selected from that list.
- Evaluation of the 'zelfstandig werk': evaluation of progress via wiki tool prior to the first examination period. Final evaluation at the end of the first exam period, based on a written report and an oral defense.
- If the student fails to submit the written report the student cannot pass.
- Student participation to the second exam period is only possible if the written report is submitted before the start of the second exam period.

Calculation of the examination mark
The evaluation of the project contributes 50% of the total mark.