MNMR Organizational Changes
MNMR Organizational Structure

Steering Committee

Director

External Advisory Committee

EDUCATION

RESEARCH
Three major NMR research areas have been identified as strengths throughout the University:

- **Structural Biology**
- **Metabolomics and Small Molecules**
- **Soft and Hard Matter (solid-state)**
MNMR Research Cores

- **Structural Biology**
  - Youlin Xia
  - 7001, 8501, 9001

- **Metabolomics and Small Molecules**
  - Todd Rappe
  - 6001, 6002, 7002
  - Satellite labs

- **Solid State**
  - T. Gopinath
  - 600SS and 700SS
Education

• The Practical NMR Workshop will continue to be offered

• Training for individuals needing immediate access will be available

• Advanced training in development
VNMFRJ 3.2 Upgrade

• Within the next couple months, all Agilent (Varian) instruments will be upgraded to VNMFRJ 3.2

• 6001, 6002, 600SS, 700SS, Med Chem 400MR, CDD 600, Chemistry
Hardware

• 6002 has been equipped with a 2H switch, so deuterium gradient shimming is possible without recabling. (6001 configuration has not changed)

• 6002 default probe is now AutoX Dual Broadband. HCN still available.
New web site – general layout

The header and footer now function in the same way as the University of Minnesota homepage. The searchbar and all the navigation links should all function in the same way.
New web site – general layout

This logo will be the main link back to the home page of the site.
New web site – general layout

We now have managers covering areas in the MNMR Center. To help point internal/external users to the right person we now have tabs corresponding to each given area.
New web site – general layout

The menu bar across the top will have the same list of dropdown menus for each of the areas. The one difference being the Education/Training tab which will have information regarding training and workshop courses.
New web site – general layout

This sidebar will always displace recent news, the main number to the MNMR Center (along with link to the staff page for contact info), and a link to our facebook page.

The “Other Links” section will be used as a quick link reference for items/pages that are relevant to that current page you are on.
New web site – general layout

At the bottom we now have quick links to pages and supporters of the MNMR Center. Things located here don’t necessarily apply to day-to-day operations, but are still useful to have accessible for the users.
New web site – general layout

Each area page will now show the manager of the area.

It will also show the instruments and probes that are available.

On the right-hand side you will also notice the rates and request training links. Each area page will have these links.
One thing we have tried to improve is the use of the cancel time request. We have updated the page that shows the canceled request so if you are trying to get on you can see who released time. If you could make sure to include the instrument in all posts that would be greatly beneficial, not just for the staff, but other users as well.
New web site – general layout

We have recently added a NMR User Wiki page.
New web site – general layout

The MNMR Center wiki page will exist to keep users updated with improvements the staff has made regarding NMR experiments.

We will also try to keep papers on setup/design of experiments for users to reference when starting a new project.
Policy update: Sign-up and Use Charges

Same:
• Schedule time through the request time page.
• Staff has discretion to make short notice changes when necessary.
• Cancel time through the cancel time page.

Different:
• Any time requests that need to be cancelled must do so 24 hours prior to the scheduled time to avoid penalty.
• Any unused time scheduled by a user that is greater than 24 hours will incur a penalty equaling half the unused time. (example: You schedule 48 hours and only use 24 hours, you would be charged for 36 hours)
• Users MUST sign in the log books when starting and ending an experiment. These are used to determine your charged time, and so can only be in your benefit. The log books are also crucial for recording problems verified by the staff, and thus affecting your billed time.

PLEASE REVIEW THE POLICY STATEMENT AND LET US KNOW IF YOU HAVE ANY QUESTIONS.
I. Rules/Attentions:

1. startnmr

```bash
cd /opt/topspin3.1
ulimit -a
ulimit -m unlimited
ulimit -s unlimited
ulimit -v unlimited
ulimit -a
./topspin

cd /opt/topspin3.1
ulimit -s unlimited
./topspin
```
2. atmm: moving wobb curve from left to right side
3. please use white spinner for 850 and 900
4. ensure water peak is less than 3.0 Hz before you log out
5. please write the usage in log book.
6. reserve the 7001, 850, 900 for more than 12 hours in one session
7. place 24 hours notice to cancel NMR time
# II. New Pulse Sequences

[https://wiki.umn.edu/MNMRCenter/WebHome](https://wiki.umn.edu/MNMRCenter/WebHome)

<table>
<thead>
<tr>
<th>Pulprog</th>
<th>Experiment setup</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>ccls</strong></td>
<td>ccls (Python script)</td>
<td>Carbonyl carbon label selective (CCLS) (^{1}H-^{15}N) HSQC. Reference: M. Tonelli, L.R. Masterson, K.Hallenga, G. Veglia, J.L. Markley, JBNMR (2007) 39:177-185</td>
</tr>
<tr>
<td><strong>ccls.x</strong></td>
<td>ccls (Python script)</td>
<td>Waltz16 replaces (^{1}H) 180° pulse for 1H-decoupling to enhance S/N by ~10%.</td>
</tr>
<tr>
<td><strong>hzqc</strong></td>
<td>hzqc (Python script)</td>
<td>Zero-quantum TROSY for methyl groups. Use AU program “shear” to process the data so that the peaks appear in normal positions. With nmrPipe, macro expY_hzqc.M must be used to recover the peak positions. Reference: V. Tugarinov, R. Sprangers, and L.E. Kay. JACS 126, 4921-4925 (2004)</td>
</tr>
<tr>
<td><strong>r1rho_adi.0</strong></td>
<td>R1R2 (Python script)</td>
<td>Measure (R_{1p}) and (R_{2p}) using adiabatic 180° degree pulses. Reference: S. Mangia, N.J. Traaseth, G. Veglia, M. Garwood, and S. Michaeli. JACS 132, 9979-9981 (2010)</td>
</tr>
<tr>
<td>methyl_hsqct2gp.x</td>
<td>methyl_T2 (Python script)</td>
<td>Methyl $^1$H-$^{13}$C single or multiple quantum relaxation dispersion experiment</td>
</tr>
<tr>
<td>-------------------</td>
<td>--------------------------</td>
<td>------------------------------------------------------------------</td>
</tr>
<tr>
<td>methyl_hmqct2gp.x</td>
<td>methyl_T2 (Python script)</td>
<td>Methyl-TROSY $^1$H-$^{13}$C multiple quantum relaxation dispersion experiment</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Reference: D.M. Korzhnev, K. Kloiber, V. Kanelis, V. Tugarinov, and Lewis E. Kay. JACS 2004, 126, 3964-3973</td>
</tr>
<tr>
<td>CH2Dtr2h.x</td>
<td>methyl_DT2</td>
<td>Methyl-TROSY 2H (deuteron) relaxation dispersion experiment</td>
</tr>
<tr>
<td>CHD22h.x</td>
<td>methyl_DT2</td>
<td>Methyl 2H (deuteron) relaxation dispersion experiment</td>
</tr>
</tbody>
</table>
| T1H2ref | T1H2 (Python Script) | Measurement of $^2\text{H} \: T_1$ and $T_{1\rho}$.
|---------|----------------------|------------------------------------------------------------------|
| nt1rhocp.x | nt1rhocp | 1D on- and off-resonance 15N $T_{1\rho}$ measurement with cross polarization for selectivity and 3919 for water suppression
Kay and coworkers, JACS 127, 713-721 (2005) |
| ct1rhocp.x | ct1rhocp | 1D on- and off-resonance 13C $T_{1\rho}$ measurement with cross polarization for selectivity
| trosyetf3gpiasi.x | rpar TROSYETF3GPI; change pulprog and a few parameters as suggested | Mapping chemical exchange in proteins with MW $>$ 50 kDa
| **wexIIltrosoy** | **wexIIltrosoy (Python Script)** | Measurements of rapid hydrogen exchange (HX) of water with protein amide sites.  
**Reference:** N.C. Fitzkee, D.A. Torchia, and A. Bax, **PROTEIN SCIENCE** 2011 VOL 20:500—512. |
|-------------------|----------------------------------|---------------------------------------------------------------------------------------------|
| **QHSQC**         | rpar HSQCETGP all getprosol pulprog QHSQC td0 4 gpz10 33.1 gpz11 66.4 | Quantitative 2D HSQC (Q-HSQC) via suppression of J-dependence of polarization transfer in NMR spectroscopy: Application to wood lignin.  
| **SpinEchoig30gp** | c13 (Python script) | Z-restored spin echo for $^{13}$C spectrum of straight baseline  
| **ipcosyesgp-tr** | ipcosy (Python script) | IP-COSY, a totally in-phase and sensitive COSY experiment  
Thank You for Your Attention!
Solid-State NMR at MNMR Center

T. Gopinath
Solution state NMR and solid state NMR

Isotropic motion

Solution state NMR

Isotopic motion

Solid state NMR

H–H J-coupling 0-20 Hz
N-H J-coupling 150 Hz

H–H Dipolar coupling 50-100 KHz
N-H Dipolar coupling 10 kHz

Hamiltonian:

\[ H = H_{CS} + H_J \]

Chemical shift:

\[ H_{CS} = \gamma (1 - \sigma_{iso}) \hbar B_o I_z \]

J-coupling:

\[ H_J = 2\pi J_{ij} \vec{I}_i \cdot \vec{I}_j \]

Order of Hz

Hamiltonian:

\[ H = H_{CS} + H_J + H_D \]

Chemical shift:

\[ H_{CS} = \gamma (1 - \sigma) \hbar B_o I_z \]

Dipolar coupling:

\[ H_D = D_{ij} (3I_z S_z - \vec{I}_i \cdot \vec{I}_j) \]

\[ D_{ij} = b_{ij} (3\cos^2 \theta_{ij} - 1) \]

\[ b_{ij} = \frac{\mu_0 \gamma_i \gamma_j \hbar}{r^3} \]

Order of kHz
Oriented- and MAS-SSNMR spectra of a dipetide (NAVL)
Magic Angle Spinning

Magic Angle

Rotor

Magic Angle Spinning = 8001 Hz

$^{13}\text{C} \text{ (ppm)}$

$^{13}\text{C} \text{ (ppm)}$
Oriented sample or static solid state NMR

$B_0$

$^{15}\text{N (ppm)}$
Solid State NMR (SSNMR)

Magic Angle Spinning (MAS) SSNMR:
(1). The sample can have molecules of multiple orientations.
(2). Anisotropy is suppressed by MAS.
(3). Anisotropy can be recoupled.

Applications
Membrane proteins, soluble proteins, Amyloid fibers, and wide range of molecules used in Material science and Pharmaceuticals.

θ\textsubscript{m} (Magic Angle) = 54.7°

Oriented SSNMR:
(1). The sample should have molecules of single orientation.
(2). Anisotropic interactions are mapped in the spectrum.

Applications
Membrane proteins, single crystals, and liquid crystalline molecules.
Solid State NMR (SSNMR)

**Magic Angle Spinning (MAS) SSNMR:**

1. The sample can have molecules of multiple orientations.
2. Anisotropy is suppressed by MAS.
3. Anisotropy can be recoupled.

**Oriented SSNMR:**

1. The sample should have molecules of single orientation.
2. Anisotropic interactions are mapped in the spectrum.

**Applications**

- Membrane proteins, soluble proteins, Amyloid fibers, and wide range of molecules used in Material science and Pharmaceuticals.

**θ_m (Magic Angle) = 54.7°**
Probes

600 Varian spectrometer:

**MAS:**
1. $^1$H/$^{31}$P-$^{13}$C/$^{13}$C-$^{15}$N 3.2mm Varian HXY BioMAS, (sample volume 36 $\mu$l)

**Static:**
2. $^1$H/$^{31}$P-$^{15}$N Varian HX BioStatic rectangular coil for glass plates (300 $\mu$L)

700 Varian spectrometer:

**MAS:**
1. $^1$H/$^{31}$P-$^{13}$C/$^{13}$C-$^{15}$N 3.2mm Varian HXY BioMAS (36 $\mu$l)
2. $^1$H/$^{31}$P-$^{13}$C/$^{13}$C-$^{15}$N 4.0mm Varian HXY T3 MAS (50 $\mu$l)

**Static:**
3. $^1$H/$^{31}$P-$^{15}$N Varian HX BioStatic rectangular coil for glass plates (300 $\mu$L)
4. $^1$H/$^{31}$P-$^{13}$C/$^{13}$C-$^{15}$N Varian HXY BiCell round coil for bicelles (150 $\mu$L)
5. $^1$H/$^{15}$N MagLab (Tallahassee, FL) HN rectangular coil for glass plate samples (300 $\mu$L)
6. $^1$H/$^{15}$N or $^1$H/$^{13}$C MagLab HX round coil for bicelles (150 $\mu$L)

700 Bruker spectrometer (Metabolomics/solids):

**MAS:**
$^1$H-$^{13}$C-$^{15}$N 3.2mm E-free probe, sample volume 30 $\mu$l
Experiments

Cross polarization

SSNMR of polylactide (PLA):

Menthol with PLA
An insensitive $^{13}$C edited experiment can be combined with two or more $^{15}$N edited experiments which are relatively more sensitive, or vice versa.
3D DUMAS-NCACX-CANCO on uniformly $^{13}$C, $^{15}$N labeled ubiquitin.

Intra residue correlations.

Inter residue ($i$, $i+1$) correlations.

Total experimental time ~ 5 days

Static solid state NMR of sarcolipin in lipid bicelles
MNMR New User Orientation
Safety

**DANGER**

**STRONG MAGNETIC AND RADIO FREQUENCY FIELDS ARE PRESENT**

**PACEMAKER AND PROSTHETIC HAZARD**

Operation of certain cardiac pacemakers can be inhibited resulting in death or serious injury to the user.

Pacemaker and metal prosthetics users should establish safety requirement with their physician and pacemaker manufacturer before entering.

**Magnetic items can suddenly fly towards the magnet and cause serious damage or personal injury.**

Keep all tools, equipment, and personal items containing steel, iron, and other magnetic material at least 2 meters (6 feet) away from the magnet.

**The magnetic field can disable credit and ATM cards and damage watches.**

Keep credit and ATM cards away from the strong magnetic field.

PLACE THIS SIGN ON ACCESS DOORS TO NMR SPECTROMETER ROOM.
Entrances and Exits

- During business hours, either entrance can be used.
- Please use lower level entrance after hours.
- Remember that the Mayo corridors are not secured after hours.
- All other doors should be treated as emergency exits. Be familiar with where all exits are.
- Lights are on auto timers.
2nd Floor Access
General Policies

• Scheduled time must be canceled at least 24 hrs. in advance or charges will apply.
• Time is scheduled through the web site and subject to change. Generally first requests accepted unless other circumstances apply.
• Completed access form required for card access.
• All new users are required to take workshop or training. No untrained users allowed.
• Pump lines on 8501 and 9001 are extremely sensitive. Stay away from these areas.
Other Amenities

• Offline computer room with offline workstations & software
• Conference room available through the center. Contact Todd or Eric for scheduling.
• Food and drink should be kept in the break room area.
• Wet lab space available for use. Biohazards may be present.
File Management

- Offline datastations are the remote gateway to your data
- Offline datastations are accessible from any .umn.edu computer
- *Users are responsible for their own data*
- Please backup any data that is important to you
File Management

Spectrometer Workstations

6001 6002 7001 7002 8501 9001

/nMNMR /backup

nfs
rsync

ssh scp

/MNMR /backup

Server

nfs

Offline Datastations

nmr1 nmr2

.umn.edu

ssh scp
File Management

• Data is saved locally on the spectrometer workstations and on the MNMR server

• Data directories from the MNMR server are mounted to datastations and spectrometer workstations in `/mnt/<hostname>`

• There may be a slight delay between collecting data and data appearing on the server
<table>
<thead>
<tr>
<th>System</th>
<th>Hostname</th>
<th>IP address</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bruker Avance III 900</td>
<td>magneto9001.mnmr.umn.edu</td>
<td>160.94.206.63</td>
</tr>
<tr>
<td>Bruker Avance III 850</td>
<td>zod8501.mnmr.umn.edu</td>
<td>160.94.206.62</td>
</tr>
<tr>
<td>Bruker Avance III 700 (2)</td>
<td>vader7002.mnmr.umn.edu</td>
<td>160.94.206.61</td>
</tr>
<tr>
<td>Bruker Avance 700 (1)</td>
<td>megatron7001.mnmr.umn.edu</td>
<td>160.94.206.65</td>
</tr>
<tr>
<td>Varian Inova 600 (2)</td>
<td>gozer6002.mnmr.umn.edu</td>
<td>160.94.206.64</td>
</tr>
<tr>
<td>Varian Inova 600 (1)</td>
<td>kahn6001.mnmr.umn.edu</td>
<td>160.94.206.66</td>
</tr>
<tr>
<td>Offline Workstation</td>
<td>nmr1.mnmr.umn.edu</td>
<td>160.94.206.11</td>
</tr>
<tr>
<td>Offline Workstation</td>
<td>nmr2.mnmr.umn.edu</td>
<td>160.94.206.12</td>
</tr>
</tbody>
</table>
File Management

• Home directory
  `/opt/users/<username>`

• Topspin data directory
  `/opt/users/<username>/data`

• Custom parameters sets, pulse sequences, etc.
  `/opt/users/<username>/lists or vnmrsys`
Experiment Setup Demos