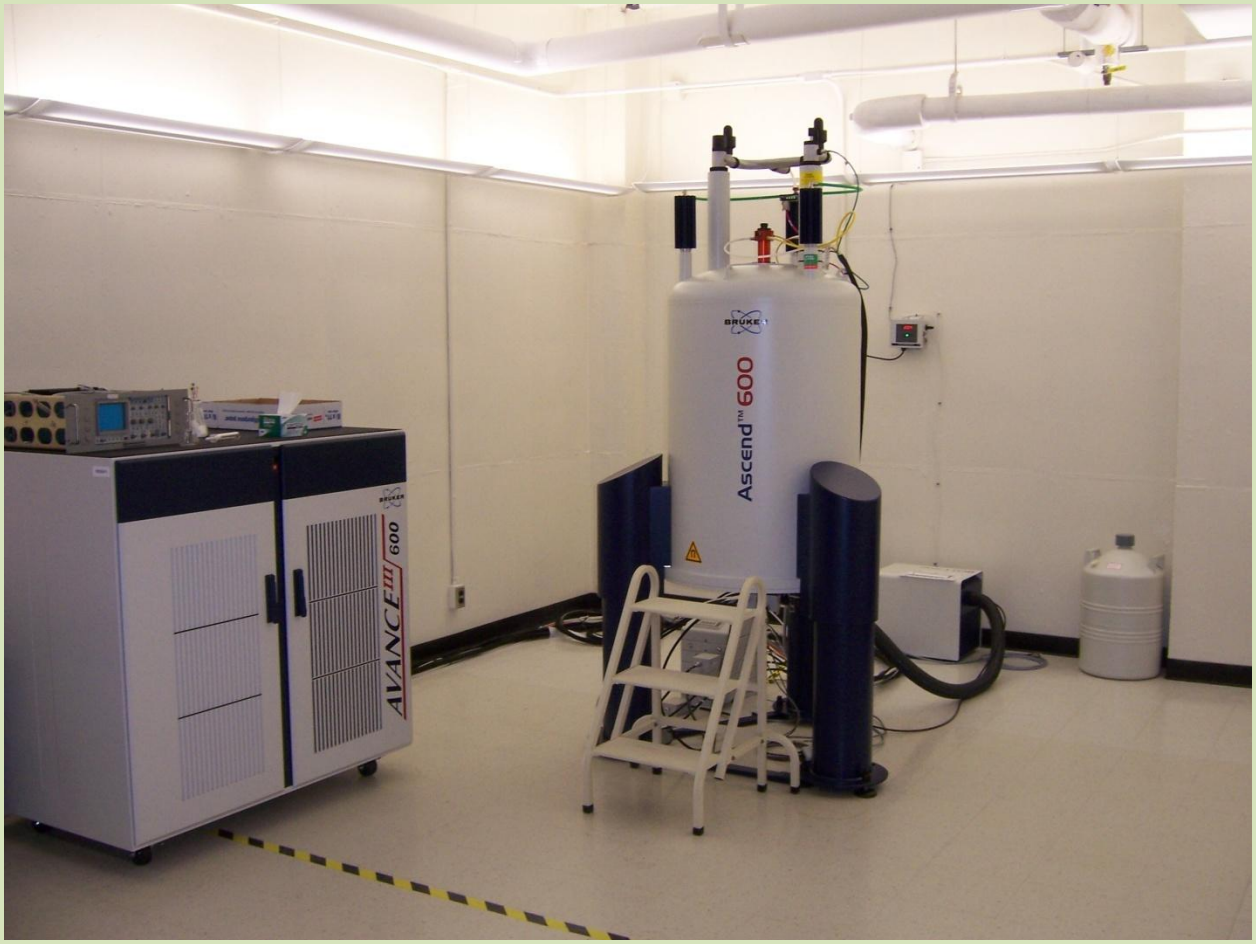
BINGHAMTON UNIVERSITY
STATE UNIVERSITY OF NEW YORK

Department of Chemistry

NMR Newsletter Fall 2012

Welcome to the first edition of the Newsletter from the NMR Facilities at Binghamton University. This newsletter was produced to keep researchers in upstate New York updated on news and developments in our NMR facilities. http://nmr.binghamton.edu



A new Bruker Avance III 600 MHz NMR spectrometer has recently been installed at Binghamton University. This instrument will serve as the cornerstone of a **Regional NMR Facility**, which is being established to provide regional colleges and universities with the means to perform liquid and solid state NMR experiments, which their own instruments were not designed to run.

We hope to welcome you soon at our NMR facility to demonstrate the instrument's capabilities. Tours of the facility can be scheduled upon request.

Binghamton University NMR Facility

http://nmr.binghamton.edu

Bruker Avance III 600 - liquids and solids capabilities

- **14.1 Tesla shielded, superconducting Magnet**
- **600 MHz Proton NMR, 3 RF Channels, z-Axis Pulsed Field Gradients**
- **5 mm Broadband/Fluorine Observe Probe**
  This probe is used in automation and manual operation for routine analysis of organic and inorganic compounds in solution. It is equipped for pulsed field gradients and can be tuned automatically to detect almost all nuclei. Typical experiments performed with this probe include many two-dimensional techniques, i.e. COSY, NOESY, HSQC, HMBC.
- **5 mm H,C,N,P Quadruple Inverse Probe**
  This probe is used for biological and biochemical samples in solution. It is equipped for pulsed field gradients and its proton sensitivity is approx. 50% better than the BBFO probe. Multinuclear correlation experiments using almost any combination of Hydrogen, Carbon, Nitrogen, and Phosphorus are possible. Excellent water suppression capabilities make this probe ideal for the analysis of proteins and nucleic acids in aqueous buffer solutions.
- **2.5 mm H,X,Y Triple Resonance MAS Probe**
  This probe is used for the analysis of organic and inorganic compounds in solid state. The probe can perform Magic Angle Spinning (MAS) at up to 35,000 Hz and it will detect almost all nuclei. Typical experiments performed with this probe include single-pulse, CP/MAS, and REDOR experiments.

Bruker AM 360 - liquids capabilities only

- **8.45 Tesla unshielded, superconducting Magnet**
- **360 MHz Proton NMR**
- **Tecmag DSPECT Spectrometer Upgrade**
- **5 mm Dual ^{13}C,^{1}H Probe**
- **10 mm Broadband Observe Probe**
- **10 mm Dual ^{19}F,^{1}H Probe**

Bruker AC 300 - liquids and solids capabilities

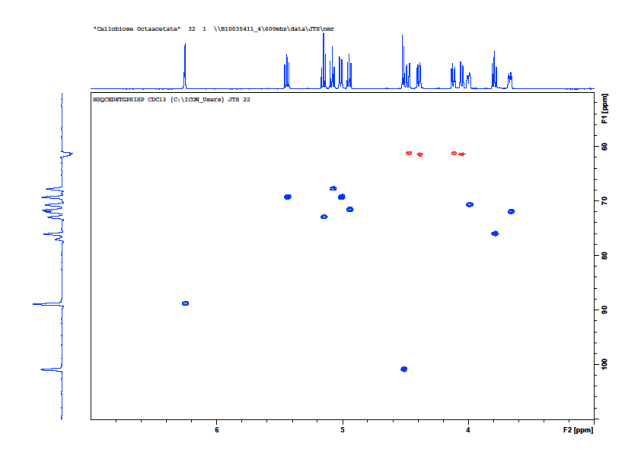
- **7.05 Tesla superconducting Magnet**
- **300 MHz Proton NMR**
- **5 and 10 mm Dual ^{13}C,^{1}H Probes**
- **5 and 10 mm Broadband Observe Probes**
- **5 mm Dual ^{19}F,^{1}H Probe**
- **Solid State NMR Accessories**
  (five 7 mm DOTY CP/MAS probes with MAS speeds up to 5 kHz)

NMR Tip: Are your samples too dilute for ^{13}C NMR experiments?

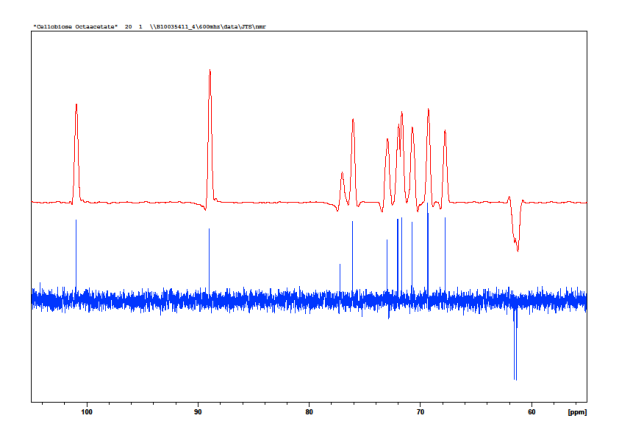
Often it is not possible to obtain ^{13}C data in a reasonable time frame, and a full set of ^{13}C, APT, and DEPT experiments could consume a prohibiting amount of spectrometer time and money. There are, however, indirect or "inverse" (=^{1}H-detected) alternatives that will allow you to get equivalent information in a fraction of the time ^{13}C and DEPT spectra would require.
2D-spectra of a 1-10 mM cellobiose octaacetate solution in $CDCl_3$ show excellent sensitivity:

1. ***^{1}H,^{13}C-HSQC experiment:***
   (1 mM, 60 min., DEPT substitute)

All proton-bearing carbon atoms show cross peaks. Peak picking can identify their shifts. CH2 signals appear as negative peaks (red).

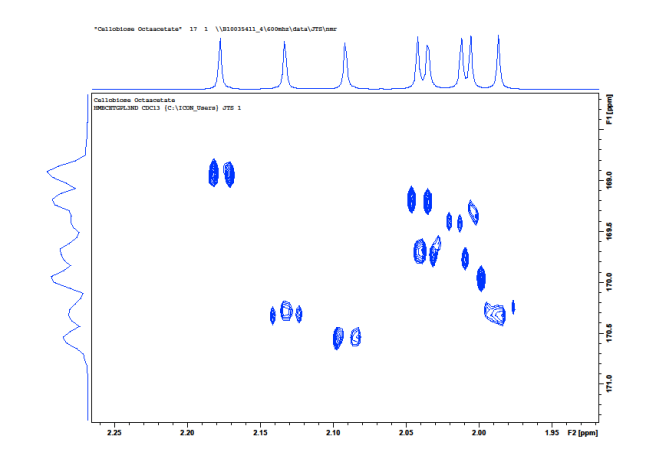


(Only the sugar ring region is displayed.)
Linear prediction of 1024 complex data points in the ^{13}C dimension gives sufficient resolution to identify signals 0.2 ppm apart:

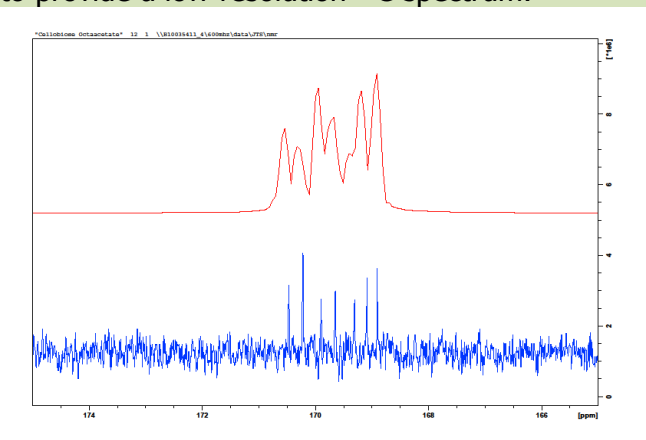


2. ^{1}H,^{13}C-HMBC experiment:
(10 mM, 5 hours, ^{13}C alternative)

All carbon atoms (incl. quarternary) show easily distinguishable cross-peaks. (Only the carbonyl-C/methyl-H cross section is shown)



After linear prediction of 4096 complex data points in the ^{13}C dimension the peak separation in the ^{13}C projection is adequate to provide a low-resolution ^{13}C spectrum:



Comparison: 12-hour DEPT and ^{13}C spectra (bottom), HSQC and HMBC ^{13}C projections (top).

HSQC and HMBC experiments can be applied to any low-abundance nucleus, i.e. ^{13}C, ^{15}N, or ^{29}Si, and they are often the only way to get any NMR data of macromolecules.

Announcement:

Chem 485f/585f:

In Spring 2013 our NMR course (Chem 485f/ Chem 585f) will be offered again. In the past this course has been very popular with students from other colleges and researchers from several regional companies.

Classes will be taught by Dr. Mark Poliks and the laboratory sessions on a variety of old and new NMR spectrometers will be organized by Dr. Jürgen Schulte.

During the course the participants will acquire basic NMR theory and practical laboratory aspects of a wide variety of NMR concepts and techniques. One-dimensional techniques include ^{1}H and ^{13}C experiments to study T1 and T2 relaxation, decoupling, and Nuclear Overhauser Enhancements. Two-dimensional experiments will include COSY, NOESY, HSQC, and HMBC. Included will also be a section about solid state NMR.

For more information go to our new NMR website:

nmr.binghamton.edu