Running nuclei other than H1 and C13.

To learn more about the properties of NMR active nuclides check:

http://chem.ch.huji.ac.il/nmr/techniques/1d/multi.html

For reasons of efficiency of the power transfer, the spectrometer is duplicated into two frequency bands, the high band (from H1 at 500 MHz to F19 at 470 MHz) and the low band (from P31 at 221 MHz to N15 at 50 MHz). A third frequency channel, H2 at 76 MHz, is used for lock.

Impedance is in AC what resistance is in DC. For efficient power transfer between the probe and the console, their impedance has to be the same (50 ohm). Since impedance depends on frequency, after changing the spectrometer frequency from say 125 MHz (C13) to 186 MHz (Sn119), you have to tune the probe, which means using some variable capacitors in the probe to make the impedance of this 50 ohm under the new frequency.

Some of the coils in the probe can be tuned to two frequencies simultaneously. The 4nuc probe, the default on m4n, has the high band coil tuned to H1 and F19, and the low band coil tuned to C13 and P31. Do not attempt to tune this probe, tuning on two frequencies is more complicated, and besides the console on m4n doesn't make any other than these four frequencies. The ATB probe, the default on mbb, has the high band channel simultaneously tuned to H1 and F19. Leave it alone, as well. You could tune the low band channel, but this is more complicated than using the default probe on i2c, the sw probe, which is a conventional one, offering the best sensitivity for nuclei in the low band. For F19, you can get routine spectra on the m4n and mbb, without the need for tuning. For best sensitivity, use i3c with the pfg probe, but you'll have to have the probe changed and tuned. For routine P31 spectra use m4n. For best sensitivity use i2c as for any other nucleus in the low band. All of the instructions bellow are written for Sn119, on i2c with the sw probe. When running a nucleus for the first time, work on a concentrated sample of a compound containing the nucleus of interest, preferably the compound used as reference for the chemical shifts, or at least a compound for which you know the chemical shift.

1. Insert the sample, lock and shim.

2. Select an experiment, select the solvent, and input a comment.

3. From the Experiments drop-down menu select Other Nucleus – Sn119. If the nucleus you want is not on the list, select Sn119 and then follow the instructions in italics. In Acquire – Channels go to Observe and set the nucleus to the one of your interest, say Li6. Note the frequency next to it*, e.g*. 186 MHz for Sn119. In the Start tab, click on Setup hardware, to put this frequency on the hardware.

4. In the black suitcase of the sw2 probe, look for the cylinder containing the tuning sticks. On the side it has a small table with the frequency ranges of the sticks. Sn119 frequency, 186 MHz is in the range of stick 13. Insert it (carefully!) in the hole at the bottom of the probe and screw it in gently.

5. Replace the quarter wavelength cable on the low band preamp with the one appropriate for the frequency. The ranges are marked on the cables.

6. Disconnect the cable from the Probe on the low band preamp (J5311), remove the filters, and connect it on the Probe on the tune interface (J5321).

7. Tune with qtune. In vnmrj, from the Tools drop-down menu, go to Probe tuning – Manually tune probe. In the panel input the frequency, 186.3 MHz, the sweep width, 150 MHz, the power and gain 30,30. Click start tuning, then auto scale.

8. Locate the tuning rod at the bottom of the probe. It is blue for the low band channel. Make sure you don't touch the red tuning rod which is for the high band. Gently turn the body of the blue rod (tune) so that the dip in the wave on the vnmrj tuning screen moves from the current frequency, 125 MHz to the target, 186 MHz. If the rod seems stuck, gently turn it back a little and continue forward. When you've reached the target, turn the tip of the tuning rod (match) in order to make the dip as low and sharp as possible. The frequency will move from the target, so bring it back with the tune. Continue to alternate between tune and match until you reach the optimum. When done, stop and close qtune.

9. Disconnect from the Probe on the tune interface, insert the appropriate filter if any, and reconnect to the Probe on the low band preamp. Look in the slot with two digit numbers on the probe and write down the value. Next time when you run this nucleus, after #5 you'll turn the tune rod to this position and skip to tuning on the reflected power, #10.

10. Now you can further optimize tuning using the reflected power. Check in Acquisition – Channels that the observe frequency is what you want, then click Setup Hardware.

11. Move the Probe cable from the low band preamp to the tune interface like in #6. Press the CHANN button to change to channel 1, and adjust the ATTN to have the reflected power, which you read on the tune interface, in the scale.

12. Use the tune and match on the low band tuning rod (see #8) to minimize the reflected power.

13. When done, change CHANN to 0, and then reconnect the Probe cable to the preamp.

14. In the Acquire – Acquisition panel set the spectral window, check gain to auto, set a small no of transients, 4-16, and acquire. You should see a signal. Check the chemical shift, or set the reference.

15. Find pw90 as follows. Change to a spectral window of ca. 1000 Hz, with the signal in the center. From the Arrays tab set New array then type in pw, and set an array of 21 starting at 5 with step 5. Acquire. From arrayed spectra on the panel on the left, display spectra horizontally, then click display value. Note the value corresponding to the second null in intensity, and divide it by 4. That is pw90 for the current transmitter power. Pw90 should be in the range 10-20. You can increase the transmitter power for shorter pw90, but keep it bellow 61.

16. Set the acquisition time at to the double the time at which the FID has decayed into noise. Now you can optimize the relaxation delay d1, if you think your sample has a typical T1 for that nucleus. Set pw to 45°, then array d1. Looking at the array of spectra, keep the minimum d1 for which the signal reaches a plateau.

17. Set the spectra window to the chemical shifts range of that nucleus, and save the parameters.