Tuning the GN3ØØ-NB, 10 MM $^{31}$P,$^{15}$N Probe

The GN-3ØØ narrow bore NMR spectrometer is a multinuclear instrument for the frequencies ranging from $^{15}$N to $^{31}$P (3Ø to 121 MHz). Since probe tuning can dramatically affect sensitivity (signal to noise), it is desirable for the user to tune on each sample. Samples of greatly different dielectric constants (different solvents), or experiments that require large variations in temperature, may require that the probe be retuned. With the aid of this handout, users are able to routinely do a variety of nuclei by retuning the probe themselves.

Both RF (observe and decoupler radio frequency) channels have one match capacitor and one tune capacitor. The wands for tuning adjustments are color-coded as given in Table 1 below, and their positions in the probe baseplate are illustrated in Figure 1. The ends of the tune and match wands (DT, DM, OT, OM) that are inside the probe act as small screwdrivers fitting into slots in the tuning capacitors. One should make certain that the wands are actually engaged in the capacitors while making adjustments as they can easily be pulled out (or drop out on their own). DO NOT ATTEMPT TO FORCE THE TUNE AND MATCH WANDS BEYOND THE END OF THE CAPACITORS' TUNING RANGE.

<table>
<thead>
<tr>
<th>FUNCTION</th>
<th>COLOR</th>
<th>ACTION</th>
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<tr>
<td>tune</td>
<td>red</td>
<td>rotary</td>
</tr>
<tr>
<td>match</td>
<td>black</td>
<td>rotary</td>
</tr>
<tr>
<td>capacitor</td>
<td>red</td>
<td>insert and snap into place</td>
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The NMR lab has two "identical" probes for the GN3ØØ-NB in order to minimize down time while a probe is in need of repair. The tuning ranges for the capacitor wands which accompany the $^{15}$N to $^{31}$P probes are represented graphically on the "map" kept under the glass of the back console. By inserting the proper red-capped capacitor wand into the probe, the range of the observe channel can be changed to accommodate tuning and matching to specific nuclei. The observe channel tune and match wands are labeled OT and OM in Figure 1. There are some variations between the two probes in the frequency ranges of the capacitor wands. A "map" for the current probe is kept under the glass of the back console. For some nuclei, more than one capacitor wand may work, but the one which most closely centers the desired frequency in the capacitor's tuning range should be used. If a frequency is near the edge of a capacitor's tuning range, arcing in the probe may occur during the RF pulse. This could damage the probe and therefore should be avoided. If it is necessary to operate near the end of a range, extra care should be taken to ensure safe transmitter power levels; these levels can be started at 53 dB and cautiously increased until acceptable $90^\circ$ pulse lengths are obtained without arcing the probe. (Obtain further instruction from lab staff if you have this situation.) The transmitter power level and pulse length set by the menu are acceptable for most situations.

By tuning and matching the decoupler channel (which also observes proton), the instrument performance is optimized for observing proton or for decoupling proton from some other nucleus. These wands are labeled DT and DM in Figure 1.
To obtain a strong lock on the deuterated solvent, the lock channel must be tuned to the deuterium frequency. The lock tune wand may be used to de-tune the lock channel in order to not interfere with the observation of deuterium or oxygen, in which case shimming will have to be done on the FID.

Figure 1. 1Ø mm $^{31}$P-$^{15}$N Probe Baseplate ("top view") and Probe Interface Module

To tune the Broadband probe, the following procedure should be observed:

1. Select the desired nucleus from the menu, >ME, and then exit to command mode.

2. Confirm that the VT and BODY air lines to the probe are connected before inserting any sample. The air flow rates can be checked via the meters inside the back console and compared to the posted recommended values. Always ensure that the outside of each sample is absolutely clean and that the sample is sealed or capped to prevent possible contamination of the probe. Use the depth gauge to be certain the sample tube is positioned correctly in the spinner. Lower your sample into the probe and enter the correct frequency if a lock solvent is being used. Make certain that the sample spins (2Ø-3Ø rps for 5 mm tubes, 1Ø-15 rps for 1Ø mm tubes.)

3. In command mode, type >XP to enter the probe tuning display. (See Figure A.) The numbers at the bottom of the screen are the sweep/display range in megahertz, and are adjustable using the multifunction control knobs A and B, respectively. A cursor appears at the frequency equal to the spectrometer frequency of your nucleus. Verify that this is correct (i.e., $^{13}$C should be at 75 MHz). Open the window with knob B to about 15Ø MHz (for $^{13}$C) initially. The up and down keys can be used to change the vertical scale from 12 to 1Ø or 9.
4. Disconnect the observe cable (spring-loaded BNC connector) from the port labeled "LB probe" on the probe interface module (see Figure 1) and reconnect it to the port labeled "TUNE". (This should never be done if the decoupler is on or if the spectrometer is acquiring data). The cable connection is of the spring loaded push-and-turn type. This should result in a display similar to Figure A. If the probe is currently tuned to a frequency outside the range of the display, the dip will not be shown until the window is moved with knob A or widened with knob B.

5. If the dip (or well) does not appear, remove the current capacitor wand and insert the one appropriate for your nucleus into the hole labeled "EXT CAP". The map of the ranges of the current probe's tuning capacitors is under the glass of the back console. Because there are two probes that can be used on the NB, and because each probe has its own set of capacitor wands that differ in their numbering system, one could get them confused. Double check that you are using the correct capacitor wand for the current probe and nucleus.) This enables the probe to be tuned to the desired frequency. Notice that there is a connector on the end of this wand that must mate with the corresponding connector inside the probe. These connectors should snap together when the capacitor wand is inserted into the probe and a slight pressure and twisting motion is exerted to the end of it. However, when removing the wand, the pulling force may seem large due to difficulty in grasping the wand end. A piece of Tygon tubing over the red end may improve one's grip. It may help to hold the probe in place with one hand while pulling down on the capacitor wand with the other hand.

6. By adjusting the observe tune and match capacitors (labeled OT and OM in Figure 1) in the probe, move the dip to the cursor location with the red tune wand and make it as deep as possible with the black match wand, as illustrated in Figure B. Again, a piece of Tygon tubing over the end of the wand may be helpful, but remember that these wands are likes screwdrivers that easily come out of the slot that they are turning against. Readjust the display to expand the dip for fine tuning and matching before quitting. If the dip wobbles too much, the spinner air can be turned off with >AF in command mode (>AN to turn it back on). If one must choose between optimum tune (dip on the cursor) or optimum match (dip at its lowest position), the match is the more important. The match dip will not necessarily touch the horizontal line; what is important is for it to be at the "relatively" lowest position.

7. Disconnect the cable from the port labeled "TUNE" and reconnect it to the port labelled "LB port" on the probe interface module.

8. The decoupler must now be tuned to the proton frequency. Disconnect the decoupler cable from the port labeled "HB probe" and reconnect it to the port labeled "TUNE". Type <return> to leave >XP. Select proton from the menu, then exit to command mode.

9. Type >XP to enter the tune routine.

10. Now, use the decoupler tune and match (DT and DM) capacitor wands (red and black) to approximate the display illustrated in Figure B above, using a procedure similar to that described in Steps 3 and 6.

11. When completed, disconnect the decoupler from the port labeled "TUNE" and reconnect it to the port labeled "HB probe".

12. Unless deuterium or oxygen are being observed, the lock channel should also be tuned to deuterium. To observe these two nuclei, it probably should be deliberately de-tuned. The transcoupler dial should normally be left at a little past 5.

13. Upon completing the tuning procedure, ensure that the cables are connected as described below: (Refer to Figure 1)

   a. The Decoupler Channel from the probe is connected to the port labeled "HB probe" on the probe interface module.
b. The Observe Channel from the probe is connected to the port labeled "LB probe" on the probe interface module.

c. The Lock Channel from the probe is connected to the port labeled "LOCK" on the probe interface module.

d. Absolutely nothing should be connected to the "TUNE" port.

14. Type <return> to exit XP. Be sure to select the proper choice from the menu for the nucleus for which you want to acquire data.

15. Record in the log book which nucleus (or nuclei) you used. Always re-tune the probe, using the correct capacitor wand, to carbon-13 before leaving the spectrometer.

NOTE: One tunes a probe to a specific radio frequency in order to observe a particular nucleus. One shims a magnet in order to obtain a homogeneous magnetic field across the volume of the sample which is currently inserted into the probe. A change in sample thus requires both a probe tuning and a magnet shimming.