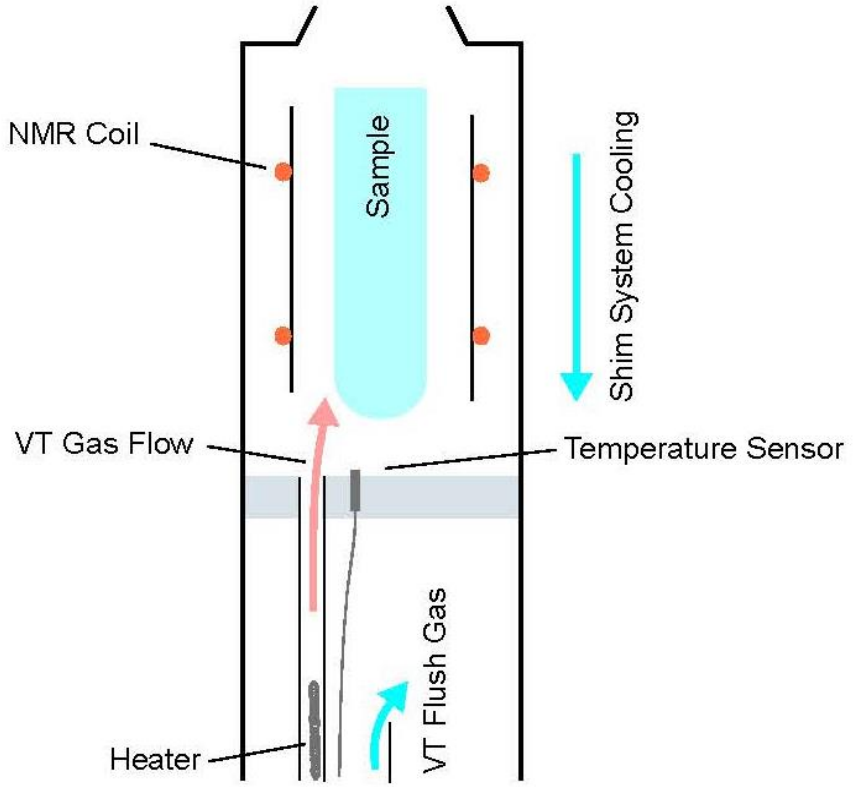
How the temperature is controlled

- 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

Wilmad LabGlass

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

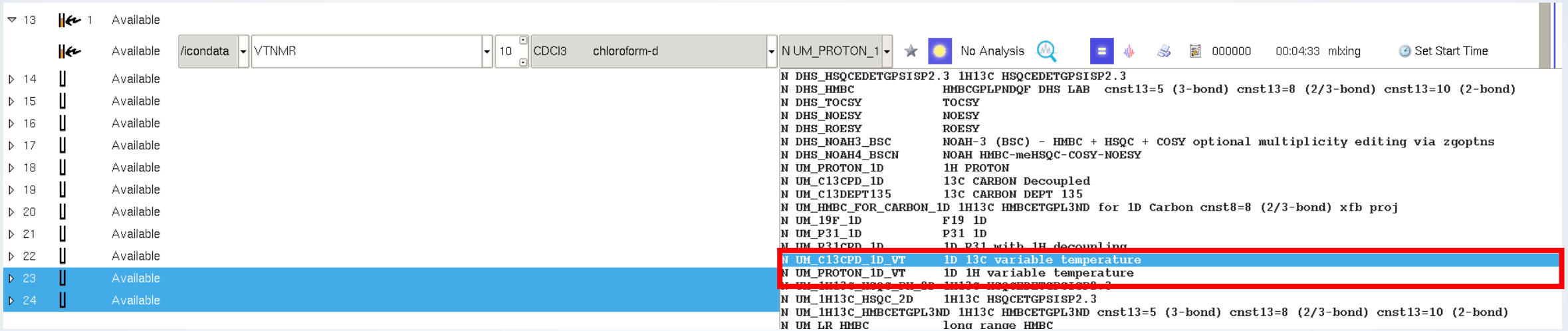
Ceramic
-150-+150 °C

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.

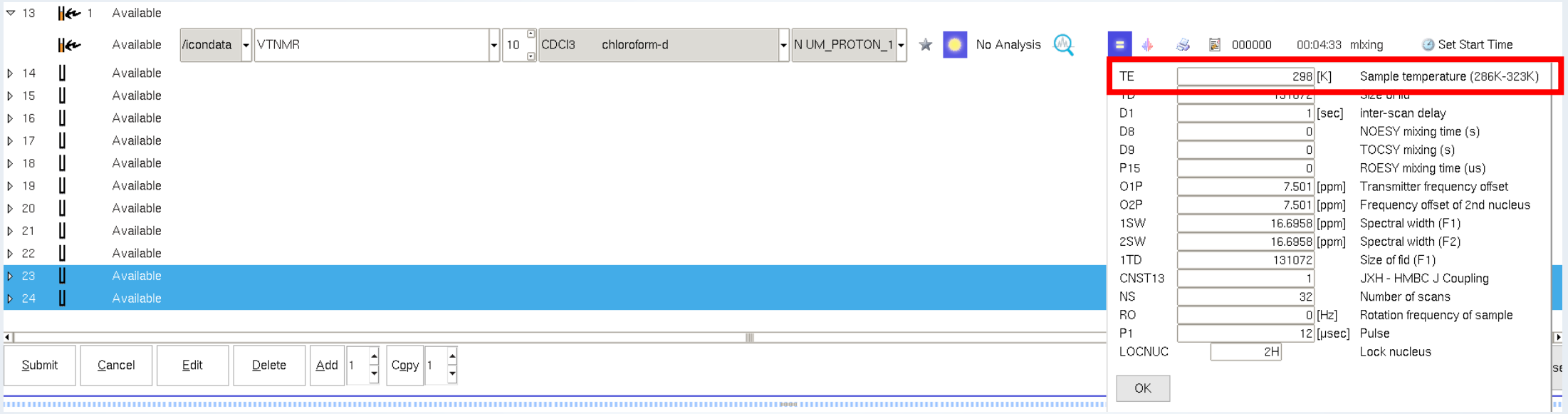
- Contact BioNMR core staff Minli Xing (mlxing@umich.edu) to add VT experiments (UM_C13CPD_1D_VT and UM_PROTON_1D_VT) into your account.
- In the parameter dropdown menu, set the temperature by changing the TE parameter value, increase/decrease the temperature incrementally by 10-20 °C. For example,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

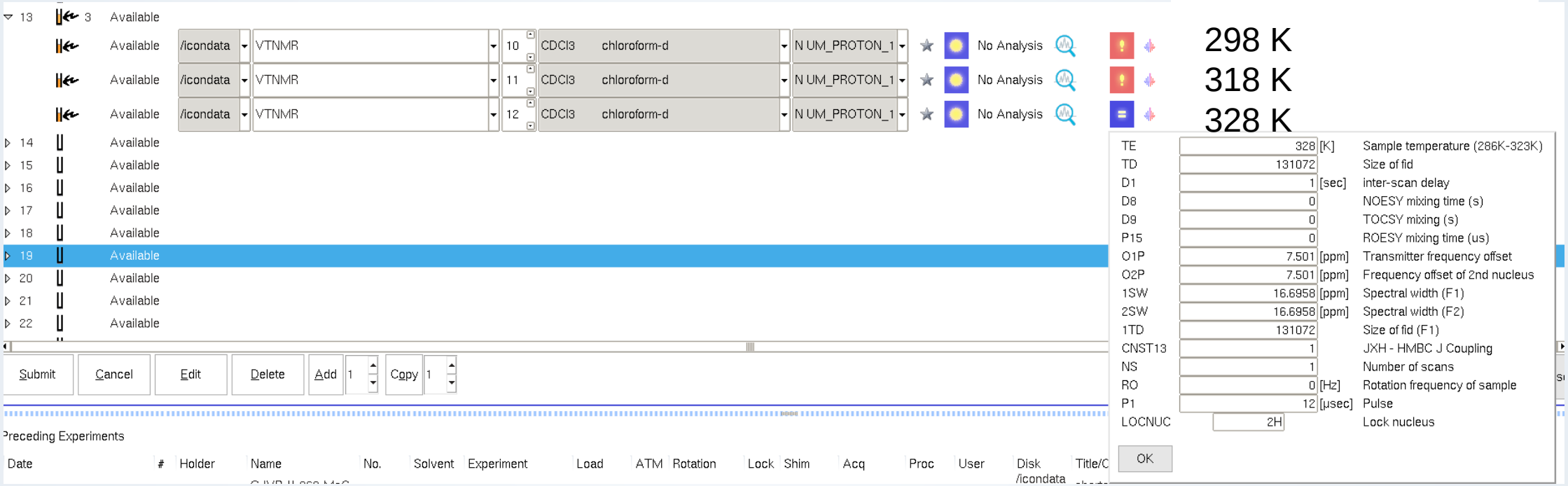


UM_C13CPD_1D_VT and UM_PROTON_1D_VT: Allows sample to equilibrate at target temperature for 5 minutes, then perform shimming and acquisition

Set target temperature ICONNMR 20-70 °C



Set up multiple VT experiments 20-70 °C

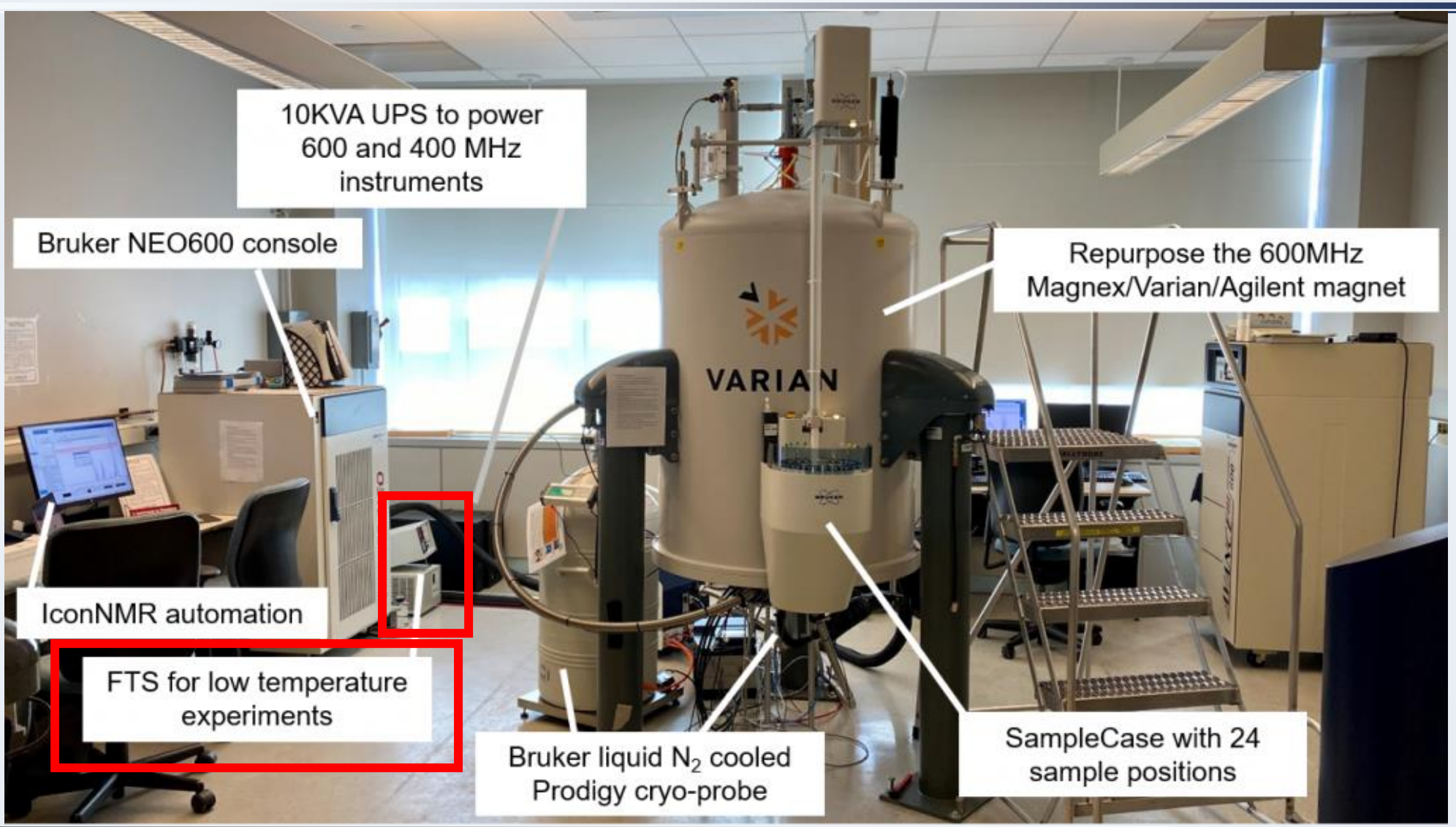


How to run VT experiment on 600 in MSCH

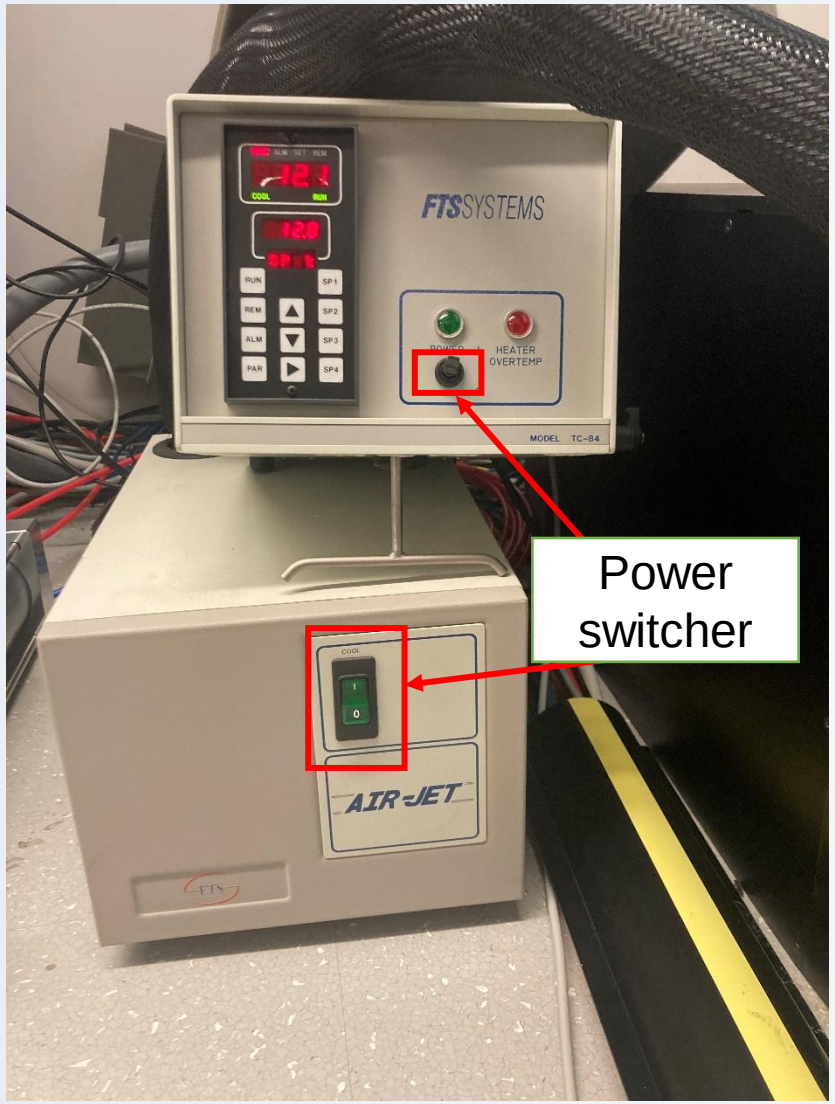
If your desired temperature is 5-20 °C, you can also use iconnmr to run VT experiments.

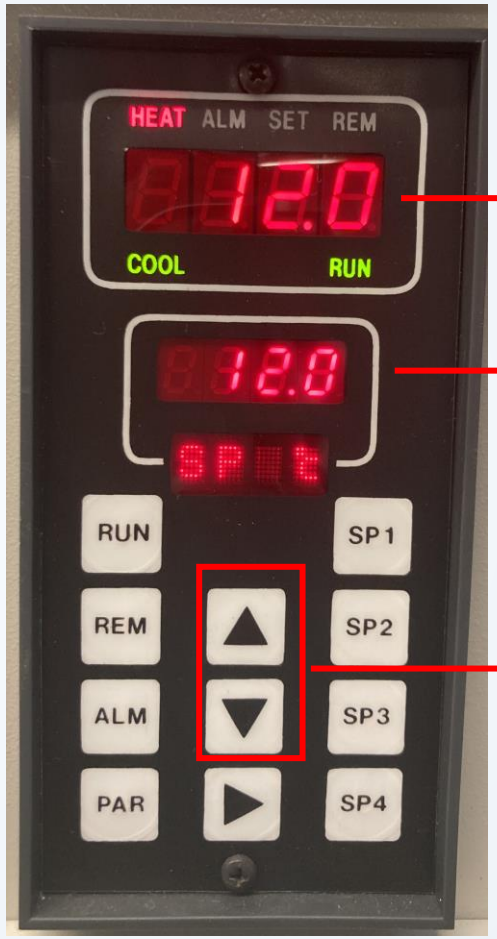
- Set FTS chiller temperature 10 °C colder than your desired experiment temperature
- Set up VT experiments on ICONNMR

600 MSCH FTS Chiller



FTS Chiller





Actual VT gas temperature

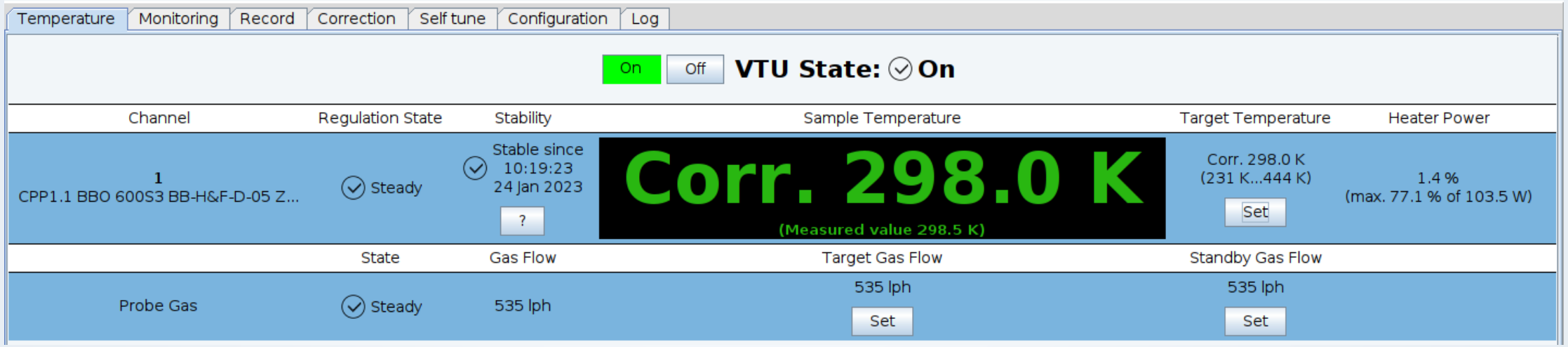
Desired VT gas temperature
Set 10 °C colder than your desired experiment temperature

Increase or decrease VT gas temperature

Manually set temperature on Topspin

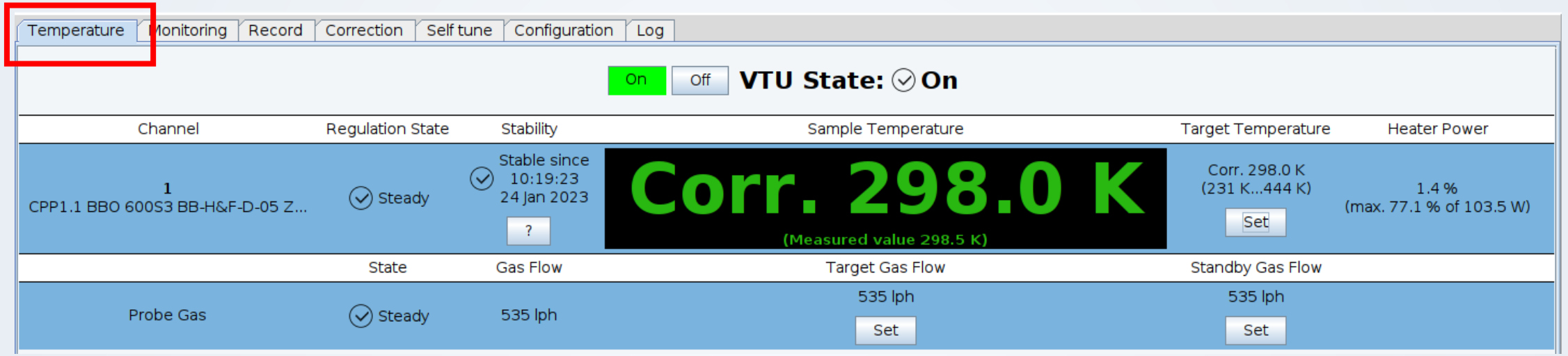
- Type EDTE in topspin command line

EDTE: temperature control suite

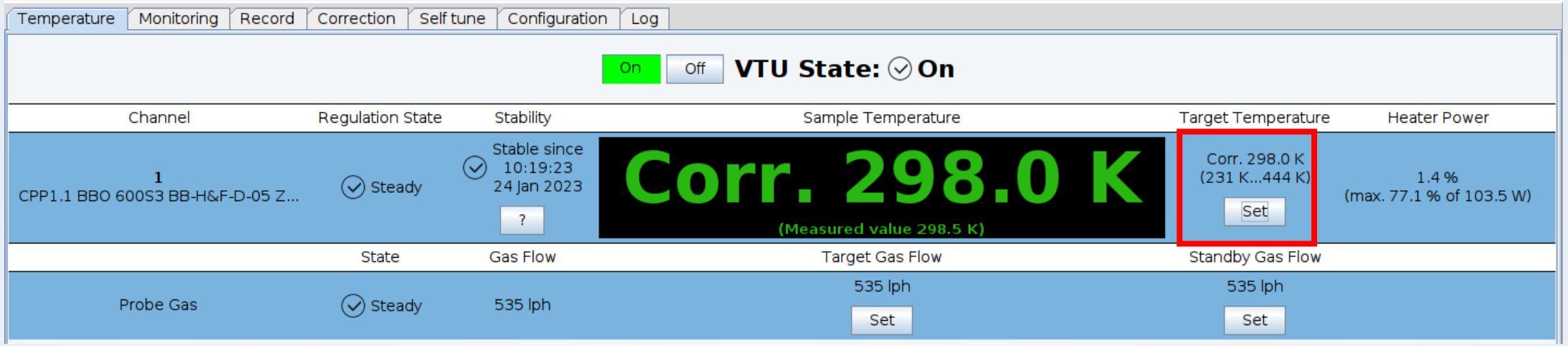


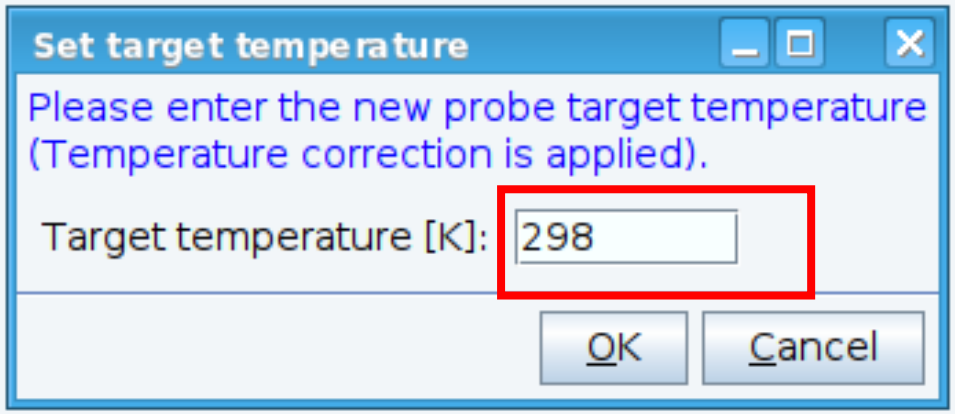
EDTE MSCH 600

EDTE: temperature control suite



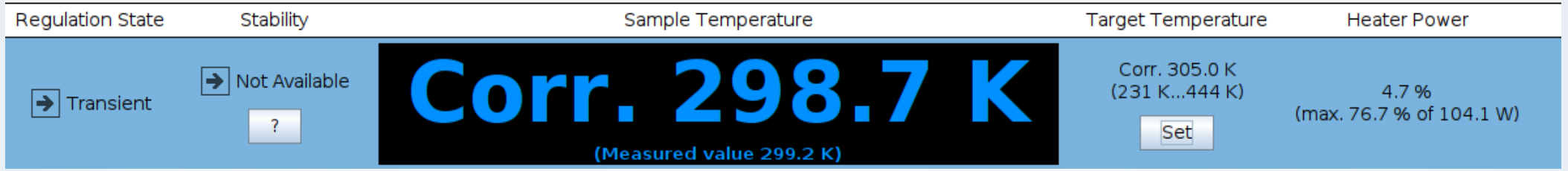
Establish high temperature MSCH 600



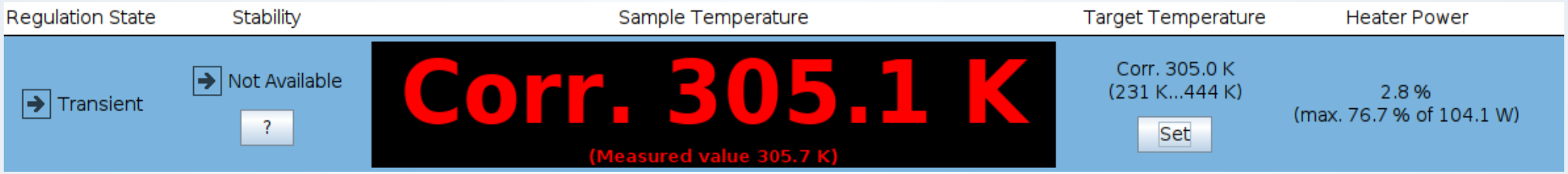


Establish high temperature MSCH 600

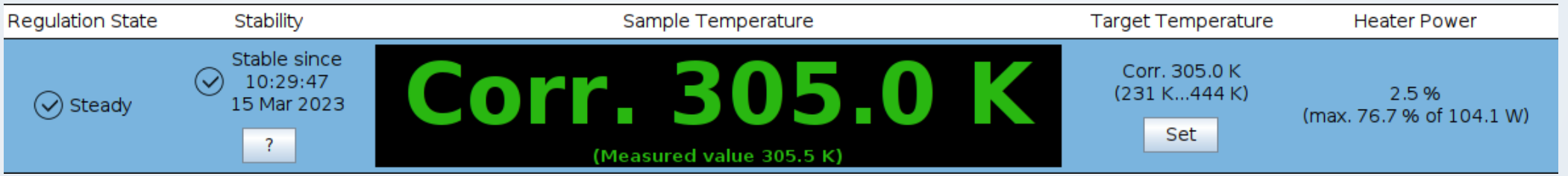
Blue: sample temperature is lower than target temperature



Red: sample temperature is higher than target temperature



Green: sample temperature is the same as target temperature



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)