BINGHAMTON UNIVERSITY
STATE UNIVERSITY OF NEW YORK

Department of Chemistry

NMR Newsletter 2021

Welcome to the **Regional NMR Facilities** at Binghamton University.
Our laboratory is currently operating three high-field NMR spectrometers at proton frequencies from 600 to 400 MHz, as well as two low-field spectrometers at 300 and 100 MHz.

The newest addition to our spectrometer lineup is a
Bruker Avance NEO 400 with solution and solid-state NMR capabilities.

This instrument will expand our **Regional NMR Facility**, which has been established in 2011 to give regional colleges and universities access to liquid and solid-state NMR instruments, which are not available at their locations.

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. For details please visit:

http://nmr.binghamton.edu

Binghamton University NMR Facility

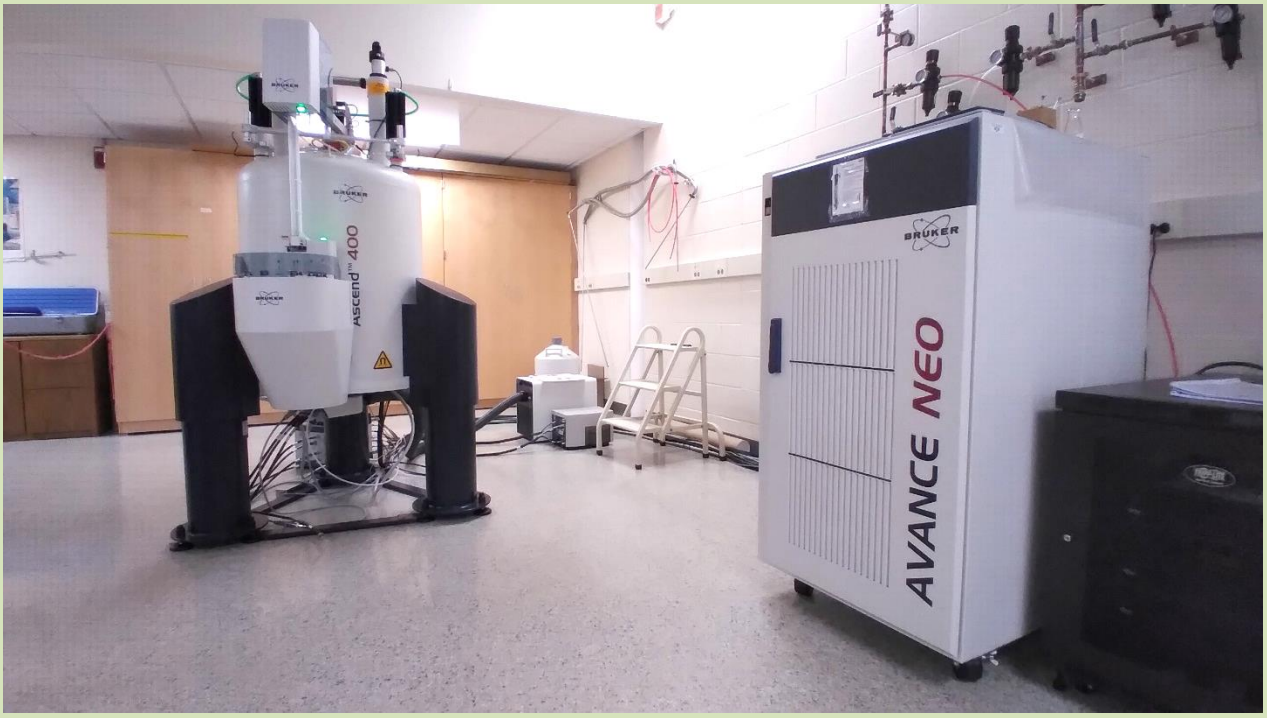
Bruker Avance III 600

Solution and Solid-State NMR 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 buffers.
- **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 Avance NEO 400

Solution and Solid-State NMR capabilities



- **9.4 Tesla shielded, superconducting Magnet**
- **400 MHz Proton NMR, 3 RF Channels, z-Axis Pulsed Field Gradients**
- **5 mm iProbe (Broadband/Fluorine Observe)**
  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.
- **3.2 mm H,C,N Triple Resonance MAS Probe**
  This probe is used for the analysis of organic and bio-organic compounds in solid state. The probe can perform Magic Angle Spinning (MAS) at up to 24,000.
  Typical experiments performed with this probe include single-pulse, CP/MAS, and REDOR experiments.
- **Automatic Sample Changer for 24 samples**
  allows for advance scheduling of all experiments and provides 24/7 availability.

Bruker Avance III HD 400

Solution NMR capabilities

This spectrometer was purchased by the Pharmacy School with funding from the Decker Foundation.

- **9.4 Tesla shielded, superconducting Magnet**
- **400 MHz Proton NMR (162 MHz ^{31}P, 100 MHz ^{13}C, 40 MHz ^{15}N)**
- **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.
- **Automatic Sample Changer for 24 samples**
  allows for advance scheduling of all experiments and provides 24/7 availability.

We are still maintaining two older NMR spectrometers for our instructional program in Science 2.

Bruker AC 300 - liquids and solids capabilities

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

Bruker AC 100 - liquids and solids capabilities

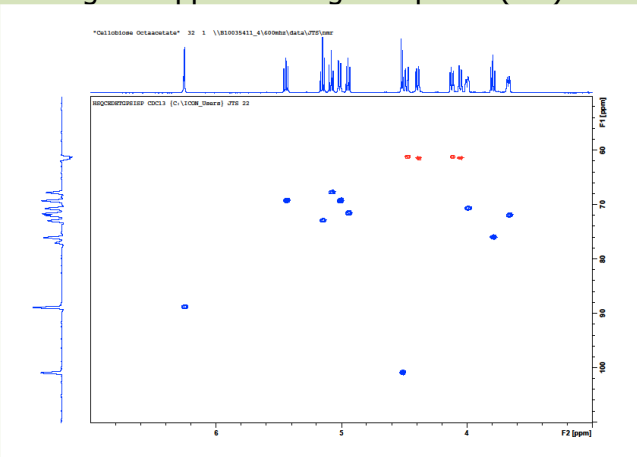
- **2.35 Tesla superconducting Magnet**
- **100 MHz Proton NMR**
- **Five Dual $^{13}C,^{1}H$ and Broadband Observe Probes (5 and 10 mm)**
- **One 7 mm DOTY CP/MAS probe with MAS speeds up to 5 kHz**

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

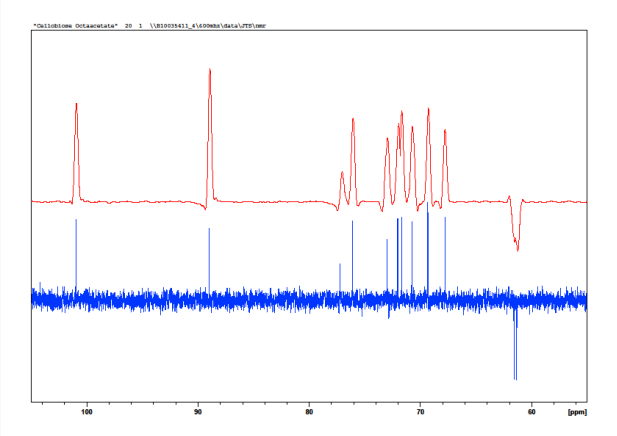
Often it is not possible to obtain ^{13}C data in a reasonable time frame, and a full set of ^{13}C, APT, and DEPT experiments could consume a prohibiting amount of spectrometer time and money. There are, however, indirect or "inverse" (=^{1}H-detected) alternatives that will allow you to get equivalent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

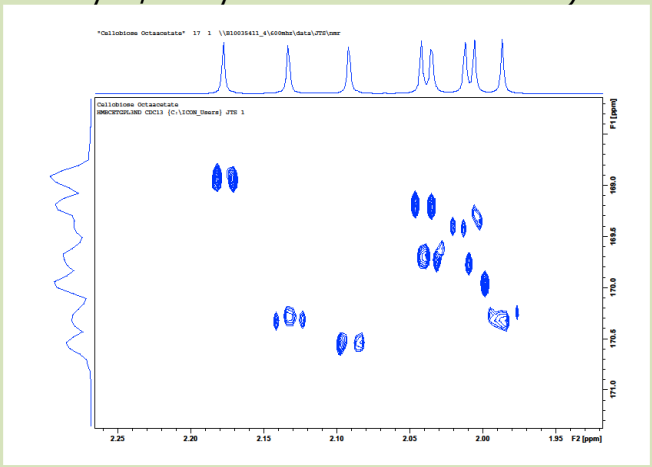


(Only the sugar ring region is displayed.)
Linear prediction of 1024 complex data points in the ^{13}C dimension gives sufficient resolution to identify signals 0.2 ppm apart:

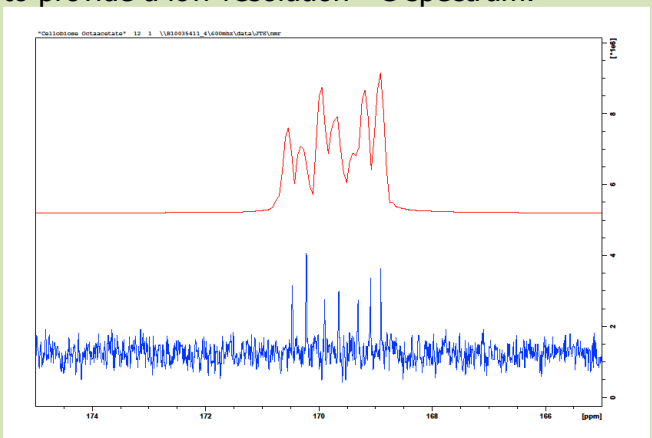


2. ***^{1}H,^{13}C-HMBC experiment:***
   (10 mM, 5 hours, ^{13}C alternative)

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.