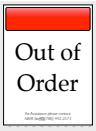
NMR NEWS

mr400 damaged probe | Reminders | Backup Infrastructure



What to do when things go wrong.

Spectrometers are used 24/7/365 and eventually things will wear out. What do you do when a machine isn't working, or when something unexpected happens?

Pg. 2 & 3 … a review

New Training Requirements

We have updated facility training requirements (pg. 4)

(Another) Reminder about Robot Samples

Please don't forget to label your important samples (*e.g.* on the cap) so that if the tape label does not get re-attached properly you can still find your sample. **Don't forget to retrieve completed samples when done.**

mr400 Probe Damaged

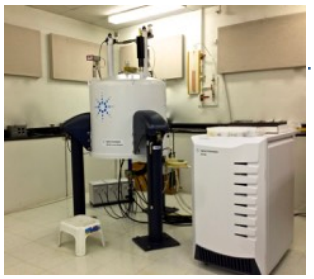
A small routine fix turned into a 21 day repair costing a lot more than we wanted. See pg. 2 for more details.

Donated NMR Equipment

The department has been extremely fortunate and received some substantial donations of NMR equipment. Aubrey Mendonca (inset center) of ChemRoutes Corp., Edmonton was the first, donating their complete 300 MHz NMR system in 2016.

Professor Pascal Legault (inset right) (Université de Montréal) donated an NMR console, probes, and a cold-cycle-chiller (CCC) which is the primary infrastructure needed for cold-probe operation on our 500 and 700.

Professor Paul Tiege (inset left) from Olds College has been very kind and donated their entire 400 MHz NMR spectrometer to the University of Alberta Chemistry department. Details of the stories are provided on Pages 5-7.

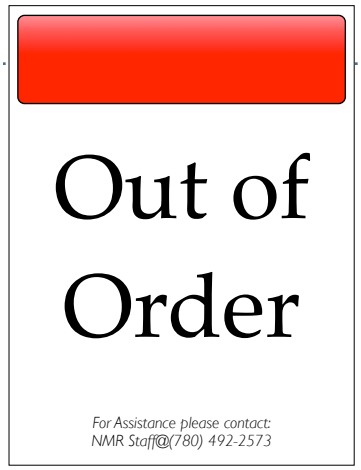


MR400 Probe Damaged - Quartz insert cracked

The mr400's "OneProbe" is presently undergoing a quartz insert repair. Agilent was kind enough to reduce the costs, but we still ended up with a $15,000 bill (after discount) for unexpected damage to our newest instrument. The cracked insert was broken at the bottom, indicating a spinner impact so either someone sent down a spinner without a sample, or didn't use the air ejection prior to inserting a sample. In either case this is a costly, and unnecessary repair. This is the type of damage we desperately need to avoid in the future.

PLEASE do not rush your samples.

What to do when things go wrong? - A review



The first thing is "*Do not panic*".

We all know that moving parts, electronics, power supplies, or pretty much anything you can name will fail eventually. Samples get stuck in magnets. Software locks up. We know this. Our goal is to make sure that everyone knows they are not going to get "In Trouble" for working properly and with care.

Often worse than the equipment failure, is people panic. They get scared that they're going to get blamed.

NMR staff aren't trying to blame. The vast majority of users are energetic, listen, and are very careful. We can solve things as long as we know about the problem. Do not hesitate to contact NMR staff.

So what's so bad about not telling staff?

Here's an example: When a sample gets stuck in the magnet, 90% of the time it's literally a 30 second fix. A student puts the "Out of Order" sign in front of the keyboard and calls the main lab. The instrument is normally back in operation in minutes. However…. when someone leaves without telling anyone, all of a sudden a 30 second fix can turn into days of work when the next unsuspecting user sends another sample down the bore of the magnet. The samples collide, break open, mix, and soak into the electronics. For a cold probe, we could be looking at months of down time and tens of thousands of dollars.

Worse, forgetting to put a sample in a spinner and inserting into the bore creates a ceramic missile heading for the probe head. If we're really lucky the impact doesn't destroy the probe. Unfortunately glass inserts can be cracked reducing instrument performance, electronics are damaged, and we are forced to shutdown an instrument or use inferior backup equipment while we send for very expensive repairs. *(continued on next page)*

Summary

So when something goes wrong ….what should we do?

1.) Don't Panic.

- Please do not try to repair or fix things yourself.
- Broken NMR tubes in depth gauges need to be carefully fixed. Trying to wipe off the solvent from the spinner damages the paint. Trying to clean the depth gauges removes the markings.
- Relax, take a breath.

2.) Contact NMR facility staff (780.492.2573 or email us after hours).

- We have a lot of floors to cover, but we'll get back to you ASAP so sit tight. Help is on the way.

3.) Set the Out of Order sign in front of the keyboard.

- If you can't find one, make one out of recycled paper or grab a fresh page out of the printer etc.

4.) That's it. That's all you need to do.

Its extremely important that users not leave the instrument without any sign of the problem. A broken sample on the table with no information means we have to pull the probe to check if the sample broke inside. Then we have to test everything. Hours of work because we don't know their harmless chloroform sample broke on the table.

Training Requirements - Reminder

The NMR facility requires that all facility users go through formal NMR facility training. For example, getting a lab member to walk you through the facility and sit down with you for a couple of times is not "training". We've found that first hand information directly from staff is more accurate, safer for the user and the equipment, up-to-date, and provides the opportunity for users to ask NMR questions, provide feedback, and get answers based on the best information available.

New users are asked to first review our facility specific safety material

http://nmr.chem.ualberta.ca/safety_manual/safety_manual.html

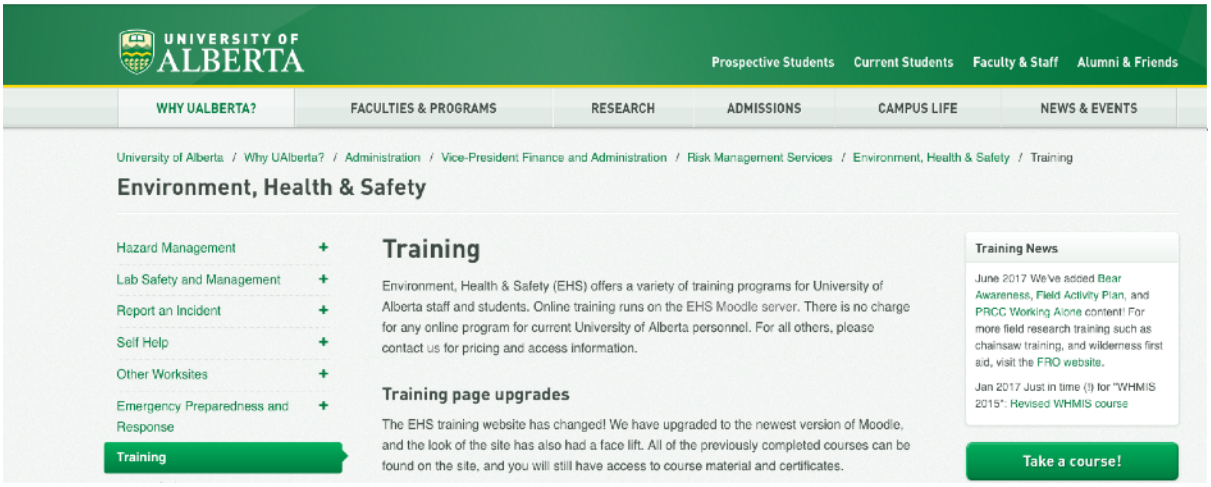
and then book a training time with facility staff (492-2573, room WB-13).

Contact Ryan (2-9950 or ryan.mckay@ualberta.ca) for any further questions or concerns.

NEW TRAINING REQUIREMENTS

In March of 2016, we began to "strongly advise" new users to first participate in the EHS "Laboratory" and "Chemical" safety training courses prior to NMR training. After a year of evaluating the impact on users, starting September 2017 we will be requiring that students complete both the *EHS Laboratory*, and *EHS Chemical Safety* training courses before they are allowed access to the NMR facility. Successful course certificates will be required prior to access approval. Updated WHMIS training is of course still mandatory for the department, and will also be checked. All 3 training courses are relatively easy, only take an hour (or so), are open book, and available at the link provided below.

https://www.ualberta.ca/environment-health-safety/training



Equipment Donations

We've been very fortunate, remembering from previous Newsletters that our liquids NMR spectrometers are all Varian, and that Varian's technology was purchased and rebranded by Agilent back in 2010 (hence some of our equipment actually says Agilent). In the fall of 2014 Agilent made the business decision to exit the NMR market, leaving only market dominant Bruker and a much smaller market share to JEOL. These are the only two remaining producers of superconducting NMR spectrometers. Bruker now offers commercial instruments in the ~300 to 1000+ MHz range, as well as desktop units while JEOL is focusing in the 400-600 MHz NMR market.

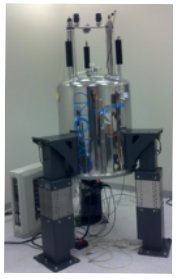
With the exit of Varian/Agilent from the NMR market, we are left with US federal law mandated product support until 2021, but only for our most modern consoles. The Mercury (m400), and Inova (i400, s400, and idb5) consoles are all official end of life (EOL) and only supported so far as existing stock piles of parts hold out.

Therefore spare parts are becoming increasingly hard to acquire, and more aggressively sought after. We are lucky to have support from local companies and organizations.

ChemRoutes Corp. Donation

The first such benefactor to the department was Aubrey Mendonca (ChemRoutes) who donated their 300 MHz Mercury NMR spectrometer to the department. The Mercury console has been helping keep our teaching lab m400 (room W1-19) running these past few years. Unfortunately we do not have any pictures of the system before decommissioning, but the inset picture shows an equivalent magnet for reference.

Olds 400 MHz NMR Donation to Chemistry



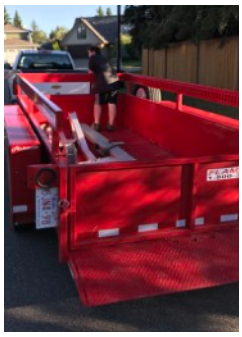
Dr. Paul Tiege at Olds had a very difficult choice to make. The 400 MHz NMR system was simply costing more for cryogens, repairs, and maintenance than his laboratory could support. The department, also under financial constraints, was unable to take over the instrument. The Varian MercuryPlus NMR system was old enough that selling simply wasn't a viable option, and so Prof. Tiege tried to think where the system might still be of the most use. Luckily for us he contacted Mark who had been helping keeping the system running over the years. We determined that the console and improved shims would be a substantial upgrade for the teaching laboratory (m400) instrument. The rest of their system, including the 9.4T (*i.e.* 400 MHz) activity shielded magnet, would be a fantastic back-up for our many 400 MHz spectrometers. (continue next page)

The problem was how to get the system properly packaged and up to Edmonton several hundred kilometres away. Initial cost estimates for professional movers started in the thousands. Another option was needed.

Working with Dirk in the machine shop, we began to evaluate the possibility of moving it ourselves. The problems were getting the 1200 pound magnet off the legs, down to a special securing transport pallet, and then the pallet into a trailer or truck bed. Luckily Olds still had the original transport pallet. A 2 ton gantry crane from United Rentals in Nisku, coupled with chains, couplers, and existing equipment from the machine shop solved getting the magnet off the legs and down to the transport pallet. This left us with ~1500 pounds to move. The catch? The building housing the magnet at Olds college doesn't have a loading dock, so how to get it off the ground and into a truck or trailer?

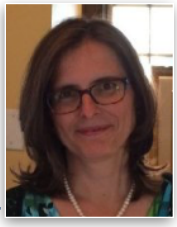
This was a major problem until we found another rental company in Nisku (Flaman Group Rentals and Equipment) that had a brand new type of trailer that was able to self lower to the ground (see left). This was the key that allowed us to move all the equipment through the building and directly onto a transport trailer. Once loaded, the trailer raised itself using a battery/air-compressor system and we were ready to go.

The last piece of the puzzle was how to move the trailer. We needed a truck capable of pulling the trailer (3600 lbs), the equipment (~1500 lbs), gantry crane, and had to be equipped with not only trailer lights, but trailer breaking controllers.



The UofA already had the solution. The motor pool was able to supply a 3/4 ton truck with all the specifications we needed, and after a short trailer-towing training course, we were able to rent the truck. The picture to the right shows the gantry crane in the trailer (our trailer turned out to be red) and truck (left), parked the following evening. We got back from Olds so late, we had to return the equipment the following morning. None of the companies charged us extra for the overnight period and we're grateful to all of them for their help. Dirk and the machine shop were instrumental in helping us prepare for the move and organize all the chains, straps, and the hoist needed to use the crane.

Mark has already installed the newer console and improved shim stack (23 shims versus the older 14 shim system) on the m400 yielding some excellent performance improvements, *e.g.* 33% increase in ^{1}H S/N, and a 50% improvement in line shape/homogeneity.



Closed Cycle Chiller and Console Donation - Université de Montréal

Closed Cycle Chiller (CCC)

Professor Pascale Legault was part of a major CFI application (2012) in collaboration with McGill university. They were fortunate enough to have their application selected for funding, and in the process were able to upgrade several of their NMR systems from the aging Agilent/Varian equipment to cutting edge Bruker instrumentation including new cold probes, and new consoles. They are mostly keeping their superconducting magnets and fitting the new equipment to the existing magnets, with a few new systems included in the total grant.

We contacted Professor Legault last year regarding their Varian/Agilent equipment that was to be replaced, as we wanted to make sure it wasn't just going to a land fill. Dr. Legault had a complete multi-channel 500 console (see example image right), and a multi-channel 600 with cold probe being moved out and upgraded. Dr. Gary Shaw (Western) was also in need of parts for his ailing 600 so requested that console. Working with Dr. Shaw, and Dr. Legault we arranged to take the 500 system (minus the magnet), probes, and the 600's cold probe support infrastructure (CCC). Dr. Shaw had a different generation of cold probe, so he did not need the version of the CCC we were interested in. Several weeks of planning, organizing ground transportation, shipping our special CCC crate out to Montreal, and finally in April flying Mark to Québec to meet with Dr. Shaw resulted in a win-win-win. Dr. Legault got two experts to assist preparing her laboratory for the new equipment and planning how to setup for the new installations (she sent an extensive thank you letter for Mark's help and suggestions). Dr. Shaw was able to get his 600 MHz spectrometer back up to full operation, and the UofA received an entire 500 MHz Inova console (including all amplifiers, frequency generators, boards, etc.), several probes, and importantly a back up CCC to support our v700 and u500 cold probes.

Why is a CCC such a big deal? Not only does this supply us with complete replacement/repair parts, but because a failure in the CCC can result in oil contamination throughout the system we now have the ability to swap the entire unit in a worst case scenario. It can cost more in time and parts to fix a contaminated system, than to buy a new one. Remember however we can't buy a new one anymore, and to get a Bruker or JEOL replacement we'd have to replace the spectrometer console, cold-probe, and other supporting equipment.

All in all we are incredibly fortunate in the support we've received and our very grateful.