

## Quick Guide CRYOGENIC



Sample temperature range 1.6K-325K, field up to 9T, vertical orientation. The cooling circuit for sample (VTI) has a separate SW (VTI FC = variable temperature insert flow controller). For the measurement SW it is advised to use the v.190317.

**!!!** At all times make sure: 1) System temperatures refer to those in table , 2) The heater of He pot is always on (i.e. green

circle, see "measure.vi" window, "diagnostic" tab, top right scheme), 3) VTI FC is on (green circle, actual time) !!! Temperatures in "measure.vi" window, tab "diagnostic":

	1st stage	2nd stage	Magnet	Switch	He pot	Hall T	Charcoal
general	≈35-40K	≈3.5-4K	≈3.5-4K	≈10K	≈4K	≈40K	≈40K
max	<50K	<4.5K	<4.5K	<15K	<4.2K	<50K	<90K

The values may differ a bit during measurement, i.e. magnet temp. can go over 4.5K up to 5-5.5K at small fields but should then cool down back under 4K in a few minutes.

## Before venting airlock (i.e. sample load and removal)/opening chamber:

Check the **field is at 0T**, make sure the **sample chamber is at room temperature (>295K**, see "TC+VTI temp.control" window, i.e. for sample load check VTI temperature in right column for **sample removal check "Sensor\_B"** temperature in right column). Proceed to sample load/removal procedure, do not forget to check He pressure and open manual valve for He inlet. **Before starting measurement (after sample loaded):** 

Make sure **pressure in chamber is below 200mbar after gate is open** (external manometer) and that **manual valve is closed**. **Before leaving after finished measurement:** 

Check/update your entry in the logbook. To ensure higher cleanliness ideally leave the gate closed with low pressure in chamber (i.e. when removing sample first move sample to airlock, close gate and vent only airlock part). At this point the external manometer will show atmospheric pressure (it is measured at airlock part) but the lower pressure will remain below gate. Put the green cap (VSM)/plastic cap (DCR) on top instead of rod.

In case the system is not in this state / you are not able to achieve these values, please contact the guarantor.

## Vibrating sample magnetometer (VSM):

- <u>Sample preparation</u>: Preferably use straws (in plane/out of plane), use the probe (available in the sample preparation box) to fix the sample in the correct position. Secure the top plug + lower end with Kapton tape and fix to VSM rod. Hold the small end part of the rod when screwing the holder in (in order to not damage the connection to the motor).
- <u>Hardware mounting and SW setting</u>: Pick-up coils (6 screws), plate with metal bellows (4 screws + gasket with clamp), manual valve (i.e. set with manometer included, gasket with clamp), wiper seal, table platform (4 screws). Cable connections: "Coils" and "Thermometry" at coil part, later (after sample load and lowered rod) connect the "VSM head" on top of rod. In SW Main menu -> Select measurement -> lowT-VSM (wait several seconds to let the SW adjust).
- <u>Sample load</u>: Lower the transparent shield of the rod over the holder and insert the shield in airlock, lock the airlock (i.e. rotate the green part). Put white plate around rod and fix black belt above it. Do not lower the rod yet, make sure field is at OT. Follow the procedure in Main menu -> Sample load, be aware of following:
  - III If the mounting was changed from DCR to VSM or if the gate was closed and you were not sure if there was low pressure in chamber then there are two possibilities to ensure the whole chamber part is clean:
    - a) Do a "fake sample load" with green top and opened gate, close the gate and then remove green top and load the sample on rod with closed gate (if holder falls it will stop at the gate).
    - b) Perform the load procedure with opened gate (if holder falls it will fall to bottom of the chamber).
  - Ill He pressure should be between 50-100mbar (manometer on wall), manual valve opened (see green circle on screw), T in sample chamber at RT (can check VTI at ≈300K, see "TC+VTI temp.control" window, right column).

Flushing is performed 3x (see pressure changing on external manometer). After flushing is finished, tighten the airlock, close the manual valve, open the gate and lower the probe (release the black belt and remove the white plate once the rod is sufficiently low). Once the rod is fully inserted, rotate it by 90° to fix it in the table platform. Connect the VSM head and check the pressure in chamber is below 200mbar after opened gate (external manometer). Further, check the indicator of vibrations is moving (on top of rod), if not, check in "DAQ control" window if the vibrator switch is on.

• <u>Sample height adjustment</u>: Set momentum on time in graph, set appropriate field and adjust the height of the sample by rotating the platform (240°≈1mm) to the maximum value of momentum (manual LargeCFMS\_VSMandAC, Fig.1-9).



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- <u>Measurement and sequences</u>: In Main menu -> Edit sequence create/load the measurement sequence. Parameters "set before measurement" can be defined saving data will start only after setting these parameters. The sequence runs all the columns in this order: Curve variable (ramp or step), Parameter1, Parameter2. Multiple frames can be in one sequence. Do not forget to adjust the data directory and file name for each frame. When measuring loops, zero must be between max and min field value, e.g. +1T -> OT -> -1T -> OT -> +1T. Save the sequence and close the "Edit sequence" window. Run the last opened sequence in Main menu -> Execute sequence.
- <u>Sample removal</u>: Make sure field is at OT, follow the procedure in Main menu -> Sample removal, be aware of following:
  - III First remove the VSM head connection (vibrations should stop when sample removal clicked, if not do so by switch in "DAQ control"). Rotate rod by 90° and move it up until the sample is seen in the transparent shield. Secure the black belt and close gate (vacuum stays below gate).
  - III He pressure should be between 50-100mbar (manometer on wall), manual valve opened (see green circle on screw), sample at room temperature (i.e. Sensor\_B. at ≈300K, see "TC+VTI temp.control" window, right column).
    This time only one He inlet is performed. Open the airlock (green part), take out the rod (hold the transparent shield).
    When taking sample holder off the rod be sure to hold the small end part of the rod.

## DC Resistance (DCR):

- <u>Sample preparation</u>: Fix sample to puck with double-sided Kapton tape, make connection (resistance, Hall effect) using wire-bonder (electro lab, note the connections made). Set the puck in sample holder: First take shield off the holder by screwing screws in, fix the puck (in-plane or out-of-plane orientation) and put the shield on by un-screwing screws.
- <u>Hardware mounting and SW setting</u>: DCR section (6 screws), manual valve (i.e. set with manometer included, gasket with clamp), wiper seal. Later, after the rod is lowered, connect the wires "DCR probe" and "Thermometry". In SW Main menu -> Select measurement -> lowT-DCR (wait several seconds to let the SW adjust).
- <u>Break-out box testing</u>: Before loading in chamber test the connections of sample. Insert the holder with sample in the break-out-box and make the connections: B1-B6 in-plane, B7-B12 out-of-plane; + red and green, black and white (Ch1 more sensitive); I outer connection (source K2400), V inner connection (NanoVolt meter). In "measure.vi" tab "measurement" set the mode "Source I Measure V" and set source I to a safe value for your sample. Switch on the current source in SW (blue circle), measured values V\_nv and R\_nv should show expected values. Switch off the current source in SW (blue circle). Disconnect the holder and fit it to the sample rod, secure shield by rotating it.
- <u>Sample load</u>: Insert the rod in airlock with brown gasket and fix it with metal clamp, fix lower black belt at the lowest position, field is at 0T. Follow the procedure in Main menu -> Sample load, be aware of following:
  - III If the mounting was changed from VSM to DCR or if the gate was closed and you were not sure if there was low pressure in chamber then perform the load procedure with opened gate
  - III He pressure should be between 50-100mbar (manometer on wall), manual valve opened (see green circle on screw), T in sample chamber at RT (can check VTI at ≈300K, see "TC+VTI temp.control" window, right column).

Flushing is performed 3x (see pressure changing on external manometer). After flushing is finished close the manual valve, open the gate and lower the probe until top black belt. Connect the "DCR probe" and "Thermometry", check the pressure in chamber is below 200mbar after opened gate (external manometer).

- <u>Measurement and sequences</u>: see comment for VSM. The current is defined in "Measurement configuration".
  - <u>Sample removal</u>: Make sure field is at OT, follow the procedure in Main menu -> Sample removal, be aware of following:
    III First remove the DCR probe and Thermometry connection, move rod up until you see a mark (approx. 107cm from top of the rod). Secure the lower black belt and close gate (vacuum stays below gate).
  - III He pressure should be between 50-100mbar (manometer on wall), manual valve opened (see green circle on screw), sample at room temperature (i.e. Sensor\_B. at ≈300K, see "TC+VTI temp.control" window, right column).
    This time only one He inlet is performed. Open the airlock (i.e. metal clamp), take out the rod. Remove the sample holder and then the sample.

## Safety and other important notes:

- Wiper seal: connect push in, remove push circle towards chamber, pull hose out
- When handling the VSM and DCR rod please have gloves on in order to keep the rod clean and greased. If you experience any problems (solved or unsolved) please contact the guarantor.