Variable Temperature Operation AVIII 400 MHz Spectrometer

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AVIII 400 User Guide: Variable Temperature Operation

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PREFACE

Purpose of This Document

This user guide aims to familiarize you with variable temperature operations of the AVIII 400 NMR Spectrometer. The document does not cover all the basic information and details of the application.

Intended Audience

This document is intended for users of AVIII 400 NMR spectrometer who are familiar with the basic operation of the spectrometer operation. Please refer to "AVIII 400 User Guide" for the basic operations of AVIII 400 NMR spectrometer. This document is *not* intended to replace any form of official Bruker user guides or related Bruker documents.

1 Introduction

The AVIII400 NMR spectrometer is equipped with a 400 MHz Bruker BBFO probe. This instrument is suitable for analyzing the time and air sensitive samples. It is also suitable for a study of reaction kinetics of chemical reactions and for a variable temperature (VT) NMR experiment. Prior user training is required to operate the spectrometer. Additional training is available for the VT experiment. The web reservation is required to use AVIII 400 NMR spectrometer. The spectrometer is running on a Bruker TopSpin software.

1.1 Safety

Iron and other ferro magnetic objects must **NOT** be brought into the vicinity of the magnets. The strong magnetic fields may erase the information of credit card, student ID card, and other magnetic media. *No admission for persons with pacemakers and other metallic implants*.

1.2 NMR Sample Preparation

- Always use clean and dry sample tubes
- Use medium to high quality sample tubes
- Filter sample solution if particles are present
- Keep the sample volume approximately 0.5 0.6 mL.
- Wipe the sample tube clean before inserting into magnet
- Use a ceramic spinner for variable temperature operation
- Use a sample depth gauge to adjust position of sample spinner

1.3 NMR Probe

The AVIII 400 MHz NMR spectrometer is equipped with a BBFO probe. The BBFO probe has two radio frequency channels (¹H and X-nucleus) with a built-in capability of automatic tuning and match (ATM). The high frequency channel is tuned to for ¹H observe and decoupling. The observe channel (X) covers a frequency range from ¹⁰⁹Ag to ¹⁹F. The probe temperature range is from -150 to 150 °C. The commonly used acquisition files for this probe are prefixed with "*bbfo*".

2 High Temperature Operation

The high temperature operation does not involve the change of the VT apparatus and accessories. However, it requires a change of the supply VT gas from the compressed air to the in-house nitrogen gas.

2.1 Preparation

2.1.1 Switch the Gas Supply to Nitrogen Gas:

It is highly recommended to use nitrogen gas for high temperature operation due to possible oxidization of probe electronic components occurring at high temperatures. The compressed air gas supply setup for room temperature operation is shown in Figure 1. In this setup, switch **S1** is **on**, **S2**, is **off**, and **S3** is pointing to the left (compressed air). To switch the gas supply to the nitrogen gas, first turn on **S2** by turning it 90° down, then turn **S3** 180° to the right, and finally turn off **S1**, as shown in Figure 1.



Compressed Air Supply

Nitrogen Gas Supply

Figure 1: Switch Gas Supply from Compressed Air to Nitrogen Gas

2.1.2 Spinner

Use a *ceramic spinner* to avoid melting of regular blue spinner for the high temperature operation.

2.1.3 Room Temperature Experiment

It is highly recommended to obtain an NMR spectrum at the room temperature as a baseline spectrum before altering the sample temperature. The procedures of acquiring a routine 1D NMR spectrum has been described in "AVIII 400 User Guide"

2.2 High Temperature Setup

 In the command line of the TopSpin Software Interface, type *edte* and a Temperature Control Suite window appears as shown in Figure 2. The Temperature Tab contains VTU State (On or Off buttons), *Sampler temperature*, *Target Temperature* with a Set button. It also shows the probe gas supply with adjustable Target Gas Flow

Т			Temperature	Control Suite		_ • ×						
Temperature	Monitoring Record	Correction Self	tune Configurat	ion Log Help								
	On Off VTU State: 🛇 On											
Channel Regulation State Stability Sample Temperature Target Temperature Heater Powe												
5 mm PABBO B	1 B/19F-1H/D Z-GRD Z	🕑 Steady	Stable since 14:20:25 08 Nov 2012 ?	Corr. 299.6 K (Measured value 299.6 K)	Corr. 299.6 K (123.0 K423.0 K) Set	0.2 % (max. 29.7 % of 195.8 W)						
		State	Gas Flow	Target Gas Flow	Standby Gas Flow							
Ρ	robe Gas	🕑 Steady	550 lph	550 lph Set	540 lph Set							
Set												
VTU: On 🕑 Samp	ole Temperature: <mark>Corr.</mark>	299.6 K Probe Regi	ulation: Steady 오 T	une: Misfit 😵 Recording: Off P	robe: 5 mm PABBO BB/	19F-1H/D Z-GRD Z104450/04						

Figure 2: Temperature Control Suite

- 2. Make sure *Target Gas Flow* is set to 550 lph. If *Target Gas Flow* is set at a different value, click on **Set** to change *Target Gas Flow* to 550 lph.
- 3. Click on Correction Tab in the Temperature Control Suite (Figure 3), select an appropriate correction parameter settings according to the desired target temperature. In this example, a file named "*bbfoht_305.1-348.1*" is selected. Click on **Set** button to assure the correction temperature calibration parameter are loaded for the high temperature run.

Note: user may also create a temperature calibration curve. An ethylene glycol sample is used for high temperature calibration. Use standard proton parameter and set rg to 1. Take a proton spectrum and measure the chemical shift difference between two peaks. Use a calibration curve or online tools to determine the actual

temperature. Please consult the NMR spectroscopist for accurate temperature calibration.

				Te	mperature Cor	itrol Su	ite				
Temperature	Monitoring	Record	Correction	Self tune	Configuration	Log	Help				
emperature co	rrection										
Jse temperature correction if you want to display the real sample temperature instead of the probe temperature sensor value. Ilease check the manual how to perform temperature measurements with NMR (to determine the real sample temperature).											
lote: Temperature correction is not applied to temperature limits (safety checks).											
✓ Enable temperature correction with these values											
Name:		RT_510_N	loCorrection								
Probe:		5 mm PAE	BO BB/19F-1	H/D Z-GRD	104450/0412						
Temperature	range [K]:	273 - 373									
Slope:		1									
Offset:		0									
Comment:		no correct	tion is applied								
vailable correc	tion settings										
△ Nan	ne			Probe			Femperature	Range	Slope	Offset	Comment
HT_670_22.3_	etgP	5 mm PAB	BO BB/19F-1	lh/d Z-gri	O Z104450/041	.2 3	03.00 - 390.0	0 0.	914826	24.984227	11May2012
T_510_NoCor	rection	5 mm PABE	80 BB/19F-1H	/D Z-GRD Z	104450/0412	2	73 - 373	1		0	no correction
bfoht_305.1-	348.1_5	5 mm PABE	80 BB/19F-1H	/D Z-GRD Z	104450/0412	3	05.10 - 348.1	.0 0.	844794	49.465422	
bfolowtemp2	73-233	5 mm PABE	80 BB/19F-1H	/D Z-GRD Z	104450/0412	2	73.1 - 233.1	0.	767754	68.647025	
t243-273_ntl		5 mm PABE	80 BB/19F-1H	/D Z-GRD Z	104450/0412	2	43.00 - 273.0	0 0.	733496	77.376528	new transfer l
									1	lew Edit	Set Delet
	I. T			Denvio	a		N	off Deck of			7 CPD 7104454

Figure 3: Select Appropriate Temperature Correction Parameter File

4. Click on Self Tune Tab (Figure 4), select an appropriate self-tune file. In this example, we use "*bbfo_550_313K*" as the self-tune file. Click on Restore to Channel 1 to activate the adaptive temperature regulation.

Note: a self-tune file contains PID parameters pre-stored to regulate sample temperature to a selected target temperature. One can generate a self-tune file by click on *Start* button in the Start Self Tune column after sample temperature is reached to the target temperature. It takes 3 to 5 minutes for self-tuning.

		Temperature	e Control Suite								
Temperature Monitoring Record Correction Self tune Configuration Log Help											
elftune											
vecute self tune to improve the regulation canabilities of the VTU											
u can self tune each channel independer	ntly (select self tune for the appropriate	channel) or self tune	all available channe	ls simultaneously (select self tune all	channels).					
o save the self tune parameters for a defined temperature, gas flow, probe and sensor press the "Get" button of the desired channel and enter a name for the settings.											
Channel	Sensor	Start self tune	Stop self tune	Get self	tune parameters	View self tune parameters					
All		Start	Stop								
1 5 mm PABBO BB/19F-1H/D Z-GRD Z.	adapter connection 1	Start	Stop		Get	View					
						Restore to channel 1 Dele					
vailable self tune settings											
Name	Probe		△ Temperature [K]	Flow [lph] S	ensor Chiller	Comment					
bfo_lt_13_470_243.1	5 mm PABBO BB/19F-1H/D Z-GRD Z10	4450/0412	243.1	545.000 1	None	low temp 13% estimate 470 L/H 243.1 K					
bfo lt 10 446 233 0	5 mm PABBO BB/19F-1H/D Z-GRD Z10	4450/0412	246.3	550.000 1	None	new transfer line					
0102112402110240010											
ofo_lt_12_446_273.1	5 mm PABBO BB/19F-1H/D Z-GRD Z10	4450/0412	273.1	545.000 1	None	low temp 12 % estimated 446 L/H					
ofo_lt_12_446_273.1 ofo_rt_545_300.0	5 mm PABBO BB/19F-1H/D Z-GRD Z10 5 mm PABBO BB/19F-1H/D Z-GRD Z10	4450/0412 4450/0412 3	273.1 302.9	545.000 1 550.000 1	None None	low temp 12 % estimated 446 L/H compressed air at 300 K					
bfo_lt_12_446_273.1 bfo_rt_545_300.0 bfo_ht_545_303.1	5 mm PABBO BB/19F-1H/D Z-GRD Z1C 5 mm PABBO BB/19F-1H/D Z-GRD Z1C 5 mm PABBO BB/19F-1H/D Z-GRD Z1C	4450/0412 4450/0412 4450/0412	273.1 302.9 303.1	545.000 1 550.000 1 545.000 1	None None None	low temp 12 % estimated 446 L/H compressed air at 300 K high temp ethylene glycol 303.1					
bfo_lt_12_446_273.1 bfo_lt_1465_300.0 bfo_ht_545_303.1 bfo_ht_550_305.1	5 mm PABBO BB/19F-1H/D Z-CRD Z1C 5 mm PABBO BB/19F-1H/D Z-CRD Z1C 5 mm PABBO BB/19F-1H/D Z-CRD Z1C 5 mm PABBO BB/19F-1H/D Z-CRD Z1C	4450/0412 4450/0412 4450/0412 4450/0412	273.1 302.9 303.1 305.1	545.000 1 550.000 1 545.000 1 550.000 1	None None None None	low temp 12 % estimated 446 L/H compressed air at 300 K high temp ethylene glycol 303.1 high temp 550 L/H					
bfo_lt_12_446_273.1 bfo_rt_545_300.0 bfo_ht_545_303.1 bfo_ht_550_305.1 bfo_ht_550_383.1	5 mm PABBO BB/19F-1H/D Z-GRD Z1C 5 mm PABBO BB/19F-1H/D Z-GRD Z1C	4450/0412 4450/0412 4450/0412 4450/0412 104450/0412	273.1 302.9 303.1 305.1 333.1	545.000 1 550.000 1 545.000 1 550.000 1 550.000 1	None None None None None	low temp 12 % estimated 446 L/H compressed air at 300 K high temp ethylene glycol 303.1 high temp 550 L/H high temp ethylene glycol 550 L/H 10 %					
bfolt.12.446.273.1 bfolt.12.446.273.1 bfolt.545.303.0 bfolt.550.305.1 bfolt.550.3331 bfolt.650.3331	5 mm PABBO 88/19F-1H/D Z-GRD Z1C 5 mm PABBO 88/19F-1H/D Z-GRD Z1C	4450/0412 4450/0412 4450/0412 4450/0412 104450/0412 4450/0412	273.1 302.9 303.1 305.1 333.1 363.0	545.000 1 550.000 1 545.000 1 550.000 1 550.000 1 670.000 ad	None None None None None Iapte None	low temp 12 % estimated 446 L/H compressed air at 300 K high temp ethylene glycol 303.1 high temp 550 L/H high temp 550 L/H = 10 % 22% max power					
bfo_tt_12_446_273.1 bfo_tt_545_300.0 bfo_tt_545_303.1 bfo_tt_550_305.1 bfo_tt_550_305.1 bfo_tt_570_363	S mm PABBO BB/19F-1H/D Z-GRD Z1C S mm PABBO BB/19F-1H/D Z-GRD Z1C	4450/0412 4450/0412 4450/0412 4450/0412 2104450/0412 4450/0412	273.1 302.9 303.1 305.1 333.1 363.0	545.000 1 550.000 1 545.000 1 550.000 1 550.000 1 670.000 ad	None None None None None Iapte None	low temp 12 % estimated 446 L/H compressed air at 300 K high temp ethylene glycol 303.1 high temp 550 L/H high temp 550 L/H high temp ethylene glycol 550 L/H 10 % 22% max power					
bfo_it_12_446_273.1 bfo_it_545_300.0 bfo_int_545_303.1 bfo_int_550_305.1 bfo_int_550_305.1 bfo_int_550_305.1 bfo_int_670_363	S mm PA8B0 88/19F-1H/D Z-CRD 21/ S mm PA8B0 88/19F-1H/D Z-CRD 21(S mm PA8B0 88/19F-1H/D Z-CRD 210 S mm PA8B0 88/19F-1H/D Z-CRD 210 S mm PA8B0 88/19F-1H/D Z-CRD 210 S mm PA8B0 88/19F-1H/D Z-CRD 210	4450/0412 4450/0412 4450/0412 4450/0412 4450/0412 4450/0412 4450/0412	273.1 302.9 303.1 305.1 383.1 363.0	545.000 1 550.000 1 545.000 1 550.000 1 550.000 1 670.000 ad	None None None None <mark>None</mark> Iapte None	low temp 12 % estimated 446 L/H compressed air at 300 K high temp ethylene glycol 303.1 high temp 550 L/H high temp cthylene glycol 550 L/H 10% 22% max power					

Figure 4: Selection of Appropriate Self Tune File

After *Correction and Self-Tune* file are selected, click on Temperature Tab (Figure 2), in *Target Temperature* column, click Set to set a sample temperature Kelvin as shown in Figure 5. In this example, the target temperature is changed from 300.1 K to 320 K. Click on OK to set the sample target temperature.

🧧 Set target temperature 🗙	Set target temperature
Please enter the new probe target temperature (Temperature correction is applied).	Please enter the new probe target temperature (Temperature correction is applied).
Target temperature [K]: 300.1	Target temperature [K]: 320
QK <u>C</u> ancel	QK <u>C</u> ancel

Figure 5: Change Target Temperature

6. Now click on **Monitoring** Tab and check the boxes in front of *Current Temperature and Target Temperature* as circled in Figure 6. The current temperature slowly approaches to the target temperature as shown in the following figure.

Note: depending on selected target temperature, it may take a few minutes or longer to for the current temperature to reach the target temperate.



Figure 6: Monitoring Current and Target Temperatures

9

7. Once the target temperature is achieved, go back to **Temperature** Tab, wait until the digits in the Sample Temperature field turning into green and the temperature stability is checked as circled in Figure 7.

Note: the Sample Temperature indicator is color coded. When the sample temperature is regulated, it turns into green. It shows blue and red with respect to the cases when the sample temperature is lower than and higher than the target temperatures.

Temperature Control Suite											
Temperature Monitoring Record Correction Self tune Configuration Log Help											
On Off VTU State: 📀 On											
Channel	Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power						
1 5 mm PABBO BB/19F-1H/D Z-GRD Z Stead Stead Stead (Measured value 317.7 K) Stead (123.0 K423.0 K) 3.8 % 7 Measured value 317.7 K) Set (max. 29.7 % of 195.8 W)											
	State	Gas Flow	Target Gas Flow	Standby Gas Flow							
Probe Gas	🕑 Steady	550 lph	550 lph Set	540 lph Set							
Probe Gas Steady 550 lph Set Set											

Figure 7: Target High Temperature Achieved

8. When sample temperature changes, the lock signal may fluctuate. Before acquiring NMR data, the shimming settings should be readjusted by typing *topshim* in the command line. Start NMR data acquisition after the auto-shimming.

2.3 Finish Up

When the high temperature measurements have completed, follow these steps to finish up:

1. After the high-temperature measurement has completed, in **Correction** Tab of the Temperature Control Suite (see Figure 3), first select the correction file of *"RT_510_NoCorrection"* and then click on **Set** to load the correction parameters.

- 2. Click on **Self Tune** Tab of the Temperature Control Suite, select the self-tune setting file of "*bbfo_rt_545_300.0*" (see Figure 4), then click on **Restore to Channel 1** button to load the self-tune parameters.
- 3. In **Temperature** Tab of the Temperature Control Suite, set *Target Sample Temperature* to 300 K as described in step 5. Wait until the target sample temperature reduces to 300 K.

Note: if the target sample temperature is higher than 320 K, make sure the target sample temperature is reduced in steps (preferably 15 to 20 K each step) to avoid extreme thermal stress in probe components.

- 4. When the target sample temperature reaches to 300 K, eject NME sample by type "*ej*" in the command line of the TopSpin NMR software interface.
- 5. Switch the gas supply from nitrogen gas to compressed air. To do so, refer to Figure 1 and the procedure described in section 2.1.1, first turn the switch **S1** on, then turn the switch **S3** by 180° such that it is pointing to the left. Finally turn off **S2**.

3 Low Temperature Operation

The low temperature operation involves installing VT apparatus and accessories, including filling the liquid nitrogen dewar, installing the nitrogen gas transfer line, and setting up target sample temperature.

3.1 Preparation

3.1.1 Liquid Nitrogen Dewar

The source of the NMR probe cooling is from the controlled evaporation of liquid nitrogen. So the first step is to fill a 25-L liquid nitrogen (LN2) dewar (Figure 8) with liquid nitrogen using a filling station in the Cylinder Storage Room (073 BRL). Place the LN2 dewar on the labeled spot near the magnet.

Note: Safety glass and face shield are required to fill liquid nitrogen.



Figure 8: Liquid Nitrogen Dewar on Labeled Spot Near the Magnet

3.1.2 Switch Gas Supply to Nitrogen Gas

It is highly recommended to use the nitrogen gas for low the temperature operation due to possible moisture condensation even iced-up inside of the probe occurring at the low temperatures. The compressed air gas supply setup is shown in Figure 9, in which S1 is at on position, S2, is at off position, and S3 is pointing to the left. To switch the gas supply to the nitrogen gas, first turn on S2, then turning S3 to the right, and finally turn off S1, as shown in Figure 9.



Compressed Air Supply

Nitrogen Gas Supply

Figure 9: Switch Gas Supply from Compressed Air to Nitrogen Gas

3.1.3 Use Ceramic Spinner

Use a ceramic spinner to avoid possible damage of the regular blue spinner for the low temperature operation.

3.1.4 Room Temperature Experiment

It is highly recommended to get an NMR spectrum at room temperature as a baseline spectrum before changing sample temperature. The procedures of acquiring a routine 1D NMR spectrum have been described in "AVIII 400 User Guide"

3.2 Low Temperature Apparatus

To connect the LN2 transfer line, follow these steps:

1. In the command line of TopSpin software interface, type "*edte*" to start the Temperature Control Suite. Click on **Off** button to turn off the Variable Temperature Unit (VTU) as shown in Figure 10. In this way, the probe heater is turned off.

Note: it is important to turn off the VTU before connecting the LN2 transfer line to the probe to prevent the overheating of the NMR probe.



Figure 10: Turn Off the Variable Temperature Unit

2. Disconnect the black VT gas hose from the probe and remove the VT adaptor as shown in Figure 11.



Figure 11: Remove the VT Gas Hose and the VT Adaptor from Probe

3. Make sure the o-rang is on the heater end of the nitrogen transfer line and remove green pressure relief cap on the transfer line (Figure 12)



Figure 12: The o-ring and Pressure Relief Cap

4. Insert the heater of the nitrogen gas transfer line into the LN2 dewar **SLOWLY** as shown in Figure 13. *Note: Safety glass is required in this operation.*



Figure 13: Insertion of N2 Transfer Line into LN2 Dewar

5. After the transfer line is in the dewar, use a clamp to seal the heater on the top of dewar. *Note: the clamp must be screwed down tightly using a butterfly nut. Otherwise it will take much longer time to cool the probe down.* After all pressure is released from the pressure relief port, screw down the green cap. These procedures are demonstrated in Figure 14.



Figure 14: Screw down the Seal Clamp and the Green Cap

6. Attach the transfer line **SLOWLY** into the probe. Keep the transfer line horizontal when inserting the transfer line into the probe. The transfer line should be inserted into the probe without any resistance if the position of the dewar and transfer line is correct. (see Figure 15). Once the transfer line is in place, screw down the cap screw to seal the transfer line to the probe. (see Figure 15)

Note: never exert force to push the transfer line into the probe. Ask for help if needed.





Figure 15: Insertion of the Transfer Line into the Probe

7. Connect the transfer line cable to the LN2 box near the base of the magnet as shown in Figure 16.



Figure 16: Connecting Transfer Line Cable to the LN2 Box

8. At this point, the Temperature Control Suite shows the **Chiller N2 Evaporator** added to the channel 2 as shown in the Temperature Control Suite (Figure 17).

T			Temperature Cor	trol Suite								
Temperature	Monitoring Record	Correction Self t	une Configuration Log	Help								
	Off VTU State: 🗢 Off											
	Channel	Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power						
5 mm PABBO B	1 5 mm PABBO BB/19F-1H/D Z-GRD Z		1 D BB/19F−1H/D Z−GRD Z Gff		Not Available	Corr. 298.0 K (Measured value 298.0 K)	Corr. 301.2 K (123.0 K423.0 K) Set	0.0 % (max. 30.1 % of 193.5 W)				
	Channel	State	Current Power	Target Power	Gas State	Gas Flow						
.: N2	2 (Chiller) Evaporator	🕑 Connected	0.0 % (max. 25.0 % of 210.0 W)	9.0 % (max. 25.0 % of 210.0 W) Set	C Estimated	150 lph						

Figure 17: N2 Evaporator Added as a New Apparatus

3.3 Low Temperature Setup

To set up NMR sample at low temperature, follow these steps:

1. Click on Correction Tab of the Temperature Control Suite, select a correction file that suitable to the target temperature. In this example, a file "*bbfolowtempertaure280-300*" is selected (Figure 18). Click on **Set** button to continue.

Note: user may also create a temperature calibration curve. A methanol sample is used for low temperature calibration. Use standard proton parameter and set rg to 1. Take a proton spectrum and measure the chemical shift difference between two peaks. Use a calibration curve or online tools to determine the actual temperature. Please consult the NMR spectroscopist for accurate temperature calibration.

T					Temperatur	e Cont	rol Suit	e			
Temperature Mor	nitoring	Record	Correction	Self tune	Configuration	Log	Help				
Temperature correction											
Use temperature correction if you want to display the real sample temperature instead of the probe temperature sensor value.											
Please check the manual how to perform temperature measurements with NMR (to determine the real sample temperature).											
Note: Temperature correction is not applied to temperature limits (safety checks).											
Enable temperature correction with these values											
Name:		No_correc	tion								
Probe:		5 mm PAB	BO BB/19F-1	H/D Z-GRD 2	Z104450/0412						
Temperature rang	ge [K]:	298 - 300)								
Slope:		1									
Offset:		0									
Comment:		compress	ed air								
Available correction	settings										
△ Name				Probe			Temp	erature Range	Slope	Offset	Comment
HT_670_22.3_etgP		5 mm PA	BBO BB/19F-	1H/D Z-GRE	Z104450/0412		303 - 3	90	0.914826	24.984227	11May2012
No_correction		5 mm PA	BBO BB/19F-	1H/D Z-GRE	Z104450/0412		298 - 3	00	1	0	compressed air
bbfoht_305.1-348.	1_550	5 mm PA	BBO BB/19F-	1H/D Z-GRE	Z104450/0412		305.10	- 348.10	0.844794	49.465422	
bbfolowtemp273-2	33	5 mm PA	BBO BB/19F-	1H/D Z-GRE	Z104450/0412		273.1 -	233.1	0.767754	68.647025	
bbfolowtemp280-	300	5 mm P.	ABBO BB/19	-1H/D Z-0	GRD Z104450/0	412	280 -	300	0.854463	43.22366	using cooling n2 gas
lt243-273_ntl		5 mm PA	BBO BB/19F-	1H/D Z-GRE	Z104450/0412		243.00	- 273.00	0.733496	77.376528	new transfer line

Figure 18: Selection of Correction Parameter Settings

2. Click on Self Tune Tab of the Temperature Control Suite, select an appropriate selftune parameter Settings. In this example, the file "*bbfo_lt_10_446_230*" is selected as shown in Figure 19. Click on **Restore to Channel 1** to load the parameters.

					Temperatur	e Control Sui	te					
Temperature	Monitoring	Record	Correction	Self tune	Configuration	Log Help]					
Self tune												
Execute self tune	to improve t	he regulati	on capabilitie	s of the VTU								
ou can self tun	ou can self tune each channel independently (select self tune for the appropriate channel) or self tune all available channels simultaneously (select self tune all channels).											
o save the self tune parameters for a defined temperature, gas flow, probe and sensor press the "Get" button of the desired channel and enter a name for the settings.												
	Channel			·	Charles and f			Canadian			16	
	Channel			sensor	Start seit	tune Stop	seir tune	Get self tu	ne parameter:	s viewse	if tune parai	neters
	All				Start		Stop					
	1		adapter	connection	1 Start		Stop		Cat		View	
5 mm PABBO	BB/19F-1H/D	Z-GRD Z	auaptei	connection	Jan		stop		Get		VIEW	
												D.L.
										Restore t	o channel I	Delete
Available self tu	ine settings											
∆ Na	me			Probe		Tempe	rature [K]	Flow [lph]	Se Se	ensor	Chiller	Comment
bbfo_ht_545_3	303.1	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	303.1		545.000	1		None	high te
bbfo_ht_550_3	305.1	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	305.1		550.000	1		None	high te
bbfo_ht_550_3	333.1	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	333.1		550.000	1		None	high te
bbfo_ht_670_3	363	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	363.0		670.000	adapter co	nnection 1	None	22% ma
bbfo_lt_10_44			ABBO BB/19		RD Z104450/0			550.000				
bbfo_lt_12_44	6_273.1	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	273.1	!	545.000	1		None	low te
bbfo_lt_13_47	0_243.1	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	243.1		545.000	1		None	low te
bbfo_lt_550_2	80_fts-40	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	282.8		550.000	adapter co	nnection 1	None	low te
bbfo_rt_545_3	00.0	5 mm PA	BBO BB/19F-	1H/D Z-GRD	Z104450/0412	302.9		550.000	1		None	compre

Figure 19: Selection of Low Temperature Self Tune Parameter Settings

3. In **Temperature** Tab and in *Target Temperature* column, click **Set** to set a sample temperature in Kelvin as shown in Figure 20. In this example, the target temperature is changed from 301.2 K to 238 K. Click on **OK** to finishing setting the sample target temperature.

Set target temperature	Set target temperature
lease enter the new probe target temperature Temperature correction is applied). 'arget temperature [K]: 301.2	 Please enter the new probe target temperat (Temperature correction is applied). Target temperature [K]: 238
QK <u>C</u> ancel	QK <u>C</u> ano

Figure 20: Set a Target Low Temperature

4. Now click on **ON** button in the top row of the **Temperature** Tab to turn on the VTU (Figure 21). The heater of the transfer line will be turned on in the liquid nitrogen dewar so that the cold nitrogen gas is introduced into the probe. The probe heater is also turned on to regulate the sample temperature if the sample temperature is lower than the target sample temperature.

T	Temperature Control Suite											
Temperature Monitoring Record	Temperature Monitoring Record Correction Self tune Configuration Log Help											
on off VTU State: On												
Channel	Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power							
1 5 mm PABBO BB/19F-1H/D Z-GRD Z	💟 Transient	Not Available	Corr. 297.2 K (Measured value 297.1 k)	Corr. 228.0 K (123.0 K423.0 K) Set	0.0 % (max. 30.1 % of 193.5 W)							
Channel	State	Current Power	Target Power	Gas State	Gas Flow							
2 (Chiller) N2 Evaporator	🕑 Connected	9.0 % (max. 25.0 % of 210.0 W)	9.0 % (max. 25.0 % of 210.0 W) Set	<mark>२</mark> Estimated	424 lph							

Figure 21: Turn ON the VTU

5. Now click on **Monitoring** Tab and check the boxes of *Current Temperature* and *Target Temperature* as shown in Figure 22. The current sample temperature slowly approaches to the target temperature.

Note: it depends on the set target temperature, it may take 15 to 20 minutes or longer to reach the target temperate.



Figure 22: Sample Temperature Monitor

6. When the sample temperature reaches to the target temperature (indicated with a check marker of the Regulation State, show in Figure 23, do an automatic shimming by type *topshim* in the command line.

T Temperature Control Suite					
Temperature Monitoring	g Record Correction Self	tune Configuration Log	Help		
On Off VTU State: 🛇 On					
Channel	Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power
1 5 mm PABBO BB/19F-1H/[D Z-GRD Z Steady	Stable since 10:54:55 15 Nov 2012 ?	Corr. 238.0 k (Measured value 246.6 k)	Corr. 238.0 K 23.0 K423.0 K) Set	3.2 % (max. 30.1 % of 193.5 W)
Channel	State	Current Power	Target Power	Gas State	Gas Flow
2 (Chiller) N2 Evaporator	Connected	9.0 % (max. 25.0 % of 210.0 W)	9.0 % (max. 25.0 % of 210.0 W) Set	🔁 Estimated	424 lph

Figure 23: Target Low Temperature Achieved

Note: the Sample Temperature indicator is color coded. When the sample temperature is regulated, it turns into green. It shows blue and red with respect to the cases when the sample temperature is lower than and higher than the target temperatures.

7. After the auto-shimming, start an NMR data acquisition as described in *AVIII 400 User Guide*.

3.4 Finish Up

When the low temperature measurements have completed, follow these steps to finish up.

- 1. Eject the NMR sample from the probe by type "*ej*" in the command line of TopSpin software interface.
- 2. In **Correction** Tab of the Temperature Control Suite (see Figure 18), first select the correction file of "*RT_510_NoCorrection*" and then click on **Set** to load the parameters.
- 3. Click on **Self Tune** Tab of the Temperature Control Suite, select the self-tune setting file of "*bbfo_rt_545_300.0*" (see Figure 19), then click on **Restore to Channel 1** button to load the self-tune parameter for room temperature operation.
- 4. In **Temperature** Tab of the Temperature Control Suite, set the sample target temperature to 300 K (refer to Figure 20 and step 3 on page 18. Wait until the target temperature research to 300 K.

Note: if the target sample temperature is lower than 280 K, make sure the target sample temperature is increased in steps (preferably 15 to 20 K for each step) to avoid the extreme thermal stress to probe components

- 5. When the temperature reaches to 300 K, click on **Off** button in the Temperature Tab of the Temperature Control Suite to turn of the VTU.
- 6. Disconnect the nitrogen gas transfer line cable from the LN2 box.
- 7. Detach the nitrogen transfer line from the probe.
- 8. Attach the VT adaptor to the probe.
- 9. Connect the black VT air hose quickly.
- 10. Click on **ON** button in the Temperature Tab of the Temperature Control Suite to turn on the VTU. Wait until the target temperature reaches and stabilizes at 300 K.
- 11. Switch the gas supply from the nitrogen gas to the compressed air. To do so, refer to Figure 9 and the section 3.1.2. First turn on **S1**, then turn the switch **S3** by 180° such that it is pointing to the left. Finally turn off **S2**.