UNIVERSITY of MISKOLC
Faculty of Materials andChemical Engineering

Antal Kerpely Doctoral School of Materials Science & Technology

Application of NMR spectroscopy in materials science

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COURSE DESCRIPTION

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Lecturer

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Recommendation

The lecture is offered for all students of the Kerpely Doctral School, especially in the field of chemistry.

Language

English

Scope

This course offers an in-depth exploration of Nuclear Magnetic Resonance (NMR) spectroscopy, focusing on its theoretical foundations, instrumental techniques, and practical applications in chemical and biochemical analysis. Topics include spin behavior in magnetic fields, chemical shift, spin–spin coupling, relaxation phenomena, and pulse sequence principles. Both 1D and 2D NMR techniques (e.g., COSY, HSQC, HMBC) will be covered, along with multinuclear NMR (e.g., ^{19}F, ^{31}P, ^{15}N). Emphasis is placed on spectral interpretation, structural elucidation, and problem-solving using real-world molecular examples. The course is designed to equip students with the skills to apply NMR in academic research, pharmaceuticals, and advanced materials characterization.

Methodology

The aim of the course is to provide in-depth and practical knowledge of the theoretical foundations, measurement techniques, and applications of nuclear magnetic resonance (NMR) spectroscopy for chemistry students. During the course, students will become familiar with the basic principles of NMR spectroscopy, methods for spectral interpretation, and its role in structure elucidation, conformational analysis, dynamic studies, and quantitative analysis. Special emphasis is placed on proton and carbon NMR, as well as modern 2D techniques and multinuclear measurements. The course also aims to enable students to independently design NMR measurement strategies and critically interpret the resulting data in the context of their own research.

Learning outcomes

By the end of the course, students will be able to:

- Understand the fundamental physical principles of nuclear magnetic resonance, including spin behavior, resonance conditions, and relaxation phenomena.
- Interpret and assign ^{1}H and ^{13}C NMR spectra, including chemical shifts, multiplicities, integration, and coupling constants.

- Apply basic and advanced 1D and 2D NMR techniques (e.g., COSY, HSQC, HMBC) for structural elucidation of organic compounds.
- Identify and analyze complex spin systems and understand first- and second-order splitting patterns.
- Utilize multinuclear NMR (e.g., ^{19}F, ^{31}P) and solvent effects in interpreting spectra.
- Design and evaluate NMR experiments for quantitative and dynamic studies (e.g., qNMR, variable temperature, kinetics).
- Use specialized software tools (e.g., MestReNova, TopSpin) for spectral processing and analysis.
- Critically assess the reliability of NMR data and formulate conclusions about molecular structure and behavior.

Topics

1. Introduction and Theoretical Foundations

This block introduces the physico-chemical principles of NMR spectroscopy, which are essential for understanding spectra. Students learn about nuclear spin behavior in a magnetic field, the conditions for resonance, and the energy transitions underlying the phenomenon.

- The role of NMR spectroscopy in modern chemistry and biomolecular research
- Magnetic properties of nuclei; concept of nuclear spin
- Magnetic field and Larmor precession
- Conditions for magnetic resonance, energy differences and population distribution
- Relaxation processes: importance of T_1 and T_2 relaxation
- Basics of Fourier transformation and pulse sequences

2. Spectroscopic Principles and Measurement Techniques

This module focuses on practical measurement parameters and their interpretation. Students will acquire the foundations of one- and multidimensional NMR techniques, as well as the specialized knowledge needed for observing different nuclei.

- ^{1}H and ^{13}C NMR: chemical shift, spin–spin coupling (J-coupling)
- Main features of NMR spectra: multiplicity, integration, fine structure
- Solvent effects, reference standards, internal and external references
- Overview of 2D NMR techniques: COSY, HSQC, HMBC
- Application of multinuclear NMR (e.g., ^{19}F, ^{31}P, ^{15}N) in special systems
- Sample preparation, concentration, pH, and temperature effects

3. Spectrum Interpretation and Practical Applications

The goal of the third part is to develop proficiency in interpreting NMR spectra. Through real examples, students practice structure elucidation and gain insight into quantitative and dynamic applications.

- Structure elucidation of simple organic molecules using ^{1}H and ^{13}C NMR
- Fundamentals of interpreting 2D NMR spectra
- Investigation of conformational and dynamic processes
- Quantitative NMR (qNMR): methodology and applications
- Spectrum interpretation with software support (e.g., MestReNova, TopSpin)
- Case studies: detailed analysis of research or industrial examples

Control Questions

1. What is the physical basis of NMR spectroscopy, and what conditions are required for resonance?
2. What is chemical shift (δ), and what factors influence its value?
3. What is the difference between T_1 and T_2 relaxation times, and how do they affect spectral quality?
4. How is multiplicity interpreted in a ^{1}H NMR spectrum, and what is the significance of the n+1 rule?

5. What is spin–spin coupling (J-coupling), and how can coupling constants be extracted from the spectrum?
6. Describe the basic principles and informational content of COSY and HSQC techniques.
7. Which nuclei, besides ^{1}H and ^{13}C, can be studied by NMR, and what are the advantages of using them?
8. What are the main steps in sample preparation for NMR measurements, and why is sample concentration important?
9. How is the structure of an unknown organic compound determined using ^{1}H and ^{13}C NMR spectra?
10. What is quantitative NMR (qNMR), and under what conditions can it be performed reliably?

References

Hungarian-language literature:

György Kéri – Gyula Mády – László Szilágyi: Szerves kémiai spektroszkópia
Semmelweis Kiadó, Budapest
Miklós Hollósi (ed.): A biomolekulák spektroszkópiája
Semmelweis Kiadó
Lecture notes / instructor-provided teaching materials

English-language literature:

T.D.W. Claridge: High-Resolution NMR Techniques in Organic Chemistry
Elsevier, 3rd Edition (2009 or later)
David G. Morris: NMR Spectroscopy in Practice: From Basics to Applications
Wiley-VCH, 2010
James Keeler: Understanding NMR Spectroscopy
Wiley, 2nd Edition (2010)
Jeremy K.M. Sanders: Modern NMR Spectroscopy – A Guide for Chemists
Oxford University Press
William Kemp: NMR in Chemistry: A Multinuclear Introduction
Macmillan, 1986

Exam

Oral exam after correctly answering some basic questions.

Complex exam topics and sample questions

1. **Describe in detail the physical principles of NMR spectroscopy, with particular emphasis on spin–magnetic field interactions, resonance, and relaxation processes.**
   How do these factors influence spectral resolution and signal intensity?
2. **Present and compare the most important 1D and 2D NMR techniques (^{1}H, ^{13}C, COSY, HSQC, HMBC).**
   What structural information can be obtained from these methods, and in what cases is each technique most appropriate?
3. **Explain in detail the phenomenon of spin–spin coupling (J-coupling).**
   How can coupling constants be used to gain structural information? What are the key differences between first-order and non-first-order systems?
4. **Choose a simple organic molecule (e.g., ethyl acetate or benzyl alcohol) and analyze its ^{1}H and ^{13}C NMR spectra.**

Explain all spectral features, including chemical shift, integration, multiplicity, and J-values.

5. **Explain the principle of quantitative NMR (qNMR), its advantages, and typical applications.**
   What requirements must be met for a reliable qNMR measurement? How are concentrations calculated?
6. **Multinuclear NMR spectroscopy: describe the basics and importance of ^{19}F, ^{31}P, or ^{15}N NMR.**
   What distinguishes these techniques from ^{1}H and ^{13}C NMR in terms of theoretical and practical considerations?
7. **How do the physical and chemical properties of a sample (e.g., concentration, solvent, temperature, pH) affect the NMR spectrum?**
   Provide specific examples and explain the observed changes.
8. **Discuss the role of Fourier transformation in NMR spectroscopy.**
   How is a time-domain signal converted into a frequency-domain spectrum, and what are the benefits of this technique?
9. **Analyze the role of NMR in studying dynamic chemical processes such as conformational equilibria, chemical exchange, or reaction kinetics.**
   What types of measurements and parameters provide information about these phenomena?
10. **Provide a critical overview of the current and future roles of NMR spectroscopy in chemical and life science research.**
    What technological or methodological advancements are emerging, and how might they improve analytical performance?