

Nanoprobe Removal / Installation Instructions for the Varian 500 & 501 (with thanks to Diana Hunt)

Starting Up:

- Log in and open the VNMR software
- type **probe?** (should be set to 'hx_pfg' of 'hcn_pfg', record this value you will need it later)
- type **probe='nano'** (loads the parameters for the nanoprobe)
- type **pfgon='nnn'** (turns off the pulse field gradients)
- type **su** (sends the preceding information to the acquisition computer)
- click **[acqi]** click **[lock]** and set both lock power and lock gain to zero (this must be done every time before a probe is removed from the magnet)
- set the pfg switch to standby inside the Inova console (lower left component in the left bay)

Swapping the Probes:

- unplug the VT control (D-sub 9); the VT and cooling air lines; the X-channel, the H-channel, and the lock channel cables
- note the alignment of the probe for later (there are two ways to put it in, one is 180° wrong. An easy way to remember is to use the VT gas inlet as a reference)
- while securely supporting the probe from the bottom, unscrew the two thumbscrews holding the probe in place and GENTLY lower it out of the magnet
- place the nanoprobe on its stand
- connect the two thin fibre optic cables (caution these are fragile) to the nanoprobe console (gray to gray; black to black)
- connect the body gas line to the nanoprobe console and to the nanoprobe (use metal clamp to secure it to the probe)
- turn on the power to the nanoprobe console (small aluminum box with wires leading to the nanoprobe console)
- clean the nanotube and place in the probe (note that the tube must be as clean as possible or you will have problems achieving the desired spin rate)
- *slowly* ramp up the air to spin the sample (knob on right-hand side of the nanoconsole) it should spin at 2500 Hz. Then *slowly* ramp the spin back to zero (you want to ensure your sample is going to spin before you put the probe in the magnet) The upper limit for the body pressure is 25psi. If you cannot reach the desired spin rate inspect your tube.
- slide the nanoprobe carefully into the magnet and tighten the thumbscrews
- attach the VT control; the cooling air line (not the black VT tube); the X-channel, the H-channel, and the lock channel cables
- again *slowly* ramp the spin up to 2500 Hz.

Taking Spectra:

- type **temp** and click **[Reset VT]** (this must be done each time the VT control cable is unplugged) Once complete close the 'temp' window.
- type **nanoshim** (analogous to bestshim for normal probe)
- setup for nucleus and solvent; type **su** (as usual)
- tune the probe for the nucleus you selected (as usual)
- turn on the lock power and lock gain and lock on solvent (as usual)
 - note the lock power will saturate at much lower values for the nanoprobe (a lock power of ~4 will be a good starting value)
- shim on the xy-shims (x1, y1, xz, yz, xy, x2y2) You may also shim on the z1 and z2 shims if you so desire.
- close acqi and type **ga** to acquire the spectrum; process as usual

Reinstalling the sw-probe:

- turn the lock power and lock gain back to zero
- slowly ramp down the spin rate to zero
- remove the cooling gas line, VT cable, H-band, X-band and lock cables

- unscrew the probe and carefully lower from magnet—place in stand
- unplug the body gas from the probe and unplug the fibre optic cables
- turn off the small box on top of the Inova console
- place nanoprobe in box
- replace the sw probe (be sure alignment is correct) and tighten the thumbscrews
- reattach the coolind and VT gas lines, the VT cable, the lock channel, the X-channel, and H-channel cables
- type **probe='hx_pfg'** (of whatever value you recorded in the 'starting up' section above) and then type **bestshim**
- type **temp** and click *[Reset VT]*
- set the pfg switch to on inside the Inova console
- type **pfgon='nny'**
- tune and shim on standard sample (ask DCIF staff for help with this)