NMR Facility Newsletter



31 August, 2013

Changes and Upcoming Events

There have been quite a few changes in the NMR facility since our last news letter. Some hard to miss (e.g. rooms reorganized), others a bit more subtle (e.g. updated forms and website), and as always a few "under the hood" tweaks that users hopefully enjoy without even realizing they've occurred.

700 MHz Magnet Swap Out



Sept.23 to Nov.8th

As mentioned in our previous newsletter the magnet for the 700 MHz NMR spectrometer (EB-44) has been scheduled for replacement with the new cryostat having arrived more than 6 months ago.

The delay has been due to global liquid helium

shortages. Agilent has finally secured a sufficient supply and we have accepted their schedule. The present magnet will be deenergized or 'ramped down' starting Sept. 23. Removal of cryogens will take several days and then warming the large mass of the magnet will take more than a week. Once at room temperature the cryostat will have to be prepared for transportation by securing internal components and a rigging crew will be required to physically pack the system up for transport.

We'll then bring the new cryostat over from storage at SMS and reverse the installation procedure for the new magnet. The vacuum space on the new magnet will require approximately a week to evacuate and test. Then begins the slow process of cooling the magnet. In previous systems liquid helium or a two stage liquid nitrogen/helium process of pouring cryogens in until cold would be incorporated. Today a "dry-cooling" procedure using ultra cold nitrogen gas is first applied then followed by slow cooling with liquid helium. The magnet will then be energized and the internal superconducting shims set. The magnet will then be allowed to settle for 10-14 days followed by: field mapping, optimization of room temperature shims, performance criteria checked, and finally hopefully acceptance signed in early November.

Once the new system is back online we will need a bit of time for our plan of installing and testing the latest VNMRJ (v4.0) software. The new software should provide the most recent pulse sequences and data processing methods available. More to come...

Things you will see



Updates to the Website, Forms, and Signage

The nmr.chem.ualberta.ca website and associated forms (e.g. Sample Submission, Pricing, Key request) have recently been refreshed. We've also altered the website and moved to simplify the look and feel of the menus and options. The most commonly accessed forms have been moved to the centre of the front page while sub-pages have been reduced in size and number with uniform colour emphasis and fonts which should assist with navigation. The final goal will be to migrate all the materials into the Universities site management software (Site Core).



You may have also noticed that the signage to the spectrometer rooms and safety information has also been updated.

New 'Out of Order' Signs

There are new Out of Order signs at each instrument. The intended goal is for students and researchers to place the sign in-front of the keyboard during a problem. This way while a NMR user goes to seek help, another user possibly unaware of the problem does not compound the issue. For example, a sample is caught in the bore of the magnet and the user correctly stops what they are doing and goes to WB-13 seeking assistance. Before they and the staff can return another user comes in finding the machine idle and attempts to insert their sample. Bad things then ensue.



Signs should be located either on the neighbouring shelf, or directly behind the computer monitors. If you see a sign like this at the spectrometer please call to the NMR staff at (780)492-2573 **before** removing the sign and attempting to use the instrument.

New NMR Pulse Sequences and Capabilities

Mark has been hard at work installing and testing new pulse sequences. Some will seem familiar



with various improvements, while others look brand new. One example is the H2BC (heteronuclear two-bond correlation) that yields a 2D spectrum specifically showing correlations between a hydrogen atom and the carbon two bonds away. In combination with gHSQC date,

the technique should allow the user to walk through the protonated ¹³C skeleton of a molecule in a fashion similar to an inadequate. Please note that the usage of 3 bond H1-H1 couplings for the technique means that correlations to quaternary carbons will not be present in the spectrum. This has been integrated in the standardized user interface for the v700, i600, ibd5, i400, mr400, and s400. Additional standardized sequences such as Te125-1D, Se77-1D, N15-1D (ibd5, i400, mr400, and s400), and the N15-gHSQC (v700, i600, ibd5, i400, mr400, and s400) have also been set up.

For those running multi-dimensional experiments, a new capability has been extracted from the *BioPack* software for determining 90° pulse angle calibration from a single scan (Nutation theory - Wu, P. S., and Otting, G. (2005) J.M.R.176, 115-119). Ask the NMR staff if you're interesting in trying it out.



Robotic Sampling Handling System Update

(m400 Room W1-19)

The controlling software on the m400 has been updated to match the version of VNMRJ (*i.e.* v3.2) running the other instruments. Most importantly it now matches the software running the u500 robotic sample handler. While the general functionality of the previous version of the software may appear to be similar to the end user, the changes bring a unified interface across both robotic systems, improved automation to the m400, and a more user friendly interface. The experience is now identical between both

robotic systems making training, documentation, and trouble shooting much easier. *Kudos Mark!*

mr400 (we call it "*Mister*") Room SB-3E

The new 400 MHz spectrometer with MR console has been happily running spectra now for several months. Both commercial and academic users have appreciated the quality of spectra. The instrument is now drifting at an exceptionally slow rate of ~0.3 Hz/hr (acceptance tests on older magnets required < 10Hz). In order to provide the most consistent work environment, the software interface has also been modified making the system identical to all other spectrometers with manual sample



handling (*i.e.* no robot). Just to mention of a few of the specifics, the mr400 has attained signal to noise (S/N) results of more than 500:1 (1H) compared to 240:1 for the i400, and the mr400 even beats the 350:1 result for the i500. The numbers for 19F are equally impressive with S/N 688:1, 300:1 and 400:1 for the mr400, i400 and u500 respectively. Just goes to show it's not always the magnitude of the magnet that counts.

Room Modifications

Room SB-3E

Along with the installation of the mr400, came the rare opportunity to completely re-organize the



accessibility of the entire room. Users will immediately recognize the changes, *i.e.* moving the host computer for the i400 out of the middle of the room and sliding the spectrometer consoles closer and over to the back wall. While the changes were small, the result is a vast improvement in the work flow of the environment. Everything from space to sit and assist facility users without impeding in/out flow, to moving cryogen dewars without disrupting users at host computers. We're confident everyone will agree it is a vast

New Steps

Rooms SB-3E (mr400 and i400) and EB-44 (i500)

New wider safer steps for the mr400 and i400, and a proper small stair case with railings for the ibd5 spectrometer have been installed. *Special thanks to Vincent in the Machine Shop for designing and fabricating a new set of stairs. We greatly appreciate the improved safety.*



Education and Direction

DOSY Training Course

Tuesday October 22nd - 9am to 4pm in Room E3-25



Ferenc Evanics is a Field Application Specialist from Agilent Technologies Canada and will be in the department Oct. 22nd to provide a training session

on DOSY. The NMR facility has arranged for

New Facility Training Policy



All users of the NMR facility must receive training and certification by NMR staff prior to using the instrumentation. This is first and foremost for the safety of the user, and secondly for the consistent/reliable operation of the instruments. The NMR facility has an extraordinary record of minimal instrument downtime, and this is due in part to the quality of training as well as the consistency and thought put into the software interface.

We respectfully ask that laboratories no longer rely on third hand training (*i.e.* a student teaching other students). Certification is presently done by NMR staff on a case-by-case basis, and new formalized training protocols and testing documentation is under development. Once complete these documents will be made available on the facility website.

Working Alone Policy



The NMR facility (working with Danny Mah, Fac. Health and Safety Advisor), has

the course to be sponsored by Agilent, and will encompass lectures on background material in the morning with real world step-by-step examples during the afternoon.

Please feel free to bring your own project details for the afternoon session and Ferenc will be more than happy to detail what our Agilent systems and software can do to answer your research needs.

Registration is free, but please RSVP to <u>ryan.mckay@ualberta.ca</u> by Sept. 23rd.

adopted university protocols for NMR staff when working during the evenings, weekends, and holidays in order to provide additional protection.

Along this line for facility users, please don't forget to add the NMR rooms that to vour laboratory's working alone policies and update the room lists and contact phone The room and numbers. phone numbers for each spectrometer are available on our website (<u>http://</u> nmr.chem.ualberta.ca/ spectrometer.htm). The University of Alberta Protective Services (780 - 492 - 5050)o r www.protectiveservices.ualbe rta.ca) offers regular telephone check-ins for researchers working after hours and this can easily become part of your working alone protocols.

Direction (cont.)

New Fees Policy - External vs. Internal Users

Always a touchy subject, but unavoidable in the present circumstances, are fees. While the NMR facility is without fees for internals users, there are reduced fees for academic users outside the department of chemistry, and full fees for commercial users. There have been a few grey areas that are now being clarified.

After discussions with the chair and some of the NMR users, we have decided that all users from outside the department of chemistry will now have fees applied. If the student/research/investigator is not in the department of chemistry, regardless of co-supervision and/or project collaboration, they will be charged as external users starting in September (see http://nmr.chem.ualberta.ca/forms/ NMR_Service_Pricing_2013-2014.pdf). In addition training (see previous page) will now be charged for external users at \$100 per individual. Competency will also be assessed at the end of training and certification given to those who meet facility requirements. Those not meeting the level of qualification will need to arrange for a repeat of training.

Expanded Access to v700

Tuesdays

The Glycomics group has asked the NMR facility to prepare the v700 spectrometer for some general access by the Department of Chemistry. After discussions we've decided that after the magnet swap out is complete, Tuesdays will be made available for general departmental booking via the same registration system that users presently enjoy for the other instruments. Details will be available on the facility website. Glycomics long term experiments may be given access priority, but should there be no long term ongoing acquisitions access will be provided for any group needing the 700 capabilities. Due to the nature and increased financial liability of the 700 MHz cold-probe (estimates for a contaminated probe cleaning range in the 20 to 30k range, with downtime in the realm of months for turn around), the NMR facility will provide additional training to interested students and researchers. Please contact the NMR group should you have any questions.

Spectrometer Resources

Agilent Inc. has recently moved to a web-based forum and repository for corporate information and spectrometer resources. The website is:

https://spinsights.chem.agilent.com/welcome

NMR users can register for free and then have access to user forums, posted problems/solutions, and most importantly the ample documentation that Agilent supplies to customers for spectrometer operation. Keen users are encouraged to browse the resources, and should come ask if something appears potentially helpful.

Note: before posting don't forget that our software here has been extensively modified so you may want to ask the NMR staff before the NMR community.

NMR Service Contact information:

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