The NMR Lab at the School of Chemical Sciences University of Illinois

Andre Sutrisno
Solids NMR

Dean Olson
Director

Lingyang Zhu
Liquids NMR

Tracie Hubert
Technical Asst.
### NMR Lab Staff

<table>
<thead>
<tr>
<th>Name</th>
<th>Position/Role</th>
<th>Duration</th>
<th>Location</th>
</tr>
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<tbody>
<tr>
<td>Ms. Tracie Hubert</td>
<td>Technical Administrator; since January 1986</td>
<td></td>
<td>55 Noyes</td>
</tr>
<tr>
<td>Dr. Andre Sutrisno</td>
<td>Solid State NMR; June 2014</td>
<td></td>
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<td>Dr. Lingyang Zhu</td>
<td>Liquids NMR; since November 2011</td>
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<tr>
<td>Mr. John Roscheck</td>
<td>Research Engineer since July 2012</td>
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<td>Dr. Dean Olson</td>
<td>Director; since December 2010</td>
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<tr>
<td>Dr. Vera Mainz</td>
<td>Director for 25 years; retired summer 2010</td>
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<tr>
<td>Prof. Martin Burke</td>
<td>Faculty advisor; since January 2012</td>
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Solution State NMR Spectrometers

146 RAL

400 MHz: Nalorac QUAD probes (H/F, C/P or B/P)

500 MHz: CB500 CryoProbe with 45 Nuclei

55 Noyes

600 MHz: AutoX BB and HCN probes

H/F, C/P probe

500 MHz: Nalorac QUAD probes

H/F, C/P w PFG Z probe

500 MHz: Varian HCN PFG Z probe
Solid State NMR Spectrometers

300 MHz WB
Liquids/Solids:
- Varian 5mm H/X probe
- Chemagnetics 7mm/4mm MAS probes

500 MHz WB:
- 3.2mm HCN Balun MAS probe
- 1.6mm HCDN Fast MAS probe
- 1.6mm HFXY Fast MAS probe

750 MHz WB:
- 1.6mm HFXY Fast MAS, HXYZ Gradient Fast MAS and HXY Fast MAS probes
- 3.2mm HCN Balun MAS and HXY BioMAS probes
- 4mm T3 HXY MAS probe

See Andre

Typically Rienstra Gp
Solution/Solid State 750 MHz NB
NMR Spectrometer

A151 CLSL

750 MHz NB:
- 5mm $^1$H/$^{13}$C/$^{15}$N PFG probe
- 10mm $^{15}$N-$^{31}$P BB probe
- 10mm $^{73}$Ge-$^{15}$N BB probe
- 3mm $^{13}$C/$^1$H probe
- 3.2mm HCN Balun MAS probe
- 3.2mm HCN MAS probe
- 4mm $^{15}$N-$^{31}$P CPMAS Probe

See Lingyang
Welcome to the NMR Laboratory

The NMR Lab of the School of Chemical Sciences offers a wide range of spectrometers, probes, and technical capabilities including multi-dimensional, multi-nuclear, and solid-state NMR. Supported by four full-time staff and two student hourlies, ten spectrometers in three locations allow walk-up and long-term NMR experiments 24/7. Spectrometers at 400 MHz, 500 MHz, and 600 MHz are available for short-to-medium length experiments; 750 MHz and 300 MHz wide-bore spectrometers with specialty probes and solids accessories can perform multinuclear and solid state NMR experiments. All are equipped for variable temperature operation. Our ten Varian spectrometers and 1 Bruker spectrometer include (in MHz): Seven liquid-state NMRs: 400 (two), 500 (four), 600; Two solid-state NMRs: 500, 750; Two liquid/solid compatible NMRs: 300 and 750. An automated Bruker CryoProbe was added in the summer of 2016. We also provide automated, on-flow NMR sample analysis (from vials) for undergrad organic chemistry courses offered via the Department of Chemistry. Electronics and IT staff provide additional support.

To make the most of NMR as an analytical tool, all newcomers receive about 4 hours of individual basic training with additional instruction available for variable temperature control, multi-dimensional NMR, and specialty spectrometers and experiments. Our primary goal is to provide the highest level of NMR performance to the students, staff, and faculty of the School and UIUC campus. The NMR Lab is also available to all outside users, both academic and industrial (contact the Director).

Spotlight on: NMR Labs
Tuesday, January 3, 2017

SCS NMR Spectrometer Status Now

Updated 3 Jan 2017 (DLO):
Working Status of Spectrometers.
Monday - Friday, 9 a.m. to 5:30 p.m., just walk in.
After business hours, use your I-Card (swipe several times as needed).

Use Mnova Version 10.0 Only.

RAL Spectrometers (Check ChemFOM for Availability):

- Carver Bruker 500 CryoProbe (CB500) - OK
  - Ask the staff for account registration, then a trained person in your group for automation instruction.
- U500 - OK
- U400 - OK H-1, C-13, P-31, F-19; No B-11. Use LN2 bucket for cooling. No spinning.
- U500NB - OK. OK for all 1D and 2D NMR, but calibrate pw90 and T1 for protons using posted modification (very minor).
  - Now optimized for BioPak.
- VXR500 - OK for H, C, P, F. VT -60 to +80 C
- SUNDS1 - OK
- Printers - Both OK

Noyes Lab Spectrometers

- UI300WB- OK; Reservations via Andre Sutrisno.
- UI600 - OK; Reservations via Lingyang Zhu.
- chem400 - OK; Chem 237 & 205 use only via DLO.

Chem Life Spectrometer

- VNS750NB - OK; Reservations via Lingyang Zhu.
  - Currently in Solution-State NMR configuration.
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NMR Training

Director e-mails 20 items:
NMR principles, Mnova, i.p. address, etc.

Get a booklet and see Tracie for paperwork & ChemFOM

UI500NB Training by Director (2D-NMR Prep)
- Tuning and matching
- pw90 calibration
- Solvent suppression
- $T_1$ calibration

Study and exercises; Training by Liaison

Special 1D, 2D/3D-NMR Training by Lingyang
- Recommended for UI500NB and required for UI600
- One key 2D document: UI500NB Advanced 1D and 2D NMR

Check-Out by Director

Temperature Control (VT) Training by Director

Solid State NMR Training by the Solids Spectroscopist
A Common Problem

- 0.3mL Positioned too low
  Solvent NOT in detected region

- 0.4mL Centered
  Solvent covers detected region

- 0.7mL Adjusted to Maximum Depth
  *Recommended*

From an MIT web document
A Common Problem
# A Few Under-Appreciated NMR Data Processing Shortcuts

**SOME UIUC VARIAN NMR MACROS OF INTEREST:**

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<thead>
<tr>
<th>Macro</th>
<th>Description</th>
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<tr>
<td>ffa</td>
<td>f full aph cdc dc</td>
</tr>
<tr>
<td>disp</td>
<td>f full aph cdc dc vsadj dscale</td>
</tr>
<tr>
<td>proc</td>
<td>wft f full aph cdc dc ds vsadj dscale</td>
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<tr>
<td>ppmh</td>
<td>wp=10p sp=-0.5p</td>
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<tr>
<td>diff</td>
<td>r1=delta r1? [displays the difference in Hertz between two cursors]</td>
</tr>
<tr>
<td>doi</td>
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<tr>
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<td>pl(‘all’) pscale pap page</td>
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</table>

p7 references the chloroform $^1$H residual signal to 7.26 ppm (select peak first)
p77 references the chloroform $^{13}$C residual signal to 77.0 ppm (select peak first)
diff displays difference between 2 cursors in Hz ($r_1 = \text{Hz}$)
nl vsadjcr adjusts selected peak to be the maximum on y scale
Bruker NMR - 500 MHz CryoProbe

Since July 2016

Carver Bruker 500: CB500

Funding from the Carver Charitable Trust: $500K

Total Cost $895K
Bruker NMR - 500 MHz CryoProbe

NMR Training Simulator
Why Visit the NMR Lab?

“I wish I had done more NMR sooner.”

- Quote from a 5th year grad student