Chem. 636 - Introduction to NMR

Lecture times are:

T 8:50 – 9:40PM

I will start promptly, and may run to 9:50am.

Chem. 636 - Introduction to NMR

This is a research directed course:

Use samples from your research group whenever possible; much more interesting! Many labs will require use of facility-provided samples.

Questions about actual research are always welcome.

Chem. 636 - Introduction to NMR

Breadth of need is large in our department:

Organic and bio-organic synthetic work:

^{1}H ^{13}C 1D, selective 1D, primary 2D:
Blackwell, Burke, Cavagnero, Gellman, Kiessling, Schomaker, Yoon, etc.

Inorganic and organometallic synthetic, or physical work:

more X (^{13}C ^{19}F), VT, metals, kinetics:
Berry, Burstyn, Dahl, Landis, Mahanthappa, Reich, Stahl, etc.

Makes focus of the class difficult. We'll center on synthesis, and specifically **structure verification**.

Grades and Access to High-Field Spectrometers

Grades: 15% on attendance in labs
20% on attendance at weekly practice sessions
35% on weekly data turn-ins
10% on midterm exam
20% on final exam

Continued access will be strongly dependent on checkout:

- any uncertainty by myself or TA will require a 2nd checkout

Course Goals

Obtain an understanding of major NMR techniques:

- ☞ For a particular chemical problem:
  - techniques that are applicable to solution NMR

    → structure verification / elucidation
    kinetics / mechanistics
    dynamics
    aggregation / MW / chromotography
    structure determination
  - which spectrometer is best to use

- ☞ Correctly setup experiments
  - proper sample prep
  - critical parameters

Course Goals

- ☞ Provide supervision and assistance to other students when in facility.

Professionalism in laboratories:

- keep a proper notebook
- read literature and use user guides; ask appropriate questions
- carefully document instrument problems, and notify staff

Literature and Reading Assignments

Recommended Texts:

Timothy D. W. Claridge, *High-Resolution NMR Techniques in Organic Chemistry* (Tetrahedron Org. Chem. Series Vol 27, **2nd ed.**, Pergamon, 2008). ISBN 0-08-042798-7.

- Up-to-date and fairly complete. Issues are organization, and many sections overly technical. Available through the library; fully downloadable as pdf sections.

Oliver Zerbe and Simon Jurt, *Applied NMR Spectroscopy for Chemists and Life Scientists* (Wiley-VCH 2013). ISBN 0-08-042798-7.

- Up-to-date and fairly complete. Perhaps not consistent in discussions; applications chapters are good (natural products, carbohydrates, peptides, etc). Some sections overly technical. Available through the library on ebook EBL (limited # hits; download sections as pdfs).

Literature and Reading Assignments

Recommended Texts:

- Jeremy K. Sanders and Brian H. Hunter, *Modern NMR Spectroscopy: A Guide for Chemists*, 2nd ed. (Oxford, 1993). Dated.
  - good examples, excellent discussion on NOE, good strategies and tactics
  - Is out-of-date on some important subjects (e.g., no NOESY2D, no gradients).

- Harald Günther, *NMR Spectroscopy: Basic Principles, Concepts and Applications in Chemistry*, 3rd ed. (Wiley-VCH, 2013).
  - excellent figures/examples; does introduce product operators and gradients; much better depth than S&H.
  - spotty on some discussions and still dated in areas; no noesy1d or other new techniques

Literature and Reading Assignments

- J. W. Akitt and B. E. Mann, *NMR and Chemistry: An Introduction to Modern NMR Spectroscopy, 4th ed.*, (Cheltenham, UK, 2000).
  - Very good examples and discussions for inorganic and organometallic chemists

- John H. Nelson, *Nuclear Magnetic Resonance Spectroscopy*, (Prentice Hall, 2003).
  - Great tables of 31P and M NMR.
  - Good for inorganic and organometallic chemists

- S. Braun, H.-O. Kalinowski, S. Berger, *150 and More Basic NMR Experiments: A Practical Course*, (Weinheim, NY 1998).
  - Excellent practical details for nearly all modern experiments (new edition, not in library, has 200 exps)

- Frank J. M. van de Ven, *Multidimensional NMR in Liquids: Basic Principles and Experimental Methods*, (VCH, 1995). Highly recommended for PChem, spectroscopist; back-in-print.
  - Excellent introduction for product operator formalism; excellent in-depth discussions of modern experiments.

Literature and Reading Assignments

- Malcolm Levitt, *Spin Dynamics: Basics of Nuclear Magnetic Resonance*, (Wiley 2008). Excellent introduction to NMR theory.

- Gordon S. Rule and T. Kevin Hitchens, *Fundamentals of Protin NMR Spectroscopy (Focus on Structural Biology)*, (Springer, 2005; ISBN 978-1402034992). Excellent resource for protein work.

- John Cavanagh, et al, *Protein NMR Spectroscopy: Principles and Practice*, (Academic Press, **2nd ed.** 2007).
  - excellent practical discussions; good balance between theoretical depth and practical application; up-to-date

- J. S. Evans, *Biomolecular NMR Spectroscopy*, (Oxford, 1995) ISBN 0-19-854766-8. Excellent resource for protein work
  - up-to-date?; less physical than Cavanagh

Week 1

Review facility website, especially the Users Guide section:

- ☞ see: http://cic.chem.wisc.edu/nmr/main.html → User Guides → Bruker AVANCE User Guides

- ✸ Install MestreNova, and setup data access (sftp or drive mounting).

- ✸ Use MNova!!

- ✸ Sample Preparation: Claridge section 3.3; Zerbe-Jurt sections 2.5.1+2.5.2

- ✸ Details on reading for processing will be posted in 2nd HW.

Lab and Practice Times

Do not change lab times without TA approval 24h prior to lab

- Lab instructor will not allow >6 students in a lab

3 students → 2.5-hours of practice time per week

- share the time at keyboard between all three!!
- You must make every attempt to show at your practice time.
- Please do not come to me about practice time changes (resolve with other students).
- Instrument problems: email description to me (try to find TA!)

  problems with plotting/printing: do the best you can, include explanation; find Heike or Zhihui

- unattended lab sessions and/or practice time will lower grade quickly