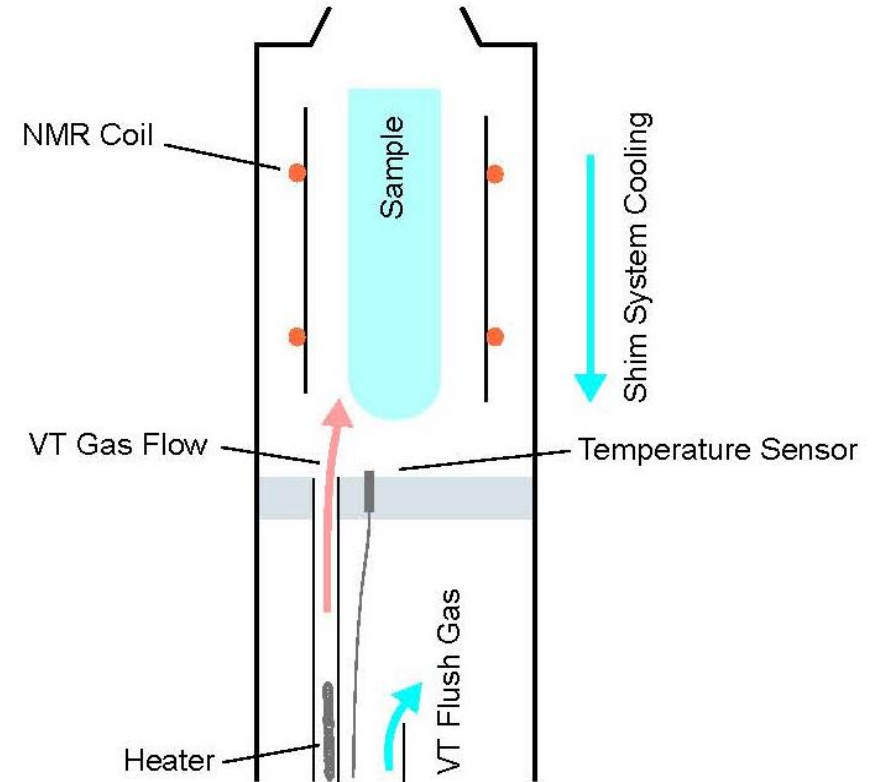


How the temperature is controlled



- The variable temperature (VT) gas flow streams through a pipe into the sample space.
- The VT gas flows along the sample tube and finally leaves the probe head at the top.
- The VT gas flow heats or cools the sample, when it streams around the sample tube.
- A temperature sensor measures the gas flow temperature and gives this measurement value to a control unit. This control unit regulates the heater power by a regulation algorithm.



Which NMR instrument to choose

Choose the proper instrument for your experiment based on the temperature range in the table

NMR Instrument	Probe	Cooling Device	Temperature Ranges
600 MHz MSCH	Nitrogen cooled	FTS chiller	5-171 °C
600 MHz Chem 1047	Helium cooled	Smart cooler BCU I	-42-171 °C
800 MHz Chem 1047	Helium cooled	Smart cooler BCU I	-40-80 °C

Sample preparation

- The highest temperature allowed should be at least 10 °C below the boiling point of your solvent. The lowest temperature should be at least 10 °C above melting point of your solvent.
- Sample should be stable at your target temperature
- Do not seal the tube
- Keep sample height short (400-500 μL)
- Use high quality NMR tube, economy tubes may deform or fracture at temperature extremes

Solvent data chart

http://isotope.com/uploads/File/new_datachart.pdf



Cambridge Isotope Laboratories, Inc.
www.isotope.com

RESEARCH PRODUCTS

NMR Solvent Data Chart

More Solvents, More Sizes, More Solutions

	¹ H Chemical Shift (ppm from TMS) (multiplicity) ●	JCD(Hz)	¹³ C Chemical Shift (ppm from TMS) (multiplicity) ●	JCD(Hz)	¹ H Chemical Shift of HOD (ppm from TMS) □	Density at 20°C ◆	Melting point (°C) ◆	Boiling point (°C) ◆	Dielectric Constant	Molecular Weight ◆
Acetic Acid-d ₄	11.65 (1) 2.04 (5)	2.2	178.99 (1) 20.0 (7)	20	11.5	1.12	16.7	118	6.1	64.08
Acetone-d ₆	2.05 (5)	2.2	206.68 (1) 29.92 (7)	0.9 19.4	2.8 *	0.87	-94	56.5	20.7	64.12
Acetonitrile-d ₃	1.94 (5)	2.5	118.69 (1) 1.39 (7)	21	2.1 *	0.84	-45	81.6	37.5	44.07
Benzene-d ₆	7.16 (1)		128.39 (3)	24.3	0.4	0.95	5.5	80.1	2.3	84.15
Chloroform-d	7.24 (1)		77.23 (3)	32.0	1.5 *	1.50	-63.5	61-62	4.8	120.38

NMR Tube



www.wilmad-labglass.com

Pyrex® Glass Precision Tubes 5 mm O.D. Precision Tubes

Good for VT NMR

Part No.	MHz Rating	O.D. (mm)	I.D. (mm)	Length (inch)	Wall Thickness (mm)	Concentricity (µm)	Camber (µm)
535-PP-7	600	4.9635±0.0065	4.2065±0.0065	7	0.38	13	6
528-PP-7	500	4.9635±0.0065	4.2065±0.0065	7	0.38	25	13
528-PP-8	500	4.9635±0.0065	4.2065±0.0065	8	0.38	25	13
527-PP-7	400	4.9635±0.0065	4.2065±0.0065	7	0.38	25	25
527-PP-8	400	4.9635±0.0065	4.2065±0.0065	8	0.38	25	25
507-PP-7	300	4.9635±0.0065	4.2065±0.0065	7	0.38	51	25
505-PS-7	100	4.9635±0.0065	4.2065±0.0065	7	0.38	76	51

N51A Glass Economy Tubes 5 mm O.D. Economy Tubes

May deform or fracture at temperature extremes

Part No.	MHz Rating	O.D. (mm)	Wall Thickness (mm)	Length (inch)	Concentricity (µm)	Camber (µm)
WG-1235-7	>400	4.93395±0.03175	0.43	7	13	6
WG-1228-7	400	4.93395±0.03175	0.43	7	25	13
WG-1228-8	400	4.93395±0.03175	0.43	8	25	13
WG-1226-7	300	4.93395±0.03175	0.43	7	51	13
WG-1226-8	300	4.93395±0.03175	0.43	8	51	13
WG-5MM-ECONOMY-7	100	4.93395±0.03175	0.43	7	76	76
WG-5MM-ECONOMY-8	100	4.93395±0.03175	0.43	8	76	76

Bulk Pack 5 mm Economy Tubes (100 tubes, no cap)

Part No.	MHz Rating	O.D.(mm)	Wall Thickness (mm)	Length (inch)
WG-1000-7	100	4.93395±0.03175	0.43	7

Select the proper spinner



Blue
0-+80 °C



Kel F
-4-+120 °C



Ceramic
-150-+150 °C

How to run VT experiment on 600 in MSCH

If your desired temperature is 20-70 °C, you can use iconnmr to automatically run VT experiments.

- Contact BioNMR core staff Minli Xing (mlxing@umich.edu) to add VT experiments (UM_C13CPD_1D_VT and UM_PROTON_1D_VT) into your account.
- In the parameter dropdown menu, set the temperature by changing the TE parameter value, increase/decrease the temperature incrementally by 10-20 °C. For example, if your target temperature is 55 °C, set up three VT experiments, running at 25 °C, 40 °C and 55 °C respectively.

Select VT Experiments 20-70 °C

The screenshot shows a software interface for NMR experiments. The top bar includes a search bar with the text "No Analysis" and a magnifying glass icon. Below the search bar, there are several dropdown menus and buttons. The main area displays a list of experiments, with two experiments highlighted in blue and enclosed in a red box:

- UM_C13CPD_1D_VT 1D 13C variable temperature
- UM_PROTON_1D_VT 1D 1H variable temperature

The interface also shows a list of other experiments, including DHS_HSQCEDETGPSISP2.3, DHS_HMBC, DHS_TOCSY, DHS_NOESY, DHS_ROESY, DHS_NOAH3_BSC, DHS_NOAH4_BSCN, UM_PROTON_1D, UM_C13CPD_1D, UM_C13DEPT135, UM_HMBC_FOR_CARBON_1D, UM_19F_1D, UM_P31_1D, UM_P31CPD_1D, UM_1H13C_HSQC_2D, UM_1H13C_HMBCETGPL3ND, and UM_LR_HMBC.

UM_C13CPD_1D_VT and UM_PROTON_1D_VT: Allows sample to equilibrate at target temperature for 5 minutes, then perform shimming and acquisition

Set target temperature ICONNMR 20-70 °C

The screenshot displays the Bruker ICONNMR software interface. On the left, a list of parameters is shown, with rows 23 and 24 highlighted in blue. The main area shows the current parameter settings for the selected row (23):

- Parameter: /icondata
- Value: VTNMR
- Unit: 10
- Sample: CDCI3
- Solvent: chloroform-d
- Channel: N UM_PROTON_1

At the bottom left, there are buttons for 'Submit', 'Cancel', 'Edit', 'Delete', 'Add', and 'Copy'. The 'Add' and 'Copy' buttons have a value of 1.

On the right side, a detailed parameter list is shown, with the 'TE' parameter highlighted in red:

Parameter	Value	Unit	Description
TE	298	[K]	Sample temperature (286K-323K)
TD	131072		Size of fid
D1	1	[sec]	inter-scan delay
D8	0		NOESY mixing time (s)
D9	0		TOCSY mixing (s)
P15	0		ROESY mixing time (us)
O1P	7.501	[ppm]	Transmitter frequency offset
O2P	7.501	[ppm]	Frequency offset of 2nd nucleus
1SW	16.6958	[ppm]	Spectral width (F1)
2SW	16.6958	[ppm]	Spectral width (F2)
1TD	131072		Size of fid (F1)
CNST13	1		JXH - HMBC J Coupling
NS	32		Number of scans
RO	0	[Hz]	Rotation frequency of sample
P1	12	[usec]	Pulse
LOCNUC	2H		Lock nucleus

An 'OK' button is located at the bottom of the parameter list.

Set up multiple VT experiments 20-70 °C

The screenshot displays the Bruker TopSpin software interface for setting up multiple VT experiments. The main window shows a list of available experiments (13-22) and a table of parameters for three selected experiments (10, 11, and 12). The parameters for each experiment are:

Exp No.	Solvent	Experiment	Lock	Shim	Acq	Proc	User	Disk	Title/C
10	CDCI3	VTNMR	chloroform-d	NUM_PROTON_1					
11	CDCI3	VTNMR	chloroform-d	NUM_PROTON_1					
12	CDCI3	VTNMR	chloroform-d	NUM_PROTON_1					

Below the table, there are buttons for **Submit**, **Cancel**, **Edit**, **Delete**, **Add** (with a value of 1), and **Copy** (with a value of 1).

The **Preceding Experiments** table is partially visible at the bottom:

Date	#	Holder	Name	No.	Solvent	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Disk	Title/C

A parameter dialog box is open for experiment 19, showing the following parameters:

TE	328 [K]	Sample temperature (286K-323K)
TD	131072	Size of fid
D1	1 [sec]	inter-scan delay
D8	0	NOESY mixing time (s)
D9	0	TOCSY mixing (s)
P15	0	ROESY mixing time (us)
O1P	7.501 [ppm]	Transmitter frequency offset
O2P	7.501 [ppm]	Frequency offset of 2nd nucleus
1SW	16.6958 [ppm]	Spectral width (F1)
2SW	16.6958 [ppm]	Spectral width (F2)
1TD	131072	Size of fid (F1)
CNST13	1	JXH - HMBC J Coupling
NS	1	Number of scans
RO	0 [Hz]	Rotation frequency of sample
P1	12 [usec]	Pulse
LOCNUC	2H	Lock nucleus

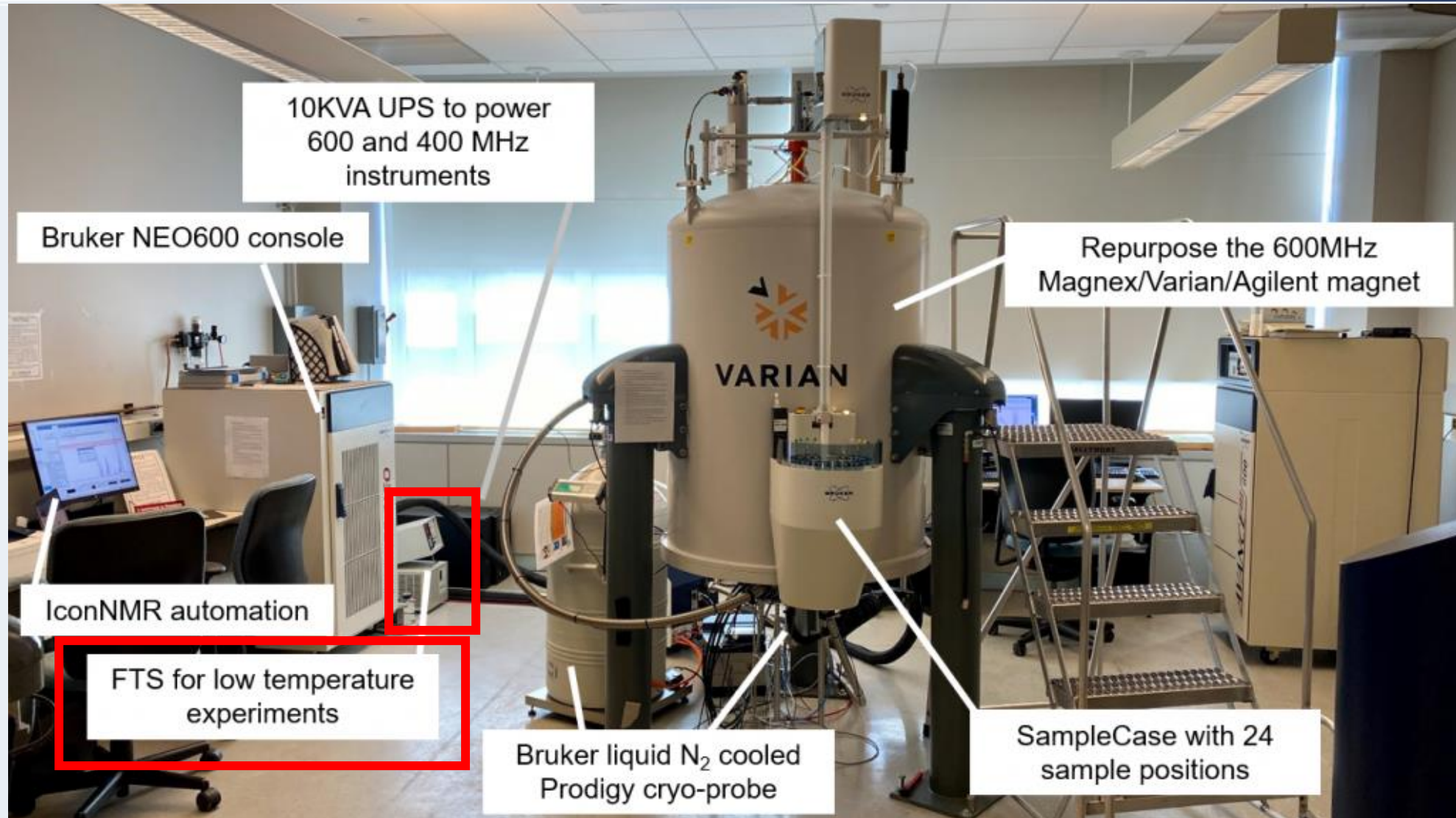
Temperature settings are displayed on the right: 298 K, 318 K, and 328 K. The dialog box has an **OK** button at the bottom.

How to run VT experiment on 600 in MSCH

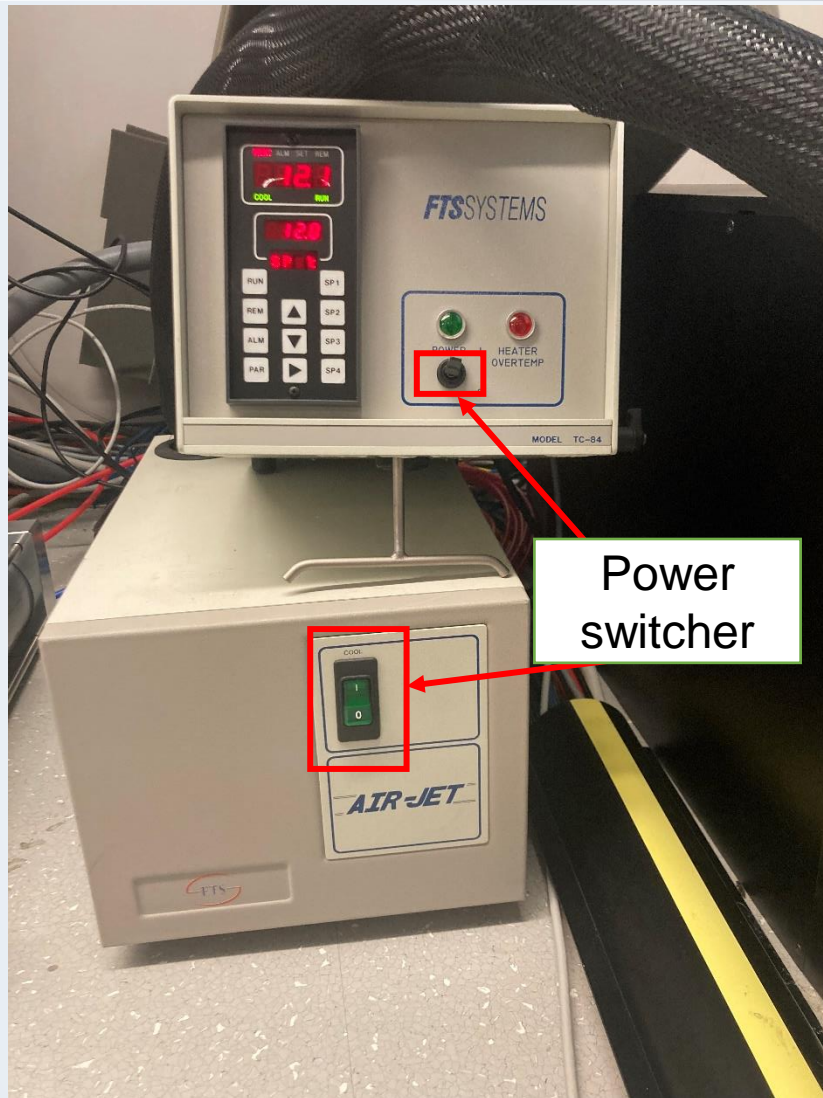
If your desired temperature is 5-20 °C, you can also use iconnmr to run VT experiments.

- Set FTS chiller temperature 10 °C colder than your desired experiment temperature
- Set up VT experiments on ICONNMR

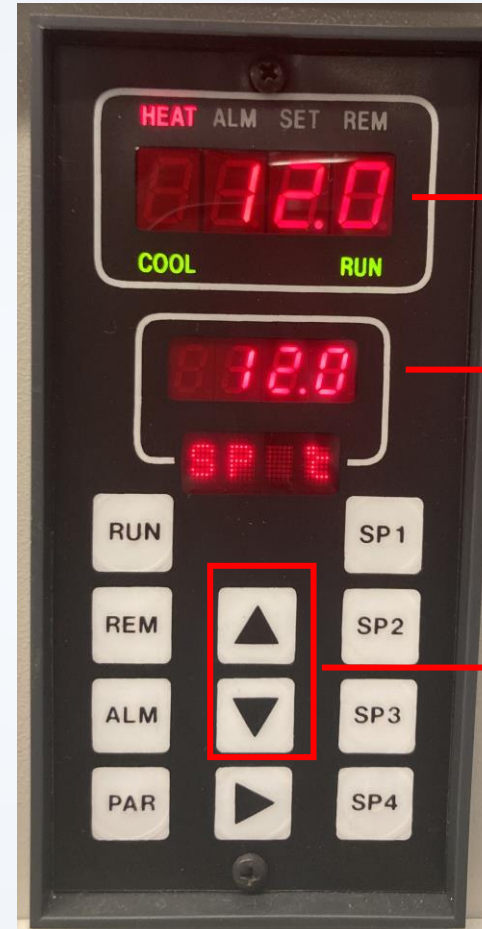
600 MSCH FTS Chiller



FTS Chiller



Power switcher



Actual VT gas temperature

Desired VT gas temperature
Set 10 °C colder than your
desired experiment temperature

Increase or decrease VT gas
temperature

Manually set temperature on Topspin

- Type EDTE in topspin command line

EDTE: temperature control suite

The screenshot shows the 'Temperature' control interface with tabs for Monitoring, Record, Correction, Self tune, Configuration, and Log. The 'VTU State' is set to 'On'. The main table displays the following data:

Channel	Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power
1 CPP1.1 BBO 600S3 BB-H&F-D-05 Z...	Steady	Stable since 10:19:23 24 Jan 2023	Corr. 298.0 K (Measured value 298.5 K)	Corr. 298.0 K (231 K...444 K)	1.4 % (max. 77.1 % of 103.5 W)
Probe Gas	Steady	Gas Flow	Target Gas Flow	Standby Gas Flow	
		535 lph	535 lph	535 lph	

EDTE MSCH 600

EDTE: temperature control suite

Temperature Monitoring Record Correction Self tune Configuration Log

On Off **VTU State:** On

Channel	Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power
1 CPP1.1 BBO 600S3 BB-H&F-D-05 Z...	<input checked="" type="radio"/> Steady	<input checked="" type="radio"/> Stable since 10:19:23 24 Jan 2023 <input data-bbox="868 664 932 699" type="button" value="?"/>	Corr. 298.0 K (Measured value 298.5 K)	Corr. 298.0 K (231 K...444 K) <input data-bbox="1898 649 1982 685" type="button" value="Set"/>	1.4 % (max. 77.1 % of 103.5 W)
	State	Gas Flow	Target Gas Flow	Standby Gas Flow	
Probe Gas	<input checked="" type="radio"/> Steady	535 lph	535 lph <input data-bbox="1370 806 1454 842" type="button" value="Set"/>	535 lph <input data-bbox="1898 806 1982 842" type="button" value="Set"/>	

Establish high temperature MSCH 600

Temperature Monitoring Record Correction Self tune Configuration Log

On Off **VTU State:** On

Channel	Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power
1 CPP1.1 BBO 600S3 BB-H&F-D-05 Z...	<input checked="" type="checkbox"/> Steady	<input checked="" type="checkbox"/> Stable since 10:19:23 24 Jan 2023 <input data-bbox="901 519 963 554" type="button" value="?"/>	Corr. 298.0 K (Measured value 298.5 K)	Corr. 298.0 K (231 K...444 K) <input data-bbox="1905 505 1982 539" type="button" value="Set"/>	1.4 % (max. 77.1 % of 103.5 W)
State	Gas Flow	Target Gas Flow	Standby Gas Flow		
Probe Gas <input checked="" type="checkbox"/> Steady	535 lph	535 lph <input data-bbox="1386 662 1462 696" type="button" value="Set"/>	535 lph <input data-bbox="1905 662 1982 696" type="button" value="Set"/>		

Set target temperature

Please enter the new probe target temperature
(Temperature correction is applied).

Target temperature [K]:

Establish high temperature MSCH 600

Blue: sample temperature is lower than target temperature

Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power
Transient	Not Available ?	Corr. 298.7 K (Measured value 299.2 K)	Corr. 305.0 K (231 K...444 K) <input type="button" value="Set"/>	4.7 % (max. 76.7 % of 104.1 W)

Red: sample temperature is higher than target temperature

Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power
Transient	Not Available ?	Corr. 305.1 K (Measured value 305.7 K)	Corr. 305.0 K (231 K...444 K) <input type="button" value="Set"/>	2.8 % (max. 76.7 % of 104.1 W)

Green: sample temperature is the same as target temperature

Regulation State	Stability	Sample Temperature	Target Temperature	Heater Power
Steady	Stable since 10:29:47 15 Mar 2023 ?	Corr. 305.0 K (Measured value 305.5 K)	Corr. 305.0 K (231 K...444 K) <input type="button" value="Set"/>	2.5 % (max. 76.7 % of 104.1 W)

Workflow for VT NMR

Open the temperature control suite (EDTE)



Set the FTS chiller temperature or BCU mode depending on your target temperature



Set target temperature (10-20 °C for about every 5 minutes)



Wait for the temperature to equilibrate, then acquire spectrum



Reset FTS chiller or BCU, return to room temperature slowly (10-20 °C for about every 1 minute)