

Bruker Avance NMR Spectrometers
UIC Chemistry / RRC-East NMR Lab

Training Guide for ^{19}F NMR Spectroscopy -
Bruker Avance Spectrometers with Bruker BBO Probes

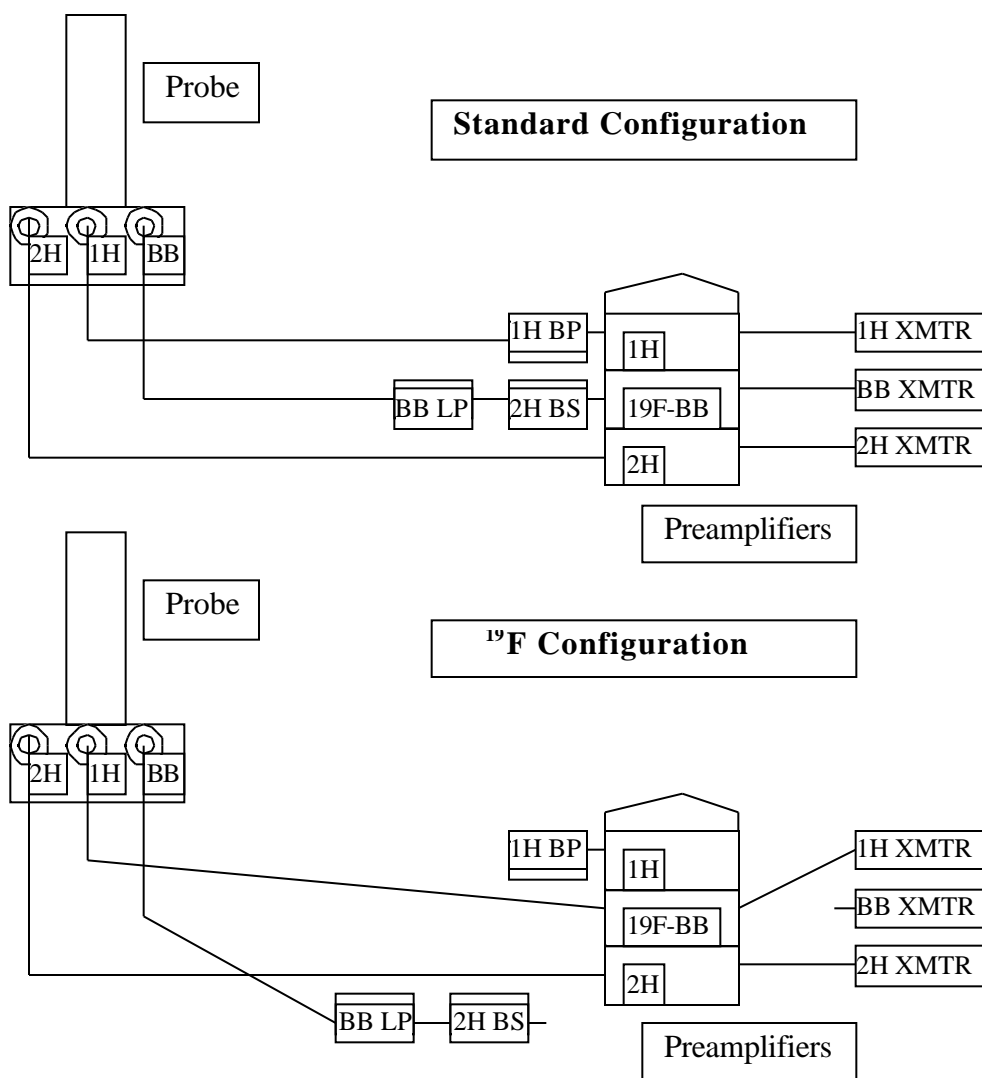
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^{19}F NMR Spectroscopy with Bruker BBO Probes - Overview

This note provides the information necessary to obtain ^{19}F spectra on routine samples using our new Bruker Avance NMR Spectrometers with Bruker 5mm BBO probes. This nucleus is not observable with the TBI or BBI probes.

The primary complication in observing ^{19}F is that, in addition to retuning the ^1H coil of the probe, the preamplifier unit must be recabled manually. This is due to the preamplifier configuration specified for our spectrometers. This note will attempt to cover the necessary recabling in a thorough and easy-to-follow fashion. The figures below show schematically the necessary recabling. This is discussed in detail on the next page.



**¹⁹F NMR Spectroscopy with Bruker BBO Probes -
Probe Tuning and Preamplifier Cabling Instructions
Bruker Avance NMR Spectrometers
UIC Chemistry / RRC-East NMR Lab**

These notes assume that the reader is checked out for 1D operation
on the Bruker Avance Spectrometers.

Prepare for ¹⁹F acquisition

1. Create a new data set. All files will be acquired using the SAME experiment NAME but DIFFERENT experiment NUMBERS. Confirm that the BBO probe is installed.
2. Create/read experiment (expno) 1. Put in your sample, lock, spin (if desired) and shim.
3. Obtain a proton spectrum in expno 1 using the standard proton parameters (e.g. h1.bbo).
4. Create/read expno 2. We will run the ¹⁹F experiment in this dataset. Type `rpar f19.bbo_all` to load the ¹⁹F parameters. Type `ii` to initialize the spectrometer interface.

Recable for ¹⁹F observation

5. Carry out the recabling as follows:
 - a) Disconnect the cable and filters that go between the ¹⁹F-BB preamplifier and the BB connection on the probe. Make the disconnection at the preamplifier connection of the ²H bandstop filter; i.e., remove the the filters (with the cable connected to them) from the ¹⁹F-BB preamplifier connection. Leave this cable/filter combination disconnected while doing ¹⁹F.
 - b) Disconnect the X transmitter cable running into the back of the ¹⁹F-BB preamplifier. Leave this cable disconnected while doing ¹⁹F.
 - c) Disconnect the cable that goes between the ¹H (upper) preamplifier and the ¹H connection on the probe. Disconnect the cable such that the ¹H bandpass filter is left connected to the preamplifier; i.e., disconnect the cable from the filter. Now connect this cable to the previously vacated connection on the front of the ¹⁹F-BB preamplifier. This connection should have no filters present.
 - d) Disconnect the ¹H transmitter cable running into the back of the ¹H preamplifier and connect it to the previously vacated connection on the back of the ¹⁹F-BB preamplifier.

Tune the probe for ^{19}F observation

6. You will be observing ^{19}F using the ^1H coil of the BBO probe. Tune the ^1H coil for ^{19}F observation using the same basic procedure described for ^1H tuning in the Avance-1D-Training Guide (these are repeated, below). However, in order to tune down from ^1H to ^{19}F you will need to turn the "tune" screw a few turns clockwise to get close to the ^{19}F resonance. Be sure to be watching the computer monitor while doing this so that you will see the dip when it appears on the screen and then gets close to the correct frequency. NOTE: when you start tuning for ^{19}F there will be no dip on the screen. This is because the probe is tuned to a frequency far away from the desired frequency. As you turn the "tune" screw clockwise you will at some point see the dip appear on the screen. You can then finish the tuning in the normal fashion.

^{19}F observation - comments

7. The ^{19}F parameters have already been loaded. ^{19}F observation is similar to ^1H except that the sweep widths are much wider since ^{19}F has a bigger chemical shift range. Some ^{19}F spectra will show a bit of roll in the baseline. This is normal and can be dealt with during processing if need be. ^{19}F sensitivity is comparable to ^1H sensitivity.

Finishing up

8. After finishing ^{19}F observation, the spectrometer MUST be recabled back to the normal configuration and the probe MUST be retuned to ^1H .
9. Recable back to ^1H by reversing the instructions given in step 5, above.
10. Retune back to ^1H by reading a ^1H dataset or loading ^1H parameters and typing `ii` . Then retune in the normal fashion, remembering that the "tune" screw will need to be turned counterclockwise to bring the dip back onto the screen for ^1H tuning.

¹H Probe Tuning Guidelines - BBO Probes
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Spectrometer setup - ¹H tuning

1. Run a quick proton experiment to determine that the sample is correct.
2. Type `acqu` to go to the acquisition window, then type `wobb` to start the probe tuning ("wobble") routine.

Tuning the ¹H coil

3. The computer screen will show a trace with a dip in it; hopefully the dip will be close to the correct frequency in the center of the screen. If there is no dip, click the `WOBB-SW` button to the left of the screen, then enter a new value in the correct field of the screen that pops up. The default `SW` is 4 MHz.; try 12 or 16 MHz. if it needs changing.
4. When you have confirmed that there is a dip present, go to the magnet and adjust the tune and match screws under the probe to bring the minimum of the dip to the center of the screen and also as close to the bottom of the display as possible. NOTE: the location of the dip left to right is referred to as the "tune" and the depth of the dip (closeness to the baseline) is referred to as the "match." The tune screw will tend to move the dip left - right and the match screw will improve the depth of the dip. The display on the preamp housing also shows the quality of the probe tuning and can be used in conjunction with or instead of the computer screen display. NOTE: on the TBI probe on the DRX-500 the proton coil tune and match is done using the small screwdriver-like rods and the special tool hanging off the probe.
5. When the tuning is OK, type `halt` to stop the wobble routine.
6. Re-acquire the proton spectrum if desired.