

Probe Tuning on the UnityPlus-500 (Anubis: Room B-7 subbasement)

Tuning the probe on a NMR spectrometer is, in essence, similar to tuning on your radio. If you have tuned your radio to the right frequency for a particular radio station, you get a crisp clear signal with little static (noise). The same is true for tuning the probe. Tuning it properly will allow you to get the best signal possible given your sample concentration. The probe is calibrated from a tuned state; therefore, a 90° pulse will be 90° only when the probe is tuned. The probe's tuning will be affected by the solvent, salt concentration, NMR tube, and to a lesser extent your solute. Therefore, it is important to tune the probe to get optimum performance from the spectrometer. The main result of poor tuning is a significant and unnecessary loss of S/N.

Tuning the Probe:

PLEASE NOTE: WHENEVER YOU ARE HANDLING INSTRUMENT HARDWARE, YOU MUST BE VERY CAREFUL. IF YOU ARE UNSURE ABOUT WHAT YOU ARE DOING, PLEASE ASK. FIRST TIME PROBE TUNING MUST BE DONE IN THE PRESENCE OF NMR STAFF.

Tuning the ¹H channel:

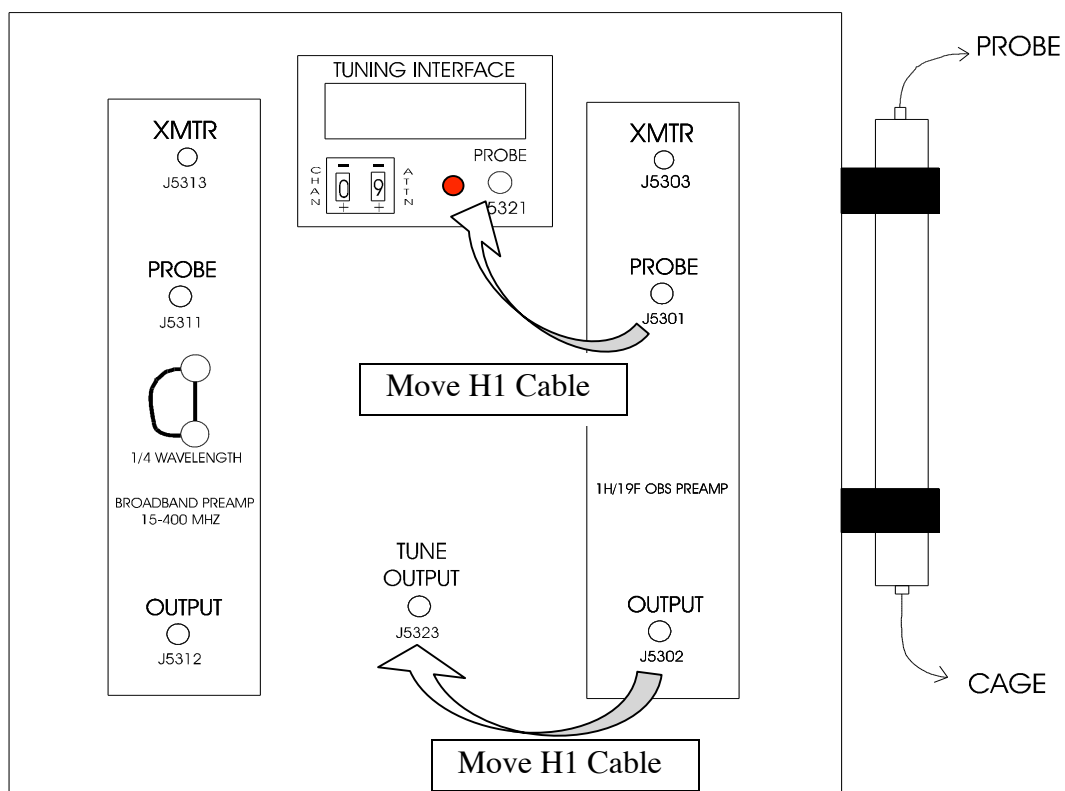
The TUNING INTERFACE is located on the PREAMP on the **left leg** of the magnet. A diagram of the TUNING INTERFACE and Probe body is on the following page.

1. Setup a standard Proton experiment (e.g. Click **Setup=>H1,CDCl3**). This sets up the instrument for tuning. Type *su* and hit **RETURN**.
2. Proceed to the Preamp to the immediate left of Magnet. Move the Cable attached to the PROBE (J5301) connector on the **1H/19F OBS PREAMP** box (see diagram on following page) to the PROBE (J5321) connector on the TUNING INTERFACE MODULE.
3. Move the cable attached to the OUTPUT (J5302) connector on the **1H/19F OBS PREAMP** box (right side) to TUNE OUTPUT (J5323).
4. Note the TUNING INTERFACE panel. CHAN should be set to 0. ATTEN should be at 9, which is the most sensitive setting. Use the + or - button to change these if necessary.
5. Change the CHAN channel from 0 to 1 by pressing the + key once. The TUNING INTERFACE readout should turn green and give a reading. If not, change CHAN to 0, close the lock/shimming window (**LC Close**), then change CHAN to 1. If the readout is green, but no numbers are shown, change the ATTEN to a lower number until numbers are seen.
6. You will try to minimize the value on the green screen. You should get a value <10 at an ATTEN value of 9.
7. Look for the ¹H tuning rods on the bottom of the probe. There is the shorter **RED** Tuning rod and a longer **BLUE** Match rod adjacent to it. Turn the short **RED** knob in the direction that gives the lowest value on

the green display. DO NOT EVER FORCE THE ROD. If you feel resistance when turning the rod, you probably have reached the end and should turn the rod in the opposite direction.

8. If you do not get <30 turning the **RED** rod, then:
 - i. Turn the longer **BLUE** rod to minimize the reading. Continue turning the **BLUE** rod in the same direction past the minimum to get a value 10-30 units higher than the minimum value.
 - ii. Turn the **RED** rod to minimize the reading. Continue turning the **RED** rod in the same direction past the minimum to get a value 10-30 units higher than the minimum value.
 - iii. Alternate turning between the **RED** and long **BLUE** rod using the procedure described in steps i. and ii. to get a minimum value.
 - iv. If this procedure results in a steady increase in the minimum value, change the direction you are turning the rods and continue to minimize.

Tuning Interface Module. Arrows indicate where cables are moved for ^1H Tuning.



9. When completed, change the CHAN channel back to 0 by pressing the - key once. The TUNING INTERFACE readout should blank, and the red light should start blinking.

10. Return the cables. Remove the free cable from the PROBE (J5321) connector and from the 'PROTON' connector on the probe. Reattach the proton cable to the 'PROTON' connector on the probe.
11. Move the cable connected to TUNE OUTPUT (J5323) connector on the TUNING INTERFACE MODULE to OUTPUT (J5302) on the 1H/19F OBS PREAMP box. When both cables are reconnected, the red light next to the PROBE connector on the TUNING INTERFACE MODULE should no longer be blinking.

Tuning the ^{13}C channel:

When performing ^{13}C NMR experiment or gHMQC, gHMBC, etc., you should tune both ^1H and ^{13}C channels.

1. Setup a standard carbon experiment (e.g. Click **Setup=>C13,CDCI3**). Type *su* and hit **RETURN**.
2. Proceed to the Preamp to the immediate left of Magnet. Move the Cable attached to the PROBE (J5311) connector on the **13C OBS PREAMP** box (see diagram on following page) to the PROBE (J5321) connector on the TUNING INTERFACE MODULE.
3. Move the cable attached to the OUTPUT (J5302) connector on the **BROADBAND PREAMP** box (left side) to TUNE OUTPUT (J5323). When both cables are reconnected properly.
4. Change the CHAN channel from 0 to **1** by pressing the + key. The TUNING INTERFACE readout should turn green and give a reading. You will try to minimize the value on the green screen.
5. Look for the **GOLD** C13 tuning rod on the bottom of the probe. Turn the rod in the direction that minimizes the TUNING readout value. The value expected should be <30. **DO NOT EVER FORCE THE ROD**. Adjust the longer match rod, if necessary.
6. If you do not get 10-30 turning the **GOLD** rod, then:
 - i. Turn the longer **BLACK** rod to minimize the reading. Continue turning the **BLACK** rod in the same direction past the minimum to get a value 10-30 units higher than the minimum value.
 - ii. Turn the **GOLD** rod to minimize the reading. Continue turning the **GOLD** rod in the same direction past the minimum to get a value 10-30 units higher than the minimum value.
 - iii. Alternate turning between the **GOLD** and long **BLACK** rod using the procedure described in steps i. and ii. to get a minimum value.
 - iv. If this procedure results in a steady increase in the minimum value, change the direction you are turning the rods and continue to minimize.

7. Change the CHAN channel back to 0 by pressing the - key. The TUNING INTERFACE readout should blank, and the red light should start blinking.
8. Return the cables. Move the cable from the TUNE OUTPUT (J5323) connector on the TUNING INTERFACE MODULE to OUTPUT (J5312) on the BROADBAND PREAMP box. Move the cable from the PROBE (J5321) on the TUNING INTERFACE MODULE to OUTPUT (J5312) on the BROADBAND PREAMP. When both cables are reconnected, the red light next to the PROBE connector on the TUNING INTERFACE MODULE should no longer be blinking.

Tuning Interface Module. Arrows indicate where cables are moved for ^{13}C Tuning.

