Vanderbilt NMR Facilities
Tuning the Probe

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Tuning the Probe

Depending on the instrument and probe, different methods for tuning are used:

1. *Automatic tuning* (400, 402, 501, 603)
2. *Manual tuning* (all CSB instruments)

- The QNP probe in the 300 requires no user tuning, since it is a direct probe and the NMR staff makes sure that the probe is tuned properly.
- Tuning on the 601 requires some re-cabling and is therefore treated in a separate section.
- Tuning is always done from the channel with the lowest frequency nucleus to the highest one!
- Tuning of **all** the channels used in an NMR experiment is essential for a successful experiment.

1. Spectrometer Routing

This sets up the spectrometer connections for the individual nuclei used and connects them with the correct amplifier and preamplifier. Without correct routing, no spectra can be measured.

a. Read in proper parameter set (***rpar***)
b. Set up routing table **(only if *rpar* is not used, more challenging!)**
   - Click on ROUTE icon or type ***edasp***
   - This brings up the routing table that has to be correct for all channels.
   - To change any connections, select the proper nucleus in the pull down list of the appropriate channel and connect the *logical channel – FCU x – amplifier – preamplifier* by clicking on the proper gray box in the routing scheme.

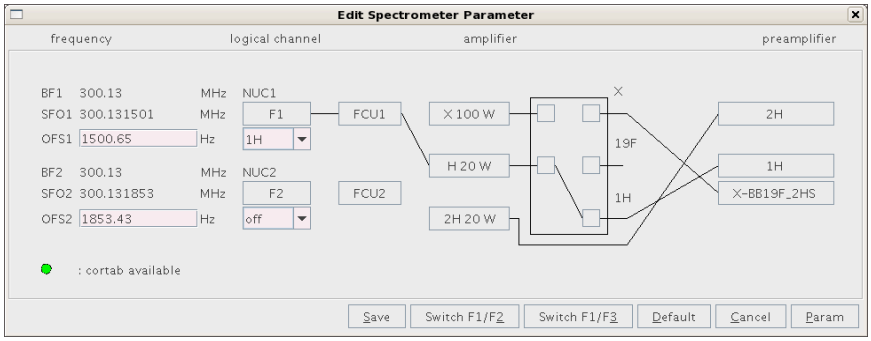


Figure 1a: Two channel instrument, routing set for ^{1}H observe. The “off” for NUC2 indicates that no decouple channel is being used.

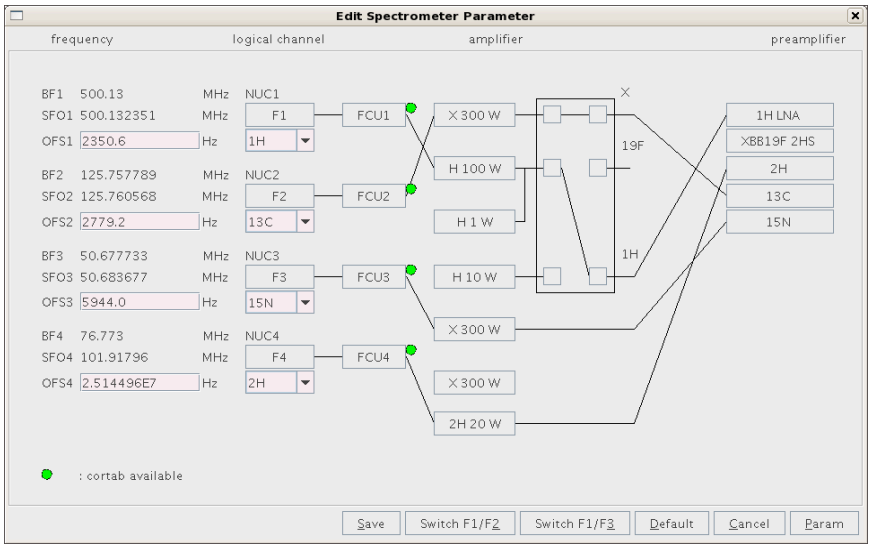


Figure 1b: Four channel instrument, routing set for ^{1}H observe, ^{13}C, ^{15}N and ^{2}H decoupling.

2. Automatic tuning

Requires special hardware on the probe (400, 402, 501, 603)

Once the routing is set correctly, type ***atma*** and all channels that are selected in the routing table will be tuned properly.

3. Manual tuning

(502, 601, 602 and 800)

a. Set the routing table as describe in 1.
b. The 3rd channel (typically ^{15}N) on the 601 requires some re-cabling, see section 4.
c. Start the tuning routine. There are several ways of doing this:
   - Click on TUNE icon or type ***wobb***
     → starts with the lowest frequency nucleus

- Type ***wobb fx*** or ***wx*** (x= channel, example *wobb f1* or *w1* for proton)
  → starts tuning on the specified channel

d. The following display will help to optimize the tuning of the current nucleus:

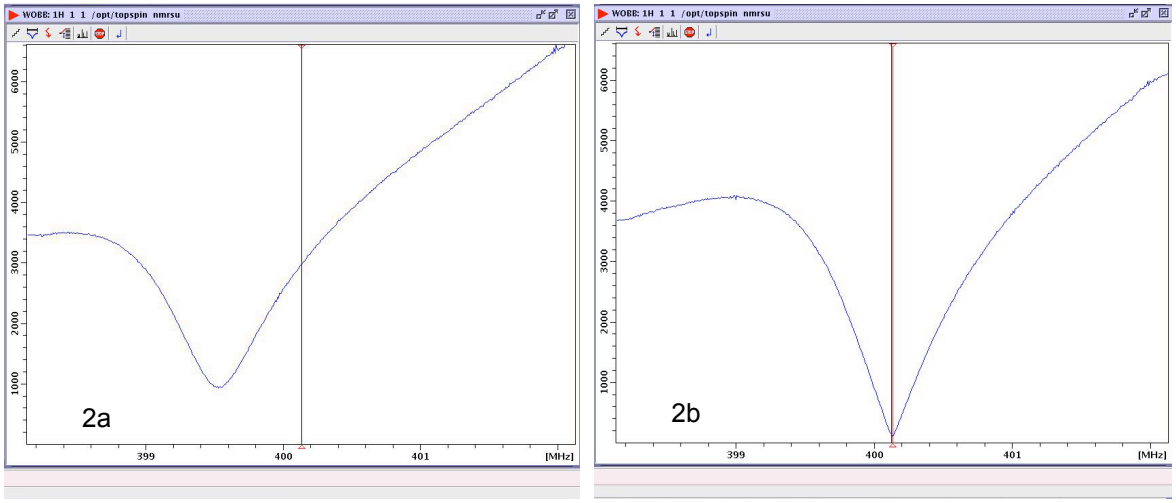


Figure 2: a) initial tuning, match and tune are not optimized, b) optimized tuning, tip of V-shaped wobble cure is on the x-axis and centered.

e. The HPPR cover module has LED's which can be used to optimize the tuning quality as well (see Fig.3)
- Horizontal LED's indicate tuning
- vertical LED's indicate matching
- arrows point direction of tune/match change
- best result is achieved, if there are only green LED's lit.

Figure 3

f. Tuning procedure:
- The V-shape curve on the monitor or the LED's on the HPPR are observed for this procedure. The goal is to have the tip of the V-shaped wobble cure is on the x-axis and centered.
- Turn on the colored rods at the bottom of the probe to move either tuning or matching.
  - M = matching
  - T = tuning
  - Color matches the color code of the nucleus on the RF –input.
- Adjust the matching first so the V is down to the x-axis
- Move the tip of the V to the center using the tuning rods

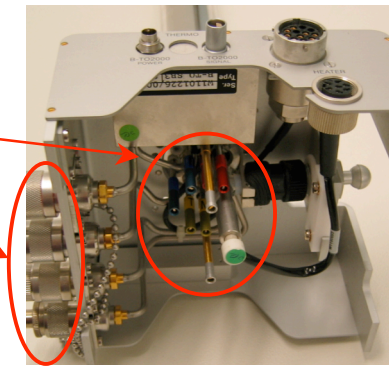


Figure 4

- If you do not see the V at the beginning, you might have to open the sweep width.
- Click on "*change wobble sweep width* " icon 
- Open to 20 MHz, "**OK**"
- Move tip of V into center using the tuning rod (CAREFUL, TO NOT APPLY FORCE ON ROD!)

- Reduce sweep width back to
  4 MHz for proton (normal setting)
  1 MHz for X-nuclei (normal setting)

➢ When the tuning is complete, type stop or click on the stop icon.

Caution

Do NOT exert excessive force when turning the rods. The tuning rods are **glued** to capacitors inside the probe and this connection can easily be broken off.

Be sure to **select the correct rod** for the nuclei to be tuned. They are color-coded.

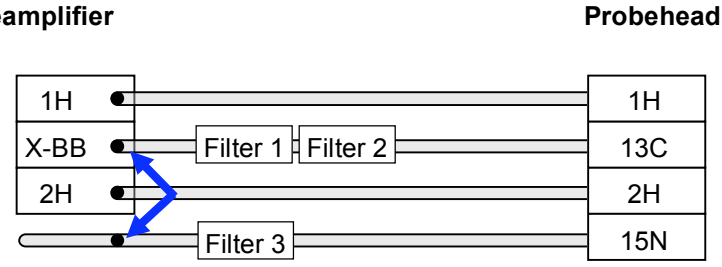
In particular on the 602, the proton rods not next to each other, but separated by a deuterium rod!!

4. Special Case: The 601 Spectrometer

The HPPR for the 601 is not equipped with a dedicated ^{15}N slice. Instead, the X-BB slice is shared for tuning both the ^{13}C and ^{15}N nuclei (or any other X-nuclei). The cabling on the HPPR configuration must be **temporarily** changed in order to tune the nitrogen channel.

a. Change cabling for ^{15}N tuning:

Schematic:



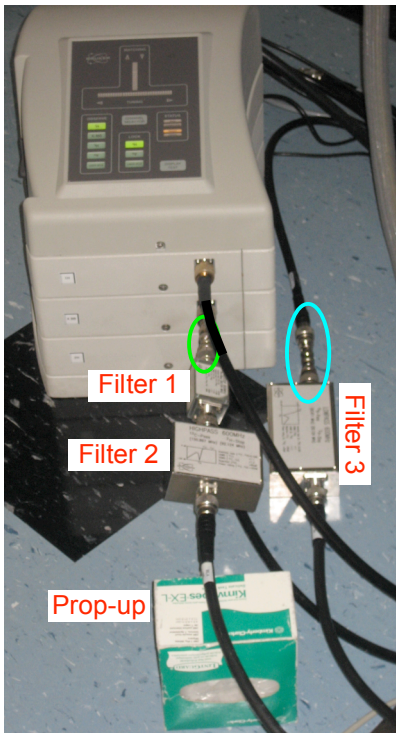


Figure 5: HPPR and filters in standard configuration

- Disconnect BNC on ^{15}N channel (indicated **blue** in Figure 5)
- Disconnect BNC on ^{13}C channel (indicated **green** in Figure 5) and leave it disconnected on the floor
- Connect ^{15}N channel to the HPPR in place of the ^{13}C channel (see Figure 6).

- MAKE SURE THE CABLE IS SUPPORTED BY THE PROP !

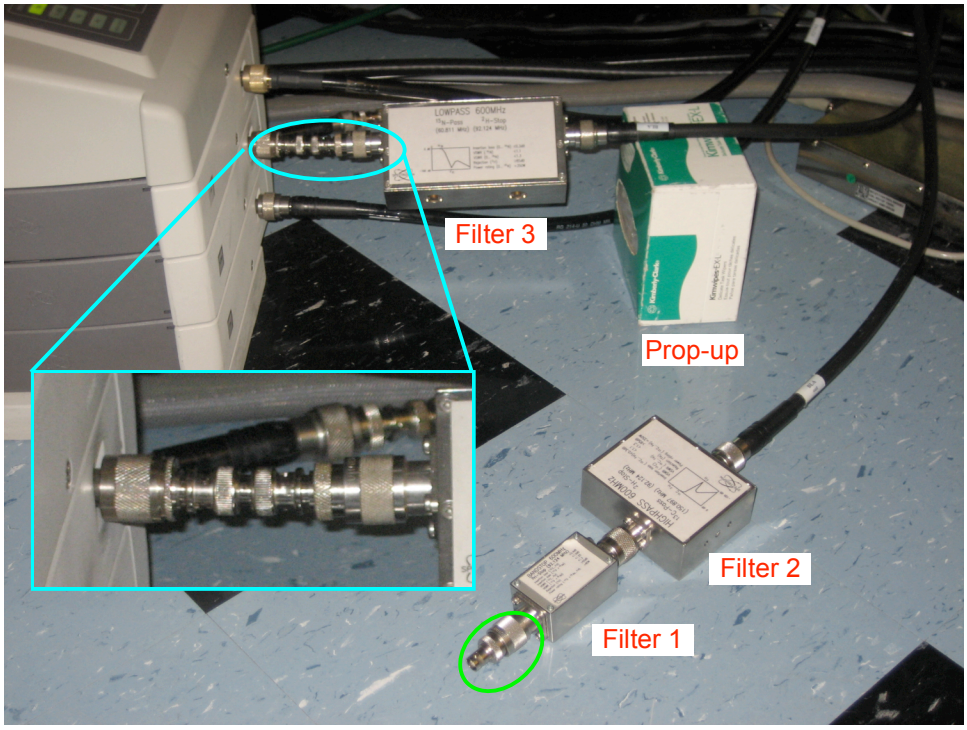


Figure 6: Cable configuration for tuning ^{15}N on 601 spectrometer with only one X-BB slice on HPPR

b. *Set-up routing:*

- Click on ROUTE icon or type ***edasp***
- Make sure the ^{15}N nucleus is selected in channel 2 as shown in Figure 7.

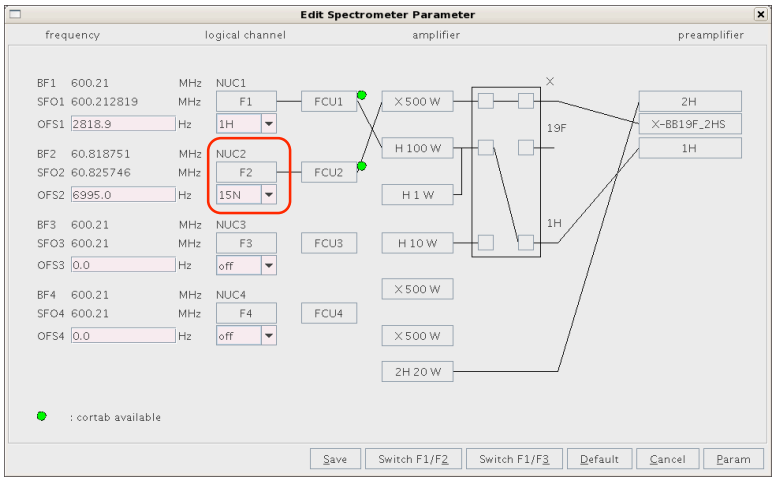


Figure 7: Routing for ^{15}N tuning on channel 2 as required on 601.

c. *Perform manual tuning on 15N channel as described in section 3 above*

When done, do not forget to stop the tuning run!

d. Return to standard configuration

- The filters have to be connected back to the original positions as shown in Figure 5.
  MAKE SURE THE CABLE FOR THE ^{13}C CHANNEL IS SUPPORTED BY THE PROP!

- The routing table has to be changed back as well, type ***edasp*** or use the ROUTE icon.
- Select ^{13}C for NUC2 and ^{15}N for NUC3, which should reconnect the routing to the standard configuration shown in Figure 8.

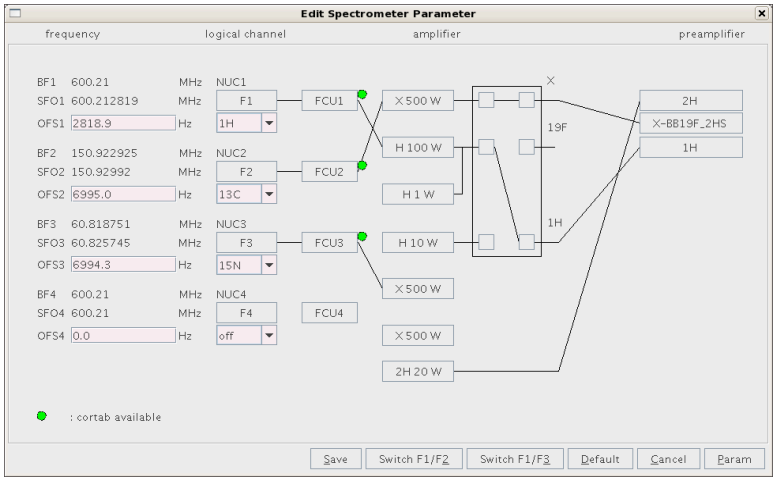


Figure 8: Standard routing for triple resonance experiments on 601.

The standard routing and connections (^{1}H in channel 1, ^{13}C in channel 2 and ^{15}N in channel 3) has to be in place when leaving the instrument