

Tuning and Matching

- (1) Load standard cross polarization sequence with ^1H and nuclei of interest enabled (eg. ^{13}C or ^{15}N)
- (2) Set acquisition time to $\sim 40\text{ms}$, contact time to $\sim 2\text{ms}$
- (3) Attenuate all power levels to $\sim 1\text{W}$ (30dBm, 20Vpp un-attenuated). At this power one will not cause damage to the high power amplifiers or probe.

NOTE: It may be convenient to set the power level associated with the ^1H $\pi/2$ pulse to 10W trigger oscilloscope.

NOTE: If power levels are high and probe is mistuned, this could cause damage to the probe and/or amplifiers.

- (4) Begin scanning.
Start tuning from highest frequency to lowest frequency (i.e. ^1H , ^{13}C , then ^{15}N)

- (5) Set the oscilloscope to trigger off of the ^1H forward voltage (V_f) from the directional coupler

NOTE: It is good practice to further attenuate forward voltage from the directional coupler by another 10dB – so as to not overload the oscilloscope.

NOTE: Never put the output of a high power amplifier directly into the oscilloscope. This WILL destroy the oscilloscope.

- (6) Set the oscilloscope time scale to 10ms/div and to trigger off of the ^1H V_f . Display reflected voltage (V_r) from the directional coupler on a 2nd channel.
- (7) To tune adjust tune and match knobs on the probe until reflected power has been minimized. Theoretical V_r is 0V – in practice this is possible for low power pulses.

NOTE: One effective strategy is to move the Tune knob in small increments, then scan the Match knob at each Tune increment to search for reflected power minimum.

- (8) Gradually increase power level of ^1H decoupling and ^{13}C and ^{15}N π -pulses by steps of 3 dB (doubling power) until desired power level is reached.
- (9) At each power level step check reflected power and tune to minimize reflected power. If you start to see flaring in the reflected power, you are near the power handling limit of the probe. If you feel you need more power, talk to a more experienced NMR spectroscopist.

NOTE: Steps larger than 3dB can be used, but if power level is increased too quickly while the probe is mistuned - this could cause damage to the probe and/or amplifiers.

(10) Check most recent notebook to determine power levels for starting powers to dial in for ^1H , ^{13}C and ^{15}N . Power levels are probe and nuclei specific.