

## Instructions for Obtaining X-nucleus ( $^1\text{H}$ , $^{19}\text{F}$ , $^{13}\text{C}$ ) Spectra on the Bruker AM/AC Spectrometers

### Background Information:

Most NMR spectrometers are designed to allow observation of most, if not all, of the NMR-active nuclei. As far as the spectrometer user is concerned, the main operations needed in switching observation from one nucleus to another involve 1) changing the RF frequency at which the spectrometer is operating and 2) adjusting the circuit in the sample probe to operate correctly at this new frequency. The NMR sample probe uses a tuned tank circuit to both irradiate and receive signal from the sample. Tuning the resonant frequency of this tank circuit for the desired nucleus generally involves changing and/or adjusting some of the elements, usually capacitors, in the tank circuit. The specifics of the operation will vary according to the type and manufacturer of the sample probe.

The NMR spectrometer will be equipped with some method to facilitate tuning of the sample probe. On the Bruker AC/AM series, this method consists of a meter that reads reflected RF power (a "reflection bridge"), which is inserted into one of the slots in the preamp housing adjacent to the magnet, in place of one of the preamps normally inserted there. After the reflection bridge is installed, the probe-tuning procedure requires that the spectrometer start pulsing rapidly at the desired RF frequency. Then, the adjustable elements in the probe tank circuit, usually variable capacitors termed "tune" and "match," are adjusted interactively until a minimum reading is shown on the reflection bridge; i.e., the reflected RF power is at a minimum. After the minimum is found, the pulsing is halted and the correct preamp is inserted in place of the reflection bridge.

**NOTE:** These instructions assume the user is checked out for  $^1\text{H}$  and  $^{13}\text{C}$  operation on the Bruker spectrometers. In the instructions below, the character(s) on the left of the page are the commands to be typed or instructions to be followed. The text in parentheses describes the function of the command or provides relevant information about the command or operation.

Note: `\r` = return key (orange), `\_` = spacebar, `\^` = "CTRL" key (orange).

**Initial Probe Tuning Setup.** Note: probe tuning is done using the standard  $\text{CDCl}_3$  sample. This is fine for standard observation of the X-nucleus. If you are doing a complex experiment, e.g. INEPT or 2D, the probe tuning should be done with your sample in the magnet.

1. Depress LOCK button on SCM to turn off the lock.
2. Depress SPIN button on SCM to turn off sample rotation.
3. `3` (go to job 3 to read tuning file)
4. `RJ_TUNEPR.XXXX` (read acquisition parameters for probe tuning)  
where XXXX = P31, P195, N15, etc.
5. `II` (initialize interfaces)

6. PO (confirm decoupler is off)  
7. tune probe (see **probe tuning procedure** below)

### Running your experiment(s).

8. Remove standard sample and insert your sample. Spin, lock (if appropriate) and shim sample.  
9. 1 (go to job 1 for proton)  
10. obtain  $^1\text{H}$  spectrum in job 1 (I always recommend obtaining a  $^1\text{H}$  spectrum, if possible, before running an X-nucleus experiment)  
11. 2 (go to job 2 to do X-nucleus observation)  
12. RJ\_XXXXDUAL.CDCL  
where XXXX = P31, P195, N15, etc.  
13. PJ (read standard parameters for X-nucleus of interest. NOTE: if your solvent is not  $\text{CDCl}_3$  or  $\text{C}_6\text{D}_6$ , the chemical shift scale may be off by a few ppm. You may run unlocked using SWEEP OFF on SCM; set the FIELD control on SCM to the  $\text{CDCl}_3$  value)  
14. TI (read the title. This tells you any important information about the nucleus of interest (e.g., if decoupling should be used, etc.))  
15. II (initialize interfaces)  
16. NS XXX (set NS)  
17. BB (turn on proton decoupling - ONLY IF NEEDED)  
18. RGA (set receiver gain; wait for "AUTO RG FINISHED" message on LCD display before proceeding)  
19. ZG (start acquisition)  
20. PO (turn off decoupling when acquisition is done)

### Processing and plotting data.

21. AQ (check acquisition time)  
22. LB XX (set  $\text{XX} = 2 * (1/\text{AQ})$ )

Rest of processing and plotting is done as for  $^1\text{H}$  and  $^{13}\text{C}$  acquisition.

If you have other samples for the same nucleus you do not need to tune the probe for each sample.

### Retuning to $^{13}\text{C}$ and finishing up.

23. Remove your sample and insert the standard sample. Do not spin or lock now.  
24. 3 (go to job 3 to read tuning file)  
25. RJ\_TUNEPR.C13 (read tuning parameters for  $^{13}\text{C}$ )  
26. II (initialize interfaces)

27. PO (confirm decoupler is off)
28. Check that the probe cable is on the selective preamp connection on the preamp housing.
29. tune probe to  $^{13}\text{C}$  (see **probe tuning procedure** below)
30. 1 (go to job 1 for proton)
31. RJ\_H1DUAL.CDCL (read parameters for  $^1\text{H}$ )
32. II (initialize interfaces)
33. Spin, lock and shim the standard sample.

## Probe Tuning Procedure for X-nuclei (X = $^1\text{H}$ , $^{19}\text{F}$ )

1. If not done already, set up the spectrometer for tuning at the nucleus of interest (steps 3 - 6, above).
2. Determine the desired operating frequency. Type SF and the spectrometer will show the frequency currently loaded in MHz.
3. Change any removable capacitor(s) as necessary to observe the desired frequency. For our Nalorac probes, the capacitor stand has a table showing which capacitor(s) or other plug-ins are needed for any frequency.
4. Install the reflection bridge in the appropriate slot in the preamp housing. Set the sensitivity control to 1.
5. Start the spectrometer pulsing (ZG command).
6. Interactively adjust the tune (YELLOW/GOLD) and match (BLACK; these colors are for our Nalorac probes) capacitor screws until a minimum reading is obtained on the reflection bridge. The sensitivity control will need to be adjusted upward as the tuning improves, to ensure that a real minimum is found. Never force the capacitor screws! If you run out of adjustment on one screw, check the other screw, and also that any removable capacitors are inserted correctly.
7. After a minimum reading is obtained on the reflection bridge, stop the pulsing (^H). The desired minimum readings for the most common nuclei using the standard sample are listed on the capacitor stand table.
8. Remove the reflection bridge and replace with the appropriate preamp. Note that the probe cable connection on the preamp housing has to be changed from "selective" to "BB" when the broadband preamp is used.