Probe Manual Addendum – H&F Tuning on NEO NMR System

PhoenixNMR probe head series PH1, PHW and PP1

PhoenixNMR

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Many of the operations described herein should be completed only by a skilled individual as damage to the equipment may occur if an inexperienced individual is left to explore without guidance. This manual provides general user specific information for the PhoenixNMR probe products which begin with a probe base part number of PB1, PP1, PBW, or a probe head part number of PH1 or PHW. The information contained herein is meant as a guide and knowledge of Solid State NMR general practices and procedures are assumed.

1 Terminology and symbols used within this manual

The following symbols will be used within this manual to bring your attention to instructions, warnings and procedures which pose a risk to the user, the equipment being described or to other equipment, or to the validity of the NMR data collected. Whenever you see one of these symbols to the left of a section, figure, table or instruction extra care should be taken to fully understand the material being presented prior to proceeding any further.

This symbol will be used when the user could be placed at risk of injury if the instructions given are not followed.

This symbol indicates that damage to the probe or other system components could occur if the instructions given are not followed.

This symbol will be used if there is a risk of explosion or explosive discharge.

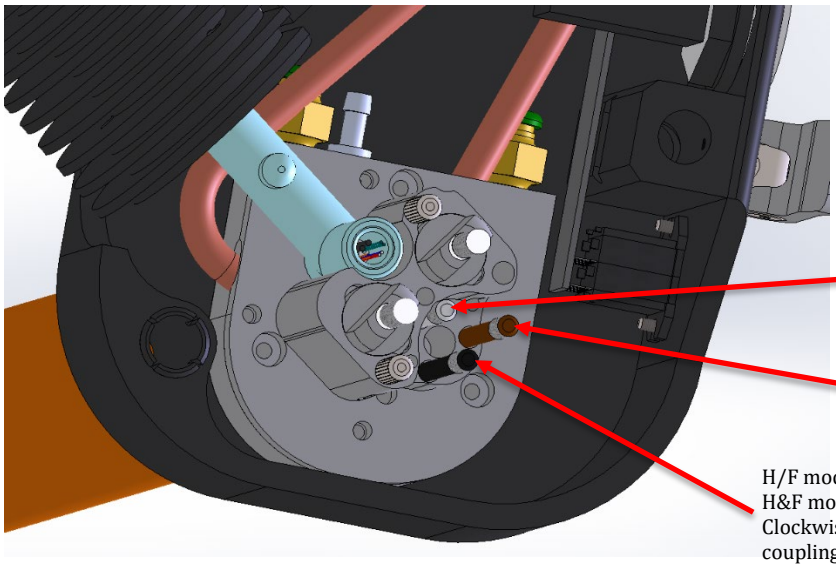
This symbol will be used when a task or process is being described that requires validation by the user.

2 ^{1}H or ^{19}F only tuning mode

The H&F assembly within the PhoenixNMR probe allows the probe to be tuned in two distinct modes, the first is ^{1}H or ^{19}F mode where the full efficiency of an HXY, FXY, HX, or FX probe is retained. In this mode, only a single resonance will be visible on the ^{1}H port of the probe. The second mode provides simultaneous tuning to both ^{1}H and ^{19}F, allowing the observation of any channel (H,F,X,or Y) while decoupling any or all of the other three channels, for example in HFCN mode, one could observe H while decoupling F,C, and N, or observe C while decoupling H,F, and N.

The instructions in Section 3 will place the probe into ^{1}H or ^{19}F only mode ready to carry out HX, FX, HXY or FXY experiments. Section 4 provides instructions to place the probe into HFXY or HFX mode ready to carry out HFXY or HFX experiments.

✓ Note: The diplexer must be removed or bypassed during tuning operation as it will provide a visual appearance to the sweep that will be confusing and/or misleading. This is normal and a result of the way the diplexer operates.



Note: The colors of the Tune/Match/Split knobs may not match this figure, the position of the knobs are what determine their function.

3 ^{1}H or ^{19}F only tuning mode

✓ Note: The diplexer must be removed or bypassed during tuning operation as it will provide a visual appearance to the sweep that will be confusing and/or misleading. This is normal and a result of the way the diplexer operates.

The following instructions assume that the probe was previously in H&F tuning mode, however if the probe is in an unknown state carrying out this procedure will return it to single resonance H/F mode.

1. Adjust the H/F mode Match knob, see figure above, fully clockwise, then return it 2 complete turns counterclockwise.
2. Adjust the H&F mode Match knob, see figure above, fully counterclockwise until the stop is reached.
3. Adjust the Tune knob fully clockwise, this will set the tuning resonance to its maximum value, approximately 10% above the ^{1}H resonance frequency.
4. While observing Wobb (on Bruker system), or TRTune (on Agilent/Varian system) or any other equipment capable of providing a visual indication of tune and match, such as a network analyzer or sweep/tune box. Adjust the tune knob counterclockwise until the resonance approaches the ^{1}H (or ^{19}F if preferred) frequency, then use the H/F mode Match knob as well to match.

4 ^{1}H and ^{19}F simultaneous tuning mode

Warning: The maximum decoupling field strengths for ^{1}H and ^{19}F are reduced in simultaneous ^{1}H & ^{19}F operation. Please refer to the specification sheet provided with the probe to prevent damage from occurring to your probe. Operation of ^{1}H and ^{19}F simultaneously at full specification from ^{1}H or ^{19}F only RF field strengths will result in damage to the probe!

Note: The diplexer must be removed or bypassed during tuning operation as it will provide a visual appearance to the sweep that will be confusing and/or misleading. This is normal and a result of the way the diplexer operates.

The following assumes you are starting from a ^{1}H only or ^{19}F only mode, see Section 3, if this is not the case please begin by tuning the probe as described in Section 3 to the ^{1}H frequency.

1. Using the actual spectrometer frequencies for ^{1}H and ^{19}F (in MHz) for Fh and Ff respectively calculate the following values –
    a. Sweep Width, S = (10/8) * (Fh – Ff)
    b. Median Frequency, M = (Fh+Ff)/2

2. Set the center of the WOBB sweep to the M value using SF02, and the sweep width to the S value, this will place the ^{19}F resonance on the 1st vertical marker of a 10-division sweep display and the ^{1}H resonance at the 9th vertical marker. For example; a 400MHz system with Fh = 399.495MHz, Ff = 375.882MHz, results in S = 29.52MHz and M = 387.69MHz.
3. Adjust the Tune knob to bring the probe resonance to the M frequency, in the 400MHz example above this is 387.69 MHz.

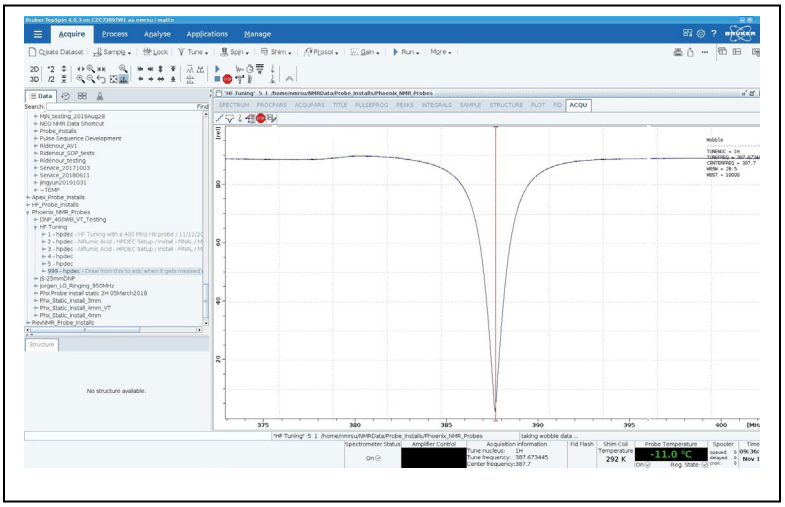


Figure: 1H channel tuned to the mean frequency, 387.69MHz.

4. Adjust the H&F mode Match knob clockwise until you see the central resonance move up towards the actual 1H resonance frequency and a second resonance will come in from the left (assuming the sweep window has lowest frequency on the left). Keep adjusting the H&F mode Match knob until the two resonances are equivalent in depth and ~ symmetric about the M frequency. The Two resonances should reside slightly above ^{19}F and slightly below ^{1}H respectively.

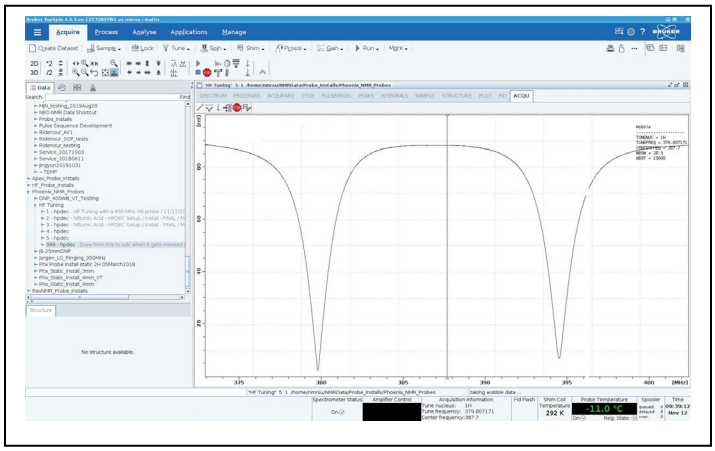


Figure: Probe in H & F mode

5. Adjust the H&F mode Split knob (this is the H/F mode Match knob) until the two resonances separation is ~= S.

6. Iterate between the H&F Match, Split and the Tune knobs until both resonances occur at 1H and 19F respectively and their depths appear equivalent.

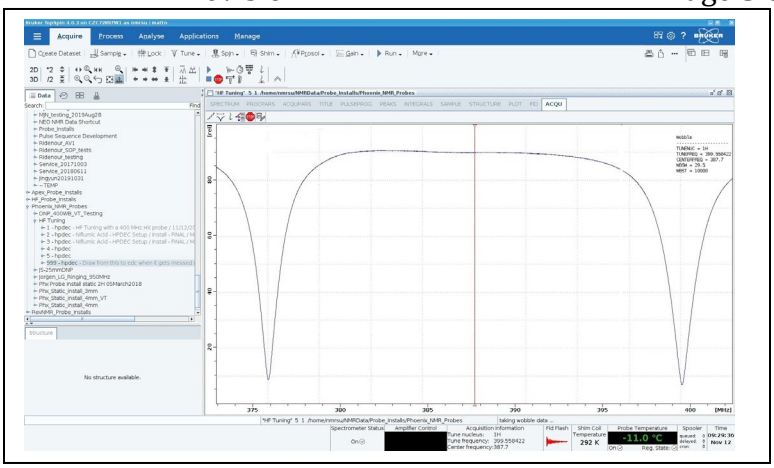


Figure: H&F frequencies tuned simultaneously.

7. Tuning is complete, reconnect the diplexer, set SF02 back to the 1H frequency (399.495MHz) and proceed with the NMR setup.