

PROBE TUNING ON THE INOVA 400

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1. INTRODUCTION

1.1. About these Notes and Related Notes

These notes describe how to tune the NMR probe on the Inova 400 in order to perform NMR experiments on nuclei other than ^1H , ^{13}C , ^{19}F or ^{31}P . All NMR active nuclei besides those four nuclei are referred to as “odd nuclei”. Common odd nuclei studied in the NMR facility include, but are not limited to, ^2H , ^{11}B , ^{29}Si , and ^{77}Se , and odd nuclei NMR experiments can be performed on either of the Inova spectrometers. Once you have chosen your spectrometer, you should consult additional notes as described in Table 1.1.

Table 1.1. Required and Recommended Notes

If you would like to...	Then you ...	
	...must consult these notes	...should consult these notes
Perform a 1D odd nucleus NMR experiment on the Inova 400 or 600	<ul style="list-style-type: none"> • NMR Sample Preparation • Reserving NMR Time using FACES • Basic Operation of the Inova 400 and 600 • Retrieving NMR Data 	<ul style="list-style-type: none"> • Processing 1D NMR Spectra using VNMRJ
Perform a 2D NMR experiment involving odd nuclei on the Inova 400 or 600	<ul style="list-style-type: none"> • NMR Sample Preparation • Reserving NMR Time using FACES • Basic Operation of the Inova 400 and 600 • Retrieving NMR Data 	<ul style="list-style-type: none"> • Processing 2D NMR Spectra using VNMRJ

2. ABOUT PROBE TUNING ON THE INOVA 400

2.1. What is Probe Tuning?

Each NMR active nuclear isotope resonates at a specific frequency. In order to obtain an NMR spectrum, radio-frequency (rf) pulses are applied to the sample at the appropriate frequency for that nucleus (eg. applied at 399.762 MHz for ^1H on the Inova 400). The correct frequencies of the pulses are generated by the frequency synthesizer of the spectrometer, the magnitude of which is increased by the spectrometer’s amplifiers. The amplified pulses are applied to the probe and then to the sample via the probe’s rf-coil. In order for the pulse to get to the sample, the frequency of the probe’s circuit must be “tuned” to match the frequency of the applied pulse from the spectrometer. If the probe is “out of tune”, the pulse is rejected by the probe and is not applied to the sample. In order to tune the probe to the correct frequency, we insert a specific capacitor stick (see section 2.2) and adjust the tune and match capacitors (see section 2.3) appropriately.

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2.2. Capacitor Sticks

A capacitor is an electrical device that stores electrical charge. In NMR, capacitors are used to change the frequency of the probe's circuit; increasing the capacitance within the probe reduces the frequency of the circuit. In order to tune a specific frequency, the appropriate capacitor must be inserted into the probe. Agilent achieves this by using different capacitor sticks, which are literally sticks with capacitors stuck onto the top of a stick.

On the Inova 400, the capacitor sticks have 2 prongs that must be aligned in a specific orientation in order to be properly inserted into the probe. Once properly aligned, the stick is screwed into the probe and secured in place. There are 4 unique sticks, labeled 1/4-wave, G0PF, G18PF, and G36PF (see Figure 2.1).

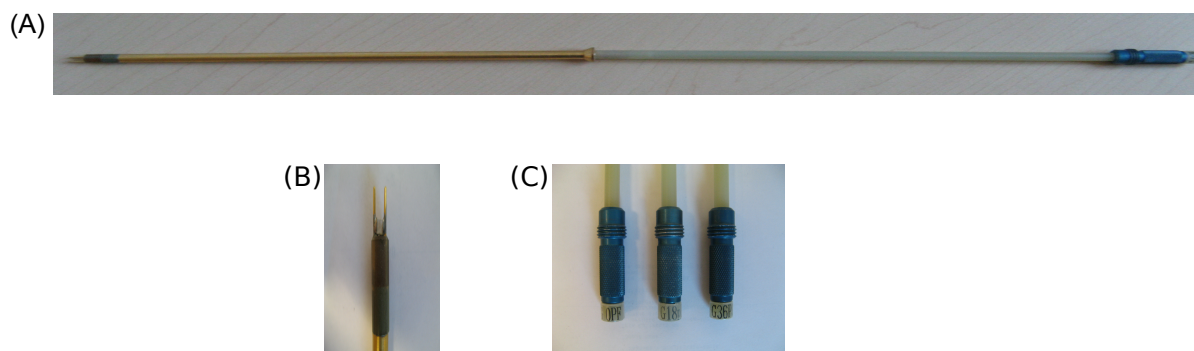


Figure 2.1. (A) A capacitor stick utilized on the Inova 400. (B) The top of a capacitor stick used by the HFPC probe on the Inova 400, showing the 2 prongs and the capacitor. The prongs must be aligned correctly within the probe in order to insert the stick. (C) The bottom of 3 of the 4 capacitor sticks used by the HFPC probe on the Inova 400. Each capacitor stick is labeled on the bottom of the stick. The blue metal portion rotates about the off-white insulator rod, allowing the stick to be screwed securely into the probe.

2.3. Tune and Match Capacitors

The capacitor sticks provide a coarse adjustment of the probe, but the adjustment is too coarse to acquire NMR spectra. To provide the fine adjustment needed, the probe contains “tune” and “match” variable capacitors within the probe. The capacitance of these capacitors, and thus the frequency of the probe's circuit, is changed by turning the tune and match knobs that are located on the bottom of the probe. The tune capacitor alters the “tune value” of the probe. This tune value ranges from 1 to 83 and is displayed on the side of the probe.

CAUTION: The adjustment of the tune and match capacitors is the most common way in which NMR probes are damaged. The knobs that adjust the capacitors can only turn a finite way in either direction. Normally, the knobs will turn smoothly and with little resistance. If you feel resistance when adjusting the tune and match, DO NOT force the knobs to turn further in that direction as doing so will damage the probe.

The Inova 400 uses what is known as an autoswitchable HFPC probe, which is shown in Figure 2.2. This probe is a 2-channel probe in which each channel can be simultaneously tuned to 2 different nuclei, thus 4

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nuclei can be studied at one time without manipulating the probe. The first channel, the HF channel, is tuned to ^1H and ^{19}F while the second channel, the CP/X-CHANNEL, is usually tuned to ^{13}C and ^{31}P . In order to independently tune each nucleus, the tune knob, which controls the tune capacitor, can slide up into the UP position or slide down into the DOWN position. For the HF channel, ^{19}F is tuned with the tune knob in the DOWN position and ^1H is tuned with the tune knob in the UP position. For the CP channel, ^{13}C is tuned with the tune knob in the DOWN position and ^{31}P is tuned with the tune knob in the UP position. In order to tune nuclei other than ^{13}C or ^{31}P , the appropriate capacitor stick must be inserted and the probe is usually tuned with the tune knob in the DOWN position.

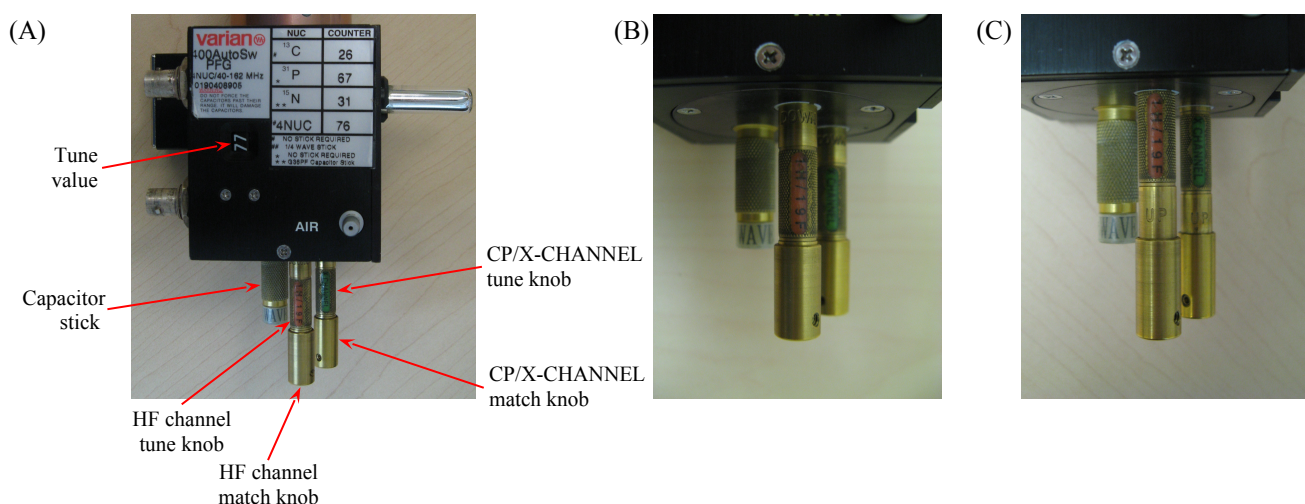


Figure 2.2. The bottom of the HF/CP probe used on the Inova 400. (A) Displayed are the tune value, capacitor stick, tune and match wands for the HF channel (red) and for the CP/X-CHANNEL (green). (B) The tune control can be in either the up or down position. In the down position, turning the tune wand will tune ^{19}F for the HF channel and ^{13}C for the CP/X-CHANNEL in 4-nucleus mode (i.e. when the $\frac{1}{4}$ -wave capacitor stick is inserted). Turning the green (CP/X-CHANNEL) tune knob in the down position also changes the tune value. (C) The tune control is in the up position, which is used to tune ^1H for the HF channel and ^{31}P for the CP/X-CHANNEL when in 4-nucleus mode.

2.4. Tuning Values for Some NMR Nuclei on the Inova 400

The table below displays the appropriate capacitor stick and tune value for several commonly studied NMR nuclei in the NMR facility.

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Table 2.1. Tune Values and Capacitor Sticks for Some Commonly Studied NMR Nuclei on the Inova 400

Isotope	Frequency, MHz	Capacitor Stick	Tune Value
² H	61.3663	G0	20
⁷ Li	155.354	None	76
¹¹ B	128.255	None	77
¹³ C	100.53	1/4-wave ^a	77
¹³ C	100.53	None ^a	33
¹⁵ N	40.5117	G36	33
²⁹ Si	79.4214	G0	51
³¹ P	161.822	1/4-wave ^a	77
³¹ P	161.822	None ^a	76
⁵⁹ Co	94.4017	None	10
⁷⁷ Se	76.2612	G0	47
¹¹³ Cd	88.6897	G0	64
¹¹⁹ Sn	149.085	None	73
¹²⁵ Te	104.782	None	68
¹⁹⁵ Pt	85.795	G0	66

^aOn the Inova 400, ¹³C and ³¹P can both be tuned at the same time using the 1/4-wave stick. If ¹³C or ³¹P can not be tuned sufficiently for your sample, you can switch to “single-resonance” mode by removing the capacitor stick completely.

3. PROBE TUNING PROCEDURE ON THE INOVA 400

3.1. Probe Tuning on the Inova 400 with a Capacitor Stick

On the Inova 400, for nuclei with frequencies less than about 90 MHz, the probe must be tuned with a capacitor stick inserted (see Table 2.1). For nuclei with frequencies greater than 90 MHz, the probe must be tuned without a capacitor stick inserted. In this case, the tuning method is slightly different (follow directions provided in section 3.2). The instructions in this section and in section 3.2 assume that the user is familiar with the basic operation of the Inova spectrometers. If not, please read the “Basic Operation of the Inova 400 and 600” notes and obtain Inova spectrometer training first.

- 1) Insert your sample.
 - 2) Disconnect the thin cable in front of the filter and connect it to the connector labeled “PROBE” on the magnet leg.
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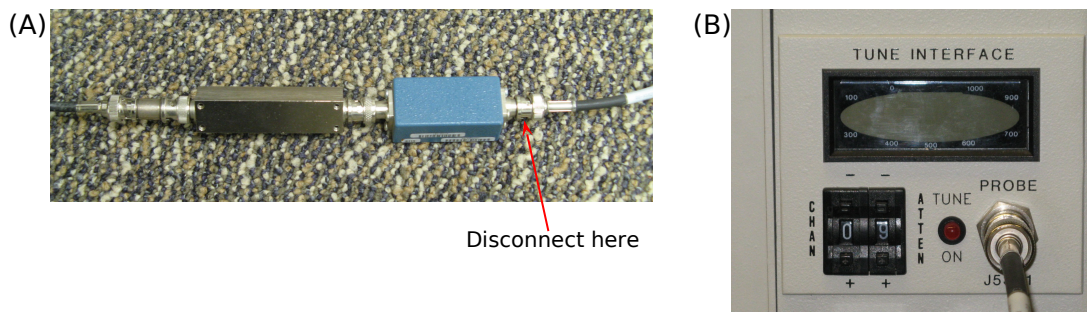


Figure 3.1. (A) The cable for the CP/X-channel on the Inova 400. The cable must be disconnected in front of the filter. (B) The TUNE INTERFACE on the face of the magnet leg. The cable must be connected to the "PROBE" port on the TUNE INTERFACE.

- 3) Remove the capacitor stick by turning the blue metal part of the stick clockwise until the stick releases. Support the stick to prevent the stick from crashing onto the floor.
- 4) Consult Table 2.1 to find the appropriate capacitor stick for your nucleus of interest.
 - a) Gently and slowly insert the appropriate capacitor stick into the probe until you feel resistance.
 - b) While very gently pushing the stick upwards, slowly rotate the stick in some direction.
 - c) When the prongs are properly aligned, you will feel the stick move up about 1 cm and you will no longer be able to rotate the stick.
 - d) Screw the stick into the probe by turning the metal part of the stick counterclockwise.

CAUTION: The prongs of the capacitor sticks used on the Inova 400 are very fragile and are thus easily damaged. Be very gentle and do not force the capacitor stick.

- 5) Consult Table 2.1 to find the appropriate tune value for your nucleus of interest.
- 6) With the CP/X-CHANNEL tune knob in the DOWN position, turn the tune knob in the appropriate direction until the correct tune value is displayed on the probe (see Figure 2.2 for a picture of the tune value).

TIP: Hold the CP/X-CHANNEL match knob still with one hand while turning the tune knob with the other.

- 7) Enter "qtune" in VnmrJ's command line. This command brings up the tuning interface known as QTune.

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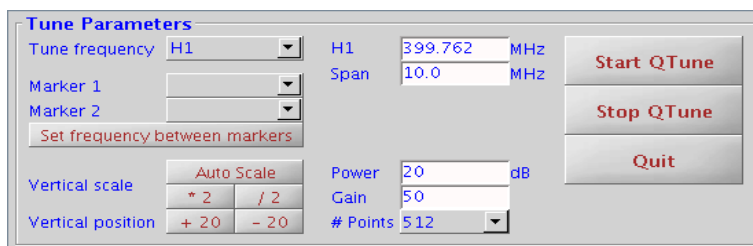


Figure 3.2: The default QTune parameter panel.

- 8) In the drop-down list next to **Tune frequency**, select your desired nuclear isotope. The frequency will automatically update after selecting the nucleus.
- 9) In the field window next to **Span**, input 50 and hit the enter key.
- 10) From the drop-down list next to **# Points**, select 1024.
- 11) Click on **Start QTune**.
- 12) Click on **Auto Scale**. The following should appear in the Graphics Display screen (Figure 3.2).

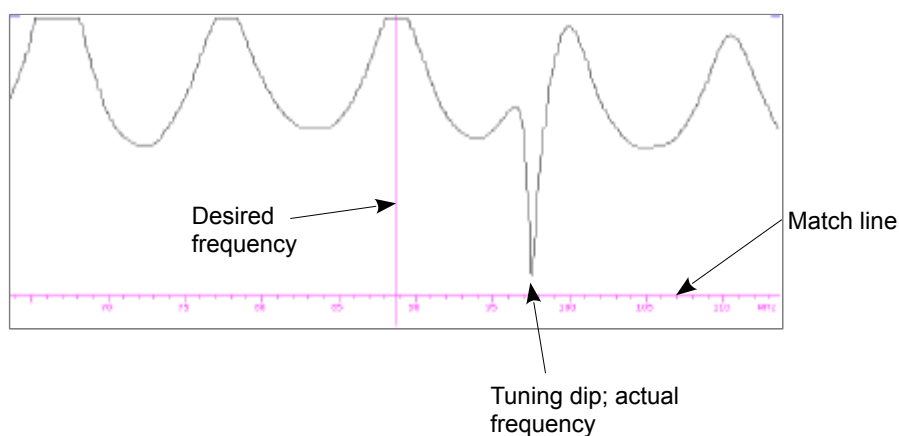


Figure 3.3. The QTune window used to tune the probe to some chosen nucleus. The dip is the current frequency of the channel. The match knob will move the dip upwards or downwards while the tune knob will move the dip left or right. The goal is to adjust the match and tune knobs iteratively until the dip is centred about the vertical pink line and also touches the match line.

- 13) Turn the match knob on the CP/X-CHANNEL in some arbitrary direction; the dip will move upwards or downwards (as well as left or right; ignore the left and right). If the dip moves upwards, reverse the direction of the match knob. Continue turning the match knob until the dip is near the match line.

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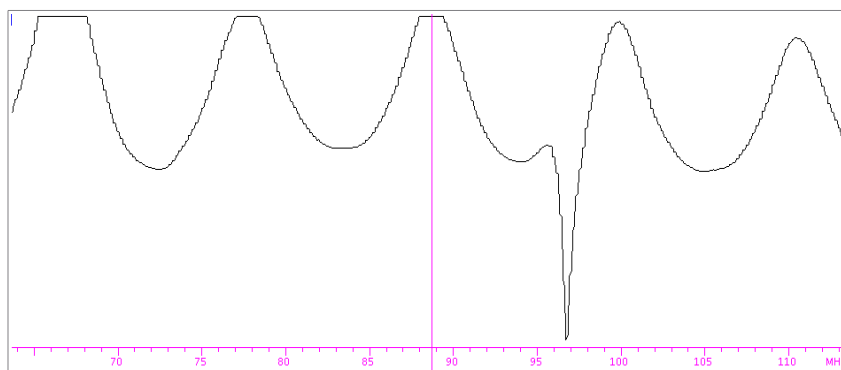


Figure 3.4. The Q-Tune window after initial adjustment of the match knob. The tuning dip is very close to the horizontal match line.

CAUTION: The match knob can only turn a finite number of times in either direction. If you feel resistance when turning the match knob, DO NOT force it to turn further. Doing so will damage the probe.

- 14) With the CP/X-CHANNEL tune knob in the DOWN position, hold the match knob still with your weak hand. Turn the tune knob in some arbitrary direction; the dip will move left or right. If the dip moves away from the vertical centre line, reverse the direction of the tune knob. Continue turning the tune knob until the dip is centred about the vertical centre line. As the dip moves left or right, the dip may move upwards away from the bottom match line; do not worry about that at this point.

CAUTION: The tune knob can only turn a finite number of times in either direction. If you feel resistance when turning the tune knob, DO NOT force it to turn further. Doing so will damage the probe.

- 15) Iteratively adjust the match and tune (in the DOWN position) knobs until the dip is aligned in the centre and also with the match line.

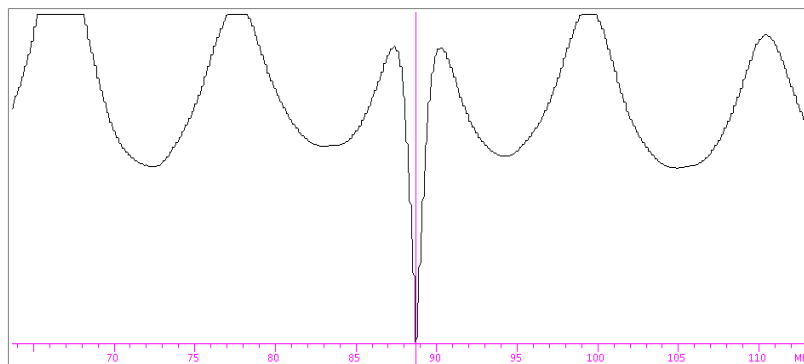


Figure 3.5. The Q-Tune window after iteratively adjusting the tune and match knobs.

- 16) In the window to the right of “Span”, input “5”, followed by enter. Iteratively make fine adjustments of the match and tune (in the DOWN position) until no further improvements can be made.

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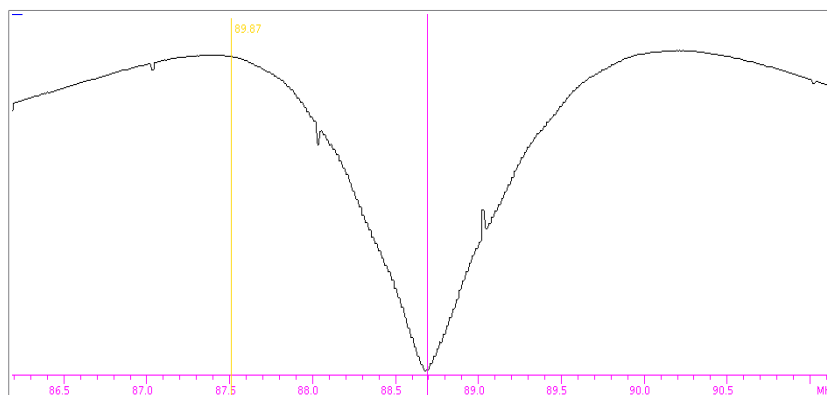




Figure 3.6. The QTune window after decreasing the span to 5 MHz and making fine adjustments of the tune and match knobs.

- 17) Click on .
- 18) Click on .
- 19) Disconnect the cable from the PROBE connection on the magnet leg and re-connect the cable to the filter.
The probe is now tuned and you can start your experiment.

3.2. Probe Tuning on the Inova 400 with no Capacitor Stick

On the Inova 400, for nuclei with frequencies greater than 90 MHz, the probe must be tuned without a capacitor stick inserted. The instructions in this section assume that the user is familiar with the basic operation of the Inova spectrometers. If not, please read the “Basic Operation of the Inova 400 and 600” worksheet and obtain Inova spectrometer training first.

- 1) Follow steps 1 through 12 of section 3.1, except do not insert a capacitor stick (i.e. skip step 4).
 - 2) Slide the CP/X-CHANNEL tune knob up and hold the tune knob in the UP position. You should feel the tune knob engage, but you may have to slightly turn the tune knob to do so.
 - 3) With the CP/X-CHANNEL tune knob in the UP position, turn the tune knob in some arbitrary direction; the dip will move left or right. If the dip moves away from the vertical pink line, reverse the direction of the tune knob. Continue turning the X-CHANNEL tune knob in the UP position until the dip is centred about the vertical pink line. As the dip moves left or right, the dip may move upwards from the bottom match line; do not worry about that at this point.
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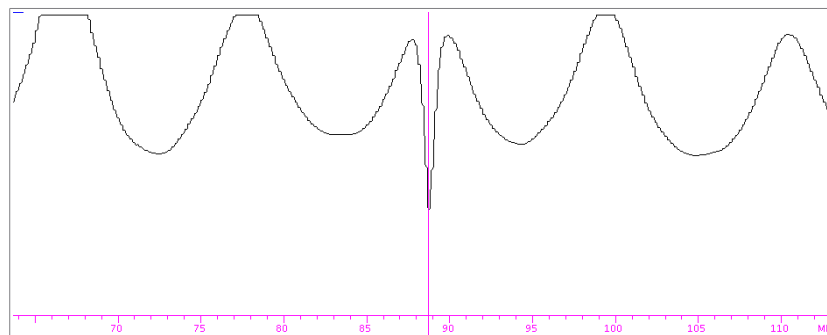


Figure 3.7: The QTune window after initial adjustment of the CP/X-CHANNEL tune knob in the UP position.

CAUTION: The tune knob can only turn a finite number of times in either direction. If you feel resistance when turning the tune knob, DO NOT force it to turn further. Doing so will damage the probe.

- 4) Iteratively adjust the match and tune (in the UP position) knobs until the dip is aligned in the centre and also with the match line.

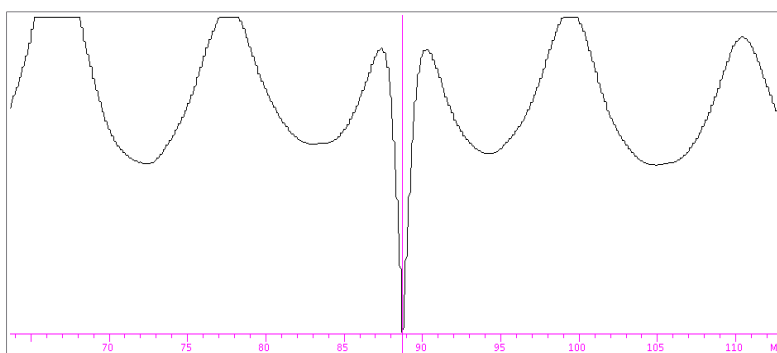


Figure 1: The QTune window after iterative adjustment of the CP/X-CHANNEL tune knob in the UP position and the CP/X-CHANNEL match knob.

- 5) In the window to the right of “Span”, input “5”, followed by enter. Iteratively make fine adjustments of the match and tune (in the UP position) until no further improvements can be made.

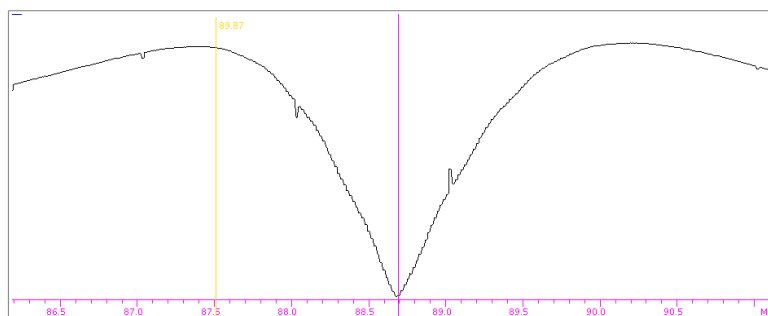
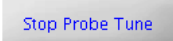
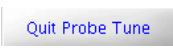


Figure 2: The QTune window after iteratively fine tuning the CP/X-CHANNEL tune knob in the UP position and the CP/X-CHANNEL match knob.

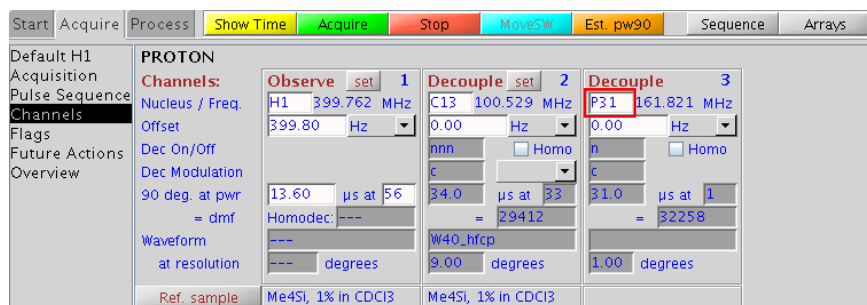
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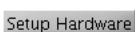
- 6) Click on .
- 7) Click on .
- 8) Disconnect the cable from the PROBE connection on the magnet leg and re-connect the cable to the filter. The probe is now tuned and you can start your experiment.

3.3. Re-tuning the Probe on the Inova 400

After completion of your experiment, the probe must be tuned back to $^{13}\text{C}/^{31}\text{P}$. This procedure is slightly different than the procedure used to tune odd nuclei.

- 1) Insert the default CDCl_3 sample.
- 2) Read in a PROTON experiment.
- 3) Go to the **Acquire** panel, **Channels** page.
- 4) In the space for channel 3, under the word **Decouple**, input “P31”, then hit the enter key.



- 5) Go to the **Start** panel and click on .
 - 6) Remove the capacitor stick (if applicable) and insert the $\frac{1}{4}$ -wave capacitor stick.
 - 7) Disconnect the thin cable in front of the filter and connect the cable to the connector labeled “PROBE” on the magnet leg.
 - 8) With the green CP/X-CHANNEL tune knob in the DOWN position, turn the tune knob until the tune value on the probe reads 77.
 - 9) On the magnet leg, next to the buttons labeled “CHAN”, press the “+” button twice to select channel 2. A number will be displayed in the TUNE INTERFACE window.
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- 10) Turn the X-CHANNEL match knob in some arbitrary direction. If the number displayed in the TUNE INTERFACE increases, or you feel resistance, reverse the direction you are turning the knob. Continue until the number displayed reaches a minimum. Note: You may have to turn the match direction many times before the number displayed changes.
 - 11) On the magnet leg, next to the buttons labeled “CHAN”, press the “+” button once to select channel 3.
 - 12) Slide the X-CHANNEL tune knob into the UP position (you will have to hold it up). With the tune knob in the UP position, turn the tune knob in some arbitrary direction. If the number displayed in the TUNE INTERFACE increases, or you feel resistance, reverse the direction you are turning the tune knob. Continue until the number displayed reaches a minimum. Note: You may have to turn the tune knob many times before the number displayed changes.
 - 13) On the magnet leg, next to the buttons labeled “CHAN”, press the “-” button once to select channel 2.
 - 14) Repeat steps 10 through 13 until the number displayed when channel 2 is selected is less than 40 and the number displayed when channel 3 is selected is less than 375.
 - 15) On the magnet leg, next to the buttons labeled “CHAN”, press the “-” button twice/thrice to select channel 0.
 - 16) Disconnect the cable from the PROBE connection on the magnet leg and re-connect the cable to the filter.
-