

CHEMISTRY

AT THE UNIVERSITY OF NORTH CAROLINA AT CHAPEL HILL

Nuclear Magnetic Resonance Laboratory Newsletter

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NMR Assignment Service

by Andrew Camp

The NMR assignment service is designed to rigorously characterize a molecule for publication and/or intralab use. NMR data will be worked up, assigned, and condensed to a report in publishable format.

The following services are available:

- **Connectivity Assignment** includes (^1H ; ^{13}C ; ^1H - ^1H COSYDQF; ^1H - ^{13}C HSQC, HSQC-TOCSY, HMBC) to show bond connectivity of molecules.
- **Absolute Assignment** (^1H - ^1H NOESY/ROESY): Uses NOE assignment to attempt to assign magnetically inequivalent protons. This can provide an absolute assignment of protons.
- **Heteronuclear Assignment** (^{11}B , ^{15}N , ^{19}F , ^{29}Si , ^{31}P): Adds experiments to characterize heteronuclear NMR and show heteronuclear connectivity.
- **Sample Preparation by NMR Staff** is divided into three tiers based on solvent cost
 - ◇ Tier 1: CDCl_3 , C_6D_6 , DMSO-d_6 , D_2O , MeOD-d_4
 - ◇ Tier 2: CD_2Cl_2 , toluene- d_8
 - ◇ Tier 3: THF- d_8

If this option is selected, staff will prepare your sample with the selected solvent (from freshly opened ampules) for NMR characterization.

Advanced Optimized NMR Training

by Marc ter Horst

The NMR Tutorial has been revamped for 2021 as the “Advanced Optimized NMR Training”. You now have the opportunity to expand your practical NMR knowledge of ...

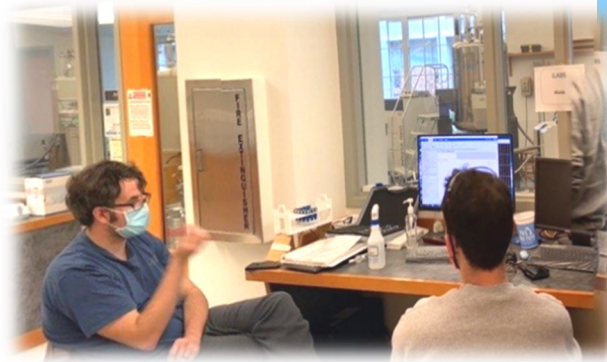
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Advanced Optimized NMR Training

by Marc ter Horst

cont. ... 1D and 2D high resolution NMR experiments for ^1H and ^{13}C . The training sessions will provide you with the understanding and hands-on experience to run high quality NMR spectra and optimize your time spent on the spectrometer.



Virtual sessions will cover topics for optimizing 1D spectra and processing those spectra in Mnova and then expand to include homonuclear and heteronuclear 2D NMR experiments. Between the five virtual sessions, you can practice the techniques on your sample(s) with support from NMR Staff.

Topics for High Resolution NMR Optimization

1. Shimming, optimizing parameters for 1D ^1H spectra and processing techniques in Mnova
2. ^1H decoupling in ^{13}C 1D spectra, parameters for ^{13}C 1D spectra and dealing with baseline roll
3. Optimizing 2D homonuclear experiments (COSY, TOCSY and NOESY), Non-uniform sampling and processing
4. Optimizing 2D heteronuclear experiments (HSQC, HMBC and HSQC-TOCSY), Non-uniform sampling and processing
5. Review of data

Proposed Weeks:

Mar 15 & Mar 22;

Apr 12 & Apr 19

Schedule would like:

Mon	Tue	Wed	Thu	Fri
March 15 Virtual Session 1: Optimize ^1H 1Ds	16 Students run NMR experiments	17 Virtual Session 2: Optimize ^{13}C 1Ds	18 Students run NMR experiments	19 Students run NMR experiments
22 Virtual Session 3: Optimize COSY, TOCSY & NOESY	23 Students run NMR	24 Virtual Ses- sion 4: Optimize HSQC, HMBC, HSQC-TOCSY	25 Students run NMR	26 Students run NMR
29 Virtual Session 5: Discussion of Re-	30	31	April 1	2

How to sign-up in iLab: under the "Request Services" tab, the form is labeled "Advanced Training: Optimizing High Resolution NMR". Users can hit "Initiate request" to get to the form.

NMR Training Request now in iLab!

by Ling Xu

Login to iLab → Request
Service → Initiate Request

Automation Training

(Up to 5 users each session):

Grant access to NEO400, NEO600

- Remote training
- Virtual tour of NMR core
- Introduction to NMR experiments and the specialty of each spectrometer
- Learn to set-up NMR experiments for automation using IconNMR

Manual Training

(1 user each session):

Grant access to 400NB, 500, B600

- In-person training with social distance
- Learn to run NMR experiments manually with TopSpin

Specific Training

(1 user each session):

Grant access to 400NB, 500, B600

- In-person training with social distance
- Variable Temperature
- qNMR
- DOSY
- Others

Automation training:

Wed 2 pm or Thu 10 am

Manual Training: MWF 10 am

Specific Training: TBD

SPECTROMETER SCHEDULE

The spectrometer schedules are changing for the two 600 MHz spectrometers starting in February to accommodate demand for long(er) runs on the B600, as research is ramping back up. The B600 schedule will now open up to allow long term reservations in addition to same-day short run reservations for directly run experiments using Topspin (and not IconNMR). At 5pm M-F, the B600 will then go into automation mode for overnight use and drop off. Since the B600 has been available for routine, automated use, we would like to continue having at least one 600 MHz spectrometer available for routine use. Therefore, the NEO600 will now run in automation mode with IconNMR like the NEO400, see schedules below.

* Note the NEO600 runs ^1H , ^{13}C , and ^{31}P experiments - it does not run ^{19}F .

Current users running the B600 in automation will have access to the NEO600 for daily automated use. If you wish to use the B600 in 'manual mode', request user training in iLab.

Drop off continues. Samples submitted by 5pm will be added to an automation queue to run after 7pm on the B600 and/or the NEO600, Monday through Friday. ^{31}P experiments will be accepted and run on the NEO600 or NEO400, as requests to use the NEO600 specifically.

		Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday
AV3400 (400NB) BBFO BACS (60)	9am-1pm	RSVP time to acquire data allow 10-minute gap for all use Short runs						
	1pm-6pm							
	6pm-9am	Long runs and short runs; allow 10-minute gap						
NEO400 Prodigy probe SampleXpress (60)	9am-1pm	In automation 24/7, RSVP to submit sample(s) allow 10-minute gap between use Each submission must be less than 20 minutes or maximum 4 1H 1Ds						
	1pm-6pm							
	6pm-9am	Automation, long runs and short runs, extended runs by request; allow 10-minute gap						
AV3500 (500)	9am-1pm	RSVP time to acquire data, allow 10-minute gap between use Priority for long runs, short runs reserved same day						
	1pm-6pm							
	6pm-9am							
NEO600 CryoQNP ^{13}C , ^{31}P , ^1H , ^{15}N SampleCase (24)	9am-1pm	In automation 24/7, RSVP to submit sample(s), allow 10-minute gap between use Each submission must be less than 20 minutes or maximum 4 1H 1Ds allow 10-minute gap between use						
	1pm-6pm							
	6pm-9am	Automation, long runs and short runs, possible drop off after 7pm; allow 10-minute gap						
AV3600 (B600) CryoQCI ^1H , ^{19}F , ^{13}C , ^{15}N SampleXpress (60)	9am-11am	RSVP time to acquire data, allow 10-minute gap between use Priority for long runs, short runs reserved same day						
	11am-5pm							
	5pm-9am	In automation, drop off has priority after 7pm, sample submission with 10-minute gap						

Publication Acknowledgements

We are seeking your help in recognizing external funding and promoting the NMR Core! Please acknowledge the **NSF Grant (1828183)** when you use the NEO600 and the NMR Core in general in publications that rely at least in part on NMR data acquired in the NMR Core. And when that paper is published, *let us know by submitting the citation at:* <https://tarheels.live/critclchem/nmr-core/submit-publication/>

For all publications resulting from the use of instrumentation in the NMR Core Laboratory, please include in the publication acknowledgements:

"We thank the University of North Carolina's Department of Chemistry NMR Core Laboratory for the use of their NMR spectrometers."

For use of the NEO600:

"This material is based upon work supported by the National Science Foundation under Grant No. CHE-1828183."

If you are not utilizing this acknowledgment in articles or papers published for scientific, technical or professional journals, you must also include the following disclaimer:

"Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation."

Further details are available at:

Home page:

<https://tarheels.live/critclchem/nmr-core/>

Acknowledgements page:

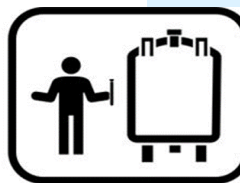
<https://tarheels.live/critclchem/nmr-core/nmr-core-helpful-info/acknowledgments/>



LUNCH



LEARN



NMR

NMR Lunch and Learn
Applied Series:

Ligand Exchange on Quantum Dot Surfaces

Hosted by: Melody Kessler
and Andrew Camp

When: Feb. 24th 12:00 pm

NMR can provide valuable insight into dynamic processes, even giving insight into the surfaces of nanocrystals!

Come chat with Melody about her work looking at quantum dot surface chemistry using NMR, alongside complementary lab methodologies like titration.

Sign Up Link: <https://www.surveymonkey.com/r/8NNMFCT>

*The NMR Lunch in Learn Applied Series is aimed to connect advanced users who use NMR routinely in their projects with other graduate students in a casual setting over lunch. The lunch and learn features a short **virtual** presentation, followed by a Q&A, and time for conversation. Feel free to bring questions on how you can apply the topic to YOUR research questions! Please sign up by following the attached link; we are capping the event size at 12 to keep the event small.*

Don't forget to sanitize and throw away used paper towels after using the computers!