

Instructions for changing and spinning DNP samples (16MAY2016)

Rotor caps:

- Zirconia caps are very fragile. It is recommended to pre-spin the rotor before inserting into the DNP MAS probe
- For Vespel caps, after sealing the sapphire rotors, immerse the **whole** rotor into liquid nitrogen (LN2) for 30-60 seconds and then check if the rotor cap can be easily removed or comes off spontaneously. If so, use another tighter cap and repeat LN2 test.
- Spinning at higher frequency increases the probability of lose caps and rotor crash. We recommend spinning higher than 8kHz only when it is absolute necessary.

Microwave:

- refer to gyrotron operation procedure for controlling the microwave
- turning on microwave wave will raise the sample temperature ~10K
- microwave should be turned off when in idling or warming up but should be kept on during sample temperature equilibration.

Inserting samples:

NOTE: always use a 'warm' sample holder that is at room temperature (i.e., not cold) to put samples into the probe

- in the Topspin 'edte' panel, set the VT gas flow to 135 L/hr. VT heater must be **ON** for VT gas flow to operate.
- on LTMAS cabinet MAS II controller,
 - | - set bearing and drive pressures to 0 mB
 - | - hit 'Eject' button to start eject gas flow
- at bottom of LTMAS probe,
 - | - remove (cold) sample holder
 - | - put packed rotor into a 'warm' sample holder with drive cap pointing upwards, i.e., the drive cap should be facing you
 - | - attach the 'warm' sample holder with the rotor to the probe
 - | - ensure sample holder is securely attached to probe
- on LTMAS cabinet MAS II controller
 - | - hit 'Stop' button to stop eject gas flow
 - | - set bearing pressure to ~200 mB; bearing flow meter should show ~10 L/min
 - | - hit 'Insert' button to start insert gas flow
 - | - if rotor inserts properly, flow value on bearing flow meter should drop to ~4 L/min
 - | - hit 'Stop' button to stop insert gas flow

Starting sample spinning:

NOTE: spinning regulation in 'Auto' mode should be used sparingly and preferably avoided if not absolutely critical to the NMR experiment

NOTE: the 'Chamber Heaters' for the VT, bearing and drive should always be 'ON' when spinning samples at any temperature! do not switch the 'Chamber Heaters' 'OFF'.

NOTE: do not switch the 'Exchanger Valves' to 'BY-PASS' mode.

- if rotor has inserted properly, the rotor should be spinning slowly, ~20-50 Hz
- on LTMAS cabinet controller panel,
 - | - in 'Run' the VT, Bearing and Drive 'Pressure setpoint (bar)' should be **1.5, 2.5, and 2.0**
- on LTMAS cabinet MAS II controller,
 - | - set bearing pressure to 400 mB
 - | - gradually increase the drive pressure until the MAS frequency goes above 1000 Hz
 - | - gradually increase the bearing and drive pressures; maintain the bearing pressure approximately three times greater than the drive pressure until bearing pressure reaches ~2000 mB
 - | - keep gradually increasing the bearing and drive pressures until the desired MAS frequency is approximately reached
- in Topspin 'edte' panel,
 - | - turn the VT, bearing and drive heaters on
 - | - set the VT gas flow to 2000 L/hr
 - | - ensure that the maximum VT, bearing and drive heater powers are set to zero
 - | - wait 15-30 minutes for the VT, bearing, drive and exhaust temperatures to stabilize. when temperature has stabilized exhaust ~ 130 K
- on LTMAS cabinet MAS II controller,
 - | - adjust the drive pressure to obtain the desired MAS frequency

Stopping sample spinning:

NOTE: when decreasing bearing and drive pressures, **slowly tap the decrease buttons instead of holding down the buttons**

NOTE: DO NOT hit the 'Stop' button (on LTMAS cabinet MAS II controller) to stop the spinning

- on LTMAS cabinet MAS II controller,
 - | - If in 'Auto' mode, switch to 'Manual' mode
 - | - **gradually** decrease the drive pressure by tapping (NOT HOLDING) the pressure decreasing button until the drive pressure is off
 - | - **gradually** decrease the bearing pressure by tapping (NOT HOLDING) the pressure decreasing button until the bearing pressure is off

Ejecting samples:

- in the Topspin 'edte' panel, set the VT gas flow to 135 L/hr
- on LTMAS cabinet MAS II controller,
 - | - hit 'Eject' button to start eject gas flow
 - | - wait 3 seconds
 - | - hit 'Stop' button to stop eject gas flow
 - | - wait 3 seconds

- | - hit 'Eject' button to start eject gas flow
- at bottom of LTMAS probe,
 - | - remove sample holder and check that the rotor has ejected
 - | - if changing samples, attach a 'warm' sample holder with another sample to the probe
 - | - if NOT changing samples, attach an empty 'warm' sample holder to the probe
 - | - ensure sample holder is securely attached to probe
- if probe will be left idle, apply bearing and drive pressures of ~400 mB and VT flow of 535 L/h. leave 'Chamber Heaters' 'ON' and 'Exchanger Valves' in 'COOLING' mode.

Warming up cabinet and probe:

- shut off microwave
- close the liquid valve on the LN2 tank, apply bearing and drive pressures of ~200 mB and VT flow 400 L/hr leave 'Chamber Heaters' 'ON' and 'Exchanger Valves' of bearing, driving and VT all in 'COOLING' mode.

Changing liquid nitrogen tanks:

- close the liquid valve on the LN2 tank
- on the LTMAS cabinet fill controller, switch from 'AUTO' mode to 'CLOSE'
- disconnect the LN2 transfer line from the LN2 tank
- replace the 'empty' LN2 tank with a full LN2 tank
(differential pressure indicator: full 25~30, close to empty 5~10)
- connect the LN2 transfer line to the liquid valve on the LN2 tank
- open the liquid valve on the LN2 tank completely
- on the LTMAS cabinet fill controller, switch from 'CLOSE' mode to 'AUTO'