01/Sept/2023 CAESR Risk Assessment -- EPR Laboratory User Form

Anyone wishing to make use of the CAESR must read, understand and strictly abide by the safety and laboratory operating rules set out in this document. You must read and fully comprehend this document and register your compliance with your signature and have it witnessed by the CAESR Scientific Applications Manager Dr. William K. Myers or Co-Director Prof. Christiane Timmel. Your Supervisor must also sign this document. Everyone wishing to use the EPR spectrometers MUST undergo the appropriate training sessions with a member of the EPR staff before using any of the instruments.

Anyone entering the CAESR facility, either Oxford frequent users or visiting users MUST read, understand and sign the CAESR Risk assessment forms and have seen the CAESR Scientific Applications Manager, the CAESR Co-Director, or the Chemistry Safety Officer (Dr. Chris Blackwell) for an introduction to CAESR safety.

No LASER work may be carried out unless the users have been given an introduction by the CAESR Scientific Applications Manager Dr. William K. Myers or Co-Director Prof. Christiane Timmel, and signed the LASER safety forms and have attended the LASER safety lectures. Dr. Kevin Henbest is the LASER manager of CAESR SRF.

All research workers' supervisors need to have read and counter-signed this risk assessment and returned a copy of the countersigned risk assessment to the CAESR Scientific Applications Manager also. *Supervisors take responsibility for the safety of their research workers* and should have regular safety meetings with their groups where the safety and regulations in CAESR are discussed.

All CAESR users must make themselves familiar with the nearest emergency exits and locations of the fire extinguishers.

All CAESR users must make themselves familiar with the CAESR laboratory oxygen level meter and evacuation procedure if the low oxygen level alarm sounds.

Users not having attended the ICL induction and safety meeting need to see the Chemistry Safety Officer (Dr. Chris Blackwell) for information.

The out-of-hours regulation for working in CAESR and access to the ICL need to adhered to strictly. See the Chemistry Safety Officer (Dr. Chris Blackwell) for full requirements.

General Lab Practice

The EPR lab hosts a number of researchers from a range of backgrounds across various departments. In addition to the specific rules given below, all the best-practice and lab rules from your main laboratory apply here, with regards to sample handling, clearing up after yourself, not bringing food and drink into the lab, etc. Do not use any equipment which you do not fully understand. Do not work while impaired by alcohol, drugs, hunger, caffeine, sleep deprivation, or are *wrecked*, prospective users in such condition must seek assistance from the Scientific Applications Manager. The daily booking change-over time is 9:00 a.m. Please allow about 30 min. of grace period. For deviations from the E580 or E680 booking schedule you are required to notify the Scientific Applications Manager as soon as possible, for the EMXmicro it is

recommended. Users must be aware of the bookings scheduled before and after theirs and follow other website instructions.

Before bringing hazardous samples or materials into the lab you must complete a 'hazardous materials' COSHH form (you must also seek permission from the CAESR Scientific Applications Manager or the CAESR Co-Director) and bring a copy of the COSHH form from your lab. All users need to have signed the risk assessment for gas cylinders.

Sample Preparation

The CAESR spectrometer labs is a characterisation facility, and should not be used for any sample preparation, which should be performed in the adjacent CAESR wet lab laboratory. Samples should be brought into the EPR laboratory in sealed or capped EPR tubes, e.g. PTFE tape. Any spillages/breakages must be immediately cleaned and recorded in to the laboratory manual for the spectrometer (and the COSHH regulations for the chemicals adhered to).

Use of the chemicals / fume cupboard / working with sharps / heat baths, etc.

If you want to use the fume cupboard in the wet lab, work with sharps, use heat baths etc. or employ any other laboratory procedure, you need to have understood and signed the corresponding risk assessment forms (some exist in the CAESR group office, others you might have to download if they are specific to your experiment and have them accepted and countersigned by the Co-Director or Scientific Applications Manager). COSHH forms need to be signed for all chemicals used.

EPR Sample Tube Handling

Safety glasses and a laboratory coat should be worn when dealing with samples, where appropriate (clean lab coats are available in the EPR laboratory; do not bring your (perhaps dirty) lab coat). EPR sample tubes can and do explode, and glass can damage your eyes, in particular. The following danger must be appreciated:

1) You must wear safety glasses and exercise caution when handling any EPR samples that are rapidly heated and cooled. The rapid cooling of some samples may result in the formation of a solid bolus in the sample tube that may make the tube prone to explosive rupture. The safety threat posed by flying glass and violently escaping gases and liquids should not be underestimated.
2) Educate yourself about the temperature at which chemicals evaporate. When a sample gets close to the temperature at which it evaporates, it may quickly become volatile.
3) Wear safety glasses, lab coat, face mask, elbow-length gauntlets and other protective clothing whenever there is any risk at all of spillage, breakage, or explosion, especially when accessing tubes stored in liquid nitrogen.
4) Follow the COSHH guidelines applicable to all chemicals within your sample (a copy of the relevant COSHH forms from your main laboratory should be given to the EPR centre at the same time as you have to read and sign all the COSHH forms applicable to any sample measured in the EPR spectrometers (even if not working on the same spectrometer) additionally.
5) Protective shields should also be employed when there is any risk of explosion or implosion.
6) Never leave unlabelled equipment and EPR tubes on CAESR premises. You are responsible for correct disposal of unused samples according to their applicable COSHH forms. Any unlabelled equipment or samples will be removed and disposed of.

7) Do not enter the EPR lab with any biological samples which have not been pre-certified and categorized by the relevant supervisors and the Scientific Applications Manager/ Co-Director. For biologically hazardous samples, a protocol of disposal and risk assessment has to be established and countersigned by the biological safety office before the sample can be taken into the EPR lab. The EPR lab is laid out for up to CAT II biological samples only. Under no circumstances should biological samples of a higher category be brought or investigated on the premises.

Magnetic Fields

There are specific safety hazards associated with the use of EPR instruments that relate primarily to the presence of intense stray magnetic fields in the vicinity of the instruments which may affect or interfere with heart pacemakers; medical implants; magnetic bank or ID cards; watches (non LCD). Thus, specific rules apply regarding access to the EPR lab:

1) Anyone with a heart pacemaker must not go beyond the magnetic field hazard warning signs posted in the ICL and will not be allowed access to any EPR laboratory.
2) Anyone with a medical implant must seek approval from the Co-Director of CAESR before entering any EPR lab or making direct use of any EPR spectrometer.
3) All users should make themselves aware of which metal objects are magnetic (whether originally in the lab, or brought in) and take suitable precautions.

WARNING: Magnetic fields attract magnetisable (permanent or temporary magnetic) objects. Strong magnetic fields will exert large forces on both large and small magnetisable objects, such as pumps, screwdrivers, or metal scissors. This can create an extremely dangerous situation if these objects are in the vicinity of a strong (defined here as on or within the 5G line) magnetic field. Large magnetisable objects should never be taken within the 5G line of either the W-band superconducting magnet or the two X-band electromagnets in the CAESR facility. Small magnetisable objects should only be taken within the 5G line where absolutely necessary whilst exercising caution. Before carrying out any work in CAESR the location of the spectrometer magnets and the 5G lines must be clearly understood.

1) Always assume the W-band magnet is at field, until you confirm it is not at field (this can be determined by inspecting the W-band magnet power supply indicators, or holding a metal paper clip near the magnet to detect a field).
2) It is best practice to position the safety fence around the W-band magnet along the 5G line, whenever the magnet is at field (ca. 3.35T).
3) The safety fence around the W-band magnet must not be removed by anyone not working on the EleXSys 680 instrument.

Microwaves

1) Do not have an open waveguide when the microwave power is on.
2) Switch the bridge to Standby Mode before you remove or change EPR cavities.
3) Never look down an open waveguide when there is microwave power. The eyes are very susceptible to damage from microwaves. Chronic or very intense exposure may lead to cataracts.

Tripping Hazard

Beware of coaxial cables, helium and nitrogen gas line tubing, water lines, LASER fibres, and electrical cords that may be disorganized, especially in temporary experimental setups. Users are required to notify others in lab of hazards and minimize dangers. Access to areas behind spectrometers is only available for agile users who are capable of stepping carefully and securely

over hazards. Please ask for help if you are unsure of your access ability or find unsafe conditions.

Cryogens

You must have had a cryogen safety induction at the equivalent level for researchers in the Chemistry Department. The University of Oxford Cryogen Safety course is strongly recommended. Before commencing work with any cryogens, check that the oxygen meter is in place and is functioning correctly. Evacuate the room immediately if the oxygen meter sounds an alarm (oxygen concentrations below 19%). Do not re-enter the room until the alarm stops and then only when you have a work-mate present and the source of the dangerous gas level has been identified and removed. While liquid nitrogen and liquid helium are subject to the Leidenfrost Effect, severe burns may happen if these liquids soak clothing, form a high pressure jet or you touch objects they have cooled. The following precautions should be adhered to:

Liquid Nitrogen dewars (open top type for sample storage, ca. < 10L)

1. Always wear safety glasses, cryogenic gloves and close-toes shoes
2. See section on EPR tube handling.

Liquid Helium dewars (for cryostat cooling)

- Always wear safety glasses and have access to cryogenic gloves
- See section on EPR tube handling. Never open dewars that are under pressure. Slowly release the pressure using the helium recovery line first.
- Never store dewars in the EPR laboratory without connecting them to the recovery line. Release any pressure slowly to ensure the return line piping does not crack from excessive cold.
- When you have finished with a dewar it is your responsibility to arrange for it to be returned, typically by e-mailing Cryogenics. If your dewar still contains LHe then you ought to write on the dewar the remaining litres (plus the date and your name).

Liquid Nitrogen dewars (for cryostat cooling)

Always wear safety glasses and have access to cryogenic gloves

See section on EPR tube handling. Never open dewars that are under pressure. Release the pressure (don't use the helium recovery line for nitrogen, rather vent out window with a hose).

1) Never open dewars that are under pressure. Release the pressure using the pressure relief valve; make sure no one is standing in front of the valve outlet.
2) Release any pressure slowly, allowing sufficient time for the pressure to reach atmospheric pressure. Vent excess N2 pressure out of a window when possible.
3) When you have finished with the LN2 dewar it is your responsibility to arrange for it to be stored safely – it must not be pressured in any way for storage.

Low-Loss Transfer Lines (LLT-600) & Cryostats

- You are not permitted to set-up the cryogen flow cryostat system without first passing the induction test (run by the Scientific Applications Manager).
- You are not permitted to set-up the cryostat systems alone under any circumstances – most users must always have a 2nd person in the room to help. Very experienced users – you must always have a 2nd person in building proximity.
- If a dewar that is under pressure is opened by mistake, don't panic. Make certain you're wearing a proper cryogenic glove. Close the ball valve calmly and open the laboratory

door. If the tank pressure exceeds the safety valves (ca. 0.5-1.5 Bar) release pressure into lab until safe level and then *slowly* vent remaining pressure to He return line (if He).
- KNOW THE TANK PRESSURE; transfer line insertion and removal must be performed with a depressurized dewar, with safety glasses and cryogenic gloves worn. Your supervisor will be notified if you shatter the He return line by rapid venting.
- A cryogenic glove must be worn on any hand removing or inserting the transfer leg.

General Safety and Operation Procedures

- You are required to seek training for all procedures new to you in the ESR labs or at any time you face procedural uncertainty. Plan ahead for required help, if possible.
- You are required to gain familiarity with all relevant spectrometer, software, and instrumentation manuals relevant to the procedures and equipment used in your experiments. Please ask for help in locating them if needed.
- You MUST wear safety glasses when changing samples.
- Retrieval or insertion of samples from/to liquid nitrogen storage requires addition of a face shield and elbow-length cryogenic gauntlets.

LASERs

See the LASER Safety Risk Assessment form. LASERs may only be used by users having signed and understood all LASER safety forms and corresponding risk assessment forms, having attended a LASER safety talk and having been instructed in the use of LASERs (contact the CAESR Co-Director for *a safety knowledge assessment and form signature*).

On the following page, instructions are given on how to access CAESR ESR labs with respect to the LASER interlock system. Further training is given to users who have completed LASER safety training and have signed necessary safety paperwork. It is <u>imperative</u> that all CAESR users respect the LASER safety interlock system. LASER experiments can last 6-20 hours and jostling of the doors by non-LASER users can inactivate the interlock and halt the experiments prematurely for other people. This is not acceptable. Please be aware of the state of the interlock prior to deciding on whether you may <u>*or may not*</u> access the spectrometer room. If an interlock is engaged, and you are not maintaining the experiment by reservation or agreement with that person, the recommended course of action is to ring the doorbell and wait to see if there is someone inside. If the keypad is operational, you have completed all LASER safety forms and training, and no response is given, you may carefully enter the room as needed.

Glowing Red LED, rm F12

Glowing Green LED, F11 & F19

If you see this sign illuminated outside of a CAESR lab door, with both red and yellow parts glowing, you may NOT enter if you don't have LASER safety training. Press the doorbell button to ring for assistance from an active user or ask for assistance from the Scientific Applications Manager. A glowing red LED means the keypad works for trained LASER users.

Wenn Sie dieses Schild mit aktiven roten und gelben Warnleuchten außerhalb einer CAESR-Labortür sehen, dürfen Sie das Labor NICHT betreten, wenn Sie keine LASER-Sicherheitseinweisung erhalten haben. Drücken Sie die Klingeltaste, um den aktiven Benutzer zu benachrichtigen, oder bitten Sie den Scientific Applications Manager um Hilfe. Eine rot leuchtende LED bedeutet, dass das Keypad für geschulte Lasernutzer zum Eintritt ins Labor verwendet werden kann.

Se i messaggi rossi e gialli del cartello di segnalazione laser all'ingresso di un laboratorio CAESR sono entrambi attivi, l'accesso per utenti che non hanno seguito il corso di formazione sulla sicurezza laser è VIETATO. Premere il pulsante del campanello per contattare l'utente attivo o chiedere assistenza al responsabile scientifico di CAESR. Un LED rosso acceso indica che la tastiera può essere usata da utenti LASER istruiti per accedere al laboratorio.

如果您在 CAESR 实验室门外看到此标志亮起，红色和黄色部分都在发光，~~，~~则且如果您没有接受过激光安全培训，则不得进入。按门铃按钮响铃以寻求~~活跃~~当前用户的帮助或寻求科学应用经理的帮助。~~。~~发光的红色 LED 表示键盘适用于经过培训的 LASER 用户。

Dark Red LED, room F12

Absent Green LED, F11 & F19

If the red LED is off, the keypad does not work. DO NOT ENTER. Alignment is in progress. Use the doorbell only if access is needed urgently.

Wenn die rote LED aus ist, ist die Tastatur nicht aktiv. BETRETEN VERBOTEN. Eine Justierung des Laseraufbaus ist im Gange. Verwenden Sie die Türklingel nur, wenn dringend Zugang benötigt wird.

Se il LED rosso è spento, la tastiera non è attiva. L'INGRESSO É VIETATO. Un allineamento laser è in corso. Utilizzare il campanello solo se l'accesso è urgente.

如果红色 LED 熄灭，则键盘不工作。此时不许进入。校准正在进行中。仅在急需进入时才使用门铃。

Without the red glowing part, you may enter.

Wenn die rote Warnleuchte nicht aktiv ist, darf das Labor betreten werden.

Se il segnale rosso non è attivo, l'accesso al laboratorio è permesso.

~~没有~~红色~~发光~~部分没有在发光时，您可以进入。

Magnet Quenching

A cryomagnet is basically just a closed loop of superconducting wire that allows electric current to flow perpetually through it without any resistive losses, thus generating a stable "permanent" magnetic field. The wires that are used to wind high field cryomagnets are only superconducting when cooled to very low temperatures. For this reason, cryomagnet coils are submerged in liquid helium to 4.2 Kelvin. Although there is no resistance in the cryomagnet coil when properly cooled and energized, there is a great deal of energy stored in the electrical current. If, for any reason, a very small portion of the superconducting wire stops being a superconductor (becomes "normal"), then the resistive heat generated in that section of wire will cause neighboring sections of wire to become normal also, resulting in a chain reaction that swiftly warms the entire coil and causes all of the stored energy to be dissipated as heat. This process takes only a few minutes, and can result in an impressive display of rapidly boiling liquid helium, similar to a geyser.

EPR Magnet Fill and Quench Safety:

In the unlikely event of the magnet quenching or of a cryogenic failure, up to 100 m^3 of helium gas may evolve over a period of several minutes. Although inert, lighter than air and non-toxic, there could be a risk of asphyxiation in a confined space. Personnel should evacuate the area in such a situation. A quench warranting evacuation would be obvious by the noise of the escaping gas and clouds of vapour. When transferring liquid nitrogen or helium, the following steps should be observed to avoid accidents:

- Gloves, eye protection, and closed shoes (NOT STEEL CAPPED) must be worn
- Doors should be propped open to increase ventilation
- Tanks on wheels must be checked or held by another person.
- Check that the oxygen meter is in place and is functioning correctly.
- Evacuate the room immediately if the oxygen meter sounds an alarm (oxygen concentrations below 19%).

Since the possibility of a quench is higher when filling the magnet, and since transfer involves manual operations, there is a remote possibility that an operator could be rendered unconscious around the time of a quench. Fills should not be done by a single operator. A second person has to be present at all times during a magnet fill.

Wet Lab

You must wear safety glasses and a white laboratory coat while in the wet lab. Work may be conducted in the CAESR wet lab (room F13), only if you have talked to the Co-Director, Scientific Applications Manager, or LASER advisor, and after you have signed the risk assessment forms for procedures and relevant chemicals in the wet lab. Separate safety regulations apply to anyone wanting to use the wet lab Schlenk line and methane/oxygen torch.

Facility Management Regulations and Reporting to Scientific Applications Manager

You must make a report as soon as possible of:

1) Any breakages of sample tubes, particularly while in the EPR resonators.
2) Any equipment, person, or circumstance that may pose a safety hazard.
3) Any safety concern.
4) Untidy laboratory conditions due to previous user(s).
5) Any user with intemperate behaviour and/or interference of your experiments.

- CAESR is a shared centre, and as such you are obligated to leave the laboratory as you have found it – neat and tidy.

- You are not permitted to store or leave any equipment or samples without arranging a suitable storage place with the Scientific Applications Manager.
- No CAESR equipment is to be removed without consent of the Scientific Applications Manager.
- If you leave samples lying around in the laboratory or equipment, the CAESR facility personnel takes no responsibility whatsoever for their well-being!

Appendix:

Volume of Rooms:

1) When there is no screen, the total volume of the F11 & F12 room is 228 cubic metres (called F11 & F12 room).
2) When the screen is down, dividing the room, the F12 E680 spectrometer is now in a room of roughly 170 cubic metres and the F11 E580 spectrometer is now in a room of roughly 58 cubic metres.
3) F19 (w/F16) is approximately 135 cubic metres.
4) B16 is approximately 73 cubic metres.

First, the calculation for Nitrogen, 50L boil-off (very unlikely indeed).

a) F11 & F12: allow liquid volume that can boil off: 28.5L
   Oxygen percentage in the room if 50L were to boil off: 17.81%
b) F12: allowed liquid volume that can boil off: 23L
   Oxygen percentage in room if 50L boil off: 16.77%
c) F11: allowed liquid volume that can boil off: 8L
   Oxygen percentage in room if 50L boil off: 8.6%
d) F19 (w/F16): allowed liquid volume that can boil off: 18L
   Oxygen percentage in room if 50L boil off: 15.6%
e) B16: allowed liquid volume that can boil off: 10L
   Oxygen percentage in room if 50L boil off: 11.2%

Second, the calculation for Helium, 50L boil-off (very similar data).

a) F11 & F12: allow liquid volume that can boil off: 28L
   Oxygen percentage in the room if 50L were to boil off: 17.55%
b) F12: allowed liquid volume that can boil off: 21.51L
   Oxygen percentage in room if 50L boil off: 16.42%
c) F11: allowed liquid volume that can boil off: 7L
   Oxygen percentage in room if 50L boil off: 7.6%
d) F19 (w/F16): allowed liquid volume that can boil off: 17L
   Oxygen percentage in room if 50L boil off: 15.2%
e) B16: allowed liquid volume that can boil off: 9L
   Oxygen percentage in room if 50L boil off: 10.3%

Glovebox Procedures

(1) Training delegated by a Supervisor is required prior to first use.
(2) Users shall read the MBraun glovebox manual, which is *reinforced* below.
(3) You must use the log book.

Insert and withdraw hands with glovebox gloves slowly to avoid large pressure changes that cannot be compensated quickly enough. Use the foot pedals to adjust the pressure as necessary. The glovebox should always be under positive pressure to avoid letting ambient atmosphere into it through small holes.
Always wear cotton gloves and wear long sleeve shirts, Tyvek forearm covers or a clean lab coat to avoid contamination of the inside of the gloves and to cover watches and jewellery. You may use the talcum powder on your cotton gloves to make hand insertions easy.

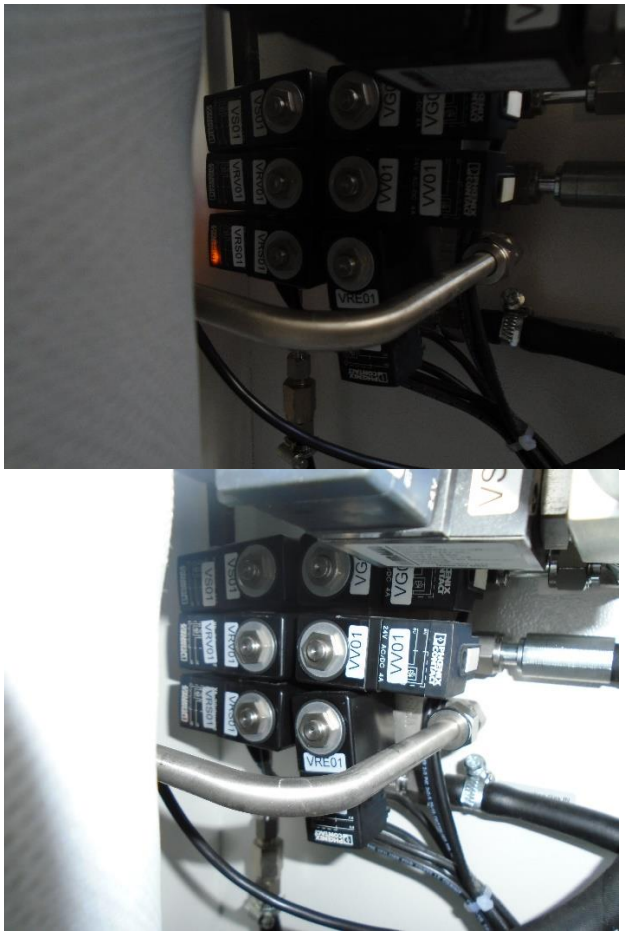
If the glovebox is <u>not in use</u>, it should be placed in **Eco Mode** and the *vacuum pump shall be turned off*.

Antechamber and transfer of materials into the glovebox
The antechamber should be evacuated fully and refilled with inert atmosphere at least three times prior to opening it from inside the glovebox. Ideally, the evacuate cycles should last 5-10 minutes (three cycles of 15 minutes for the large antechamber).
When closing antechamber doors, make sure the seals are free of dirt and other objects. Never attempt to open an antechamber placed under vacuum.
Only keep the antechamber open for the minimum time necessary to insert the items that need to be transferred into the box.
When moving material into the glove box, open closed containers (boxes, bottles, vials, etc.) far enough to allow exchange of gasses to make sure the enclosed ambient air does not contaminate the box atmosphere. All liquids transferred into the box have to be degassed and dried, newly purchased anhydrous solvent bottles with Sure/Seal™ cap can be transferred into the box. Chemicals under nitrogen can be transferred into the box in closed vials or containers, any closed transport containers should be suitable for vacuum to avoid the risk of explosion.
When transferring paper or other porous materials (e.g. cork rings) into the glovebox, keep them under vacuum in the antechamber for at least 12 hours (to avoid bringing adsorbed water into the glovebox).
The antechamber should be kept under vacuum at all times if not in use.

Working inside the glovebox
When working with solutions inside the glovebox, disposable gloves may be used on the inside to avoid contamination of the gloves. A piece of aluminium foil is recommended to facilitate clean-up and reduce contamination of the glovebox workspace.
The amount of solvent used inside the glovebox should be kept to a minimum. Turning the circulation off while solvents are being used and purging the box before turning the circulation back on is a good solution to prevent damage to the catalyst. This procedure is <u>*required*</u> if larger amounts or volatile chemicals and solvents are used.
Any vials or bottles containing solvents should be completely sealed at all times if not actively used. Only Sure/Seal™ or other septa-cover solvent bottle tops are allowed for long term solvent storage in the glovebox. Please remove solvent bottles and dissolved materials at the project conclusion.

Clean up the work space after use and remove any waste from the glovebox, especially if contaminated with solvent. Immediately clean up any broken glass and do not leave open needles on the glovebox workspace to avoid puncturing the gloves by accident.
Everything should be clearly labelled at all times and stored on the shelves if not currently in use. Avoid loose vials, use vial racks or trays where possible.
Any malfunction or spills should be logged in the log book and an experienced user should be notified as soon as possible.

Glovebox molecular sieves & catalyst regeneration – READ MBRAUN MANUAL FIRST
This must be performed in communication with the CAESR Scientific Applications Manager
Solvent vapour filter (molecular sieves) is to be regenerated on a three month timescale using normal working gas and procedures of the *supplemental manual in manual binder*. Catalyst regeneration shall take place whenever the O2 increases to 3-5 ppm when left on overnight, or every six months, whichever comes first.



The red light on lower VRS01 indicates valve is open, photo taken during regeneration.

Between regenerations, VRS01 can fill with debris and jam open and the subsequent check valve will also be full of debris if that is the case. During a regeneration, the hot catalyst is put under vacuum and atmosphere can come back into the system via the jammed VRS01. Regeneration will fail in this case.

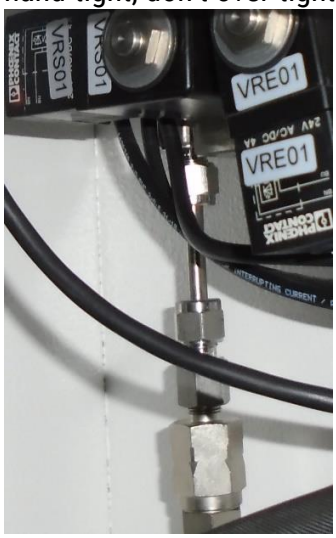
EXTRA Procedure *prior* to Regenerations with respect to VRS01 valve, to do once or twice per year.

1. If the procedure is executed, regeneration MUST follow. Set Circulation off. Remove VRS01 internal components. Be very careful to not scratch or mark smooth end surfaces of long cylinder part.

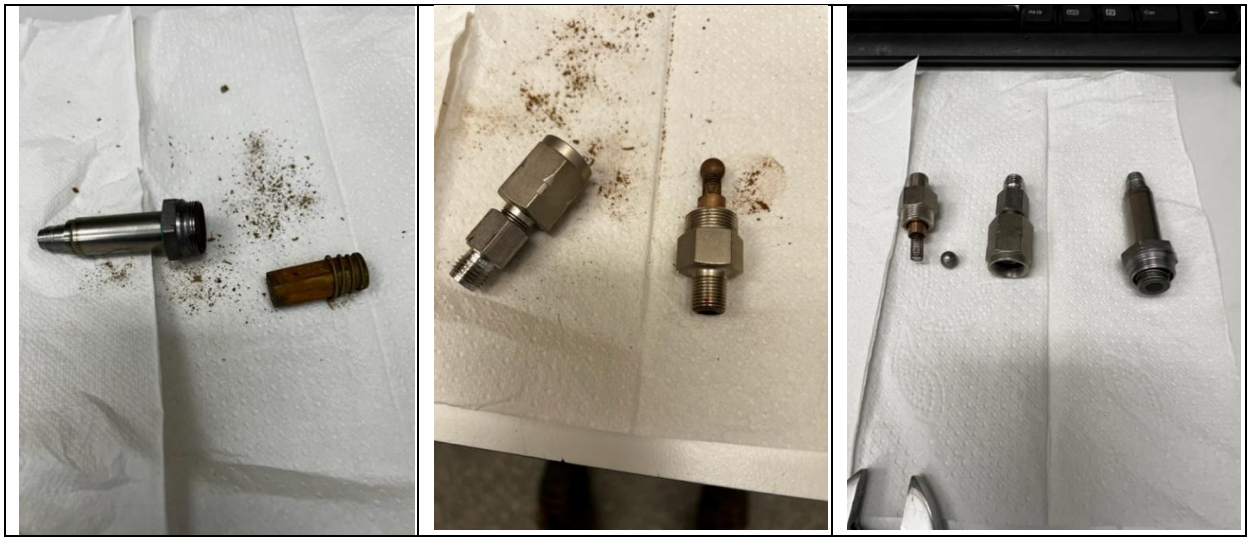
2. Use a bronze brush or Scotch-Brite® pad to wipe away any debris. Isopropyl alcohol (IPA) may be used but not strong chemicals to clean parts.

3. If debris is found inside the VRS parts, then you must also disassemble and check the One-way valve shown in the photograph below. Follow step 2 above. Be very careful to not scratch or mark smooth surfaces that ball bearing contacts.

4. For reassembly, first check that spring action is smooth and free. Nuts should be simply hand-tight, don't over-tighten. Please note the arrow direction.



VRS01 valve and one-way valve.



Before and after for VRS01 (left) one-way valve (centre), and cleaned parts (right).

Please note that the solvent vapour system also features a VRS01 valve and check valve. Currently there is no recommendation to clean those parts.

Apart from above, a **Catalyst Regeneration** procedure *in the Manual* takes 960 minutes and should ideally be started in the late afternoon and left to run over night. The glovebox should not be used and the antechamber should not be operated during the regeneration.

Setting up the catalyst regeneration

1. Set up regeneration gas cylinder (N_2:H_2 90:10 max 10% H_2).
2. Open cylinder valve, check that sufficient regeneration gas is present (min 80 bar on 50 l gas bottle).
3. Insert the regeneration gas exhaust output into the sink within the fume hood, fix it securely and clearly mark it as a trip hazard.
4. Stop the circulation on the touch screen, make sure the vacuum pump is on and press Regeneration on the Glovebox control.
5. A message asking you to confirm that a regeneration should be performed appears on the touch screen, select Yes.
6. At this point the input valve for the regeneration gas opens and the message "Prove flow of the regeneration gas! If OK -> Confirm this button" appears on a red background. Adjust the pressure regulator on the regeneration gas cylinder to achieve a flow rate of 15-20 l/min (ca. 0.5 bar). (The correct pressure and flow rate are only displayed when the regeneration gas input valve is open, i.e. when the mentioned message shows up, after confirming, the valve is closed again and no flow is observed. Do not adjust the output pressure at this point.) Once the correct flow rate is established, confirm this by touching the red button on the touch screen.
7. The regeneration starts. To monitor the progress, go back to the main menu, press on the symbol for the reactor (purple, REG) and then open the STATUS tab. This will show the remaining time.
8. The circulation will automatically be turned back on after the regeneration program has finished. It typically takes about 1 h for the oxygen and water readings of the analyser to be accurate.

Once started, the regeneration program cannot be interrupted. If the regeneration is aborted after the regeneration gas input valve has been opened (after the red message asking to confirm regeneration gas flow), a 300 min program is started that purges the regeneration gas still present in the reactor. The glovebox cannot be used during this time.

The regeneration program automatically runs through the following steps:

- t = 0 min: Purge valve (VRS) open and heating (EH) on
- t = 180 min: Regeneration gas inlet valve (VRE) open
- t = 345 min: Heating (EH) off
- t = 360 min: Regeneration gas inlet valve (VRE and VRS) closed
- t = 480 min: Regeneration vacuum/gas ballast valves (VRV/VGB) open
- t = 540 min: Gas ballast valve (VGB) closed, Refilling for 20 sec, then evacuating
- t = 570 min: Refilling for 20 sec, then evacuating
- t = 600 min: Refilling for 20 sec, then evacuating
- t = 660 min: Regeneration vacuum valve (VRV) closed and pressure compensation valve (VS1) open
- t = 960 min: Pressure compensation valve (VS1) closed, program completed