

Struktureinheit/Arbeitsbereich:  
ZQB / Bau M26 / Raum 1106

Tätigkeit: Arbeiten mit einem 600 MHz NMR-Spektrometer  
Stand 28.05.2024

### BEZEICHNUNG

## NMR-Gerät Oxford Instruments - 600 MHz

### GEFAHREN FÜR MENSCH UND UMWELT



- Auf Betriebsmittel in der unmittelbaren Nähe des NMR-Magnetsystems können große Anziehungskräfte ausgeübt werden. Die Kraft kann so groß werden, dass die Geräte unkontrolliert in Richtung des NMR-Magnetsystems bewegt werden.
- Große Geräte (z.B. Gasflaschen, Netzteile) können dazu führen, dass Körper oder Gliedmaßen zwischen dem Gerät und dem Magnetsystem eingeklemmt werden: je näher am NMR-Magnetsystem, desto größer ist die Kraft; je größer die magnetisch wirksame Gerätemasse (proportional zum Magnetfeldgradienten), desto größer die Kraft.
- Durch die effektive Abschirmung der supraleitenden Spule, werden die Auswirkungen des magnetischen Streufeldes minimiert. Die horizontale 5 Gauss-Linie befindet sich nur rund 1m (96 cm) vom Magnetmantel entfernt.
- Dennoch ist zu beachten, dass direkt über und direkt unter dem Magneten das Streufeld und damit der Magnetfeldgradient sehr hoch ist und die Anziehungskräfte auf magnetische Gegenstände sehr stark sind!
- Kryogene Flüssigkeiten, auch wenn sie in isolierten Lagerbehältern (Dewar-Behältern) aufbewahrt werden, bleiben auf einer konstanten Temperatur um ihren jeweiligen Siedepunkt und verdampfen allmählich. Die sehr große Volumenzunahme, die mit dem Verdampfen der Flüssigkeit zu Gas und dem anschließendem Erwärmungsprozess einhergeht, beträgt bei Helium und Stickstoff etwa 700:1.
- Ein Sauerstoffmangel unterschiedlichen Ausmaßes kann auftreten, wenn der Magnetraum nicht richtig belüftet wird.
- Bei verschlossenen für kryogene Substanzen (Dewars, Kryostaten, ...) Behältern ohne Überdruckventil besteht Explosionsgefahr.
- Kryogene Stoffe in flüssiger oder dampfförmiger Form (oder als Tieftemperaturgase) erzeugen verbrennungsähnliche Wirkungen auf der Haut (Kälteverbrennungen). Freiliegende oder unzureichend geschützte Körperteile, die mit nicht isolierten Entlüftungsrohren oder Behältern in Berührung kommen, verkleben sofort und das Körpergewebe wird beim Entfernen des betroffenen Körperteils verletzt.
- Kondensierender Sauerstoff kann die Sauerstoffkonzentration lokal erhöhen und es kann in Kontakt mit vielen Substanzen (Öle, Fette, Lösungsmittel, ...) - auch solchen die sich unter normalen Bedingungen nicht entzünden lassen - zu einer Selbstentzündung kommen.
- Materialien, die bei starker Kühlung spröde werden, können in Kontakt mit kryogenen Substanzen beschädigt werden und Verletzungen verursachen.
- Es besteht eine erhöhte Gefahr eines Quenches.
- Überlaufende Behälter können zu Verspritzen von kryogenen Flüssigkeiten führen.
- Es ist möglich, dass während des Ladevorgangs einige Trainings-Quenches auftreten, um die Spannungsänderungen im Magneten auszugleichen. Diese Quenches sind in der Regel unbedenklich, müssen aber als vorübergehend hohe thermische und mechanische Belastung für den Magneten angesehen werden und sollten möglichst vermieden werden.
- Ausführliches Studieren der vollständigen Betriebsanleitung des NMR ist zwingend nötig.

## SCHUTZMASSNAHMEN UND VERHALTENSREGELN



- Die elektrischen Geräte sind regelmäßig nach Niederspannungsrichtlinie 2014/35/EU von qualifiziertem Personal zu testen.
- Der Betrieb von medizinisch-elektronischen Implantaten, wie z.B. Herzschrittmachern, kann entweder durch statische oder wechselnde Magnetfelder beeinträchtigt werden. Herzschrittmacher können auf nicht vorhergesehene Weise reagieren, wenn sie Feldern über 5 Gauss ausgesetzt sind. Der Gefahrenbereich ist somit durch die 5 Gauss-Linie eingegrenzt.
- Andere medizinische Implantate, wie z.B. Aneurysmen-Clips, chirurgische Clips oder Prothesen, können ferromagnetische Materialien enthalten und daher in der Nähe des NMR Magnetsystems starken Anziehungskräften ausgesetzt sein. Dies könnte zu Verletzungen oder zum Tod führen. Zusätzlich können in der Nähe von schnell wechselnden Feldern (z.B. gepulste Gradientenfelder) im Implantat Wirbelströme induziert werden was zu einer Wärmeentwicklung führt.
- Die 5 Gauss-Linie des NMR Magneten ist mit einer Trassierband-Markierung gekennzeichnet.
- Da das Feld des NMR-Magnetsystems dreidimensional ist, müssen sowohl die Etagen über und unter dem Magneten als auch der umgebende Raum auf gleicher Höhe berücksichtigt werden.
- Die 5 Gauss-Linien des Oxford Instruments 600 MHz Magneten liegen bei 175 cm vom Mittelpunkt in vertikaler Richtung und 250 cm in horizontaler Richtung.
- Der Magnet in Raum 1106 steht im Kellergeschoss, somit befinden sich keine Räume unter dem Magneten. Nach oben ist die Deckenhöhe über 4 m. Somit ist auch nach oben ein Streufeld von deutlich kleiner als 5 Gauss außerhalb des Labors garantiert.
- Im Gefahrenbereich dürfen keine ferromagnetischen Gegenstände oder Werkzeuge benutzt werden.
- Störungsanfällige Geräte und Geräte, bei denen ein Ausfall Auswirkungen auf die Sicherheit hat oder die durch magnetische Felder beschädigt werden, sind aus dem Gefahrenbereich fern zu halten.
- Bei Besteigen des Magneten besteht die Gefahr des Umkippens. Ein Besteigen oder Beklettern des NMR-Magneten ist verboten.
- Der Zugang zum Gefahrenbereich ist auf geschultes Personal zu beschränken, indem das NMR-Labor verschlossen gehalten wird.
- Zur Aufbewahrung und zum Transport von kryogenen Substanzen dürfen ausschließlich Niederdruck-Dewars verwendet werden. Mit Drücken von  $> 2$  bar beaufschlagte Dewars sind für die Benutzung mit dem NMR untersagt.
- Behälter für kryogene Flüssigkeiten dürfen nicht vollständig verschlossen werden, da dies zu einem starken Druckaufbau führen würde. Dies stellt eine Explosionsgefahr dar.
- Helium kann in den oberen Bereichen eines Raumes die Luft verdrängen und kalter Stickstoff kann in den unteren Bereichen die Luft verdrängen.
- Große Mengen an Gasen verdampfen bei einem Quench und wenn größere Mengen Flüssigkeiten verschüttet werden. Um einen Quench zu vermeiden, ist auf stetiges ausreichendes Vorhandensein der kühlenden Flüssigkeiten (Stickstoff und Helium) zu achten. Helium wird elektronisch überwacht und mindestens wöchentlich kontrolliert. Stickstoff ist täglich zu kontrollieren. Bei der Befüllung ist auf hohe Sorgsamkeit zu achten, um versehentliches Verschütten zu vermeiden.
- Kryogene Substanzen dürfen nur in Labore mit technischer Lüftung und einer Mindestluftumwälzung von  $25 \text{ m}^3/\text{h}$  pro  $\text{m}^2$  genutzt werden.
- Das maximale Gasvolumen im expandierten Zustand bei 1 bar darf einen Wert von  $1/3$  des Raumvolumens nicht überschreiten.
- Sollte ein starker Austritt von Gasen (z.B. bei einem Quench) festgestellt werden, muss das Labor sofort verlassen werden und der zuständige Sicherheitsbeauftragte ist zu kontaktieren. Das Labor darf erst wieder betreten werden, wenn ein sicherer Zustand festgestellt, und das Labor vom zuständigen Sicherheitsbeauftragten freigegeben wird. Die Nutzer der Nachbarlabore sind auch zu warnen und deren Labor sind ebenfalls zu räumen, bis eine Freigabe erfolgt.

## SCHUTZMASSNAHMEN UND VERHALTENSREGELN (Fortsetzung)

- Das Labor ist mit drei festinstallierten Gaswarnsensoren zur Überprüfung des Sauerstoffgehaltes mit akustischem und optischem Alarm ausgestattet. Zwei Sensoren sind über den NMR-Geräten angebracht, um die Sauerstoffabnahme durch verdampfendes Helium zu erfassen. Ein Sensor ist in Bodennähe angebracht, um die Sauerstoffabnahme durch verdampfenden Stickstoff zu erfassen. Bei Auslösen einer der Sensoren ist das Labor von allen Anwesenden zu verlassen, bis ein sicherer Zustand wiederhergestellt ist und das Labor vom zuständigen Sicherheitsbeauftragten freigegeben wird.
- Der Kontakt mit kryogenen Flüssigkeiten mit Luft ist zu minimieren.
- Da flüssiger Stickstoff und Helium kälter sind als flüssiger Sauerstoff, kondensiert der Sauerstoff der Luft aus. Wenn dies über einen längeren Zeitraum geschieht, kann die Sauerstoffkonzentration in den Behältern so hoch werden, dass die Handhabung genauso gefährlich wird, wie die von flüssigem Sauerstoff. Dies gilt insbesondere für Weithals-Dewars aufgrund der großen Oberfläche.
- Im Gefahrenbereich der kryogenen Flüssigkeiten, sind: Feuer, Flammen offene Zündquellen und das Rauchen verboten. Dies gilt insbesondere beim Füllvorgang des Magneten.
- Während Stickstoff und Helium keine Verbrennung unterstützen, kann ihre extreme Kälte dazu führen, dass Sauerstoff aus der Luft an kalten Oberflächen kondensiert und die Sauerstoffkonzentration lokal erhöht ist. Besondere Brandgefahr besteht, wenn die kalten Oberflächen mit Ölen oder Fette bedeckt sind. Es kann zu einer Selbstentzündung kommen!
- Flächen, die mit kryogenen Flüssigkeiten in Kontakt kommen, sind vorher insbesondere von brennbaren Ablagerungen zu reinigen.
- Zur Befüllung mit Stickstoff sind entweder Schläuche aus einem Material zu verwenden, dass bei  $-196^{\circ}\text{C}$  nicht versprödet (Gummi-, Teflonschläuche) oder es sind isolierte Leitungen zu verwenden.
- Zur Befüllung mit Helium sind ausschließlich isolierte Rohrleitungen (Transferline) zu verwenden.
- Auch die O-Ringe am Magnetsystem sind empfindlich gegen tiefe Temperaturen. Achten sie deshalb beim Füllen darauf, dass kein flüssiges Helium oder flüssiger Stickstoff auf die O-Ringe gelangt.
- Das Funktionieren der Lüftung ist vor Antritt der Arbeiten zu überprüfen und die Arbeiten sind bei fehlerhafter Lüftung unverzüglich einzustellen. Indikatoren für eine fehlerhafte Lüftung sind:
  - o Angekündigte Abschaltung der Lüftung
  - o Stromausfall / Stromabschaltung
  - o Pfeifgeräusche der Lüftung
  - o Indikatoren vor der Lüftung zeigt keine Strömung an
  - o Schnelle Temperaturänderungen im Labor oder Temperaturen über  $30^{\circ}\text{C}$
  - o Kopfschmerzen oder Konzentrationsschwierigkeiten im Labor
- Bei einer Funktionsstörung der Lüftung ist der Befüllprozess sofort abzubrechen und der zuständige Sicherheitsbeauftragte zu kontaktieren. Das Befüllen darf erst fortgesetzt werden, wenn ein sicherer Zustand festgestellt und das Labor vom zuständigen Sicherheitsbeauftragten freigegeben wird.
- Das Stickstoffgefäß sollte täglich auf Abdampfen und Stickstoffstand überprüft werden. Diese Werte sollten aufgezeichnet werden. Wenn das Abdampfen auf Null fällt, sollten die Türme sofort auf das Vorhandensein von Eis überprüft und evtl. Verstopfungen entfernt werden, um einen Druckaufbau zu vermeiden.

## SCHUTZMASSNAHMEN UND VERHALTENSREGELN (Fortsetzung)



- Bei signifikant steigendem Verbrauch an kryogener Flüssigkeit, besteht der Verdacht, dass die thermische Isolation des Kryostaten reduziert ist. In diesem Fall sollte der Druck in dem Vakuumgefäß überprüft werden.



- Überlaufventile an den Behältern sind mit einem Schlauch zu versehen oder die Öffnung ist so anzuordnen, dass kein Nutzer von austretenden Flüssigkeiten getroffen werden kann.



- Beaufschlagen Sie den Stickstoff-Dewar niemals mit einem Überdruck von mehr als 350 mbar (5 psi) und achten Sie immer darauf, dass alle Auslassventile am NMR-Magneten vollständig geöffnet sind.

- Lassen Sie die Heliumventile nicht länger als 5 Sekunden zur Atmosphäre geöffnet, es sei denn, es ist ein großer Gasstrom vorhanden.

### Persönliche Schutzmaßnahmen



- Augenschutz: Gestellbrille mit Seitenschutz.
- Handschutz: geeignete Kälteschutzhandschuhe. Die Handschuhe müssen locker sitzen, damit sie im Falle eines Flüssigkeitsaustritts leicht zu entfernen sind.
- Kleidung: Es ist geschlossene Kleidung und ein Labormantel zu tragen, die ein Eindringen der kryogenen Flüssigkeiten verhindern. Der Labormantel verhindert im Falle einer Kontamination, dass die Kleidung am Körper festfriert.

## VERHALTEN BEI STÖRUNGEN – LEITWACHE: 22222



- Gefahrenbereich räumen und absperren, Vorgesetzten und Sicherheitsbeauftragten informieren. Die Kontaktmöglichkeiten zu den vorgenannten Personen können dem Alarmplan entnommen werden
- Sollte ein starker Austritt von Gasen (z.B. bei einem Quench) festgestellt werden, muss das Labor sofort geräumt und der zuständige Sicherheitsbeauftragte kontaktiert werden. Das Labor darf erst wieder betreten werden, wenn ein sicherer Zustand festgestellt und das Labor vom zuständigen Sicherheitsbeauftragten freigegeben wurde.
- Bei einer Funktionsstörung der Lüftung ist der Befüllprozess sofort abzubrechen und der zuständige Sicherheitsbeauftragte zu kontaktieren. Das Befüllen darf erst fortgesetzt werden, wenn ein sicherer Zustand festgestellt und das Labor vom zuständigen Sicherheitsbeauftragten freigegeben wurde.
- Notruf Leitwache: 22222
- Brand: 112

## VERHALTEN BEI UNFÄLLEN – ERSTE HILFE – NOTRUF: 112



- Bei jeder Erste-Hilfe-Maßnahme: Selbstschutz beachten, Vorgesetzten und Sicherheitsbeauftragten informieren. In der Regel umgehen medizinisches Fachpersonal hinzuziehen.
- Bei Kontakt von kryogenen Flüssigkeiten mit Augen oder Haut: spülen Sie den betroffenen Bereich sofort mit großen Mengen kaltem oder lauwarmen Wasser und legen Sie anschließend kalte Kompressen an. Verwenden Sie niemals heißes Wasser oder trockene Hitze. Es sollte sofort ärztlicher Rat eingeholt werden.
- Bei Erstickungssymptomen sofort künstlich beatmen.
- Ersthelfer: siehe Alarmplan
- Unfälle mit Verletzten: 112

## SACHGERECHTE ENTSORGUNG

- Fragen zur sachgerechten Entsorgung richten Sie bitte an den Verantwortlichen im Dez. V-5, Telefon +49 (0)731 50-22137/38396, Telefax +49 (0)731 50-22102 Uni-Ost, M25/227

## FOLGEN DER NICHTBEACHTUNG

### Gesundheitliche Folgen

- Leichte bis tödliche Verletzung durch Ohnmacht

### Sachschäden

- Durch Brände bei Selbstzündung von Sauerstoff können hohe materielle Schäden verursacht werden

### Rechtliche Folgen

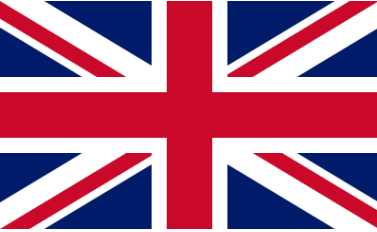
- Betriebsanweisungen sind verbindlich und stellen eine schriftliche Arbeitsschutzanweisung an die Beschäftigten dar.
- Die Nichtbeachtung kann juristische Folgen haben.
- Das Nichtbeachten dieser Anweisung ist ein Verstoß gegen gegebene Weisungen und wird entsprechend geahndet.

Erstellungsdatum: 28.05.2024 – Ersteller: M. Ferner

10/12/2024



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The following pages were translated using Google Translate. For this reason, their correctness cannot be guaranteed. The German version of the operating instructions is legally binding.

Structural unit/work area:  
ZQB / Building M26 / Room 1106

Activity: Working with a 600 MHz NMR spectrometer  
As of 28 May 2024

### DESIGNATION

## NMR device Oxford Instruments - 600 MHz

### DANGERS FOR HUMANS AND THE ENVIRONMENT



- Large forces of attraction can be exerted on equipment in the immediate vicinity of the NMR magnet system. The force can become so large that the equipment is moved uncontrollably towards the NMR magnet system.
- Large devices (e.g. gas cylinders, power supplies) can cause bodies or limbs to become trapped between the device and the magnet system: the closer to the NMR magnet system, the greater the force; the larger the magnetically effective device mass (proportional to the magnetic field gradient), the greater the force.
- The effective shielding of the superconducting coil minimizes the effects of the stray magnetic field. The horizontal 5 Gauss line is only about 1m (96 cm) away from the magnet casing.
- However, it should be noted that directly above and directly below the magnet the stray field and thus the magnetic field gradient is very high and the forces of attraction on magnetic objects are very strong!
- Cryogenic liquids, even when stored in insulated storage containers (Dewar vessels), remain at a constant temperature around their respective boiling point and gradually evaporate. The very large increase in volume that accompanies the evaporation of the liquid to gas and the subsequent heating process is about 700:1 for helium and nitrogen.
- Oxygen deficiency of varying degrees can occur if the magnet room is not properly ventilated.
- There is a risk of explosion in sealed containers for cryogenic substances (Dewars, cryostats, etc.) without a pressure relief valve.
- Cryogenic substances in liquid or vapor form (or as low-temperature gases) produce burn-like effects on the skin (cold burns).  
Exposed or inadequately protected body parts that come into contact with uninsulated vent pipes or containers will immediately stick together and body tissue will be injured when the affected body part is removed.
  - o Condensing oxygen can increase the oxygen concentration locally and can cause spontaneous combustion when it comes into contact with many substances (oils, fats, solvents, etc.) - even those that do not ignite under normal conditions.
  - o Materials that become brittle when subjected to extreme cooling may be damaged when in contact with cryogenic substances and cause injury.
  - o There is an increased risk of quenching.
  - o Overflowing containers can cause cryogenic liquids to splash.
  - o It is possible that some training quenches will occur during the charging process to compensate for the voltage changes in the magnet. These quenches are usually harmless, but must be considered as a temporary high thermal and mechanical stress on the magnet and should be avoided if possible.
  - o A thorough study of the complete NMR operating manual is mandatory.



## PROTECTIVE MEASURES AND RULES OF CONDUCT



- Electrical equipment must be regularly tested by qualified personnel in accordance with the Low Voltage Directive 2014/35/EU.
- The operation of medical electronic implants, such as pacemakers, can be affected by either static or changing magnetic fields. Pacemakers can react in an unpredictable manner when exposed to fields above 5 Gauss. The danger zone is therefore limited by the 5 Gauss line.
- Other medical implants, such as aneurysm clips, surgical clips or prostheses, may contain ferromagnetic materials and therefore be subject to strong attractive forces in the vicinity of the NMR magnet system. This could result in injury or death. In addition, eddy currents can be induced in the implant in the vicinity of rapidly changing fields (eg pulsed gradient fields), leading to heat generation.
- The 5 Gauss line of the NMR magnet is marked with a tracing tape.
- Since the field of the NMR magnet system is three-dimensional, both the floors above and below the magnet and the surrounding space at the same height must be taken into account.
- The 5 Gauss lines of the Oxford Instruments 600 MHz magnet are located at 175 cm from the center in the vertical direction and 250 cm in the horizontal direction.
- The magnet in room 1106 is in the basement, so there are no rooms underneath the magnet. The ceiling height is over 4 m. This guarantees a stray field of significantly less than 5 Gauss outside the laboratory.
- No ferromagnetic objects or tools may be used in the danger area.
- Equipment that is susceptible to failure and equipment whose failure has an impact on safety or which is damaged by magnetic fields must be kept away from the danger area.
- There is a risk of tipping over when climbing onto the magnet. Climbing onto or onto the NMR magnet is prohibited.
- Access to the hazardous area must be restricted to trained personnel by keeping the NMR laboratory locked.
- Only low-pressure Dewars may be used for storing and transporting cryogenic substances. Dewars pressurized to pressures of  $> 2$  bar are prohibited for use with the NMR.
- Containers for cryogenic liquids must not be completely closed, as this would lead to a strong build-up of pressure. This represents a risk of explosion.
- Helium can displace air in the upper areas of a room and cold nitrogen can displace air in the lower areas.
- Large quantities of gases evaporate during a quench and when large quantities of liquids are spilled. To avoid a quench, it is important to ensure that there is a constant supply of sufficient cooling liquids (nitrogen and helium). Helium is monitored electronically and checked at least weekly. Nitrogen must be checked daily. Great care must be taken when filling to avoid accidental spillage.
- Cryogenic substances may only be used in laboratories with technical ventilation and a minimum air circulation of  $25 \text{ m}^3/\text{h}$  per  $\text{m}^2$ .
- The maximum gas volume in the expanded state at 1 bar must not exceed  $1/3$  of the room volume.
- If a large amount of gas is released (e.g. during a quench), the laboratory must be left immediately and the responsible safety officer must be contacted. The laboratory may only be re-entered once a safe condition has been established and the laboratory has been cleared by the responsible safety officer. Users of neighboring laboratories must also be warned and their laboratories must also be evacuated until clearance is given.



## PROTECTIVE MEASURES AND RULES OF CONDUCT (continued)

- The laboratory is equipped with three permanently installed gas warning sensors to check the oxygen content with acoustic and visual alarms. Two sensors are installed above the NMR devices to detect the loss of oxygen due to evaporating helium. One sensor is installed near the floor to detect the loss of oxygen due to evaporating nitrogen. If one of the sensors is triggered, everyone present must leave the laboratory until a safe condition has been restored and the laboratory has been released by the responsible safety officer.
- Contact of cryogenic liquids with air must be minimized.
- Because liquid nitrogen and helium are colder than liquid oxygen, the oxygen in the air condenses. If this happens over a long period of time, the oxygen concentration in the containers can become so high that handling them becomes just as dangerous as handling liquid oxygen. This is especially true for wide-neck Dewars due to the large surface area.
- In the danger area of cryogenic liquids, fire, flames, open ignition sources and smoking are prohibited. This applies in particular when filling the magnet.
- While nitrogen and helium do not support combustion, their extreme cold can cause oxygen from the air to condense on cold surfaces and the oxygen concentration to increase locally. There is a particular risk of fire if the cold surfaces are covered with oil or fat. Spontaneous combustion can occur!
- Surfaces that come into contact with cryogenic liquids must be cleaned beforehand, particularly of any flammable deposits.
- To fill with nitrogen, either hoses made of a material that does not become brittle at  $-196^{\circ}\text{C}$  (rubber, Teflon hoses) or insulated lines must be used.
- Only insulated pipes (transfer lines) may be used for filling with helium.
- The O-rings on the magnet system are also sensitive to low temperatures. When filling, make sure that no liquid helium or liquid nitrogen gets on the O-rings.
- The functioning of the ventilation must be checked before starting work and work must be stopped immediately if the ventilation is faulty. Indicators of faulty ventilation are:
  - Announced shutdown of ventilation
  - Power failure / power outage
  - Whistling noises from the ventilation
  - Indicators before ventilation show no flow
  - Rapid temperature changes in the laboratory or temperatures above  $30^{\circ}\text{C}$
  - Headaches or difficulty concentrating in the laboratory
- If the ventilation malfunctions, the filling process must be stopped immediately and the responsible safety officer contacted. Filling may only be continued once a safe condition has been established and the laboratory has been released by the responsible safety officer.
- The nitrogen vessel should be checked daily for flash off and nitrogen level. These values should be recorded. If the flash off drops to zero, the towers should be checked immediately for the presence of ice and any blockages removed to prevent pressure build-up.

## PROTECTIVE MEASURES AND RULES OF CONDUCT (continued)



- If the consumption of cryogenic liquid increases significantly, it is suspected that the thermal insulation of the cryostat is reduced. In this case, the pressure in the vacuum vessel should be checked.
- Overflow valves on the containers must be fitted with a hose or the opening must be positioned in such a way that no user can be hit by escaping liquids.



- Never pressurize the nitrogen Dewar to more than 350 mbar (5 psi) and always ensure that all exhaust valves on the NMR magnet are fully open.
- Do not leave the helium valves open to the atmosphere for more than 5 seconds unless there is a large gas flow.



### Personal protective measures

Eye protection: Frame glasses with side protection.

Hand protection: suitable cold-resistant gloves. The gloves must be loose-fitting so that they can be easily removed in the event of a liquid spill.



Clothing: Closed clothing and a lab coat must be worn to prevent the penetration of cryogenic liquids. The lab coat prevents clothing from freezing to the body in the event of contamination.

## BEHAVIOR IN CASE OF FAULTS – CONTROL STATION: 22222



- Clear and cordon off the danger area, inform your supervisor and safety officer. Contact details for the aforementioned persons can be found in the alarm plan
- If a large amount of gas is released (e.g. during a quench), the laboratory must be evacuated immediately and the responsible safety officer contacted. The laboratory may only be re-entered once a safe condition has been established and the laboratory has been released by the responsible safety officer.
- If the ventilation malfunctions, the filling process must be stopped immediately and the responsible safety officer contacted. Filling may only be continued once a safe condition has been established and the laboratory has been released by the responsible safety officer.
- Emergency call control station: 22222
- Brand: 112

## WHAT TO DO IN THE EVENT OF ACCIDENTS – FIRST AID – EMERGENCY CALL: 112



- Whenever you administer first aid, pay attention to self-protection, inform your supervisor and safety officer. As a general rule, call in medical professionals.
- If cryogenic fluids come into contact with eyes or skin: immediately flush the affected area with large amounts of cold or lukewarm water, followed by cold compresses. Never use hot water or dry heat. Medical advice should be sought immediately.
- If symptoms of suffocation occur, provide artificial respiration immediately.
- First aid: see alarm plan
- Accidents with injuries: 112

## PROPER DISPOSAL

- If you have any questions about proper disposal, please contact the person responsible in Dez. V-5, Telephone +49 (0)731 50-22137/38396, Fax +49 (0)731 50-22102 Uni-Ost, M25/227

## CONSEQUENCES OF NON-COMPLIANCE

### Health consequences

- Minor to fatal injury due to fainting

### Property damage

- Fires caused by spontaneous combustion of oxygen can cause significant material damage

### Legal consequences

- Operating instructions are binding and represent written occupational safety - instructions for employees.
- Failure to comply may result in legal consequences.
- Failure to follow this instruction is a violation of the given instructions and will be punished accordingly.

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- There is a risk of explosion in sealed containers for cryogenic substances (Dewars, cryostats, etc.) without a pressure relief valve.
- Cryogenic substances in liquid or vapor form (or as low-temperature gases) produce burn-like effects on the skin (cold burns).  
Exposed or inadequately protected body parts that come into contact with uninsulated vent pipes or containers will immediately stick together and body tissue will be injured when the affected body part is removed.
- Condensing oxygen can increase the oxygen concentration locally and can cause spontaneous combustion when it comes into contact with many substances (oils, fats, solvents, etc.) - even those that do not ignite under normal conditions.
- Materials that become brittle when subjected to extreme cooling may be damaged when in contact with cryogenic substances and cause injury.
- There is an increased risk of quenching.
- Overflowing containers can cause cryogenic liquids to splash.
- It is possible that some training quenches will occur during the charging process to compensate for the voltage changes in the magnet. These quenches are usually harmless, but must be considered as a temporary high thermal and mechanical stress on the magnet and should be avoided if possible.
- A thorough study of the complete NMR operating manual is mandatory.

## PROTECTIVE MEASURES AND RULES OF CONDUCT



- Electrical equipment must be regularly tested by qualified personnel in accordance with the Low Voltage Directive 2014/35/EU.
- The operation of medical electronic implants, such as pacemakers, can be affected by either static or changing magnetic fields. Pacemakers can react in an unpredictable manner when exposed to fields above 5 Gauss. The danger zone is therefore limited by the 5 Gauss line.
- Other medical implants, such as aneurysm clips, surgical clips or prostheses, may contain ferromagnetic materials and therefore be subject to strong attractive forces in the vicinity of the NMR magnet system. This could result in injury or death. In addition, eddy currents can be induced in the implant in the vicinity of rapidly changing fields (eg pulsed gradient fields), leading to heat generation.
- The 5 Gauss line of the NMR magnet is marked with a tracing tape.
- Since the field of the NMR magnet system is three-dimensional, both the floors above and below the magnet and the surrounding space at the same height must be taken into account.
- The 5 Gauss lines of the Oxford Instruments 600 MHz magnet are located at 175 cm from the center in the vertical direction and 250 cm in the horizontal direction.
- The magnet in room 1106 is in the basement, so there are no rooms underneath the magnet. The ceiling height is over 4 m. This guarantees a stray field of significantly less than 5 Gauss outside the laboratory.
- No ferromagnetic objects or tools may be used in the danger area.
- Equipment that is susceptible to failure and equipment whose failure has an impact on safety or which is damaged by magnetic fields must be kept away from the danger area.
- There is a risk of tipping over when climbing onto the magnet. Climbing onto or onto the NMR magnet is prohibited.
- Access to the hazardous area must be restricted to trained personnel by keeping the NMR laboratory locked.
- Only low-pressure Dewars may be used for storing and transporting cryogenic substances. Dewars pressurized to pressures of  $> 2$  bar are prohibited for use with the NMR.
- Containers for cryogenic liquids must not be completely closed, as this would lead to a strong build-up of pressure. This represents a risk of explosion.
- Helium can displace air in the upper areas of a room and cold nitrogen can displace air in the lower areas.
- Large quantities of gases evaporate during a quench and when large quantities of liquids are spilled. To avoid a quench, it is important to ensure that there is a constant supply of sufficient cooling liquids (nitrogen and helium). Helium is monitored electronically and checked at least weekly. Nitrogen must be checked daily. Great care must be taken when filling to avoid accidental spillage.
- Cryogenic substances may only be used in laboratories with technical ventilation and a minimum air circulation of  $25 \text{ m}^3/\text{h}$  per  $\text{m}^2$ .
- The maximum gas volume in the expanded state at 1 bar must not exceed  $1/3$  of the room volume.
- If a large amount of gas is released (e.g. during a quench), the laboratory must be left immediately and the responsible safety officer must be contacted. The laboratory may only be re-entered once a safe condition has been established and the laboratory has been cleared by the responsible safety officer. Users of neighboring laboratories must also be warned and their laboratories must also be evacuated until clearance is given.

## PROTECTIVE MEASURES AND RULES OF CONDUCT (continued)

- The laboratory is equipped with three permanently installed gas warning sensors to check the oxygen content with acoustic and visual alarms. Two sensors are installed above the NMR devices to detect the loss of oxygen due to evaporating helium. One sensor is installed near the floor to detect the loss of oxygen due to evaporating nitrogen. If one of the sensors is triggered, everyone present must leave the laboratory until a safe condition has been restored and the laboratory has been released by the responsible safety officer.
- Contact of cryogenic liquids with air must be minimized.
- Because liquid nitrogen and helium are colder than liquid oxygen, the oxygen in the air condenses. If this happens over a long period of time, the oxygen concentration in the containers can become so high that handling them becomes just as dangerous as handling liquid oxygen. This is especially true for wide-neck Dewars due to the large surface area.
- In the danger area of cryogenic liquids, fire, flames, open ignition sources and smoking are prohibited. This applies in particular when filling the magnet.
- While nitrogen and helium do not support combustion, their extreme cold can cause oxygen from the air to condense on cold surfaces and the oxygen concentration to increase locally. There is a particular risk of fire if the cold surfaces are covered with oil or fat. Spontaneous combustion can occur!
- Surfaces that come into contact with cryogenic liquids must be cleaned beforehand, particularly of any flammable deposits.
- To fill with nitrogen, either hoses made of a material that does not become brittle at  $-196^{\circ}\text{C}$  (rubber, Teflon hoses) or insulated lines must be used.
- Only insulated pipes (transfer lines) may be used for filling with helium.
- The O-rings on the magnet system are also sensitive to low temperatures. When filling, make sure that no liquid helium or liquid nitrogen gets on the O-rings.
- The functioning of the ventilation must be checked before starting work and work must be stopped immediately if the ventilation is faulty. Indicators of faulty ventilation are:
  - o Announced shutdown of ventilation
  - o Power failure / power outage
  - o Whistling noises from the ventilation
  - o Indicators before ventilation show no flow
  - o Rapid temperature changes in the laboratory or temperatures above  $30^{\circ}\text{C}$
  - o Headaches or difficulty concentrating in the laboratory
- If the ventilation malfunctions, the filling process must be stopped immediately and the responsible safety officer contacted. Filling may only be continued once a safe condition has been established and the laboratory has been released by the responsible safety officer.
- The nitrogen vessel should be checked daily for flash off and nitrogen level. These values should be recorded. If the flash off drops to zero, the towers should be checked immediately for the presence of ice and any blockages removed to prevent pressure build-up.



## PROTECTIVE MEASURES AND RULES OF CONDUCT (continued)



- If the consumption of cryogenic liquid increases significantly, it is suspected that the thermal insulation of the cryostat is reduced. In this case, the pressure in the vacuum vessel should be checked.



- Overflow valves on the containers must be fitted with a hose or the opening must be positioned in such a way that no user can be hit by escaping liquids.
- Never pressurize the nitrogen Dewar to more than 350 mbar (5 psi) and always ensure that all exhaust valves on the NMR magnet are fully open.
- Do not leave the helium valves open to the atmosphere for more than 5 seconds unless there is a large gas flow.



### Personal protective measures

- Eye protection: Frame glasses with side protection.
- Hand protection: suitable cold-resistant gloves. The gloves must be loose-fitting so that they can be easily removed in the event of a liquid spill.
- Clothing: Closed clothing and a lab coat must be worn to prevent the penetration of cryogenic liquids. The lab coat prevents clothing from freezing to the body in the event of contamination.



## BEHAVIOR IN CASE OF FAULTS – CONTROL STATION: 22222



- Clear and cordon off the danger area, inform your supervisor and safety officer. Contact details for the aforementioned persons can be found in the alarm plan
- If a large amount of gas is released (e.g. during a quench), the laboratory must be evacuated immediately and the responsible safety officer contacted. The laboratory may only be re-entered once a safe condition has been established and the laboratory has been released by the responsible safety officer.
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- Emergency call control station: 22222
- Brand: 112

## WHAT TO DO IN THE EVENT OF ACCIDENTS – FIRST AID – EMERGENCY CALL: 112



- Whenever you administer first aid, pay attention to self-protection, inform your supervisor and safety officer. As a general rule, call in medical professionals.
- If cryogenic fluids come into contact with eyes or skin: immediately flush the affected area with large amounts of cold or lukewarm water, followed by cold compresses. Never use hot water or dry heat. Medical advice should be sought immediately.
- If symptoms of suffocation occur, provide artificial respiration immediately.
- First aid: see alarm plan
- Accidents with injuries: 112

## PROPER DISPOSAL

- If you have any questions about proper disposal, please contact the person responsible in Dez. V-5, Telephone +49 (0)731 50-22137/38396, Fax +49 (0)731 50-22102 Uni-Ost, M25/227

## CONSEQUENCES OF NON-COMPLIANCE

### Health consequences

- Minor to fatal injury due to fainting

### Property damage

- Fires caused by spontaneous combustion of oxygen can cause significant material damage

### Legal consequences

- Operating instructions are binding and represent written occupational safety - instructions for employees.
- Failure to comply may result in legal consequences.
- Failure to follow this instruction is a violation of the given instructions and will be punished accordingly.

Structural unit/work area:  
ZQB / Building M26 / Room 1106

Activity: Working with a 600 MHz NMR spectrometer  
As of 28 May 2024

### DESIGNATION

## NMR device Oxford Instruments - 600 MHz

### DANGERS FOR HUMANS AND THE ENVIRONMENT



- Large forces of attraction can be exerted on equipment in the immediate vicinity of the NMR magnet system. The force can become so large that the equipment is moved uncontrollably towards the NMR magnet system.
- Large devices (eg gas cylinders, power supplies) can cause bodies or limbs to become trapped between the device and the magnet system: the closer to the NMR magnet system, the greater the force; the larger the magnetically effective device mass (proportional to the magnetic field gradient), the greater the force.
- The effective shielding of the superconducting coil minimizes the effects of the stray magnetic field. The horizontal 5 Gauss line is only about 1m (96cm) away from the magnet casing.
- However, it should be noted that directly above and directly below the magnet the stray field and thus the magnetic field gradient is very high and the forces of attraction on magnetic objects are very strong!
- Cryogenic liquids, even when stored in insulated storage containers (Dewar vessels), remain at a constant temperature around their respective boiling point and gradually evaporate. The very large increase in volume that accompanies the evaporation of the liquid to gas and the subsequent heating process is about 700:1 for helium and nitrogen.
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