Oxford Triton Dilution Fridge
Standard Operating Procedure

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Updated 12/2017
Update 2018.6 Put details related to Elsa operation including magnet operation etc. by Guang
Update 2019.3 make it into SOP.

Section 1: Overview and Risk Assessment

Users must have enough knowledge on vacuum system, compressed gas cylinder operation and cryogenics. DRS Compressed Gases and Cryogens online training AND hand on training by master user will meet this requirement.

The cold trap in the system has liquid nitrogen in it, fill nitrogen in well ventilation area, wear cryogenic gloves, and safety glasses. Don't leave while transferring nitrogen, and don't overfill.

The system contains Helium -3 gas which is extremely expensive. Don't touch the helium mixer tubing without permission. Follow the maintenance schedule according to Oxford.

Read Oxford manual for all the details of the system, maintenance etc.

Hand on training by master user for each DR system is required.

1. If Thou are ever in doubt, Thou shalt not!
2. If Thou are ever in doubt, Thou shalt not!
3. Thou shalt not open V10 unless ALL the mixture is in THE Tank!
4. Thou shalt not move the Red mobile pump while the turbo is running.
5. Thou shalt not move the Leak checker while the turbo is running.
6. Thou shall Respect the Fridge.
7. The person must be trained and approved to use the fridge.
8. Safety first!
9. Wear proper PPE for liquid nitrogen handling.
10. Compressed air cylinder/bottle should be secured while in use, and capped while in storage or transport. The transport cart is used for temporary move of the cylinder, NOT for storage or regular cylinder usage.
11. Magnetic materials like screw drivers, pump stations, dewars should stay away from the magnet to avoid magnetic field harzard. Certain field line boundary is marked on the floor.
12. Remember the magnet is a high current and high inductance device. Changing or disconnecting the current leads circuit can be fatal. Make sure the magnet is zero energy and the power supply is off before you touch the circuit.

Section 2: Training Documentation

Signing this document means that you have read and understand all aspects of this Standard Operating Procedure. The supervisor (master user) is the person that acknowledges you took the training and understand the procedure.

Name (Printed)	Name (Signed)	Supervisor	Date

Section 3: Operation Procedure

Accessing the Sample Space

1. The fridge should be at room temperature, all mixer is collected, magnet controller is off.
2. Remove the blank over the OVC venting port at the top of the fridge (bottom for Elsa).
3. Slowly leak atmosphere into the OCV over the course of 2-5 min. Or the OVC is already 1Bar depending on warm up method.
4. Remove the outer 3 shields from the fridge using the Triton tools.
    - Minimize the Net Torque on the nuts when unscrewing them to prevent damage to the supports that hold the fridge up
    - The shields should be lowered onto the labjack which is situated <1" **BELOW** the bottom of the shield. The lab jack should never push up on the fridge support
    - Slowly lower the labjack once the shield is unscrewed to prevent hitting the side of the shields that are still attached to the fridge
    - Elsa fridge has special screws to hang the shield after the regular screws are removed. This feature DOES NOT apply to the magnet flange. Use the lab jack to hold the magnet while unscrewing. The screws used to hang the shield need to be inspected each time to make sure they are not too loose or too short.

For Magneto magnet detachment:

- Cut the white wire ties using the Triton wire tie cutters.
- Position the magnet Support stand under the magnet <.5" from the labjack.
- Remove the nuts, and lower the magnet to the magnet support rods.
- Gently unscrew the 3 inner shields from the fridge.
- Load/Remove the sample using the 0-80 nut driver.

For Elsa:

- Gently detach the D-connector from the magnet
- Use a hex screw driver to unscrew the magnet leads
- When the leads are disconnected, put back the screws/nuts/washers to the magnet.
- Unscrew the butterfly leads holder to detach it, such that the magnet can be removed easily without scratching the leads.
- Use the lab jack to hold the magnet.
- Remove the screws holding the magnet
- Detach the magnet.

Buttoming up the Fridge

1. Load your sample and remove the grounding cap.
2. Gently screw on the first Cu shield watching out for Cu on Cu gumming up.
3. Test the continuity of the devices.
4. Gently screw on the other 2 Cu shields.
5. Clean the thermal-n grease from the magnet flange, then apply a generous coat.
6. Lift the magnet up far enough so that nuts can be put on the screws.
    - No pressure should be applied to the fridge support by the labjack.

- Minimize the Net Torque on the nuts when unscrewing them to prevent damage to the supports that hold the fridge up.
- Watch out for the magnet current leads, they should not be bended too much or scratched by the shield.

7. Tighten the nuts down sequentially so that the magnet is slowly lifted to be in thermal contact with the flange.

For Magneto:

- Smoothly move the magnet leads to a place they can be tied down easily.
- Secure the magnet leads to the support bars well enough to eliminate movement.
- Tape the Magnet thermometer lead down, and check the magnet temp.
- If the magnet temp reading is bad, unplug and replug the D-sub 25 connector.

For Elsa:

- Connect the magnet current lead, using the Oxford provided torque wrench. See oxford manual picture if not sure the order for each part.
- Connect the d connector for the magnet.
- Put on the butterfly leads holder.
- Zip tie the leads or cables if needed.

8. Mount the 3 outer shields using the labjack.
    - The shields should be raised using the labjack until it is situated <1" **BELOW** the bottom of the flange. The lab jack should never push up on the fridge supports.
    - For the outer shield, clean the O-ring and slot with IPA, then apply a thin coat of vacuum grease to O-ring before mounting the shield.

Pulling Residual Mixture out the DU/PCC

1. Turn on the turbo pump cooling water a little less than 1/8 of a turn. The water level should just be visible in the drain below the valve.
2. Open V14 then V4, V5, V6 before turning on the compressor and the Floor pump.
3. Open V11 to pull any extra mixture from the cold traps and the DU into THE Tank.
4. Then Open V7, V8 to also pull mixture from the Precool Circuit into THE Tank.
5. When P3 reads 1e-1 mB, turn on the Turbo Pump. and let it run for >30 min.
    - Go to Lunch

6. When the P3 is down to 1.5e-4 mB, Close V7, V8 to isolate the PCC, then close V14
7. Then turn off the turbo pump and close V11.
8. Close V4, V5 , then turn the Compressor and the Floor Pump off.
9. Let the water run until the Turbo Pump has spun down all the way to 0 RPM.
10. If you are done, turn the Turbo Pump water off, otherwise keep up the good work!

Cleaning the Cold Trap (Method I)

1. Collect all mixture from the DU and the PCC, the cold trap needs to be liquid nitrogen cold at this step.
2. Put the Gas handling system in proper status (Close V14, V11 to prevent losing mixer).
3. Detach the cold trap push-on connector

4. Pull out the cold trap from dewar
5. Connect the cold trap to a dry rough pump or pump station, and start pumping
6. Use a heat gun to gently and uniformly warm up the cold trap while pumping. To avoid overheating, keep heat gun at distance or use cold air, make sure the cold trap temperature is only warm to touch or cooler.
7. When the cold trap warms up to room temperature, keep pumping until good vacuum is reached.
8. Detach the cold trap from the pump.
9. Put cold trap back into the nitrogen dewar, and connect the push-on connectors.
10. Make sure the push-on connector is well connected and no leak.

Cleaning the Cold Trap (Method II)

1. Collect all mixture from the DU and the PCC, the cold trap needs to be liquid nitrogen cold at this step.
2. Position the Red mobile pump next to the Magnet Power Supply.
3. Connect the Red mobile pump ball valve to the V10 vent port using the long thin oil free pumping line. Plug in the Red mobile pump and pump on the line in standby mode.
4. Once the Turbo has spun down to 0 RPM, pull the LN2 cold trap from the dewar, and secure it with the C clamps.

    - Be careful to not stress the quick connects at the top of the cold trap
    - The cold traps may need to be twisted to help it come out of the Dewar
    - Do not open the safety release valve at the top of the cold traps

5. Make sure V11 is closed, and then open V10. P3 should read around 2e-2 mbar.
6. Let the cold trap warm slowly on its own until the ice has melted away.
7. Once the ice is gone, use a heat gun to warm the stainless steel on all sides until it is warm to the touch.
    - It will take several cycles of warming and thermalizing to get the Cold trap warm, be patient. It will take around 1 hour. Do not heat it to over 100 C.
    - Stainless Steel is a bad thermal conductor, so heat it on all sides.

8. Once the LN2 cold trap is warm, wait until P3 read below 1e-1 mbar, then change the Red mobile pump out of standby mode and start pumping out the traps.
    - Pump on the trap for >1 hour

9. Close V10 and replace the blank before turning off the pump and letting it spin down.

Pumping Down and leak Checking the OVC

Caution: Dry pump station is preferred. If wet pump with turbo has to be used to pump the OVC, the operator has to keep an eye on the pumping. In the case of power failure, the pump oil can back flow into the OVC which is very bad.

1. Remove the labjack to the pump room for storage.

2. Position the Red mobile pump in front of the fridge so that the ball valve can be connected to the leak checker in the future.
3. Connect the large oil free pumping line between the Red mobile pump and the OVC venting port.
4. Open OVC venting port valve and turn on mobile pump
5. The pump station should reach max rpm within 30min otherwise there is a big leak.
6. After pumping overnight, the OVC pressure should be <1e-2 mbar.
7. Refill the LN2, which should take about 30min of transfer.
8. Connect the leak checker to the Red mobile pump, but keep the ball valve closed.
    a. Close the OVC vent port and the black screw valve on the Red mobile pump.
    b. Pump down the leak checker line, the He level should be ~10^-9 mbar*l/s. when the OVC valve opens it should read ~10^-8 mbar*l/s
9. Leak check all newly made joints starting at the leak checker and ending with the o-ring on the lower vacuum shield on the fridge.
10. If cooling down is next, then keep the leak checker attached to the Red mobile pump.

Cooling the fridge to base

1. Load your sample.
2. Clean the cold traps if needed. Cold trap need to be cleaned once every year.
3. Pump Down and Leak Check the OVC.
4. Turn on the He3 compressor cooling water to 7.5 L/min in the pump room, turn on the cooling water for turbo pump.
5. On the Triton Computer, go to to the Logging Menu, and start a new log file.
6. Open V14 and V11.
7. On the Triton Computer, go to Refrigerator menu, and start full cool down.
8. Watch for a spike on the leak checker that would signal a PCC leak to the OVC.
9. after 5 minutes without an obvious leak (>1e-7 Torr He level) remove the leak checker.
    a. Close the ball valve to the leak checker, open the screw valve to the Red mobile pump.
    b. Turn off the leak checker, close the ball valve and disconnect it from the Red mobile pump. Wait for the leak checker to spin down before moving it.
    c. Remove the vacuum line, and pump a blank down onto the intake port of the leak checker, and leave the leak checker under vacuum to keep it clean.
10. Watch the cooling process for another 30 minutes to make sure cooling water temperature is in the normal range.
11. Let the Red mobile pump run for the first 12 hours of the cooldown process.

- Go to lunch/dinner but not out for drinks in case of an emergency shutdown by the Red mobile pump due to power fluctuations from lighting (or other sources). If this happens, close the OVC as soon as you can (before it vents).

12. After 12 hours, close the OVC venting valve, and cover the port with a blank.

13. Turn off the Red mobile pump and let it spin down. Per usual, do not move it until the turbo is completely spun down to 0 Hz.
14. Wait for 40 hours from starting the cool down process and your fridge will be at 20 mKelvin.

- The fridge spits out an error “It took unexpectedly long to condense the mixture”. This is fine, and the message can be cleared without issue. It is most likely due to the large thermal mass of the sample stage we have.
- If have the function, the by-pass circuit can be opened such that the KNF compressor is bypassed, then the KNF compressor can be closed if the measurement does not generate too much heat. This function only works as long as the condenser pressure is below 600mBar, otherwise the KNF compressor should be used.

Using the Fridge at elevated temp<1K

1. Run the Fridge to Base temperature
2. Once the fridge has stabilized at base temp, turn off all of the thermometers except for the MX RuO thermometer
3. In the temperature control menu, chose the temperature (below 1K) that the fridge should be run at. Select a heater option (10mA is a good place to start) and click close loop.
4. Wait for the fridge to use the PID heater feedback loop to stabilize at your temperature.
5. When done, change the setpoint to 10mK, and close the loop to have the heater system turn off, and run back to base temp.

Using the Fridge at elevated temp <2K, >1K.

1. Run the fridge to 1K
2. Turn off the Turbo Pump
3. Now by adjusting the set point and the heater range run the fridge up to the desired temperature.
4. To run the fridge back to base, turn off the heater, and change the setpoint to 20mK or less. As the MX thermometer drops below 1K, turn the turbo pump back on.

Using the Fridge at Elevated temp >2K

1. Run the fridge to 2K
2. Close V9 and open V4 so that the Tank is on the other side of the compressor.
3. Disable PID loop, switch thermometer, set temperature, Enable PIC loop.
4. When done, run the fridge to 2K, and then change the valve configuration back so that V9 is open, and V4 is closed.
5. Once the fridge is below 2K, turn the heater off, and change the setpoint to base temperature. As the MX thermometer drops below 1K, turn on the turbo pump

Warm up the fridge

1. Make sure the temperature PID loop is off and turn on all the thermometers.
2. Make sure the magnet is zero energy and properly powered off.

3. If has the function, the by-pass circuit should be closed such that the KNF compressor is connected back to the fridge circuit.
4. click start a warm up, then wait for about 50 minutes for the mixtures circulates back into the mixture tank.
5. Close V14, and V11.
6. After the turbo Pump has spun down to 0 rpm turn off the Turbo Pump water in B09.
7. When the compressor temp cools below 30 C, turn off the Compressor water cooling in pump room.
8. When the coldest part of the fridge warms above 77K add exchange gas to the OVC.
9. Wait for 2 more days, or until the fridge is over 290K. Waiting this long will reduce/eliminate condensation collecting on parts that should not get wet.

Add exchange gas to assist warm up

For Fridge without big magnet:

- Add small amount of pure helium gas into the OVC as exchange gas.

For Fridge with big magnet:

- The fridge needs to be at least 77K on all thermometers.
- pure nitrogen gas maintained at 1Bar is used as exchange gas.
- To add nitrogen gas into the OVC and maintain the pressure to be 1 Bar. A transparent rubber tube is used to connect the OVC to a metal tube. The metal tube will be inserted into liquid nitrogen to generate nitrogen gas. The OVC valve is then opened barely and slowly such that only gas, not liquid nitrogen is put into the OVC. The process can take 30min, close the valve more if liquid nitrogen is seen or sounded in the rubber tube. No liquid nitrogen should pass the rubber tube, and the OVC relief valve should not ice up. This process must be monitored to avoid putting liquid nitrogen into the OVC or freezing the OVC valve. A heat gun can be used if the valve become cold.
- When the OVC pressure reach 1Bar (the OVC pressure gauge will read 210mBar for Elsa), the OVC valve can be fully opened. Keep the metal tube in liquid nitrogen during the warm up. This will make sure the pressure is always maintained at 1Bar, and avoid overpressure when system get warm.

Appendices

Abbreviations used in this document

- PCC - Pre Cool Circuit
- DU - Dilution Unit

To enter/exit standby mode on the Red mobile pump

1. Toggle left and right to move to the menu item called “standby”
2. Press both arrows to enter the menu that toggles between on and off “standby”
3. Toggle standby on or off using a single arrow
4. Press both arrows to set the new mode.
5. move to channel 309 to see the speed of the turbo.

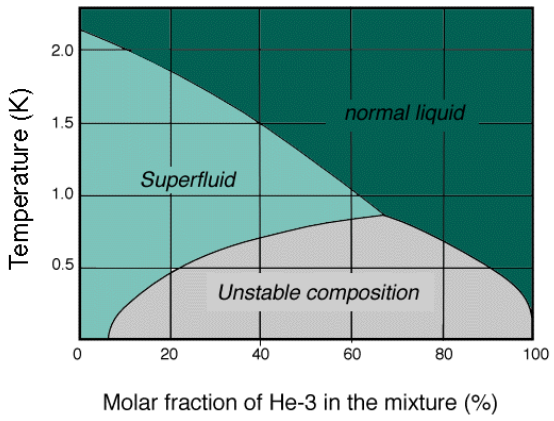
Changing Water Filters

1. Get the needed items from the pump room (white bucket, new filter)
2. Open the bypass valve to the pump.
3. Put the bucket under the filter, and release the pressure
4. Unscrew the clear outer casing that holds the filter in.
5. Carry the bucket/filter combo over to the sink and dump the water out.
6. Throw the old (brown, dirty, gunky) filter in the trash, and rinse out the clear casing.
7. Place the new filter in the clear casing, and put it back into the water filter base.
8. Tighten the clear casing with the appropriate wrench until it is moderately tight
9. Holding the pressure release button, slowly open the entry valve to the filter.
10. Once water comes out of the pressure release, let go, and open the exit valve
11. Close the bypass valve to force water to go through the water filter.
12. Put the bucket back in the Pump room.
    - The small filters can be purchased in the MRL store room
    - Large filters need to be purchased from McMaster Carr as
        - High Capacity Filter Cartridge, Polyester, 20” Height, 30 Micron

Using the Leak Checker

1. Plug in and turn on the leak checker. It will take about 2 min to run through its calibration process before it is ready to use.
2. Click the vent button (looks like filled in >< with an arrow pointing at it)
3. Remove the Blank, and connect the leak checker to the vacuum space to check.
4. Close the venting valve, and change it out of standby by pressing the button labeled with 2 arrows pointing around each other. It should pump down the vacuum space.
5. Spray He4 around the joints/vacuum space to check for leaks.
6. Put the leak checker in standby, and vent the chamber.
7. Remove the pumping line, and replace the blank over the port.
8. Close the venting port, and Pump the system down before turning it off.
9. Let the Leak checker spin down before moving it after use.

Notes on Dilution Refrigerators



http://cdms.berkeley.edu/UCB/75fridge/inxsrc/dilution/

Parts of the cryostat cool down to such cryogenic temperatures as 20 milliKelvin (0.02 K) using a technique called "3Helium - 4Helium Dilution." For this reason another name for the cryostat is "dilution refrigerator." This process relies on certain thermodynamic characteristics of 3He and 4He. The 3He-4He dilution has the following phase diagram:

- At temperatures below the triple point , the 3He-4He mixture will separate into two liquid phases, divided by a phase boundary.
    - One phase we'll call the 3He rich phase, because it contains mostly 3He. This corresponds to a point in the diagram below and to the right of the triple point, along the equilibrium line.
    - The second phase we'll call the 4He rich phase, because it is mostly 4He -- it will, however, always be composed of at least 6% 3He, no matter what temperature. This corresponds to a point in the diagram below and to the left of the triple point, along the equilibrium line.
- The two phases are maintained in liquid-vapor form. Since there is a boundary between both phases, extra energy is required for particles to go from one phase to another.
- A good example of this state would be what happens when you mix together oil and water. If you maintain the mixture at a high temperature they will stay mixed. But, if you were to lower the temperature (this effect can be seen at room temperature) the oil would separate from the water and float to the top, giving you two different phases in the liquid mixture. Not only that, but if you were to take a sample of the oil you would find a small amount of water present and vice-versa.

When you pump (we use a rotary pump) on the 4He rich phase you will remove mostly 3He (a move to the left off the equilibrium line in the diagram), destroying the equilibrium. To restore equilibrium, 3He will have to cross the phase boundary from the 3He rich side to the 4He rich side. However, it needs energy to get past the boundary. The 3He rich phase will provide the 3He and get the energy in the form of heat, from the walls of the mixing chamber; the walls are in thermal contact with whatever you're trying to cool down. Then the 3He will cross the phase boundary and join the 4He rich phase, restoring equilibrium. Finally, the atoms lost by the 3He rich phase are replenished by a constantly circulating flow of 3He.

Another way of thinking about this process is in terms of expansion. 4He is inert, in that it does not react with other molecules and thermodynamically can be thought of as a vacuum in some situations. Thus when the 3He moves from the 3He rich phase to the 4He rich phase, it expands into an almost vacuum. This expansion takes heat out of the walls of the mixing chamber, reducing the temperature of whatever you're trying to cool.