Safe Operating Procedure (SOP):
Hazards, Considerations, and Advice on the Decommissioning of a Superconducting Magnet

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[1] See Acknowledgements for list of contributors

Intended Audience

This document is intended as a summation for people not familiar with superconducting magnets (*e.g.* NMR, MS, MRI) but who've been made responsible for the administration, upkeep, and/or decommissioning. It is also intended to provide a reminder for those deeply familiar with spectrometers, but who may never have faced an controlled (or uncontrolled) quench. NMR managers and experts may also find this useful to provide answers to administrations, offices such as Environmental Health and Safety, and any other groups involved in supporting infrastructure. The descriptions and vocabulary are meant to facilitate the broadest audience possible. While in some cases the examples and analogies may be a bit over general, they are meant to provide useful "rules-of-thumb" wherever possible.

Usual LEGALESE

– No liability or responsibility assumed. Information provided as is, intended as a general aid so use at your own discretion.

Now seriously: NEVER MESS WITH A CRYOSTAT UNLESS YOU KNOW WHAT YOU"RE DOING.
The information in this document is meant to help people make informed decisions, and to seek further information.

The amount of electrical energy in a magnet is substantial and easily has enough amperage to KILL YOU.

The cryogens in a superconducting magnet are dangerous and can cause serious harm and even death. Major parts of the hazards are detailed below.

Please do not try anything referred to in this document without getting more information and finding help from people who know what they're doing.

Table of Contents

SOP for Superconducting Magnet Decommissioning

Brief Background

Superconducting magnets (also commonly referred to as cryostats) use coils of specific composite materials (*e.g.* Niobium Tin or Niobium Titanium) that obtain close to zero electrical resistance when cooled to extremely low temperatures. Liquid helium is most commonly used to cool the superconducting material down to 4.2K or -269°C. A great deal of work has been done and continues regarding development of materials which can sustain superconductivity at temperatures above liquid helium levels (*i.e.* high-temperature superconductors or HTS). Unfortunately, nothing has been produced to-date which can be readily wound into a coil and/or contains the current stability necessary for NMR experiments. This field is progressing rapidly though so take into consideration the date of this document.

Relatively cheap and abundant liquid nitrogen is often utilized in between vacuum insulated spaces (see Figure 12) around the liquid helium to prevent radiant energy transfer to the more expensive liquid helium. The vacuum spaces and insulation are placed around and between the cryogens to minimize heat transfer between the various levels.

The more electrical current forced into the coil, the greater the produced magnetic field though current wire technology can only sustain a limited amount of charge density. The lower the temperature the greater the wire capacity and liquid helium in the cryostats can be actively refrigerated (or pumped) down to below 2.2 K. This allows even stronger and stable magnetic fields to be produced. Presently the most powerful commercial research spectrometers being ordered top off at ~1.2 GHz (*i.e.* several ordered but none yet installed) with multiple 900 MHz, 950 MHz and even 1 GHz systems installed and functional[2]. Commercially available NMR systems can now be purchased including self-recycling of helium[3], and very recent developments have introduced a new cryostat design utilizing highly cooled helium gas and evacuated cryostats requiring no liquid cryogens[4]. While these developments are exciting, they are not the systems that will pertain to this document and will not be covered further. We will deal with traditional 4.4K and 2.2K pumped cryostat systems without helium recycling capabilities.

When dealing with the cryostats it’s important to remember that all superconducting coils bleed off a relatively tiny portion of their energy (drift) due to factors such as imperfections in the coil production, and joints between sections. However, if the cryogens and temperature is maintained cryostats can be useably stabile for decades. The rate of magnetic drift over the years of instrument operation, along with records of cryogen consumption can be employed to gauge the health of a magnet. High field drift, high or increasing cryogen consumption, and poor spectrometer performance indicate deep problems that will necessitate the eventual replacement and decommissioning. Typically, cryostats are considered to have a 20-year performance/life expectancy, though of course many have examples of much older fully functional installations.

The magnetic field is generated when energy is deposited into a superconducting coil. Initially this is done by connecting a controlled active circuit to the coil, and adding a tiny amount

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[2] http://www.businesswire.com/news/home/20150917005142/en/

[3] www.bruker.com

[4] www.oxford-instruments.com

of heat to a small intervening section of the coil. This makes the external circuit the “path of least resistance” and energy can be slowly added to, or removed from the superconducting coil. Once complete the tiny applied heating is removed, the internal circuit becomes a complete/low resistance path, and electrical energy circulates internally through the (virtually) resistance free loop indefinitely.

This pathway can of course be reversed by a trained individual with the right equipment. The only other way of removing the electrical energy is to dump it into the surrounding cryogens, but much more on that later.

Decommissioning

Checklist (NMR decommissioning)

Communicate with everyone involved – while seemingly straight forward it’s harder than you think to not miss anyone and to make sure everyone has the same data.
Inform Building Services (Security, Fire Response, Custodial Staff, building maintenance, *etc.*) of the event as early as possible.
Be Flexible, something unexpected always comes up. An initial time window of a week is a good place to start, though even 2 weeks be prudent.
Inform Users well in advance (*i.e.* months if possible) when decommissioning will happen so they can plan experiments.
Check magnet local environment for pipes, wiring, electrical panels, tubes, *etc.* anything temperature **and condensation** sensitive. Remember moisture effects.
Check for fire suppression (e.g. sprinkler heads, smoke detector) equipment
Move items out of the way if/when possible
Prepare an established exit route clear of obstructions for all participants
Have a plan where the gas will go (see below) – stated before but VERY important

Checklist (*Preparing the Magnet*)

Remove probe and samples
Remove shims
Remove bore tube
Disconnect all wiring
Remove any unnecessary cryostat sensors
Arrange for fans to blow down bore and on cryostat
Condensation will happen be ready (*e.g.* bucket under magnet, paper towels *etc.*)

Planned Decommissioning

There are essentially two controlled and one uncontrolled pathway for decommissioning a superconducting magnet. Of the controlled methods, the first involves trained professionals usually from an equipment supplier or 3rd party company using a reversal of the charging process. They would slowly remove the energy from the coil using the same equipment (*e.g.* power supply, leads etc.) at a scheduled time. Each of the original equipment manufacturers (OEMs) can be hired to send experienced people with the appropriate power supplies and equipment to decommission a system. This should always be the preferred route, **especially** if the magnet is to be re-used in a future application. The only disadvantage is price. Good people and good equipment cost money, but if you want to use the magnet again don't cheap out here.

For the decommissioning of a magnet scheduled for replacement or retirement, the sustained viability of the magnet afterwards is not a factor and the price of professionals can become more of an issue. The second controlled method can be done locally, but requires details of how the magnet was charged, *i.e.* current achieved, direction of DC current, and most importantly specifications of the connecting pins through the charging apparatus (often called a demountable lead, charging stick, or charging rod). Without the cryostat specifics, wiring information, and/or appropriate equipment this method of decommissioning is more challenging. There is rarely established funding for decommissioning, and with funding growing ever more scarce the options presented here become more applicable.

If one has the original equipment: shorting plug removal tool (Figures 1-3), demountable lead (Figures 4-5), operator for cryostat vacuum (Figure 6), and information on the pin functions (*e.g.* detailed manual) it is possible to slowly drain the energy from the magnet and purge the vacuum.

Figure 1 Example of a Shorting plug removal tool with ruler for scale

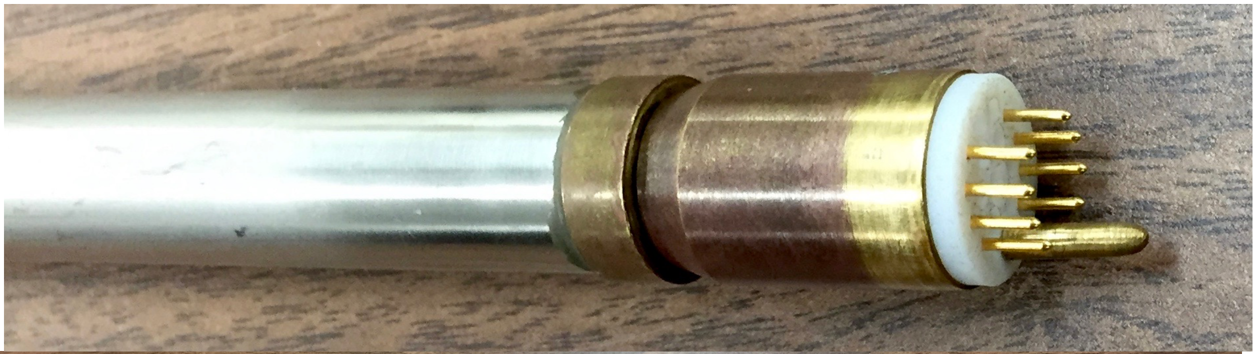


Figure 2- Expansion of a shorting plug that must be removed from the cryostat prior to decommissioning.

Figure 3– Shorting Plug beside Tool

- **If you do not have this extraction tool or if the pins are broken inside the cryostat, draining the energy slowly is not an option by any method.**

Figure 4– Demountable Lead example from Oxford 400 MHz NMR

Figure 5 – Demountable Lead Head

Figure 6 – Operator for Cryostat Vacuum.

With all of the equipment for your cryostat, it is possible to either hire a professional with the appropriate power supply, or use a home-made solution. We will give some details of the in-house solution.

Draining the Energy

Please note: Introducing equipment and connections into the core of the magnet comes with electrical risks to the operator, possible damage to the magnet, and the risk of a quench (see next section).

Our basic steps when decommissioning are to: remove the shorting plug, connect the correct demountable lead, and finally connect a diode (* ours is a double diode so current direction is not an issue, single diodes must be connected in the correct direction or a quench will result) and heat sink (Figure 7). Once this is all in place and double checked, we then use a modified 12V power supply (500 mA) (Figure 8), connected to the correct pin connectors (Figure 5 - small multi-pins) on the demountable lead. Once plugged in, the small transformer supplies heat to an internal section of wire effectively closing the new external circuit (*remember that a "closed" circuit is functional). The power then drains slowly through the diode and into a constant load (*e.g.* heat sink). Without heating the small section of internal coil the external path is so relatively resistive that no functional circuit exists.

Figure 7 – Double Diodes for either current flow direction, connected into heat sink.

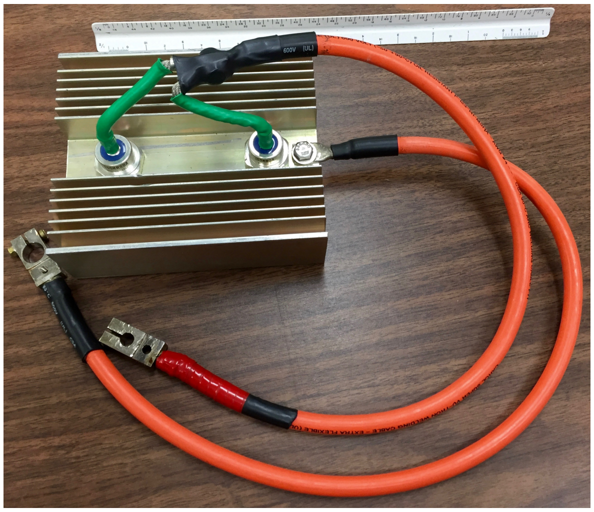
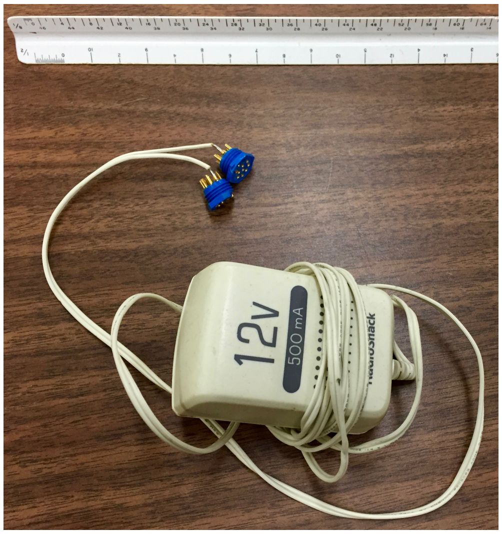


Figure 8 – Power Supply and Pin Adaptor



While this may sound easy, having the correct extraction tool, successfully removing the shorting plug without damaging any pins or freezing a rod in place, possessing and successfully connecting the right lead into the magnet core, knowing the pin functions, having an appropriate controlling power supply or the correct diode/heat sink is all non-trivial. As noted above and in the figure legend, the load we use has a dual channel diode so knowing the direction of current is not absolutely critical. Below is an example of a unidirectional diode heat sink which will cause a quench if not correctly used. We include this to help recognize a potential problem if you should have one available.

Figure 9 – Single Direction Diode

In our experience attempting the home-made solutions have about a 50% chance of success (*i.e.* spontaneous quench, damaged pins so can't use, missing demountable leads, no documentation *etc.*).

Talking with several commercial magnet installers, they have been very successful using the diode and heat sink combination but emphasized that the correctly sized diodes (*i.e.* break down voltage capacity) are absolutely necessary. They also relayed a situation where the correctly chosen diodes had lost their effectiveness over time and caused several quenches before being identified and replaced. This emphasized that even equipment successfully used before is not a guarantee. Appropriate precautions must be taken before attempting any solution.

For magnets that are to be reused or sold, it is our recommendation that professionals with professional equipment be utilized whenever possible. For magnets being permanently decommissioned the information provided should act as a guideline and one should seek help from people with equipment and experience.

The final option for decommissioning is to force a quench manually. This can be done either by letting all the cryogens boil off naturally (but no set time therefore scheduling is not possible), or force the cryogens out (can still take many hours depending on magnet and cryogens), or use an emergency "quench" capability if provided with the instrument. The third option is relatively rare.

QUENCH

Assuming one does not have a "quench" capability included with the instrument, there are then only two possibilities left. Either one lets the helium level naturally drop down so low the coil is exposed and the temperature is not maintained (slow and random quench), or one forces the cryogens out by spoiling the vacuum insulation and/or pushing the cryogens out reversing the procedures used to fill the magnet (quicker).

It should be noted that quenches can occur spontaneously (*i.e.* without external intervention), and while very rare this is certainly the most dangerous situation. In an unplanned quench, personnel will not have a chance to vacate the area before the event. People may even be in close proximity and/or dangerous positions (*e.g.* up on a ladder switching samples, or under changing probes). Automatic ventilation systems (see section below) will hopefully already be in place. For example, duct work should be in place to move the majority of gasses outside as quickly as possible.

Another rare but possible situation is a building fire. In this case the vacuum space might be suddenly compromised once the cryostat has reached a particular temperature and the O-ring fails. In another example, multiple ice blockages might restrict or halt the release of vaporized cryogens allowing pressure and temperatures to rise. In these cases, the quench and subsequently release of gasses can exceed the exhaust capacity and over pressurization can occur. These situations while extremely rare, do occur and interested readers are directly to an online search where multiple videos are available.

So what happens in a Quench?

A portion of the magnet coil becomes non-superconducting and the energy of the coil is dumped via a safety circuit into the surrounding cryogens to save the very expensive coil material. To my knowledge, every system has some sort of quench protection system in place to release the energy through the cryogens[5].

While the safety circuitry may save the coil and possibly allow the magnet to be re-cooled and re-energized later, it does not protect the occupants of the cryostat area. Depending on the size of the instrument and the cryogens on board, there can be a sizeable volume of vaporized gasses emitted in a very short period of time.

How does one cause a Quench?

As stated, the magnet will quench if the solenoid coil rises above the stable operating temperature. The stable temperature limit depends on the coil size, cryostat design, and most importantly the charge density deposited in the coil. A rise in temperature can result from many factors such as: insufficient cryogens, mechanical shock, local magnetic field changes, and contaminated cryogens. It can take very little physical energy transferred to the coil to cause a problem. A quench can occur from something seemingly benign, or a magnet can survive events that would be expected to cause a quench; there is a great deal of variability. In our experience, magnets have quenched from a small insult (*e.g.* heating and stopping heating of the switch closure during decommissioning), though others have survived presumably catastrophic events (*e.g* purposefully spoiling the vacuum space with no effect) that were thought to immediately destabilize a magnet. It is impossible to accurately predict what a cryostat will do. We often spend a great deal of time and effort to safe guard the system sometimes for decades and attempting to force a critical event is certainly counter-intuitive. Spectroscopists can develop an emotional attachment for systems, therefore arranging for a decommissioning can require some empathy, patience, and understanding. Communication helps all of these.

If there are no other options (*e.g.* shorting plug pins broken off in cryostat core, no demountable lead available *etc.*) and a forced quench is the only option for decommissioning, then one must do something rather decisive such as remove the cryogens protecting the magnet core. In the past we have done this by briefly spoiling the vacuum space with a small burst of dry good quality (don't bother with highest purity) nitrogen gas, and observing the cryogen boil-off rate. Dry cylinder nitrogen gas is used as we want to avoid ice build up inside the vacuum space. We want to prevent freezing large quantities of nitrogen or other gases inside the vacuum space as the liquid helium will "cryopump" the heat out of any gas, freezing it to the sides.

Spoiling the vacuum space can cause an instantaneous quench, as presumably the introduced heat will boil off some helium gas inside the cryostat. However, an immediate quench might not occur. Instead we have observed slowly increasing helium boil off. To speed things up, the cryogens can be pushed out with the appropriate gas using a reversal of the filling processes. Specifically, we have removed the liquid nitrogen, and blown out the majority of helium liquid in a system before a quench was finally achieved. A far longer process than thought. A quench from lack of helium can burn out the magnet internal protection network, reportedly

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[5] If you know of common cryostats without this capability, please let me know so we can include.

causing black smoke to be ejected (we have never experienced this but an AMMRL contributor has). A quench from lack of $He_{(liq)}$ is sometimes referred to as a "black quench".


Figure 10 – Quench Button

For one of our systems a "Quench Button" was included (Figure 10). This device heats a section of the wire, and the lead is permanently installed so no other equipment is required. Activation caused the expected dumping of the electrical energy into the safety circuit. No vacuum spoilage was necessary. Quench capability is added to higher field and larger stray field spectrometers as a safety feature in the event someone is pinned against the magnet by a large metal object. Not all systems have this addition, or even the option for inclusion. The hazards presented by the quench itself however must be controlled or the individual at risk may be in even great peril.

Identifying Hazards and the Magnet Contents

Asphyxiation - Oxygen Depletion Hazard

The most immediate hazard is vaporized helium gas filling the room and displacing available oxygen. As both helium and nitrogen gases are invisible and pretty much non-reactive they are hard to identify, but fortunately only the physical hazards are concerning (i.e. no corrosive or toxicity issues). However, even small changes in oxygen levels can have immediate effects on room occupants long before an asphyxiation danger. Major gas suppliers have excellent articles available online detailing oxygen displacement effect hazards on people and we will summarize briefly below.

For example, oxygen levels between 18-21% are considered normal with no expected effect on people. However even dropping down a few percent can interfere with a person's cognitive abilities and physical stamina. Once down below 11% people will begin to pass out depending on conditioning and physical activity. This can occur without warning and posses a real danger of injury, *e.g.* a person could strike their head "on the way down". While spectrometer user asphyxiation is extremely uncommon and would require a large amount of vaporized cryogen gas in an enclosed small space, passing out and injuries due to falling are much more likely. Once below 11% feinting becomes much more likely regardless of conditioning and even if remaining still. Very low oxygen levels for extended periods (*e.g.* 0-7% oxygen) can result in brain damage even if resuscitation is performed. Remember, the brain does not do well in low oxygen environments, and everything is worse for extended periods.

Extreme Cold Hazard

The second hazard is potential cold damage from the very cold, rapidly escaping, and expanding cryogens. The helium gas will be absorbing a substantial amount of heat energy and expanding rapidly as it exits the cryostat. While individuals can hopefully leave the area safely, infrastructure in and around the spectrometer will be effected. In a laboratory environment this can easily include domestic hot and cold water pipes, chilled water lines for equipment cooling, electrical, natural gas, vacuum, steam, distilled water, fume hoods, and normal building heating/cooling air handling. Even when full quench ventilation is in place care must be taken in regards to what is outside the building but in the exhaust path. For example, we have experienced a large unscheduled quench resulting in frosting of car windshields in the neighbouring parking lot across from the quench discharge vent on a warm fall day.

Also, alcohol vial type sprinkler heads can activate from the cold gas. All of these possibilities should be communicated to the Fire and Safety team ahead of time. They should be told that the chance of an actual fire is null, but the cost of their sprinklers dousing a console or flooding the entire building is considerable!

Over Pressurization from Ice Blockage(s) – Potential or Existing

The final significant hazard is the trapping of expansion gasses and potential for explosion should the pressure be allowed to build. Helium and nitrogen expand ~700 fold from liquid to gaseous form (*i.e.* cold liquid to room temperature and pressure). Normally pressure relief valves are in place when liquid helium and nitrogen are handled, but in the case of a rapid unscheduled release of cryogens freezing and blockages can occur. In other cases, de-icing heat-sinks and one-way check valves can be accidently left off of exhaust stacks (*e.g.* after fills) allowing back pressure of atmosphere into the cryostat. Moisture from the air can then form ice blocks. The internet has several dramatic examples of dewars and even cryostats failing catastrophically. These videos are great for teaching purposes. Blockage of the cryogen chambers is not the only pressurization hazard.

Counterintuitively pressure can also build up in the vacuum space if contamination has frozen inside over the decades of operation. This may be more common with systems facing decommissioning as they are presumably at the ends of their service lifetimes. Many modern systems have a pressure release mechanism or “drop plate”, but older systems may not. The purpose of the plate is to avoid any possible pressurization of the vacuum chamber. Check your system, know what is available, and insure that the plate is not interfered with or sealed.

Volume and Type of Cryogens

The physical size of the magnet usually correlates to the volume of cryogens inside the instrument. However, this can also depend on the bore size (*e.g.* wide bore versus narrow), and type of cryostat (*i.e.* actively shielded and/or passively shielded). Wide bore instruments tend to have more cryogens to accommodate the wider coils and typically have larger stray magnetic field space around the cryostat. Actively shielded magnets tend to have larger cryostats to contain the passive shielding and/or active components. Actively shielded magnets can essentially be visualized as having an outer superconducting coil charged in the opposite direction to “squish” the main field and contain the horizontal spread. This containment however usually causes the field lines to extend vertically. Take note of what is being effected above and below the magnet in neighbouring floors as these will be effected by the quench. The sudden loss of a pre-existing magnetic field can perturb surrounding objects and especially other superconducting magnets. Magnets in close proximity have been reported to sequentially quench in a domino like effect when one system fails.

Larger cryostats do not always mean more cryogens as passive shielding can take up a great deal of cryostat volume. A reasonably exact determination of cryogen types (usually liquid helium and liquid nitrogen) and volumes is important when determining hazard potentials. This information should be available in the initial installation documentation. In the event this material has been lost, internet searches and/or magnet forum requests (*e.g.* AMMRL) can be a fantastic source of information. Table 1 is an attempt to supply some general guidelines.

Table 1 – General Common Cryostats and their Cryogen Volumes[6]

Magnet Field/Bore (MHz/mm)	$He_{(liq)}$ (& Coil Amps[7])
300/54	30
300/54 LH235[8]	87
300/54 LH365	136
300/89	76
400/54 LH330	123
400/54 S[9]	123
400/89 S	69
400/Horizontal WB (9.4T MS magnet)	700 (~270Amps)[10]
500/51 S	130
500/54 PS[11]	188
500/89 S	285
500/89(?) WB	283 (105Amps)
600/51	130
600/51 S	198
600/54 PS	275
600/89	142
700/54 S	370
800/54 S	1351
800/51 2.2K pumped gen II	700 (151A)
900 Aeon (self recycling)	690 (?A)
950 Bruker US^2 (2.2K shielded)	700 (?A)
800 & 850 4.2K non-pumped[12]	1350? (?A)
1000 to 1200/54	1350? (TBD)

Hazard Mitigation

All the hazards can be readily handled by either automatically or manually controlling the gas discharged, and directing the gas to the nearest outside space. Once external, the gas possess no practical threat as it should rise and disperse rapidly. Again, assuming a ventilation system is in-place it is important to make sure the discharge area is clear and safe (*e.g.* not a common

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[6] Originally taken from *Varian, Inc. NMR Systems Pub. No. 01-999262-00, Rev. B 1005* with additions as available from installation materials.

[7] Amps in coil when available

[8] LH stands for 'long-hold' with a maximum number of days between fills

[9] S stands for Shielded (not indicating passive or active). AS usually stands for actively shielded.

[10] ~570 litres of helium is minimum before quench

[11] PS is premium shielded. Smaller footprint, longer hold time on the cryostat, *etc.*

[12] Ascend Bruker are actively shielded non-pumped systems

gathering place outside, *e.g.* smoke break, benches *etc.*). The ventilation system needs to be checked regularly (we check annually) and especially before a known event.

For situations where no ventilation was provided (or possible) there are further complications.

Room Size/Capacity

One of the first items on the check list should be determining the approximate volume of the gas being released in relation to the room. This should give a fairly good approximation of the danger. Also the normal building air circulation flow rate should be taken into account. If the air is regularly replaced the hazard level is reduced. *However many buildings recycle conditioned internal air in order to reduce costs. We had an example where 80% of the sub-basement air was being recycled where a decommissioning was being planned. In these cases, building services should be approached to see if recirculation can be turned off, and 100% fresh air used to supply the area. Normal building air circulation is rarely sufficient and access to external space for the gas should be a priority.

Informing People before Exposure

The purpose of this section is to remind everyone regarding the need to communicate as widely, accurately, and as often as possible regarding the event timing, expected outcome, and potential hazards. We need to have essential and non-essential people well out of harms way. Potential harm comes not only from the magnet cryogens being released as a gas, but also possible secondary effects, *e.g.* domestic water pipes being frozen and bursting exposing electrical outlets to water. This can be easily guarded against with directing gas flows and/or simple insulation, but needs to be recognized before the event occurs.

Groups that need to be Notified

There are many groups that will need to know about the event, and know how to properly respond. What we want to avoid is an emergency response to a situation that is under control. Having people rush to the area may end up causing problems. These include (but are not limited to):

Campus/Building Security

This will often be the group who will contact Police and Fire Rescue. If not, Police and Fire Rescue will need to be informed directly. They need to know the date and range of times. Always plan in additional time (usually as a range) for unexpected complications. Also you may want to consider reserving an entire day if using a diode energy drain system is not available.

All building services staff

Including janitorial, maintenance, and services. It's important to make sure all groups know so they can avoid the area. They often have building keys so locking doors and signage is not sufficient. We want to avoid scaring anyone and possible injury.

Appropriate and highly visible signage for unexpected visitors (*i.e.* the public)

While signage is not sufficient by itself, it is necessary to help stop unexpected visitors. Signage needs to simple and distinct from any other forms commonly present. Adequate number of signs, especially covering entrance is essential.

Staff Using Facility

Research staff, students, post-doctoral fellows, associates, and primary investigators all need to be informed and have time to pass only information their co-workers. They also have to be able to plan experiments around the disruption. We chose an early Sunday morning for our scheduled quench to minimize the number of people who might be wanting to access the facility.

Active Ventilation

One important aspect is triggering of the exhaust system. This should ideally be automatic based on sensors in the room and not dependent on manual activation. That said, there should be a manual override available both to activate the system in the event of sensor failure, and also to deactivate in the event of a false positive. Room oxygen sensors are a common trigger, but have their limitations.

Oxygen Sensors

While oxygen sensors (both permanent and personal portable models) are an excellent avenue for detecting and automatically responding to an unscheduled (or scheduled) quench, they have several important problems. The first is the chemical nature of the sensor. The detector wears out in only a year or two. This requires constant expenditures to replace, and calls into question the reliability or accuracy getting close to replacement times. In addition to sensor replacement, calibration is also required usually by a skilled technician increasing the cost. Hand held individual units can cost several hundred dollars while room installations can be thousands (and up). Sensor failure usually results in false positive alarm, *i.e.* low oxygen readings, and have a tendency to occur between 2-4 AM[13]. False positives usually result in disabling the alarm and often the detectors are not serviced for extended periods (if ever again). Oxygen sensors should not be purchased unless the long term costs are understood and a commitment is made to maintain them. Also keep in mind that many companies offer rental units which can be very cost effective for single events such as a decommissioning.

Another point to consider is unit placement. While it might seem logical to place an oxygen sensor directly in the helium exit path to detect a quench as soon as possible, this is completely wrong. Oxygen detectors can easily freeze. Once frozen they will not deliver a low oxygen reading. We have experience with an unscheduled quench that froze the oxygen sensor before a subsequent exhaust fan trigger could be activated. Exhaust fans had to be manually activated. Moving the sensor to the other side of the room away from the quench path allowed the sensor to not only detect quench events, but also any slow quenching of the magnet which could present a hazard without the more obvious signs. Redundancy is key and other sensors using different triggers are a great idea (see below).

Temperature Sensors

A useful additional automatic trigger is a temperature sensor. These have the advantage of no annual detector replacement, and can be placed in the quench blast-path for rapid detection and subsequent exhaust activation. They are small and relatively cheap providing a nice backup for the primary detection systems. Testing is also easy using a small amount of gas off a cryogen dewar for example.

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[13] Personal observation (whining), no basis in real fact.

Manual Activation Switch

We strongly recommend having a well marked and easily accessible manual control switch for any exhaust system. This can allow user fan activation prior to alarms from detectors sensing an issue. For example, in situations where cryogens will be heavy used such as cooling a new spectrometer cryostat, or even during standard fills for poorly ventilated rooms. Vaporizing cryogens in these situations can pose all the risks of a quench, and manual activation of the exhaust system can be useful to prevent problems and false positive alarms. A manual override to prevent fan can also be important. During fills, building service repairs, constructions *etc.* it is possible to accidently trigger a fan activation. In these cases, it is useful to have the override close by. In one installation we had a very large fan leading to a 16-inch vent which would pull hard enough to make opening doors difficult. Even testing the fan annually would suck in all loose materials such as: dust, dirt, tiny pebbles, staples, and a surprising amount of metal fillings would end up on the bottom of the magnet. We would have to clean the metal "beard" from the bottom of the magnet regularly, and make sure both ends of the magnet bore are covered to prevent contamination.

Helium

Helium is a finite world resource with the majority of production centered in the United states, though recently other international facilities are coming online (*e.g.* Qatar, Algeria, and Russia[14] with China actively seeking their own strategic resources).

Figure 11- HELIUM PRODUCTION AND USAGE[15]

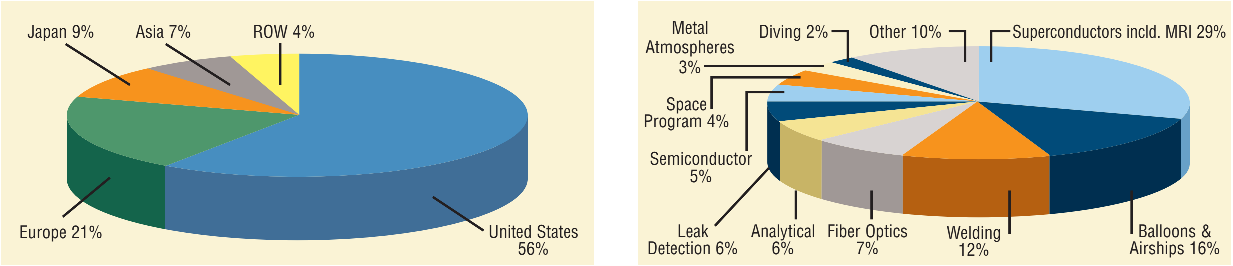

Fig 2 The U.S. alone consumes more than half of the world's supply of helium (2002)

Fig 3 Helium use in various applications

Additionally there is great article from 2012 describing expected helium production and usage along with predicting the "helium crisis" we experienced in 2014[16]. Specifically, in 2014 there were distributor shortages resulting in quotas for almost all helium users. A quota system of course doesn't make any sense when considering NMR and MRI instruments as the usage of cryogens is consistent, and required. One can not "turn down" or "shut-off" a spectrometer and "turn it back up" at a later point with the hope of conserving cryogens. Not acquiring data on a spectrometer does not effect the cryogen boil off, while mistreatment can certainly increase the boil off rate (*e.g.* no filling the liquid nitrogen shield). **Any spectrometer not continuously supplied with cryogens will lose superconductivity and "quench" resulting in hazards, possible**

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[14] http://www.gazprominfo.com/articles/helium/

[15] Figure taken from "Helium Recovery and Recycling Makes Good Business Sense" by
Donald J. Bowe, Air Products & Chemicals Inc., Allentown, Pa. (2004)

[16] http://www.popularmechanics.com/science/health/med-tech/why-is-there-a-helium-shortage-10031229

magnet damage, and inevitably large monetary and cryogen costs to re-cool and re-energize the instrument later. The relatively slow small boil-off from a stable cryostat is insignificant compared to the cryogens required to cool a magnet, so any consideration of a repeated decommissioning and re-commissioning at a later time is well beyond impractical.

Helium Recycling

Many people do not realize that helium released to the atmosphere (*i.e.* balloons, industrial/welding use, or boiling off from cryostats) makes its way to the upper atmosphere and then leaves the earth. The earth's gravity is insufficient to retain the gas and therefore we have no way to recapture helium. This inevitably leads to the consideration of a helium recycling/re-liquefaction facility.

While it is always desirable to recycle, one has to also pragmatically consider the cost of the initial equipment and then operating a helium re-liquefaction facility. Even when a grant is available for equipment, in terms of salaries alone this can make recycling economically impossible without support. The financial problems with running a recycling facility are now amplified as administrations are requiring "cost-recovery" via user fees and self sustaining budgets. This makes it harder to justify to users the costs of getting their gas back in liquid form. It is speculative to put a dollar value on the present worth of helium, even in consideration of a finite quantity available to us. For example, do we set the dollar amount based on cost of isolation, purification, and transport? Do we factor in some form of cost of production if we were forced to take higher mass unstable atoms and through radioactive decay isolate new helium? One can only imagine the cost that would entail.

Unfortunately, from a purely short term point of view it is cheaper today to blow the evaporating gas off and buy liquid at market value. Even including transportation costs, and additional costs of rushes when shipments are late (or missing), the market cost is cheap compared to running a recycling facility. This simple comparison does not even take into consideration the electrical costs (both financial and environmental) of running recycling compressors for gas liquefaction. Though ideally a renewable source of electricity such as solar or hydroelectric power would be used, to my knowledge this not the case for any facility. For example, in Alberta we would predominantly use coal generated electricity to run the compressors. While the electrical costs are usually hidden from the end user and absorbed by the institution, in good conscious we must take this into consideration. So the end result boils[17] down to how much does liquid helium cost to buy, versus how much does it cost an institution to re-liquefy gas? Too often the financial answer is to buy market helium, blow it off through evaporation and let someone else worry about 20-30 years down the road.

However, for some areas with extremely high helium costs and/or non-secure delivery (*e.g.* >$50/litre on the Hawaiian Islands circa 2014) the economic factor tips the other way and recycling becomes not only prudent, but essentially insurance against supply disruptions such as a loss of shipping access during a storm.

For yet other groups, the total usage may be so high that a recycling system makes even short term economic sense. In these cases, the correct capacity of the recycling unit becomes essential. Too large a recycling system can require so much input gas just to maintain operational temperature. These can require vast quantities of gas just to get down to operational

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[17] Forgive the pun.

temperature, only to shut down before liquefaction can begin. Too small a system can't liquefy the gas quickly enough to keep up with evaporation. Extensive high pressure storage and/or bulk low pressure collection bags can help, but even then the excess storage will exceed compressor capacity. Luckily many manufacturers exist with several options and interested readers are directed to a simple web search or NMR conference vendor suites to discover more. Equipment ranges greatly in space foot-print, capacity, volumes utilized, speed, and cost. The Association of Managers in Magnetic Resonance Laboratories (AMMRL) also has fairly extensive documentation from discussion groups on the subject including real-world installation results. Interested readers are highly recommended to take advantage of those resources before making a purchase decision.

Self Cryogen Recycling Magnets

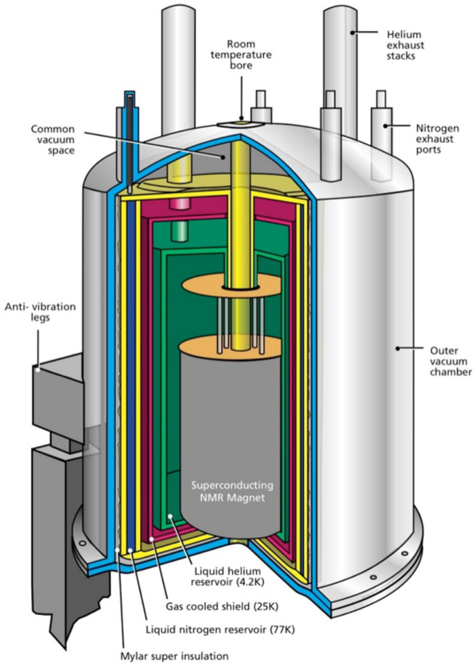
There is a recent design push for "cryogen free" magnets. Unfortunately, there are two definitions of cryogen free causing some confusion. The first are actually cryogen free cryostats and use cooled superconducting coils in a vacuum. The act of pumping on the system maintains the temperature. Complications arise if pumping is lost (*e.g.* electrical failure) or the compressor needs regular servicing. These are relatively new additions to the area and the methodology is quickly expanding.

The second type are not actually cryogen free, but instead self recycling and would better be described as filling-free or maintenance reduced, but obviously this isn't as catchy for sales. The newer cyrostats contain a cold-finger style cooling head at the top of the cryostat operated by a compressor. Cold helium gas condenses on this cold finger and drips down back into the spectrometer cryogen bath. In order to try and further reduce maintenance time and personnel costs the liquid nitrogen shield is removed in the design as the machine can maintain itself. Therefore, weekly nitrogen and semi-annual helium fills are not needed. Only occasional top-ups of helium are required throughout the year. At time of writing it is not entirely clear if helium gas and/or helium liquid is required to top-up the systems periodically, or if this is system dependent. While not entirely new, the biggest advancement for self-recycling came when vibration suppression finally reached a point where the recycling cold-finger did not interfere with data acquisition. Up until then substantial vibrations from the compressor unit disrupted high quality NMR spectra. MRIs seemed less sensitive and have had self-recycling for some time.

Self Cryogen Recycling Magnet Maintenance Costs

A major point to note is that the helium condensing unit (pulse tube) needs regular minor servicing every two years, and a major service every four years. Spectrometers would have one (*e.g.* 400, 500 MHz NMR), two (*e.g.* 600, and 700 MHz NMR), or even larger units (*e.g.* 800-1000 MHz) of the pulse tubes to re-liquefy helium, and possibly an additional compressor for the cooled NMR probe. The cost of an NMR probe cold-head refurbishment is ~16-18k every 18 months at present. The cold-finger 2-year minor and every 4-year major servicing for pulse tubes total (over the entire 4 years) is about ~40k for 400/500 NMR, ~80k for 600/700 and ~240k for 800-1000 MHz instruments. Remember that the 800+ instruments are 2.2K pumped systems consuming a great deal of additional helium. Having several of these units to maintain might be financially disastrous for a facility.

Figure 12 Cryostat Cross-Section showing cryogen chambers. Non-pumped system.



The cost savings of any recycling would have to take these maintenance points into consideration and then need to evaluate the electrical power consumption into calculations which could be significant.

Any equipment grant application should take this into consideration how future maintenance costs will be handled.

Quick Facts about Helium

Liquid helium (-268.9°C or 4.2K) has an expansion ratio of 740:1 (*i.e.* gas:liquid) going from the liquid to gaseous state at standard temperature and pressure. Helium gas has an impressive expansion as it warms, and gas at 5-10K can be roughly treated as a liquid in terms of volume due to the rapid change of density with heating to room temperature. The expansion of course presents the possible hazard of over pressurization if not properly vented.

The cooling hazard is substantial. According to the CRC handbook, helium gas has a thermal conductivity of 0.142 W/(meter•K) which is substantially higher than air (0.02) or water vapour (0.016 - while this suggests damp air is worse at transfer and should feel warmer, don't forget that heat capacity also comes into play). This indicates that helium gas while not an excellent conductor of heat (*e.g.* copper 385 W/m•k, water 0.5 W/m•k, or ammonia 0.5 W/m•k) will strip heat energy from the room or room contents faster than air. Despite helium gas having a relatively low heat capacity, *e.g.* $He_{(g)}$ 12.8 J/mol•K, $He_{(liq)}$ (type I) 20.78 J/mol•K, and water 75 J/mol•K, the volumes present in cryostats can still pose a problem to occupants and infrastructure (*e.g.* pipes, containers, sensors *etc.*).

Helium is misleadingly quoted as having an astounding thermal conductivity, *i.e.* >100000 W/(meter•K), but this only occurs in the form of liquid helium II and only below the lambda point of 2.17 K. Of note, there are excellent videos online for anyone interested in seeing the remarkable transition behaviour of helium liquid to the helium II state. Helium II also has other remarkable properties such as no bubbling during boil off (evaporation occurs only at the surface), and produces a "Rollin's film" 30nM wide that will literally climb the container walls against the force of gravity.

Nitrogen

Nitrogen does not have the superfluid transition of helium, the thermal conductivity (*i.e.* less desirable for industry cooling), nor quite the temperature extreme in liquid form though it does still have a sizable thermal capacity (~20 J/mol•K). Liquid nitrogen boils at -196°C or 77 K, but it is not sufficiently cold to stabilize the charge density of present day superconducting wire (*e.g.* review on high temperature superconductors[18]). Liquid nitrogen does have one substantial advantage; it's cheap (~$1.5/liter) compared to helium (~$14/liter locally). This is due to the abundance in air (~71%) and the ability to purchase small local liquefiers as well as storage towers for larger distribution. In NMR we use liquid nitrogen jackets around the outer shell of the cryostat to protect the expensive liquid helium (see Figure 12, from Oxford Magnetics Inc.).

Nitrogen is typically topped-up weekly for NMR spectrometers. This provides adequate heat shielding for the nitrogen and provides continuous positive pressure from the slight boil off. While $N_{2\ (liq)}$ fills can be put off for longer durations than a week (typically 14 days is a manufacturer recommended maximum), we never want to expend all the nitrogen as this can result in warming of the nitrogen vessel and room air being sucked back into the magnet. Air entering the space has a strong potential for ice formation with inherent blockage hazards (*i.e.* can't fill liquid N_2 later and pressurization problems). Sufficient contamination can even negate the vessel's efficiency allowing a thermal "touch" between the outer and inner vessel walls. Magnets without a cooled nitrogen shield will experience dramatically increased, and often accelerating helium boil off and great difficulty cooling the nitrogen shield back down. If the nitrogen vessel is suspected of being contaminated, then removing liquid nitrogen and blowing dry high quality nitrogen gas through should help. Removing ice or water can be very difficult. Ice blockages can be carefully penetrated using a warmed wooden doweling while blowing room temp nitrogen gas across the blockage. We want to slowly melt and evaporate our way back into the chamber. Patience and care are essential. We also want to determine of the source of the initial problem (*e.g.* heat exchanger left off, nitrogen vessel empty, *etc.*) and remove the problem otherwise the problem will simply repeat soon.

Liquid nitrogen, as the most frequently filled cryogen posses the most likely hazard in terms of freezing "burns" and oxygen displacement. Transfer lines (*e.g.* braided steel, evacuated, or simple latex tubing) should be regularly inspected, replaced as needed, and technicians performing fills should be prepared at all times for a transfer line failure. Gloves, eye protection, closed toe shoes, and full length pants are mandatory for our facility and highly recommended. Technicians should also be present at all times during fills in case of over filling or transfer line failure. Leaving a fill, even for a few minutes, is not allowed in our facility. In an emergency (e.g. fire alarm), we halt the fill by removing external supplied pressure, and remove transfer lines from the magnet replacing heat exchangers. At bare minimum external nitrogen gas pressure is halted and lines left connected to prevent back flow of external air.

Failure to be present during fills can result in an overfilling magnet and/or rupture of the transfer line without stopping of the liquid nitrogen flow. We have seen a report of a magnet O-ring on the main cryostat vacuum space fail when cooled by overflowing liquid nitrogen. The magnet quenched, but was luckily not damaged further and was able to eventually be re-energized, albeit at loss of usage for several weeks and the associated costs for cryogens to re-cool and an engineer.

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[18] Anderson, P. W. (2013) Twenty-five Years of High-Temperature Superconductivity? A Personal Review, *Journal of Physics: Conference Series 449*, 012001.

Video of Preparations and Quench of

An associated video was prepared for a recent quench in our department. The magnet was a 9.4 T horizontal wide bore mass spectrometry cryostat installed in a sub-basement without adequate access to windows or ventilation. The video was prepared to show safety preparations and ducting introduced to move the escaping gasses to the nearest external access. This document and the video are intended to help inform and answer common questions non-NMR experts in case a magnet decommissioning is needed

Request Access to Quench Video

The video is presently privately shared on YouTube. Please email ryan.mckay@ualberta.ca and we'll send you the link ASAP.

Grateful acknowledgements:

We would like to acknowledge all the contributions from the many members of the AMMRL and NMR community who made this document possible. Special thanks to the following who contributed to the document and/or to the subsequent Safety/Quench video: Jerry Dallas (UC Davis retired), Jerry Hirschinger (Purdue), David Badger (Ashland Performance Materials), Neil Jacobson (Arizona), Bob Berno (McMaster), Megan Kirk (Sasol technology), Chris Rithner (Colorado State), Jürgen Schulte (Binghamton), Beverly Ostrowski (Global Analytical), Martha Morton (Nebraska-Lincoln), Joe Dumais (Boise State), Luo Rensheng (Missouri – St. Louis), Alfred Kwan (HK Polytechnic), Dee-Hua Huang (Scripps), Rainer Wechselberger (ITS-JNJ), George Furst (Pennsylvania), David VanderVelde (CalTech), Steve Peurifoy (Millersville), Ian Luck (Sydney), Norman Chu (Piramel Healthcare). We would also like to thank significant contributions from vendor associates Knut Mehr, Tim Laker, Claude Major, and Brian Tilson who all provided personal anecdotes, facts, advice, and reality checks for the document.

Please forgive if I missed anyone as it's my mistake and not a purposeful omission. There were so many contributors and I tried to keep good notes but if you've been missed drop a line and it'll be fixed in subsequent versions.