

# PROBE TUNING ON THE INOVA 600

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## 1. INTRODUCTION AND BACKGROUND INFORMATION

### 1.1. About these Notes and Related Notes

These notes describe how to tune the NMR probe on the Inova 600 in order to acquire the best possible  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra and also perform NMR experiments on other nuclei such as  $^{19}\text{F}$ ,  $^{29}\text{Si}$ , and  $^{31}\text{P}$ . Before tuning the probe, 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 600	<ul style="list-style-type: none"> <li>• NMR Sample Preparation</li> <li>• Reserving NMR Time using FACES</li> <li>• Basic Operation of the Inova 400 and 600</li> <li>• Retrieving NMR Data</li> </ul>	<ul style="list-style-type: none"> <li>• Processing 1D NMR Spectra using VnmrJ</li> </ul>
Perform a 2D NMR experiment involving odd nuclei on the Inova 600	<ul style="list-style-type: none"> <li>• NMR Sample Preparation</li> <li>• Reserving NMR Time using FACES</li> <li>• Basic Operation of the Inova 400 and 600</li> <li>• Retrieving NMR Data</li> </ul>	<ul style="list-style-type: none"> <li>• Processing 2D NMR Spectra using VnmrJ</li> </ul>

### 1.2. About NMR Probes

#### 1.2.1. What is an NMR Probe?

An NMR spectrometer consists of three major components: a console, a magnet and a probe. The NMR probe is suspended within the NMR magnet and is connected to the console by electrical cables. During an NMR experiment, the NMR sample solution sits within the coil of the NMR probe. A rf-pulse is generated by the spectrometer console and travels through electrical cables into the probe and to the probe's coil where it is applied to the NMR sample. In the same coil, a resulting NMR signal is picked up from the sample, and that signal is then transferred to the console through electrical cables for digitization and conversion into an FID from which an NMR spectrum is generated.

#### 1.2.2. NMR Probe Channels

There are numerous types of NMR probes, with each probe suited for a specific purpose. Probes are thus classified by various characteristics, with one of the most important being its number of channels. The number of channels dictates how many different nuclei can be probed at one specific time. A typical NMR probe will have a minimum of a two channels - a high-band and a low-band channel - and is termed a double-resonance NMR probe. Typically the high-band channel will tune to either  $^1\text{H}$  and  $^{19}\text{F}$ , while the low-band channel will tune to any nucleus that has a Larmor frequency that falls between that of  $^{15}\text{N}$  and  $^{31}\text{P}$  (eg.  $^{11}\text{B}$ ,  $^{13}\text{C}$ ,  $^{29}\text{Si}$ ,  $^{119}\text{Sn}$ , etc.). Such a probe would enable you to acquire a  $^{13}\text{C}\{^1\text{H}\}$  spectrum or a  $^1\text{H}$ - $^{31}\text{P}$  HSQC, for example.

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## 1.3. About Tuning NMR Probes

### 1.3.1. About Frequency, Impedance, and Capacitance

Before discussing probe tuning, it is first necessary to introduce some basic electrical terms and concepts. Consider the alternating current (AC) used by NMR spectrometers for sample irradiation. The AC signal has a frequency ( $\nu$ ) and an amplitude. The frequency is in the radiofrequency region of the electromagnetic spectrum and the amplitude is generally in the mV. The AC signal of the desired frequency is generated and amplified by the spectrometer's various components and is transmitted to the probe by electrical cables.

These components and cables have a natural resistance to the transmission of the AC current. This resistance is termed a characteristic impedance. In NMR, the characteristic impedance of the spectrometer components is 50 ohms and in order for the AC signal to transmit without loss between the spectrometer components, it is imperative that all components have the same characteristic impedance.

Impedance is dependent on frequency and also another electrical characteristic known as capacitance. Capacitance (C) is a measure of an electrical device's ability to store electrical charge. In an AC circuit, impedance (Z) is related to frequency ( $\nu$ ) and capacitance (C), such that:

$$Z \propto (\nu C)^{-1}$$

### 1.3.2. What is Probe Tuning?

The easiest way to understand probe tuning is to envision that the spectrometer console is one electrical circuit and the probe is another electrical circuit. The spectrometer components that produce and transmit the rf-pulse have an impedance of 50 ohms, while the frequency of the rf-pulse is determined by the nuclear isotope of interest. To ensure the rf-pulse is transmitted with minimal loss from the spectrometer to the probe channel, the probe's channel must be of the same frequency and impedance as that of the spectrometer. Manipulating the probe channel to ensure this condition is met is known as "probe tuning".

If the probe is "out of tune" when attempting to perform NMR experiments, the rf-pulses are not efficiently transferred to the NMR probe. This results in significant reductions in the signal-to-noise ratio for 1D NMR experiments and can lead to the absence of any signal in 2D NMR experiments.

### 1.3.3. Tune and Match Capacitors

The adjustment of the probe channel's impedance and frequency is done by manipulating the probe's variable capacitors. Each channel has at least two such capacitors, which are generally labeled "tune" and "match" capacitors. Their capacitance is adjusted by turning the appropriate tune and match knobs located at the base of the probe. In general, manipulating the tune capacitor mostly affects the probe channel's frequency, with small changes in the probe channel's impedance, while the match capacitor mostly affects the probe's channel's impedance, with small changes in the probe channel's frequency. Because of this relationship, the probe must be tuned in an iterative manner, alternating between the adjustment of the tune and match capacitors until the desired frequency and 50 ohm impedance is obtained.

### 1.3.4. Sample Dependence of Probe Tuning

The NMR sample is part of the probe's circuit and the impedance and frequency of a probe's channels are therefore affected by the nature of the NMR sample solution. As a result, the probe's tuning depends explicitly on the NMR sample solution and the sample solution's impedance. The solution's impedance is predominantly determined by its solvent. In general, the more polar the solvent, the lower its impedance. When comparing

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deuterated solvents, the solvent's ability to conduct electricity and therefore its impedance is reported as its dielectric constant (see Table 2).

To simplify whether the probe needs to be tuned from its default  $\text{CDCl}_3$  tuning, the solvents have been separated into two classes: class I solvents have a low dielectric constant and class II solvents have high dielectric constants. More details about how the solvents affect the probe tuning can be found in section 1.4.5.

**Table 1.2.** Some commonly utilized NMR solvents and their dielectric constants

Solvent	Dielectric Constant	Solvent Class
Benzene- $d_6$	2.3	I
Toluene- $d_8$	2.4	I
Chloroform- $d$	4.8	I
Dichloromethane- $d_2$	8.9	I
Acetone- $d_6$	20.7	II
Methanol- $d_4$	32.7	II
Acetonitrile- $d_3$	37.5	II
Dimethyl sulfoxide- $d_6$	46.7	II
Deuterium oxide	78.5	II

### 1.4. About Tuning the NMR Probe on the Inova 600

#### 1.4.1. The NMR Probe on the Inova 600: The AutoX-DB Probe

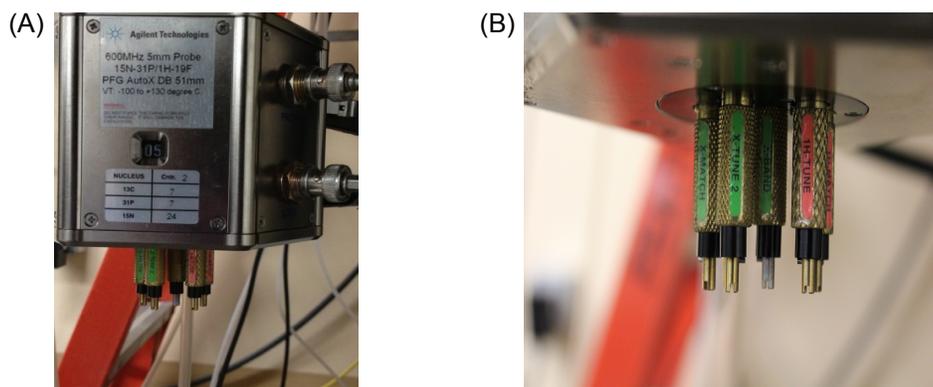
The probe on the Inova 600 is referred to as the AutoX-DB NMR probe. This probe is a double-resonance probe and therefore has two channels: a high-band channel and a low-band channel. The high-band channel can be tuned to either  $^1\text{H}$  or  $^{19}\text{F}$ , while the low-band channel can be tuned to any nucleus that has a Larmor frequency between that of  $^{15}\text{N}$  (60.74 MHz) and  $^{31}\text{P}$  (242.64 MHz). Some commonly studied nuclear isotopes within that frequency range include  $^2\text{H}$  (92.01 MHz),  $^{13}\text{C}$  (150.74 MHz),  $^{29}\text{Si}$  (110.09 MHz), and  $^{119}\text{Sn}$  (223.54 MHz).

#### 1.4.2. High-band Tune and Match Capacitors

The probe has one high-band tune and one high-band match capacitor (see Figure 1.1). The knobs that adjust these capacitors have labels with a red background and are labeled 1H-MATCH and 1H-TUNE. They can be found on the back of the probe base.

**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.

## PROBE TUNING ON THE INOVA 600

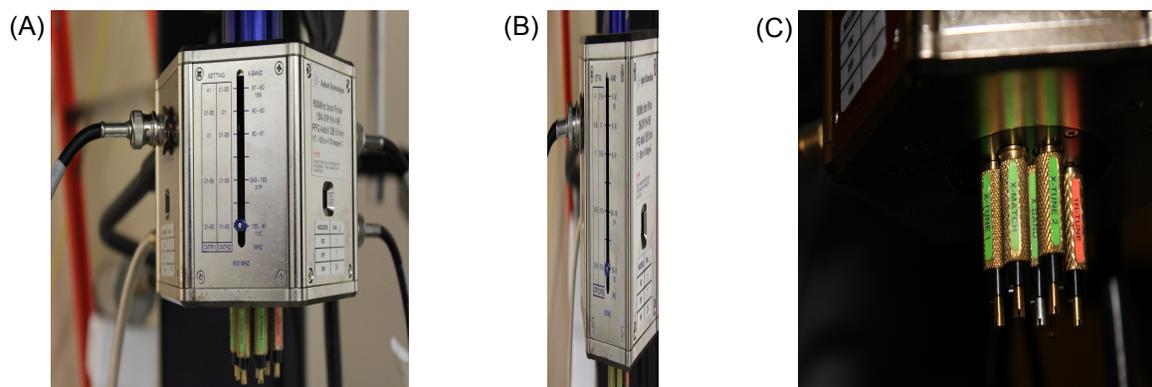


**Figure 1.1:** (A) A side view of the base of the AutoX-DB probe on the Inova 600 showing the high-band channel's match and tune knobs, which are labeled 1H-MATCH and 1H-TUNE. (B) A close-up of showing the 1H-TUNE and 1H-MATCH knobs.

### 1.4.3. Low-band Tune, Match and Band Capacitors

Tuning the low-band channel is more complicated than the high-band channel because of the much larger frequency range that the low-band channel covers. The low-band channel has four knobs that may need adjustment in order to tune the probe. All of the knobs have labels with green backgrounds and are on the front of the probe base.

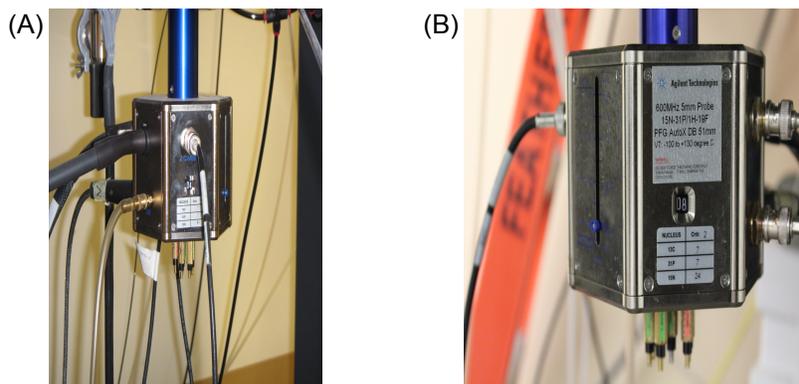
The first knob that must be adjusted is the X-BAND knob (see Figure 1.2). The X-BAND sets the coarse frequency range on the probe. In total, there are five frequency ranges, which starting from the top of the probe base and moving downwards are: 61-60 ( $^{15}\text{N}$ ), 90-80, 80-61, 243-155 ( $^{31}\text{P}$ ), and 155-90 ( $^{13}\text{C}$ ). Turning the X-BAND knob will move the indicator on the front of the probe base up or down to the appropriate frequency range for your desired nucleus.



**Figure 1.2:** (A) The base of the AutoX-DB probe, showing the X-BAND indicator and frequency ranges, with the various tune and match knobs as well. (B) A close-up of the frequency ranges. (C) A close-up of the low-band channel tune, match and band knobs, plus the high-band channel tune knob.

To provide the fine adjustment needed, the probe has two tune knobs, labeled “X-TUNE 1” and “X-TUNE 2”, and a match knob, labeled “X-MATCH”, at the base of the probe (see Figure 1.3). Each tune knob has its own associated “tune value”, with the X-TUNE 1 tune value displayed on the left-hand side of the probe and the X TUNE 2 value displayed on the right-hand side of the probe. The tune value for each tune knob ranges from 1 to 55, which must be set appropriately in order to observe the NMR nucleus of interest.

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**Figure 1.3:** The base of the AutoX-DB probe on the Inova 600, showing (A) the X-TUNE 1 tune value and (B) X-TUNE 2 tune value.

### 1.4.4. Tuning Values for Some NMR Nuclei on the Inova 600

The table below displays the appropriate X-BAND, X-TUNE 1 and X-TUNE 2 values for several commonly studied low-band NMR nuclei in the NMR facility.

**Table 1.3.** X-BAND, X-TUNE 1 and X-TUNE 2 Values for some Commonly Studied NMR Nuclei on the Inova 600.<sup>a</sup>

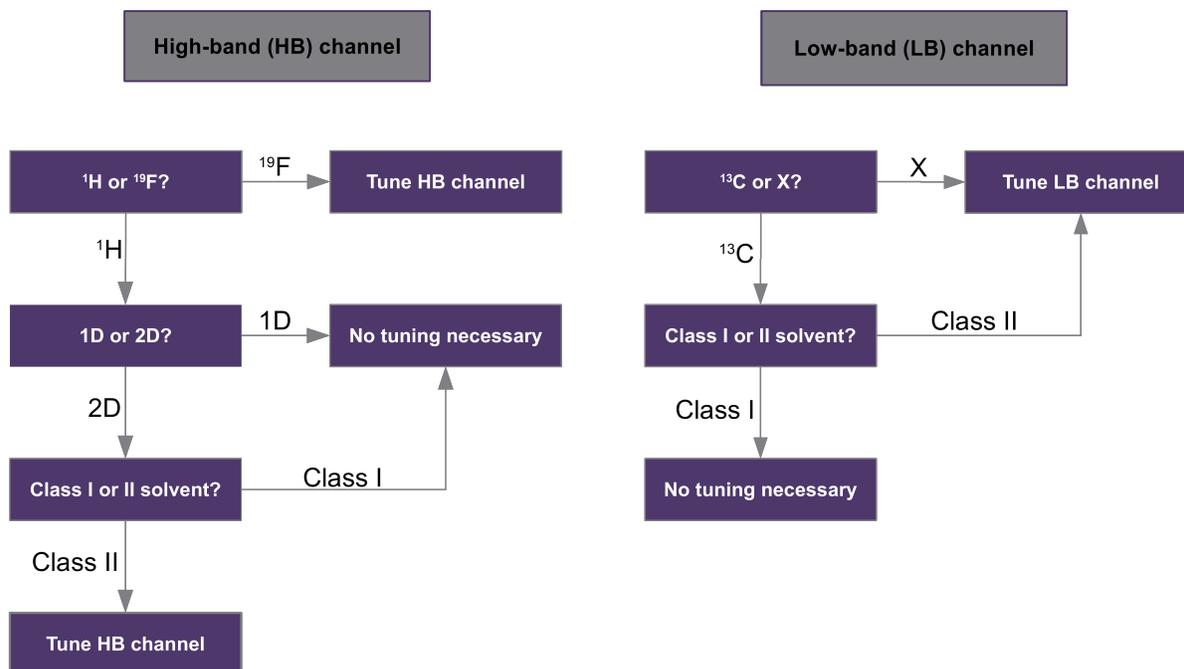
Isotope	Frequency, MHz	X-BAND position	XTUNE 1 Value	XTUNE 2 Value
<sup>11</sup> B	192.36	243-155 ( <sup>31</sup> P)	21	22
<sup>13</sup> C	150.78	155-90 ( <sup>13</sup> C)	4	4
<sup>15</sup> N	60.76	60-61 ( <sup>15</sup> N)	41	21
<sup>29</sup> Si	119.12	155-90 ( <sup>13</sup> C)	22	23
<sup>31</sup> P	242.71	243-155 ( <sup>31</sup> P)	4	4
<sup>71</sup> Ga	182.79	243-155 ( <sup>31</sup> P)	26	25.5
<sup>77</sup> Se	114.38	155-90 ( <sup>13</sup> C)	26	25
<sup>119</sup> Sn	223.61	243-155 ( <sup>31</sup> P)	12	12
<sup>125</sup> Te	189.40	243-155 ( <sup>31</sup> P)	23	23

<sup>a</sup>The X-TUNE 1 and X-TUNE 2 values are for samples dissolved in CDCl<sub>3</sub>. Samples with solvents having higher dielectric constants than CDCl<sub>3</sub> will have slightly lower X-TUNE 1 and X-TUNE 2 values.

### 1.4.5. When do I need to Tune the AutoX-DB Probe?

The AutoX-DB probe supposed to remain tuned to CDCl<sub>3</sub> at all times, with the high-band channel tuned to <sup>1</sup>H and the low-band channel tuned to <sup>13</sup>C. Whether or not the probe needs to be tuned depends on the type of experiment you are performing, the nucleus or nuclei involved and the solvent. Use the flow-chart below to determine if you need to tune the probe, but keep in mind it never hurts to check if the probe is tuned, regardless of what you are doing!

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**Figure 1.4:** The flow chart shows whether or not the probe must be tuned. In the flow chart, Class I solvents are those with low dielectric constants, while Class II solvents have high dielectric constants. See Table 2 for a list of commonly used deuterated solvents and their dielectric constants.

## 2. TUNING THE PROBE ON THE INOVA 600

These instructions 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. Please go to the section that suits what you are doing:

1. tweaking the probe to adjust the  $^1\text{H}$  or  $^{13}\text{C}$  tuning; see section 2.1
2. tuning the high-band channel to  $^{19}\text{F}$ ; see section 2.2
3. tuning the low-band channel to a nucleus other than  $^{13}\text{C}$ ; see section 2.3.

### 2.1. Method for fine tuning $^1\text{H}$ and $^{13}\text{C}$

The probe will be tuned to a  $\text{CDCl}_3$  sample,  $^1\text{H}$  (high-band channel) and  $^{13}\text{C}$  (low-band channel) by default. If you need to improve the tuning of these nuclei, follow the instructions in this section.

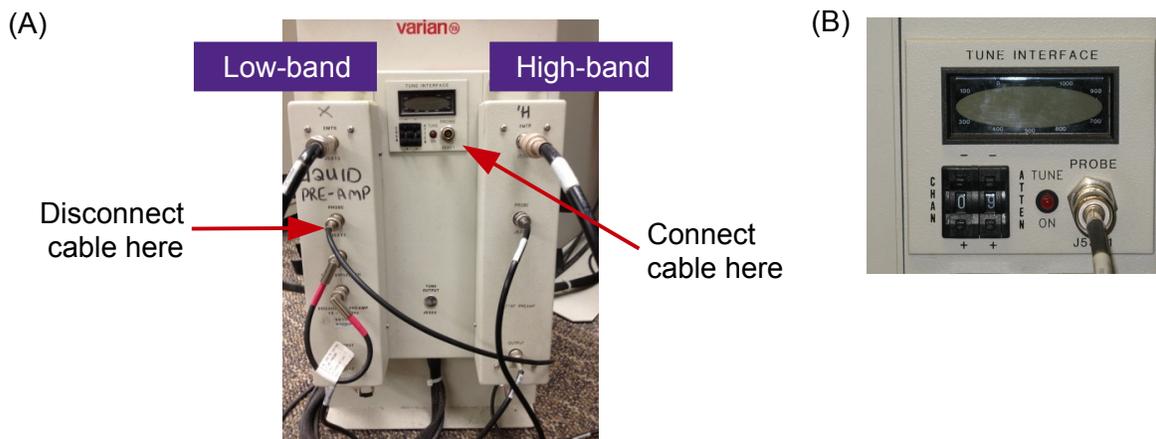
#### 2.1.1. Fine tuning $^{13}\text{C}$ on the low-band channel

- 1) Insert your sample.
- 2) Read in a PROTON experiment.
- 3) In the VnmrJ command line, enter `su`, followed by enter key. The messages `expX: Experiment started` and `expX: Setup Complete` should appear in the message window.

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**NOTE:** After entering `su`, DO NOT do any other software manipulations or the tuning will not work!

- 4) Disconnect the cable from the port labeled PROBE on the low-band pre-amplifier and attach it to the port labeled PROBE in the Tune Interface.



**Figure 2.1:** The I600's low-band pre-amplifier, high-band pre-amplifier and Tune Interface. (B) A close-up of the tune interface. When fine tuning the low-band channel to  $^{13}\text{C}$ , disconnect the cable from the PROBE port on the low-band pre-amplifier and connect it to the PROBE port on the Tune Interface. Press the + button next to the CHAN label twice to select channel 2 ( $^{13}\text{C}$ ).

- 5) Press the + button next to CHAN twice to select channel 2. A number should appear in the Tune Interface window.

**NOTE:** The ATTN value should always be 9 and never needs to be adjusted.

- 6) Using the red tuning stick, turn the X-TUNE 2 knob as gently as possible in some arbitrary direction. The number displayed in the tune interface will either get smaller or larger. If it gets larger, you will need to turn the X-TUNE 2 knob the other direction.

**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.

- 7) Continue turning the X-TUNE 2 knob in the direction that resulted in a decrease in the number until the value displayed is 15 or lower. If you turn the knob too far, the tune value will begin to increase again; you will then need to turn the X-TUNE 2 knob in the opposite direction.
- 8) Press the – button next to CHAN twice to select channel 0. The number in the tune interface should disappear.
- 9) Disconnect the cable from the PROBE port of the Tune Interface and re-connect it to the PROBE port on the low-band pre-amplifier.
- 10) If necessary, tune the high-band channel (see section 2.1.2).
- 11) Lock, shim and acquire your spectra.
- 12) When your experiments are complete, insert the  $\text{CDCl}_3$  sample and tune the low-band channel as described in section 3.1.

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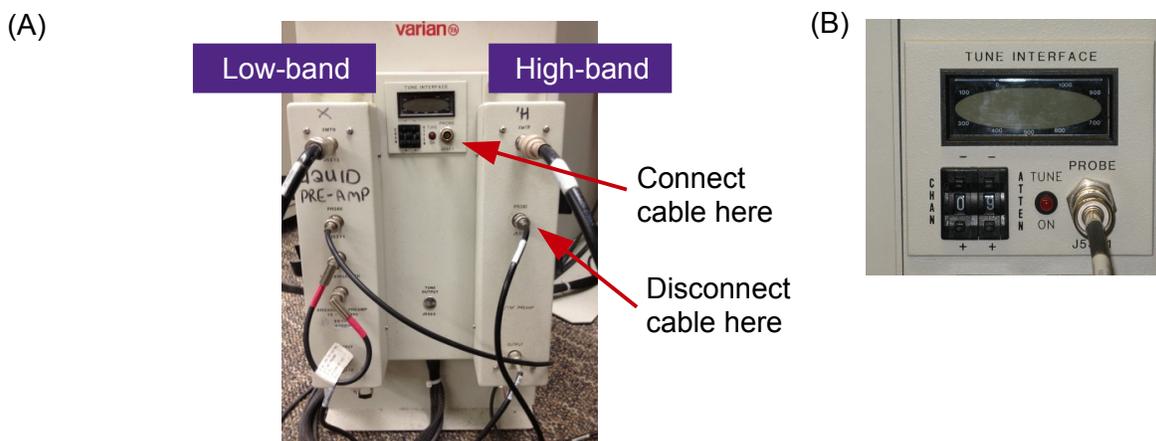
### 2.1.2. Fine tuning $^1\text{H}$ on the high-band channel

If you have just tuned the low-band channel as described in section 2.1.1, you can skip steps 1 – 3 below. If not, start at step 1.

- 1) Insert your sample.
- 2) Read in a PROTON experiment.
- 3) In the VnmrJ command line, enter `su`, followed by enter key. The messages `expX: Experiment started` and `expX: Setup Complete` should appear.

**NOTE:** After entering `su`, DO NOT do any other software manipulations or the tuning will not work!

- 4) Disconnect the cable from the port labeled PROBE on the high-band pre-amplifier and attach it to the port labeled PROBE in the Tune Interface.



**Figure 2.2:** The I600's low-band pre-amplifier, high-band pre-amplifier and Tune Interface. (B) A close-up of the tune interface. When fine-tuning the high-band channel to  $^1\text{H}$ , disconnect the cable from the PROBE port on the high-band pre-amplifier and connect it to the PROBE port on the Tune Interface. Press the + button next to the CHAN once to select channel 1 ( $^1\text{H}$ ) for tuning.

- 5) Press the + button next to CHAN once to select channel 1. A number should appear in the TUNE INTERFACE.

**NOTE:** The ATTEN value should always be 9 and never needs to be adjusted.

- 6) Using the red tuning stick, turn the 1H-TUNE knob as gently as possible in some arbitrary direction. The number displayed in the tune interface will either get smaller or larger. If it gets larger, you will need to turn the 1H-TUNE knob the other direction.

**NOTE:** The 1H-TUNE wand is quite sensitive and it is easy to overshoot the tuning minimum.

- 7) Continue turning the 1H-TUNE knob in the direction that resulted in a decrease in the number until the value displayed is 15 or lower. If you turn the knob too far, the tune value will begin to increase again; you will then need to turn the 1H-TUNE knob in the opposite direction to get back to the minimum.
- 8) Press the – button next to CHAN once to select channel 0. The number in the tune interface should disappear.
- 9) Disconnect the cable from the PROBE port of the Tune Interface and re-connect it to the PROBE port on the high-band pre-amplifier.

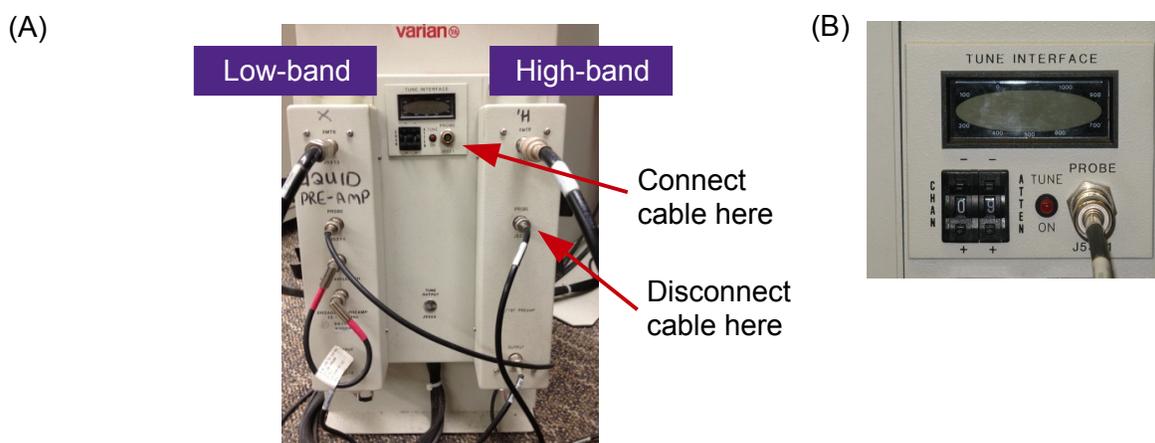
## PROBE TUNING ON THE INOVA 600

- 10) Lock, shim and acquire your spectra as normal.
- 11) When your experiments are complete, insert the  $\text{CDCl}_3$  sample and tune the high-band channel as described in section 3.2.

### 2.2. Method for Tuning the High-Band Channel to $^{19}\text{F}$

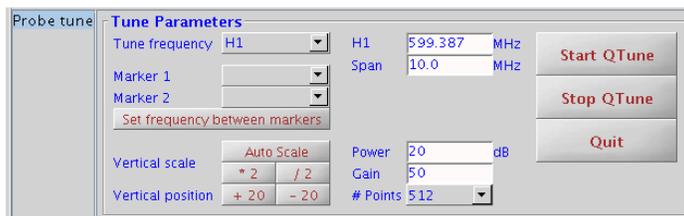
- 1) Insert your sample.
- 2) Disconnect the cable from the port labeled PROBE on the high-band pre-amplifier and attach it to the port labeled PROBE by the Tune Interface window.

**NOTE:** Do not use the CHAN button to select a channel. For Qtune to work, channel 0 must be selected.



**Figure 2.3:** The I600's low-band pre-amplifier, high-band pre-amplifier and Tune Interface. (B) A close-up of the tune interface. When tuning the high-band channel to  $^{19}\text{F}$ , disconnect the cable from the PROBE port on the high-band pre-amplifier and connect it to the PROBE port on the Tune Interface.

- 3) Enter `qtune` in VNMRJ's command line. This command brings up the tuning interface known as QTune.

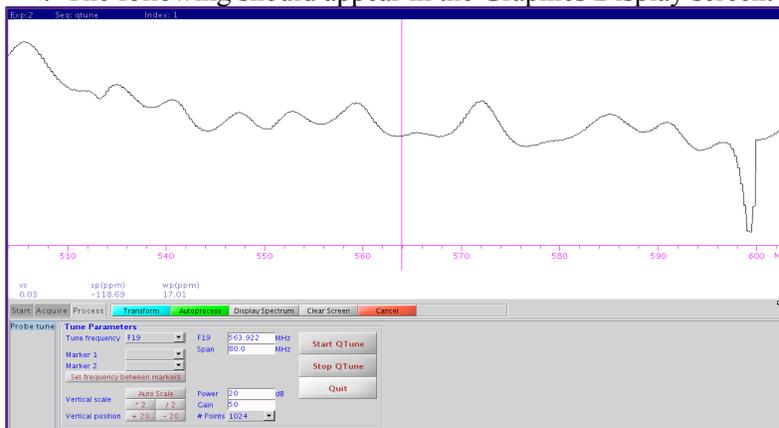


**Figure 2.4:** The initial QTune panel before modifying the necessary parameters.

- 4) From the drop-down list next to **Tune frequency**, select F19. The frequency will automatically update.
- 5) In the window next to **Span**, enter 80 and hit the enter key.
- 6) From the drop-down list next to **# Points**, select 1024.
- 7) Click on **Start QTune**.

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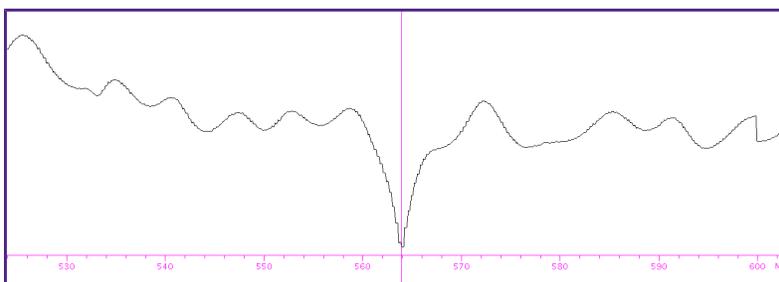
- 8) Click on **Auto Scale**. The following should appear in the Graphics Display screen:



**Figure 2.5:** The Q Tune window after setting the appropriate parameters for  $^{19}\text{F}$ . The dip on the right-hand side of the screen shows the initial tuning frequency and impedance of the high-band channel.

- 9) Turn the 1H-TUNE knob left (counterclockwise); the dip will move left. Continue turning the 1H-TUNE knob until the dip is near the vertical line.

**CAUTION:** The 1H-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.



**Figure 2.6:** The Q Tune window after adjusting the 1H-TUNE knob. The tuning dip is now centred about the vertical line (the  $^{19}\text{F}$  frequency).

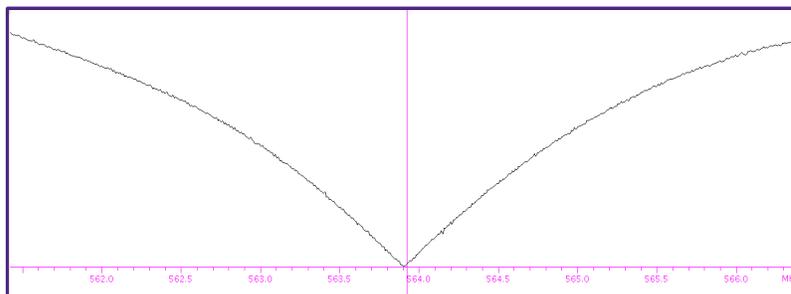
- 10) In the window to the right of **Span**, input “5”, followed by enter.
- 11) Turn the 1H-MATCH knob in some arbitrary direction; the dip will move up or down (and also a little left or right). If the dip moves upwards, reverse the direction you are turning the 1H-MATCH knob. Continue turning the 1H-MATCH knob in the appropriate direction until the dip is touching the horizontal line. As the dip moves downwards, the dip may move away from the vertical line; do not worry about that at this point.

**CAUTION:** The 1H-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.

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- 12) Iteratively adjust the 1H-TUNE and 1H-MATCH knobs until the dip is centred about the vertical pink line and touches the match line.



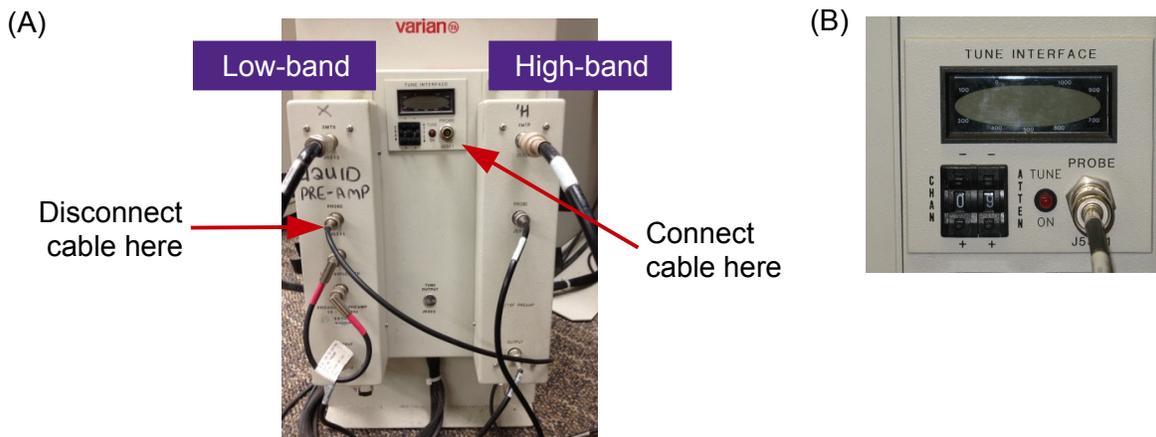
**Illustration 2.7:** The Q Tune window after iteratively adjusting the 1H-TUNE and 1H-MATCH knobs. The probe is now perfectly tuned to acquire  $^{19}\text{F}$  NMR spectra.

- 13) Click on .
- 14) Disconnect the cable from the PROBE port by the tune interface and re-connect the cable to the PROBE port on the high-band pre-amplifier.
- 15) If necessary, tune the low-band channel as described in section 2.1.1 (fine tuning  $^{13}\text{C}$ ) or section 2.3 (low-band nuclei other than  $^{13}\text{C}$ ).
- 16) Click on .
- 17) Lock, shim and acquire your spectra.
- 18) When your experiments are complete, insert the  $\text{CDCl}_3$  sample and tune the high-band channel as described in section 3.2.

### 2.3. Method for Tuning the Low-band Channel to Nuclei other than $^{13}\text{C}$

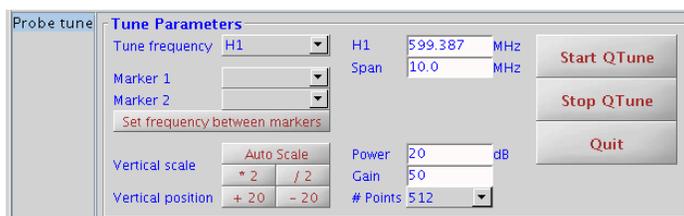
- 1) Insert your sample.
  - 2) Disconnect the cable from the port labeled “PROBE” on the low-band pre-amplifier and attach it to the port labeled “PROBE” by the Tune Interface window.
-

## PROBE TUNING ON THE INOVA 600



**Figure 2.8:** The I600's low-band pre-amplifier, high-band pre-amplifier and Tune Interface. (B) A close-up of the tune interface. When tuning the low-band channel to a nucleus other than  $^{13}\text{C}$ , disconnect the cable from the PROBE port on the low-band pre-amplifier and connect it to the PROBE port on the Tune Interface.

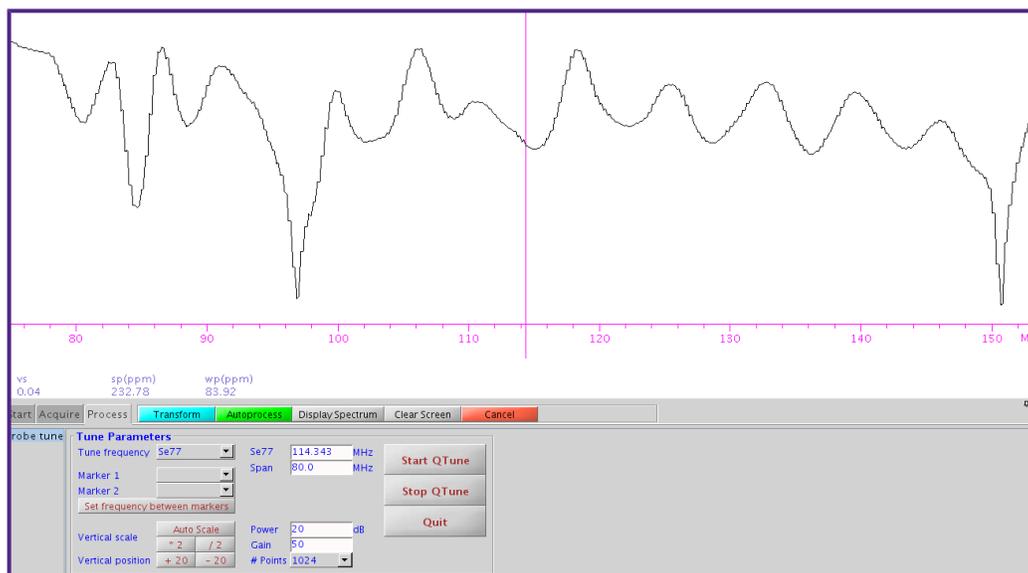
- 3) Enter “qtune” in VNMRJ’s command line. This command brings up the tuning interface known as QTune.



**Figure 2.9:** The initial Qtune panel before modifying the necessary parameters.

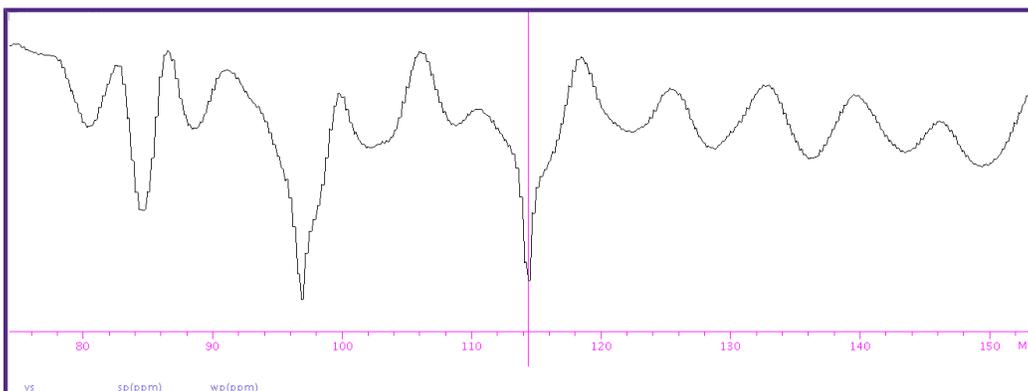
- 4) From the drop-down list next to **Tune frequency**, select your desired nucleus. The frequency will automatically update.
- 5) In the window next to **Span**, enter 80 and hit the enter key.
- 6) From the drop-down list next to **# Points**, select 1024.
- 7) Click on .
- 8) Click on . The following should appear in the Graphics Display screen:

## PROBE TUNING ON THE INOVA 600



**Figure 2.10:** The Qtune window and parameter panel after modifying the parameters to observe  $^{77}\text{Se}$  on the probe's low-band channel.

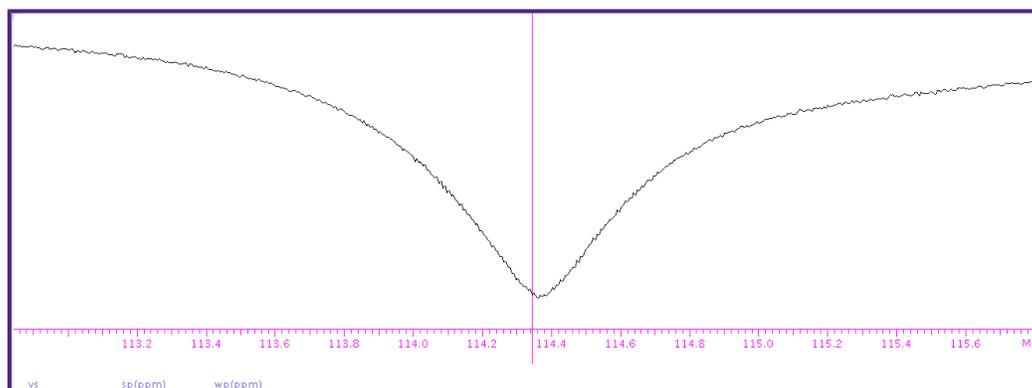
- 9) See Table 1.3 for the appropriate X-BAND, X-TUNE 1 and X-TUNE 2 values for your nucleus of interest.
- 10) Turn the X-BAND knob in whichever direction needed to move the indicator to the appropriate frequency range for your nucleus.
- 11) Turn the X-TUNE 1 and X-TUNE 2 knobs to the values shown in Table 1.3 for the nucleus of interest.



**Figure 2.11:** The Qtune window and parameter panel for the low-band channel after adjusting the X-TUNE 1 and X-TUNE 2 values to 26 and 25, respectively, in order to tune the probe to observe  $^{77}\text{Se}$ .

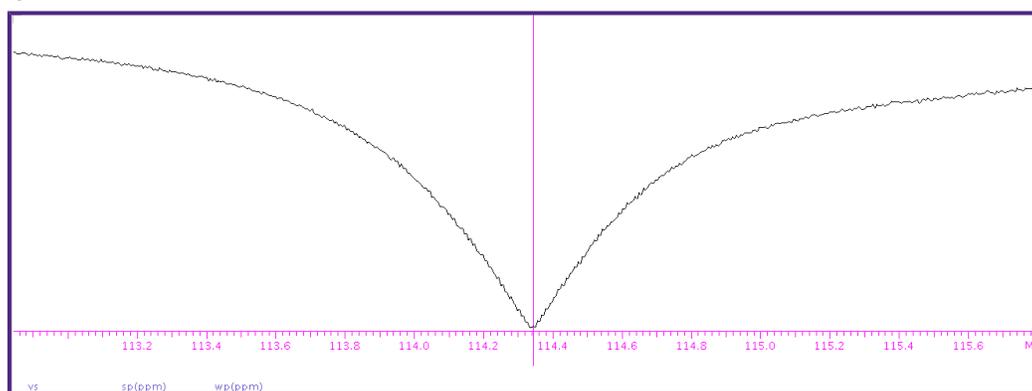
- 12) In the window next to **Span**, enter 3 and hit the enter key.

## PROBE TUNING ON THE INOVA 600



**Figure 2.12:** The Qtune window and parameter panel for the low-band channel after reducing the span to 3 MHz.

- 13) Turn the X-MATCH knob in some direction. If the dip moves downwards, continue turning the knob in the same direction until the dip touches the horizontal line. If the dip moves upwards, turn the knob the opposite direction until the dip touches the horizontal line.
- 14) Turn the X-TUNE 1 knob in some direction. If the dip moves towards the vertical line, continue turning the X-TUNE 1 knob in the same direction until the dip is centred about the vertical line. If the dip moves away from the vertical line, turn the X-TUNE 1 knob in the opposite direction until the dip is centred about the vertical line.
- 15) Iterate between the X-TUNE 1 and XMATCH knobs until the dip is centred about the vertical line and is touching the horizontal line.



**Figure 2.13:** The Qtune window and parameter panel for the low-band channel after fine tuning the X-MATCH and X-TUNE 1 knobs. The probe is now tuned correctly to observe  $^{77}\text{Se}$ .

- 16) Click on .
- 17) Disconnect the cable from the PROBE port by the TUNE INTERFACE window and re-connect the cable to the PROBE port on the high-band pre-amplifier.
- 18) Tune the high-band channel as described in section 2.1.1 or section 3.2.
- 19) Click on  to exit Q-Tune.

## PROBE TUNING ON THE INOVA 600

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- 20) Lock, shim and acquire your spectra.
- 21) When your experiments are complete, insert the  $\text{CDCl}_3$  sample and tune the low-band channel as described in section 3.

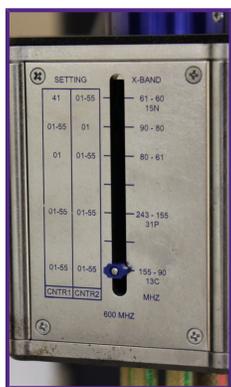
### 3. Re-tuning the Probe back to its Default Configuration

After completion of your experiment, the  $\text{CDCl}_3$  sample must be inserted, the low-band channel of the probe must be tuned back to  $^{13}\text{C}$  and the high-band channel back to  $^1\text{H}$ .

#### 3.1. Re-tuning the Low-band Channel

If you tuned both the low and high-band channels, you should re-tune the low-band channel first.

- 1) Eject your sample and insert the default  $\text{CDCl}_3$  sample.
- 2) On the low-band pre-amplifier, disconnect the cable from port labeled “PROBE” and attach it to the port labeled “PROBE” by the Tune Interface window.
- 3) Enter “qtune” in VNMRJ’s command line.
- 4) From the drop-down list next to **Tune frequency**, select **C13**. The frequency will automatically update.
- 5) In the window next to **Span**, enter 10 and hit the enter key.
- 6) From the drop-down list next to **# Points**, select 1024.
- 7) Click on .
- 8) Click on .
- 9) On the probe, turn the X-BAND knob until the indicator points to 155-90 (13C).



**Figure 3.1:** The X-BAND indicator on the Inova 600’s NMR probe after adjusting the X-BAND knob to select the  $^{13}\text{C}$  tuning range.

- 10) Turn the X-TUNE 1 knob and set the X-TUNE 1 value to 4.
  - 11) Turn the X-TUNE 2 knobs and set the X-TUNE 2 value to 4.
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## PROBE TUNING ON THE INOVA 600

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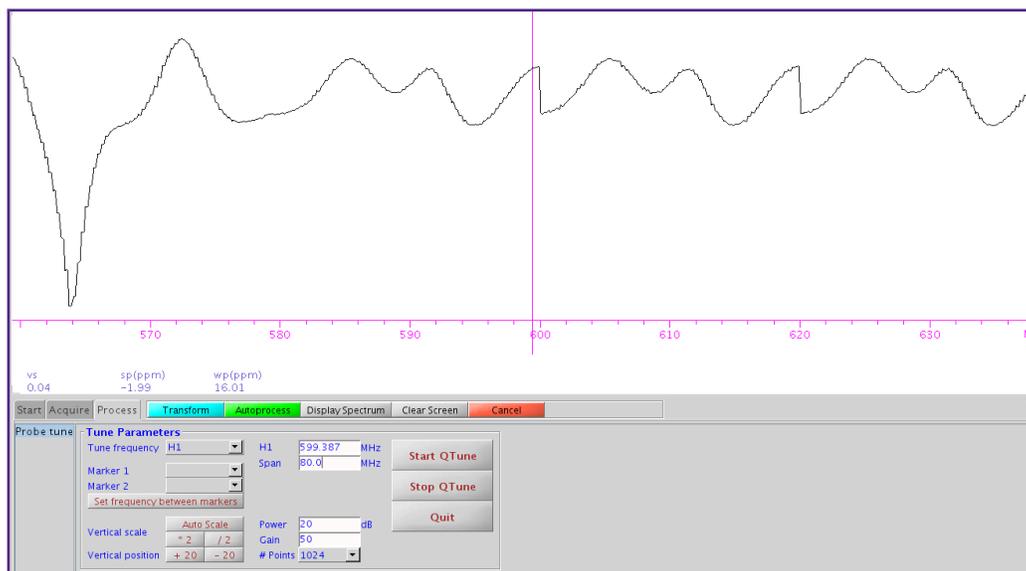
- 12) Turn the X-MATCH knob in some direction. If the dip moves downwards, continue turning the knob in the same direction until the dip touches the horizontal line. If the dip moves upwards, turn the knob the other way until the dip touches the horizontal line.
- 13) Iterate between the X-TUNE 1 and X-MATCH knobs until the dip is centred about the vertical line and is touching the horizontal line.
- 14) Click on .
- 15) Disconnect the cable from the PROBE port on the magnet leg and re-connect the cable to the PROBE port on the low-band pre-amplifier.
- 16) If necessary, tune the high-band channel as described in section 3.2. If not necessary, click on  to exit QTune.

### 3.2. Re-tuning the High-band Channel

If you tuned both the low and high-band channels, you should re-tune the low-band channel first (see section 3.1).

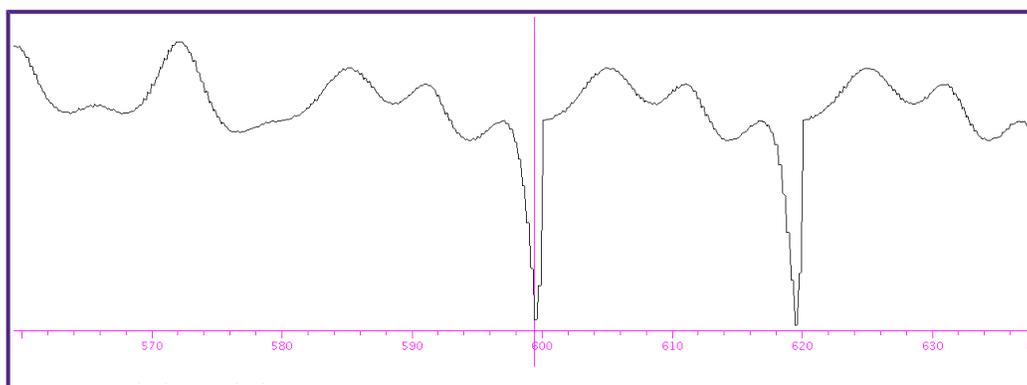
- 1) Eject your sample and insert the default  $\text{CDCl}_3$  sample. Skip this step if you just tuned the low-band channel as described in section 3.1.
  - 2) On the high-band pre-amplifier, disconnect the cable from port labeled “PROBE” and attach it to the port labeled “PROBE” by the Tune Interface window.
  - 3) Enter “qtune” in VNMRJ’s command line. This command brings up the tuning interface known as QTune. Skip this step if you just tuned the low-band channel as described in section 3.1.
  - 4) If necessary, from the drop-down list next to [Tune frequency](#), select **H1**. The frequency will automatically update.
  - 5) In the window next to [Span](#), enter 80 and hit the enter key.
  - 6) From the drop-down list next to [# Points](#), select 1024.
  - 7) Click on .
  - 8) Click on . The following should appear in the Graphics Display screen.
-

## PROBE TUNING ON THE INOVA 600



**Figure 3.2:** The Qtune window and parameter panel for tuning the high-band channel after setting the necessary parameters to tune  $^1\text{H}$ . Currently, the high-band channel is tuned to  $^{19}\text{F}$ .

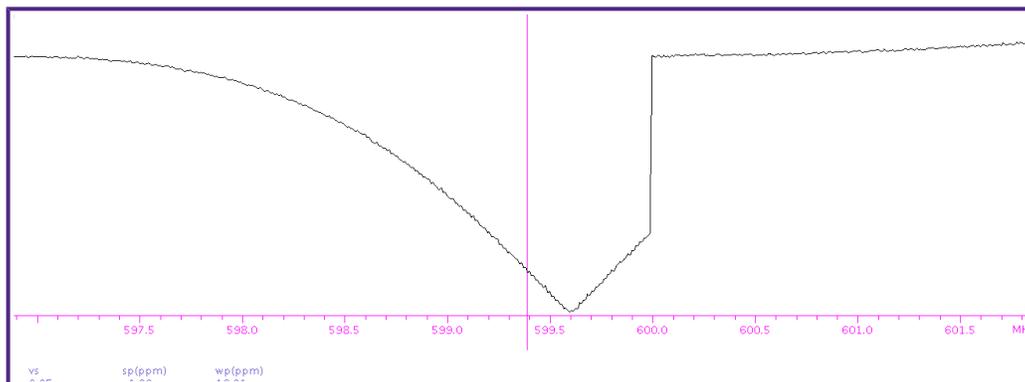
- 9) Turn the 1H-TUNE knob right (clockwise) and continue turning until the dip is close to the vertical pink line.



**Figure 3.3:** The Qtune window and parameter panel for tuning the high-band channel after adjusting the 1H-TUNE knob. The tuning dip is centered about the vertical pink line and is close to the horizontal axis.

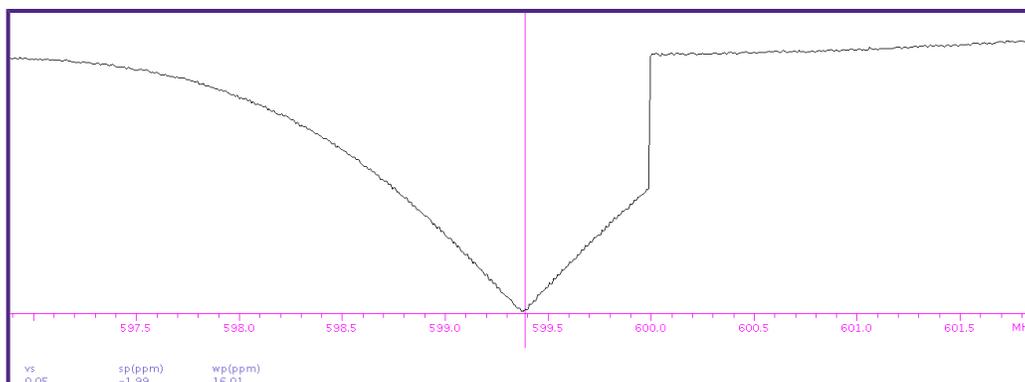
- 10) In the window next to **Span**, enter 5 and hit the enter key.

## PROBE TUNING ON THE INOVA 600



**Figure 3.4:** The Qtune window and parameter panel for tuning the high-band channel after initial adjustment of the 1H-TUNE knob and reduction of the span to 5 MHz.

- 11) Fine tune the adjustment of the 1H-TUNE knob until the dip is centred about the vertical line. If the dip rises off the horizontal axis, adjust the 1H-MATCH knob to bring the dip back down to the horizontal axis. Continue iteratively adjusting the 1H-TUNE and 1H-MATCH knobs until complete.



**Figure 3.5:** The Qtune window and parameter panel for tuning the high-band channel after final adjustment of the 1H-TUNE and 1H-MATCH knobs.

- 12) Click on .
- 13) Disconnect the cable from the PROBE port on the Tune Interface of the magnet leg and re-connect the cable to the PROBE port on the high-band pre-amplifier.
- 14) Click on  to exit QTune.