Quantum-I*Plus* NMR Spectrometer

STM 300~600MHz Probe User Manual

WMR/3-YF/P-T/SC/B0

Q.One Instruments Ltd.

Nov 2022

Quantum-I^{Plus} NMR Spectrometer STM 300~600MHz Probe User Manual

Version: B0

No.: WMR/3-YF/P-T/SC/B0

Supporting documents:

SpinStudioJ User Manual (WMR/3-YF/P-C/YHCZ/B0)

SpinStudioJ Command and Parameter Manual (WMR/3-YF/P-C/MLCS/B0)

Installation Preparation Instructions for Quantum-I^{Plus} NMR Spectrometer (WMR/3-YF/P-C/ZB/C0)

Quantum NMR Spectrometer Health and Safety Notice(WMR/3-YF/P-C/JA/A0)

Author:Chai Shigan, Li Zhenggang

Modification history:

A0: 2022.2, initial version.

B0: 2022.11, 2nd version.

Q.One Instruments Ltd. reserves the right to change the technical specifications. The contents of this specification may be updated without informing the customer.

WMR, Q.One Inst, Q.One is the abbreviation of Q.One Instruments Ltd.

WNMR, BIXI and Quantum are the model codes of NMR spectrometer products of Q.One Instruments Ltd. WNMR-I, BIXI-I and Quantum-I refer to the same products.

Technical Support and Services

Service concept

Your satisfaction is our greatest success!

Scope of Service

1) Installation and maintenance of NMR spectrometer.
2) Customization of NMR Spectrometer.
3) User training (experimental operation, strong field safety, high-order nuclear magnetic resonance, etc.).
4) Spectrometer calibration and maintenance services during the life cycle of NMR spectrometer.
5) During the life cycle of NMR spectrometer, the following free services are provided:
   - SpinSdudioJ, the control and data processing software, is upgraded free of charge.
   - Major defects or major safety defects of the functions agreed in the contract shall be handled free of charge.

Please help us do better!

We are committed to providing excellent NMR spectrometers to continuously meet your current and future needs in using NMR spectrometers. Your feedback is very important to us. If you find any problems with spectrometer, software or manuals, or have any suggestions for improvement, please feel free to contact us.

Product consultation, sales, repair and technical support

Q.One Instruments Ltd..
Address: No. 128, Guanggu 7th road, East Lake high-tech developing zone, Wuhan city, Hubei province
Postcode: 430075
Tel: 0086 27 8177 3999

Security Summary

All manuals of Quantum series NMR spectrometer products use the following warning or prompt icons:

WARNING Warning: this icon is used to prompt you to strictly adhere to these rules. For breach of these principles, it may result in personal injury or death, or significant property damage.

CAUTION Caution: this icon is used to prompt you for these considerations, and failure to comply with these prompts may result in device damage or loss of data.

Prompt: this icon is used to indicate information you can refer to.

Contents

Technical Support and Services III
Security Summary IV
1. Introduction 1
2. Contents of delivery 1
3. System overview 5
3.1 Working with VNA and PLC 5
3.2 Working with PLC, but without VNA 6
4. Probe description 7
4.1 Overview 7
4.2 Probe size 8
4.3 RF channels 10
4.4 Temperature sensors 12
4.5 VT system 12
4.6 Flush gas system 12
4.7 Gradient system 12
5. Probe installation 13
5.1 Probe mounting 13
5.2 Connecting cables 14
6. Operation parameters and application 16
6.1 Power limits 16
6.2 VT gas and heater 17
6.3 Flush gas 18
7. SpinstudioJ settings 18
7.1 With PLC and VNA 19
7.2 With PLC, but without VNA 20

STM 300~600MHz Probe User Manual

1. Introduction

The purpose of this manual is to guide users and service technicians through the installation process, maintenance, service, and overall use of a Q.OneTec NMR probe.

With a proper installation and use, the probe will work at peak performance and damages are prevented. Carefully read all relevant chapters before working with the probe.

Q.One reserves the right to change specifications of the products at any time. It is Q.One's policy to implement state-of-the-art technology to improve the products. Every economically reasonable effort has been made to keep this manual up to date.

2. Contents of delivery

Q.OoneTec NMR probes are delivered in a solid cardboard case containing the probe, probe documentation and the standard items related to the probe.

Figure 1: Delivery contents: Case with probe and probe accessories

Table 1. Delivery content in solid cardboard case

1	NMR Probe	STM 300/400/500/600
2	Accessories	VT gas adapter，Coaxial sleeve, Coaxial ring，Dust cap, Probe Mounting adapter
3	Documentation	This manual

Figure 2. The appearance of Q.OneTec NMR probe

Figure 3. Probe accessorie, VT gas adapter

The adapter has two sizes, which are suitable for 6mm and 8mm air tubes respectively. Please insert the corresponding air tubes into it when controlling temperature regularly, Or insert the outlet end of cryogenic accessories into it when we need control low temperature.

Figure 4. Coaxial sleeve with upper tube

This coaxial sleeve is very important, it can make probe and upper tube in a line with vertical direction. If the coaxial sleeve's size is not proper, the spinning of sample in the upper tube maybe not stable. So, different probe should has it's own coaxial sleeve. When the suitable coaxial sleeve is selected, we can lock it into the upper tube with two screws.

Figure 5. Coaxial rings with shimcoil(2P)

Almost all probes of Q.OneTec have the similar sizes with external diameter, but the internal diameter of shimcoils may have different sizes , as the bore of magnet usually has 51 or 54mm size. If the bore of magnet is 54mm, the internal diameter of shimcoil is smaller than 51mm's. In order to let the probe don't wobble in the shimcoil, two plastic coaxial rings are used. The position of coaxial rings in the probe is shown in the picture above. If the bore of magnet is 51mm, we needn't use these two coaxial rings.

Figure 6. Dust cap

The dust cap is used only in the storage and delivery of probe, it can protect the probe from dust and moisture. It must be taken off when the probe is installed in the magnet.

Figure7. Probe mounting adapter/ supporting bracket

The probe mounting adapter/supporting bracket is used to fix the probe to shimcoil. In order to prevent the probe fall down or moving, we should tighten the two screws in the ears of it . And we can also adjust the position of probe in order to let the probe's RF coil and the shimcoil's center are in the same horizontal position.

3. System overview

3.1 Working with VNA and PLC

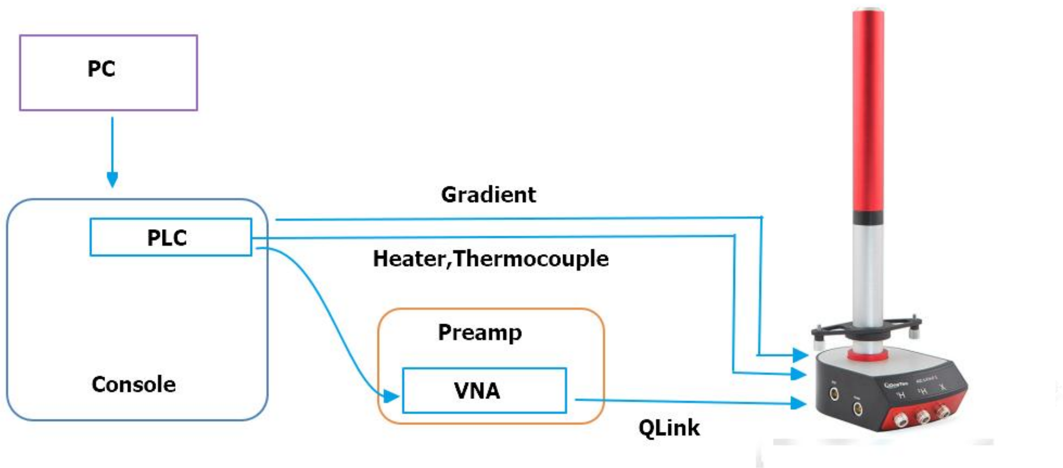

Figure 8. System overview with VNA and PLC

PLC is responsible for tune/match and controlling temperature of probe. VNA board, which is in the bottom of preamplifier, has the function of RF of transmit and receive. It can get S11 parameter of current channel and send the S11 data to PLC. The CAN Bus communication is used between PLC and VNA as it has high efficiency. It needn't communicate with others, so the tune/match is very fast by this way.

3.2 Working with PLC, but without VNA

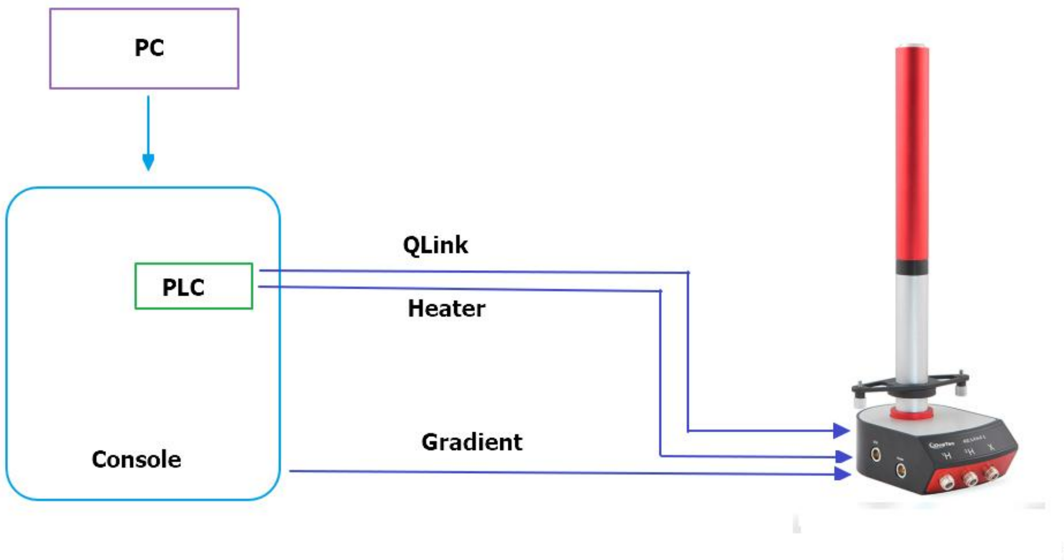

Figure 9. System overview with PLC, but without VNA

It is the traditional tune/match way to work only with PLC. PLC is responsible for tune/match and controlling temperature of probe. As it has no VNA board in the preamplifier, so PLC can only get S11 data from console.

4. Probe description

4.1 Overview

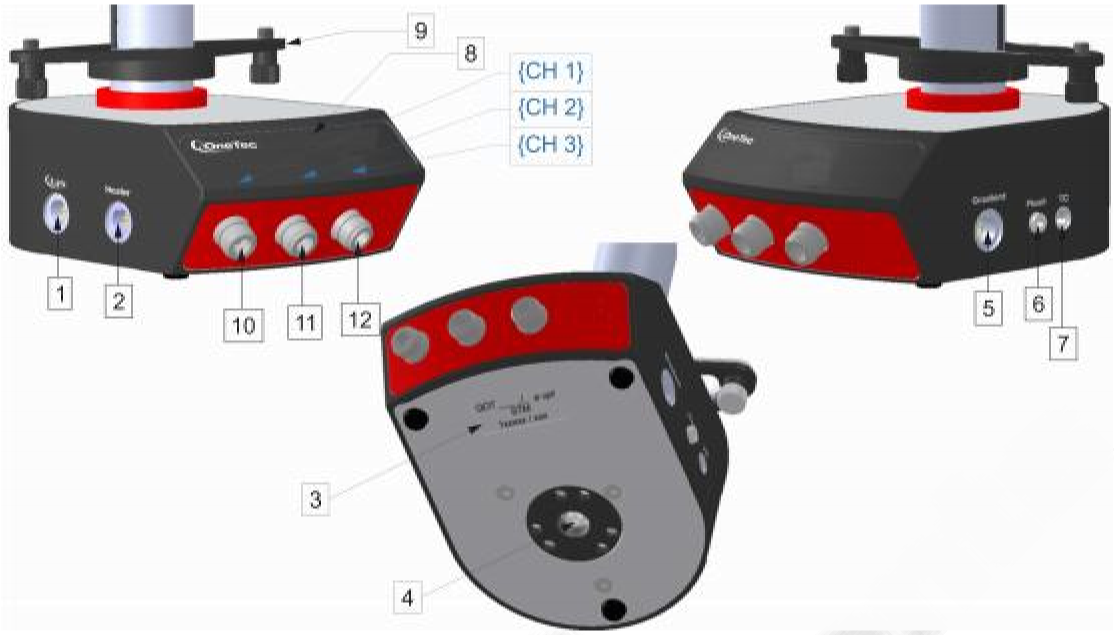

Figure 10. Q.OneTec STM probe in detail

The individual components and connections of the Q.OneTec STM probe are shown in figure 10 and explained in the next table.

Table 2. Description of probe parts

1	Q.Link socket for STM	Connects the probe with the Q.Link cable to the STM unit. This socket is equipped with an LED. If the LED is blinking, the probe is connecting to the STM unit. If the LED is permanently on, the probe is ready for operation.
2	Heater socket	Connects the heater cable to the spectrometer.
3	Label	Indicates the name, part number, and serial number of the probe. For example: **QOT 400 X,F/H,F 5** **STM** **140006 /001**
4	VT Gas socket	Connects VT gas adapter with dry gas.
5	Gradient socket	Connects the gradient cable.

6	Flush Gas socket	Connects a 4 mm tubing with dry gas pressurized with more than 1 bar. The usage of flush gas is mandatory.
7	Thermocouple socket	Reserved for thermocouple cable .
8	Probe code	The current probe code, for example, **400 X,F/H,F 5,** is printed in this area of the front plate starting with **400(300/400/500/600)** (base frequency) followed by the first RF element broad band channel **X, F** (inner coil, range ^{19}F, ^{31}P - ^{15}N) and the second RF element **H,F** (outer coil, ^{1}H,^{19}F). The sample diameter for the probe is **5** mm.
9	Supporting bracket	The bracket, as shown in Figure 11, is functionally responsible for the position and the orientation of the probe in the shim system. The position of the bracket is preset at factory to **-4 to -6 mm.** Fine adjustments in orientation and position are explained in chapter 5.1.
10	RF socket H channel	Connects the ^{1}H RF cable to the channel 1 of preamp.
{CH1}	Channel label 1	The channel label 1 is always **H**, if present. If channel 1 can be additionally tuned to e.g. ^{19}F, it will not be indicated.
11	RF socket D channel	Connects the ^{2}H (=LOCK) RF cable to the D channel of preamp.
{CH2}	Channel label 2	The channel label 2 is standard for LOCK, if present. The possible labels are **D** or **F**. If the probe has no lock, the next circuit will be at this position, if present.
12	RF socket X channel	Connects the X channel RF cable to the channel 3 of preamp.
{CH3}	Channel label 3	The channel label 3 is standard for X, if present. The pos- sible labels are **X** or abbreviation for a selective nucleus.

4.2 Probe size

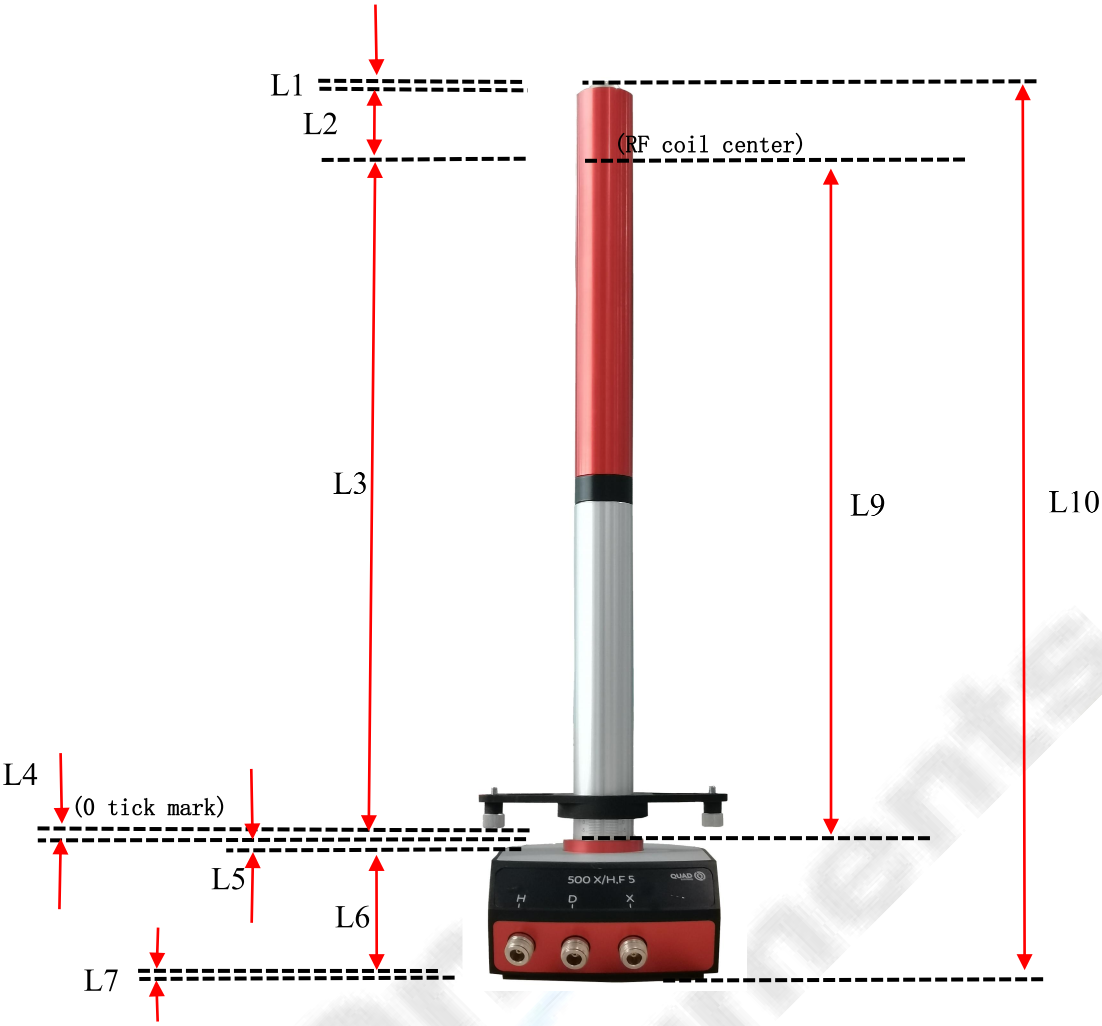

Figure 11. Probe size

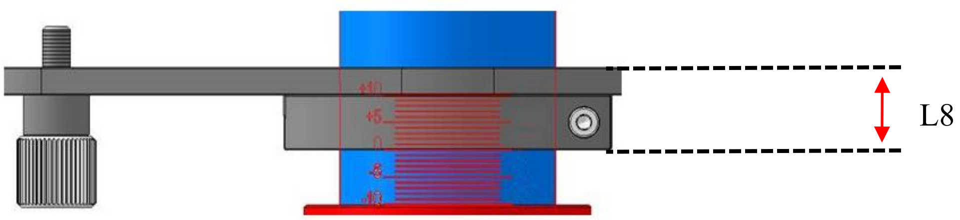

Figure 12. Probe mounting adapter’s size

Some important sizes of probe are labeled in the Figure 11 and 12, and the result is shown in table 3. L1 and L2 are important for NMR sample depth. It is easier to get a better shim if we assure that the NMR sample's center is consistent with the RF coil's center. L3 is important for when we install the probe into the shimcoil, this data can help us calculate the position of the probe.

Table 3. Probe size

Size（mm） / Probe	L1	L2	L3	L4	L5	L6	L7	L8	L9	L10
STM 300	10	49.5	374.5	10	10	67	3.3	15	384.5	524.3
STM 400	10	49.5	374.5	10	10	67	3.3	15	384.5	524.3
STM 500	10	49.5	444.5	10	10	67	3.3	15	454.5	594.3
STM 600	10	49.5	499.5	10	10	67	3.3	15	509.5	649.3

4.3 RF channels

Usually, the probe has 3 RF channels and can be tuned to the following nuclei (base frequency ^{1}H of 400.0 MHz):

Table 4. RF channels of H,F/X series

Channel number	Channel label	Nuclei	Remarks
1	H	^{1}H,^{19}F	^{1}H and ^{19}F channel
2	D	^{2}H	The deuterium channel is the dedicated for locking and can also be used for direct measurement.
3	X	^{15}N ...^{31}P	The range between 54.0 MHz and 75.0 MHz(for 400MHz) is not tunable. The nuclei ^{153}Eu, ^{137}Ba, ^{175}Lu, ^{181}Ta, ^{123}Sb,^{133}Cs, ^{138}La, ^{17}O, ^{9}Be and ^{139}La are therefore excluded.

Table 5. RF channels of H/X,F series

Channel number	Channel label	Nuclei	Remarks
1	H	^{1}H	^{1}H channel
2	D	^{2}H	The deuterium channel is the dedicated for locking and can also be used for direct measurement.
3	X	^{15}N....^{31}P, ^{19}F	The range between 54.0 MHz and 75.0 MHz(for 400MHz) is not tunable. The nuclei ^{153}Eu, ^{137}Ba, ^{175}Lu, ^{181}Ta, ^{123}Sb,^{133}Cs, ^{138}La, ^{17}O, ^{9}Be and ^{139}La are therefore excluded.

Table 6. RF channels of H,F/X,F series

Channel number	Channel label	Nuclei	Remarks
1	H	^{1}H,^{19}F	^{1}H and ^{19}F channel
2	D	^{2}H	The deuterium channel is the dedicated for locking and can also be used for direct measurement.
3	X	^{15}N ...^{31}P, ^{19}F	The range between 54.0 MHz and 75.0MHz(for 400MHz) is not tunable. The nuclei ^{153}Eu, ^{137}Ba, ^{175}Lu, ^{181}Ta, ^{123}Sb,^{133}Cs, ^{138}La, ^{17}O, ^{9}Be and ^{139}La are therefore excluded.

As the channel 3 must have a large frequency range, from ^{15}N to ^{31}P, even to ^{19}F. It's very hard to have so large continuous frequency. So, usually, there are 3 switch position for channel 3. For H,F/X,F and H/X,F series, there is a ^{19}F nuclei in channel 3, ^{15}N is in the position 1, ^{13}C and ^{31}P are in the position 2, and ^{19}F is in the position 3. For H,F/X series, there is no ^{19}F nuclei in channel 3, ^{15}N is in the position 1, ^{13}C is in the position 2, and ^{31}P is in the position 3.

Besides, 300, 500, 600MHz STM probe's frequency range is similar to 400MHz.

4.4 Temperature sensors

This probe is equipped with four temperature sensors:

- Thermocouple 1: Sensor closest to the sample (VT temperature)
- Thermocouple 2: Overheating protection sensor
- PT100 1: Temperature of the tuning and matching parts
- PT100 2: Overheating protection sensor (only for Agilent-Varian)

4.5 VT system

The sample temperature system, including temperature sensors, dewar, heater and VT gas, is regulated by PLC or Eurothermo 3508. For the regulation a constant flow of dry gas or nitrogen needs to be provided.

4.6 Flush gas system

In order to keep the sensitive parts in the probe free of contamination (e.g. moisture) and at ambient temperature, a constant flow of dry gas or nitrogen has to be provided.

4.7 Gradient system

The probe is equipped with a gradient system which can be used in experiments and for shimming. The gradient strength is about 5 G/cm*A.

5. Probe installation

5.1 Probe mounting

Insert the probe into the shim system and tighten the hand screws completely to the bottom plate of shim system(figure 13). Q.One recommend to do course adjustments of supporting bracket's position before inserting the probe into the shim system.

In the next step, the height and orientation of the probe can be adjusted by changing the position of the probe adapter. Carefully loosen the Allen screw of the bracket slightly (figure 14), orient the probe, if needed, and guide the probe to the proper position. Then tighten the Allen screw again.

Figure 13. Insert the probe and tighten the two hand screws.

The correct position is normally **-4 mm** in AS400 magnet and standard shim systems. In order to get the exact position, we can make a shim map with SpinstudioJ, and we can judge whether the center of probe is coincident with the shim coil and then finely adjust the probe. This step require an engineer must has much experience. Then record this actual position after all adjustments from the scale laser inscribed into probe body at the bottom of the bracket (figure 14).

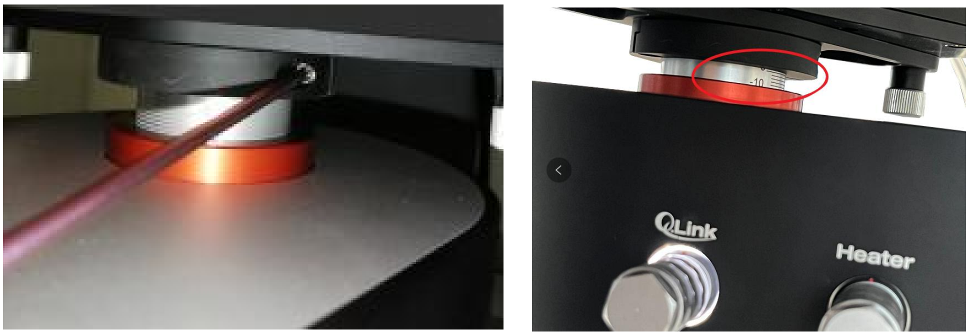

Figure 14. Adjust the height of the probe.

CAUTION
The use of magnetic tools in the vicinity of the magnet can be dangerous and injury or the permanent damage to the magnet can occur. So nonmagnetic tools are strongly recommended.

5.2 Connecting cables

After the probe is correctly mounted, all cables should be connected, including high power RF cable, Q.Link, Heater, Gradient, VT gas and Flush gas rubber tube. As shown from Figure 15 to 18.

Figure 15. Connection of RF cables

Figure 16. Connect the Gradient cable and the flush gas(4mm)

Figure 17. VT gas connection

Figure 18. Connect Q.Link and Heater cable

6. Operation parameters and application

The descriptions and values given in this chapter apply to the probe part number 140008 with the serial number 001. Tiny difference may exist in each probe.

6.1 Power limits

The power limits in Watt are as follows:

Table 7. Peak power levels in Watt

Nucleus	Peak Power [W]
^{1}H	25
$^{19}F_{(^{1}H\ coil)}$	30
^{19}F[X coil]	70
^{31}P	80
^{13}C	90
^{15}N	180
^{2}H[LOCK]	20

NOTICE

Do not exceed these power limits. Higher power values might damage the probe.

6.2 VT gas and heater

It is recommended to use a flow of 500 l/ h of VT gas. Usually, you can use dry air as VT gas. But if you are using a high sensitive probe, in order to get a good shim, you can use nitrogen. If using a cooler for the VT gas, make sure the VT gas meets the specification of the cooler.

NOTICE

Never turn on the probe heater without using VT gas. The heater might overheat and damage parts in the probe.

6.3 Flush gas

Flush gas with a flow of 5-10 LPM should always be provided. Generally, the dry air can be used as flush gas, but in order to get higher sensitivity and better shim, it's recommend to use nitrogen as flush gas. The temperature of the flush gas needs to be at room temperature. Flush gas not only prevents from overheating but also keeps the probe free from contamination or background signal due to moisture. It's important to keep the temperature of flush gas between 10°C and 30°C to ensure the reliability of the probe functionality.

NOTICE
Always use flush gas in order to prevent contamination (e.g. moisture) in the probe.

7. SpinstudioJ settings

In this section , we use the SpinstudioJ Ver2.6.0 as the example, it has the similar settings in different versions of SpinstudioJ.

Q.One usually uses complex tune algorithm, so parameter "*doCalPhase*" should be set to 1. If we use PLC to tune and match, we should set parameter "*use OPCUA*" to 1. If there is a VNA board in the bottom of preamplifier, which can make tune/match process in a fast way, we should set parameter "*ExtentVNA*" to 1. If the tune algorithm is controlled by PLC, set parameter "*method*" to 0. Otherwise, if the tune algorithm is controlled by SpinstudioJ, set parameter "*method*" to 1.

7.1 With PLC and VNA

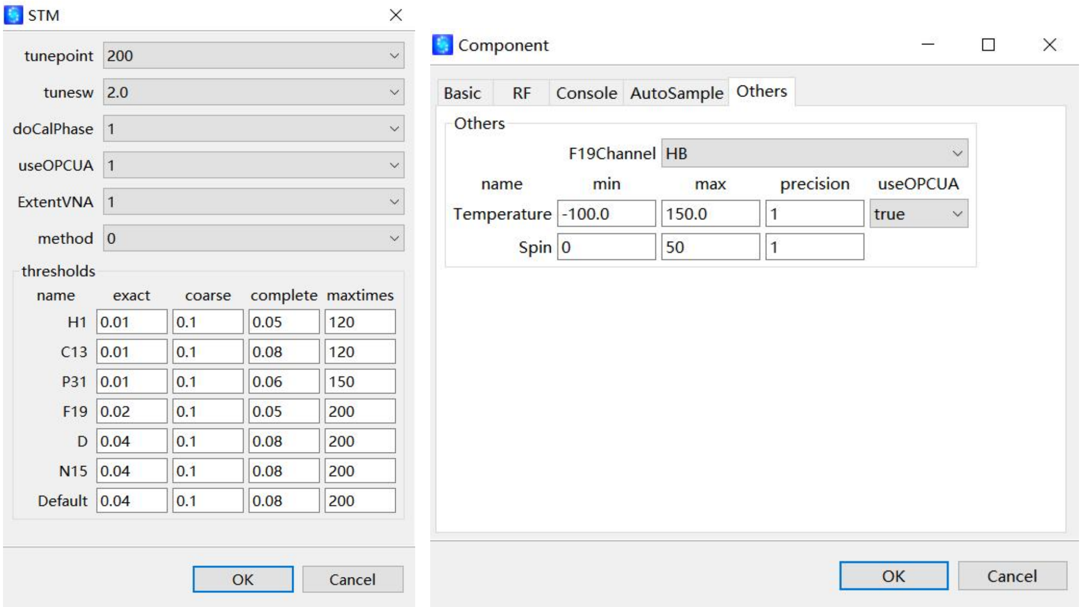

Figure 19. Settings with PLC and VNA

Q.One uses complex tune algorithm, so parameter “*doCalPhase*” should be set to 1. As there is a PLC and a VNA board in the bottom of preamplifier, so we should set parameters “*useOPCUA*” and “*ExtentVNA*” to 1. And the tune algorithm is controlled by PLC, so parameter “*method*” should be set to 0. The temperature is controlled by PLC, so set parameter “*useOPCUA*” to true as shown in the picture above(right).

7.2 With PLC, but without VNA

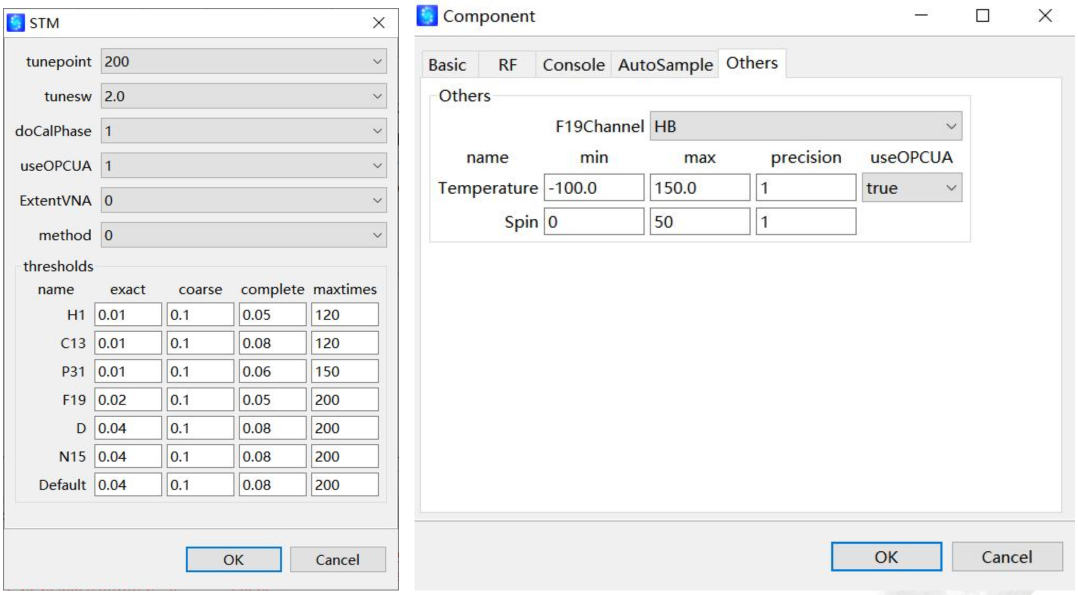

Figure 20. Settings with PLC ,but without VNA

This is the traditional way of tuning. As it has PLC, but has no VNA board in preamplifier, so we set parameters "*doCalPhase*", "*useOPCUA*" to 1, and set parameters "*ExtentVNA*", "*method*" to 0. The temperature is controlled by PLC, so set parameter "*useOPCUA*" to true as shown in the picture above.