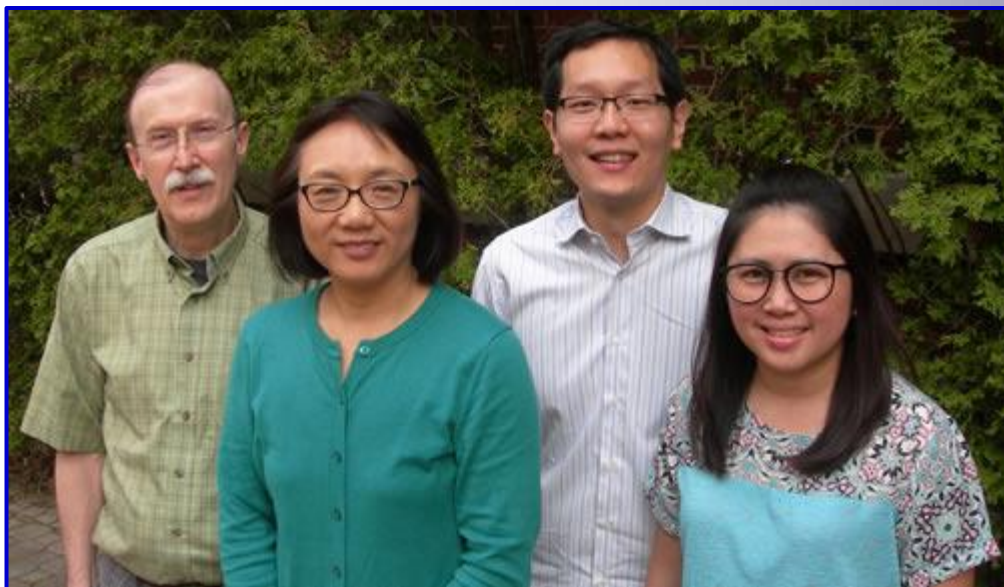


The NMR Lab at the School of Chemical Sciences University of Illinois

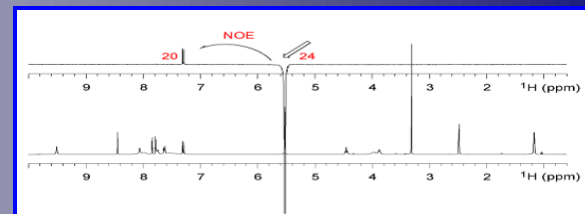
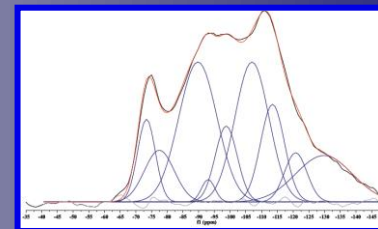


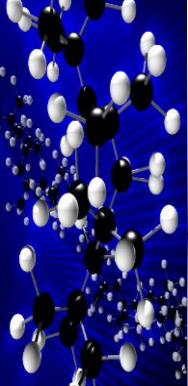
Dean Olson
Director

Lingyang Zhu
Liquids NMR

Andre Sutrisno
Solids NMR

Nikki Duay
Technical Asst.





Nikki Duay

Dean Olson

Lingyang Zhu

Andre Sutrisno

– Technical Assistant since April 2018

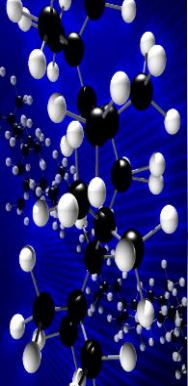
– Director since 2010

– Liquids NMR since November 2011

– Solid State NMR since June 2014

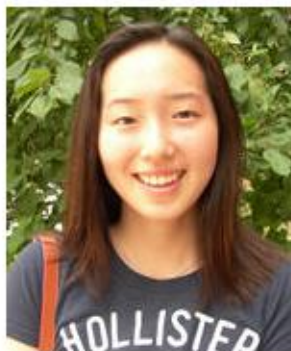
55 Noyes

55 Noyes



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

UNDERGRADUATE HOURLY



Damee Moon

dmoon8@illinois.edu

Responsibilities: Liquid nitrogen fills, spinner cleaning, bore swabs, odd jobs

UNDERGRADUATE HOURLY

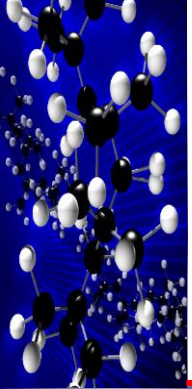


Aliza Siddiqui

Undergraduate Hourly

asiddi48@illinois.edu

Responsibilities: Liquid nitrogen fills, spinner cleaning, bore swabs, odd jobs



Solution State NMR Spectrometers

146 RAL

55 Noyes

NMR Lab Overview



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

500 MHz: CB500
CryoProbe with 45 Nuclei

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

55 Noyes

A151 CLSL

NMR Lab Overview

300 MHz WB Liquids/Solids:

- Varian 5mm H/X probe
- Chemagnetics 7mm/4mm MAS probes



750 MHz WB:

- 1.6mm HFX Y 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



500 MHz WB:

- 3.2mm HCN Balun MAS probe
- 1.6mm HCDN Fast MAS probe
- 1.6mm HFX Y Fast MAS probe



Typically Rienstra Gp

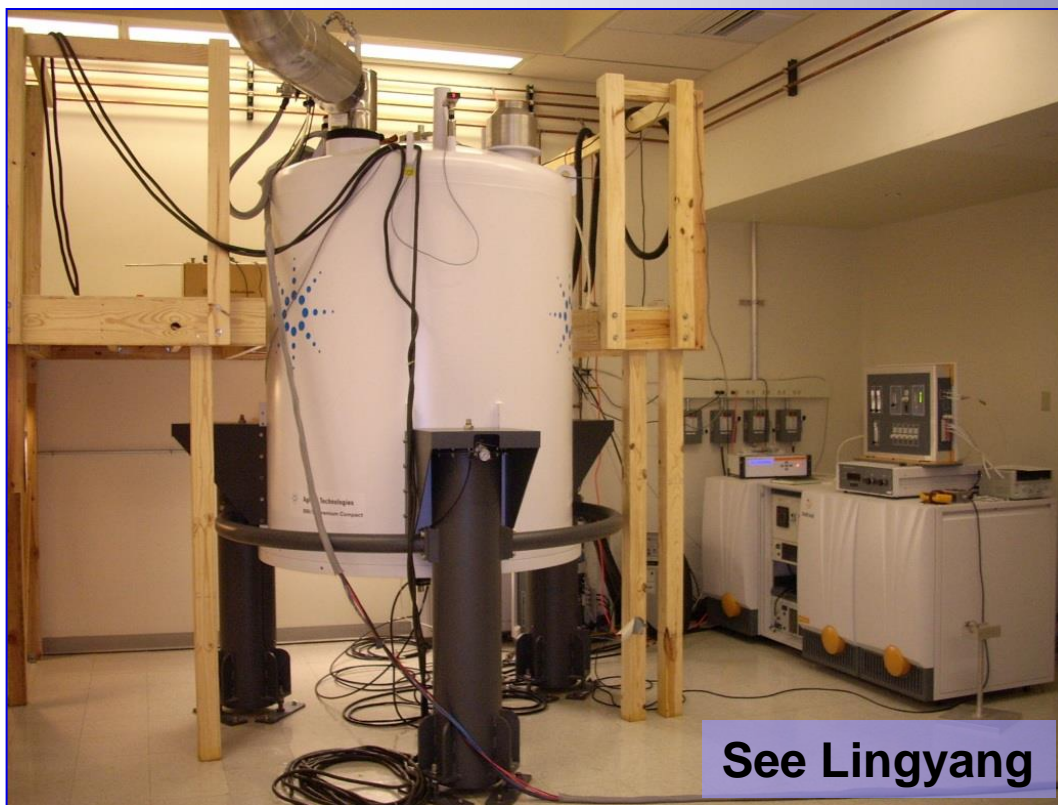
Typically Rienstra Gp



Solution/Solid State 750 MHz NB NMR Spectrometer

A151 CLSL

NMR Lab Overview



750 MHz NB:

- 5mm $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ PFG probe
- 10mm ^{15}N - ^{31}P BB probe
- **10mm ^{73}Ge - ^{15}N BB probe**
- 3mm $^{13}\text{C}\{^1\text{H}\}$ 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 (Nuclear Magnetic Resonance) 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 hourly, 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](#)).

NMR LAB

[Home](#)[Spectrometer Status Now](#)[Carver B500 Sample Queue](#)[ChemFOM](#)[Mnova Software](#)[NMR Staff](#)[Instructional Handouts and Tools](#)[Basic Training Packet](#)[NMR Rate Table](#)[Instrument Information](#)[Schedules/Protocols](#)[Location Maps](#)[55 NL Activities](#)[A151 CLSL Activities](#)[Helium Shortage](#)[Directors' Presentations](#)

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**
- UI400 - **OK** H-1, **C-13**, P-31, F-19; No B-11. Use LN2 bucket for cooling. No spinning.
- UI500NB - **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.

SCS NMR CB500 Status



Automation - Running - Busy until : Tue 13:12 - Day : 00:34 - Night : 05:55

Init OK Field Locked Shimmed RGA Done ZG In Progress Process Data

Help
Logoff

Stop Run
Pause

Logged in as
olsond


Instrument Name
cb500: spect

Holder	Type	Status	Disk	Name	No. Solvent	Experiment	Par	Title/Orig Pri	Time	User	Start Time
15	2	Available									
15	2	Available	/home/data/Hergenrother	RLS-4-35-01-clean1H	10 MeOD	PROTON				svecr	
15	2	Completed	/home/data/Hergenrother	RLS-4-35-01-clean13C	10 MeOD	13C			00:27:12	svecr	10:10 Tue Jan 31 2017
16											
17	1	Completed									
17	1	Completed	/home/data/Denmark	RML-EXP-17-A29-Crude	10 CDC13	PROTON			00:03:59	leisingr	10:47 Tue Jan 31 2017
18	1	Completed									
18	1	Completed	/home/data/Denmark	RML-EXP-17-A30-Crude	10 CDC13	PROTON			00:03:59	leisingr	10:54 Tue Jan 31 2017
19											
20											
21											
22	2	Queued									
22	1	Completed	/home/data/Mitchell	170131_N15Ktryp_1H	10 CDC13	PROTON			00:01:17	schwalench	10:42 Tue Jan 31 2017
22	1	Queued	/home/data/Mitchell	170131_N15Ktryp_COSY	10 CDC13	COSYGPSW			03:56:19	schwalench	21:30 Tue Jan 31 2017
23	1	Running									

Date	Time	Holder	Name	No.	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Title	Remarks
2017-01-30	17:33:39	24	2017-01-30_JYL_5_34_1H_HSCQ_dMeOH_CB	10	HSQCEDETGP	✓	✓				✓	✓	leejuyeon		
2017-01-30	17:27:27	24	2017-01-30_JYL_5_34_1H_COSY_dMeOH_CB	10	COSYGPSW	✓	✓				✓	✓	leejuyeon		
2017-01-30	17:22:14	24	2017-01-30_JYL_5_34_1H_dMeOH_CB	10	PROTON	✓	✓	✓	✓	✓	✓	✓	leejuyeon		
2017-01-30	17:07:01	6	01302017CJR4-113F21-25'	11	13C	✓	✓				✓	✓	reinhardt	sref: refe	
2017-01-30	17:01:29	6	01302017CJR4-113F21-25'	10	PROTON	✓	✓	✓	✓	✓	✓	✓	reinhardt	sref: refe	
2017-01-30	16:43:44	50	ZW5-51A-CNMR	10	13C	✓	✓				✓	✓	wuz	sref: refe	
2017-01-30	16:39:02	50	ZW5-51A-FNMR	10	F19	✓	✓	✓	✓	✓	✓	✓	wuz	sref: NU	
2017-01-30	16:22:47	8	2017-01-30_JYL_5_36_deBoc-sulfoxide_1H_HSQC_dMeOH_CB	10	HSQCEDETGP	✓	✓				✓	✓	leejuyeon		
2017-01-30	16:17:43	8	2017-01-30_JYL_5_36_deBoc-sulfoxide_1H_dMeOH_CB	10	PROTON	✓	✓	✓	✓	✓	✓	✓	leejuyeon		
2017-01-30	15:59:48	10	PCK_AmAzButane	11	13C	✓	✓				✓	✓	zhouc	sref: refe	
2017-01-30	15:54:59	10	PCK_AmAzButane	10	PROTON	✓	✓	✓	✓	✓	✓	✓	zhouc	sref: refe	
2017-01-30	15:49:59	9	kar-17-A32-f4-6-31P	10	P31	✓	✓				✓	✓	robbk	sref: NU	

NMR Training

Director e-mails 26 items:
NMR principles, Mnova, i.p. address, etc.



Get a booklet
and see Nikki
for paperwork
& ChemFOM

Study and exercises;
Training by Liaison

Check-Out by
Director

Gateway Instrument

UI500NB Training
by Director
(2D-NMR Prep)

Special 1D,
2D/3D-NMR Training
by Lingyang

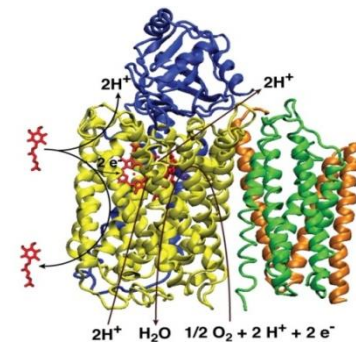
Temperature Control
(VT) Training
by Director

- pw90 calibration
- T₁ calibration
- Solvent suppression

- Required for UI600 and 750NB
- One key 2D document:
UI500NB Advanced 1D and 2D NMR

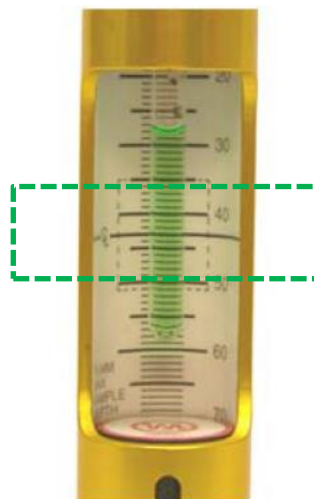
Solid State NMR
Training
by the Solids
Spectroscopist

A Common Problem



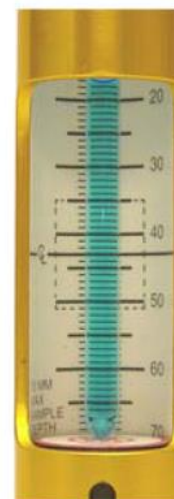
0.3mL
Positioned too low

Solvent NOT in
detected region



0.4 mL
Centered

Solvent covers
detected region



0.7mL
Adjusted to
Maximum Depth

Recommended

From an MIT web document

A Common Problem - getting this backwards

LOCKED SPIN: 20 VT: OFF

[757]				
20	-1+	-4+	-16+	-64+
[9]				
lockpower	-1+	-4+	-16+	-64+
[44]				
lockgain	-1+	-4+	-16+	
[103]				
lockphase	-1+	-4+	-16+	-64+
[20]				
spin	-1+	-4+	-16+	-64+

Best if Lower

Best if Higher

Temp. See lab staff.

A Few Under-Appreciated NMR Data Processing Shortcuts

SOME UIUC VARIAN NMR MACROS OF INTEREST:

ffa	f full aph cdc dc
disp	f full aph cdc dc vsadj dscale
proc	wft f full aph cdc dc ds vsadj dscale
ppmh	wp=10p sp=-0.5p
diff	r1=delta r1? [displays the difference in Hertz between two cursors]
doi	f full intmod='partial' cz cdc dc isadj
ipart	intmod='partial'
plot	pl pscale pap page
plotT	pl pscale pltext page
plotI	vp=12 pl pscale pir pap page
plotA	pl('all') pap page
plotAs	pl('all') pscale pap page

p7 references the chloroform ^1H 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 (r1 = Hz)
nl vsadjcr adjusts selected peak to be the maximum on y scale

Bruker NMR - 500 MHz CryoProbe

Since July 2016

BRUKER



Carver Bruker 500:

CB500

**Funding from the
Carver Charitable
Trust: \$500K**

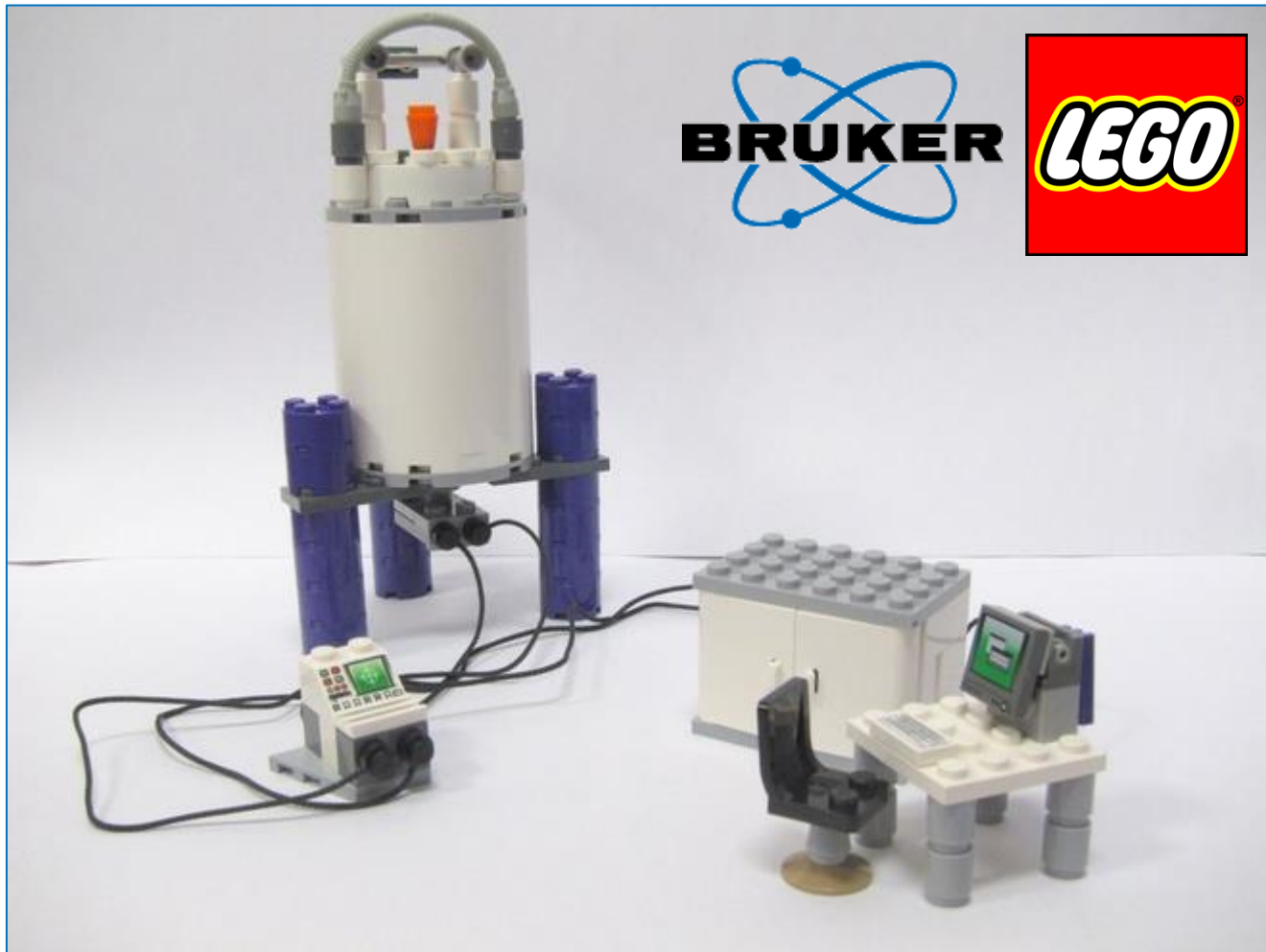
**Total Purchase
Price: \$895K**

Today: \$1.1M

Bruker NMR - 500 MHz CryoProbe

NMR Training Simulator

NMR Lab Overview



Why Visit the NMR Lab?

“I wish I had done more NMR sooner.”

- Quote from a 5th year grad student