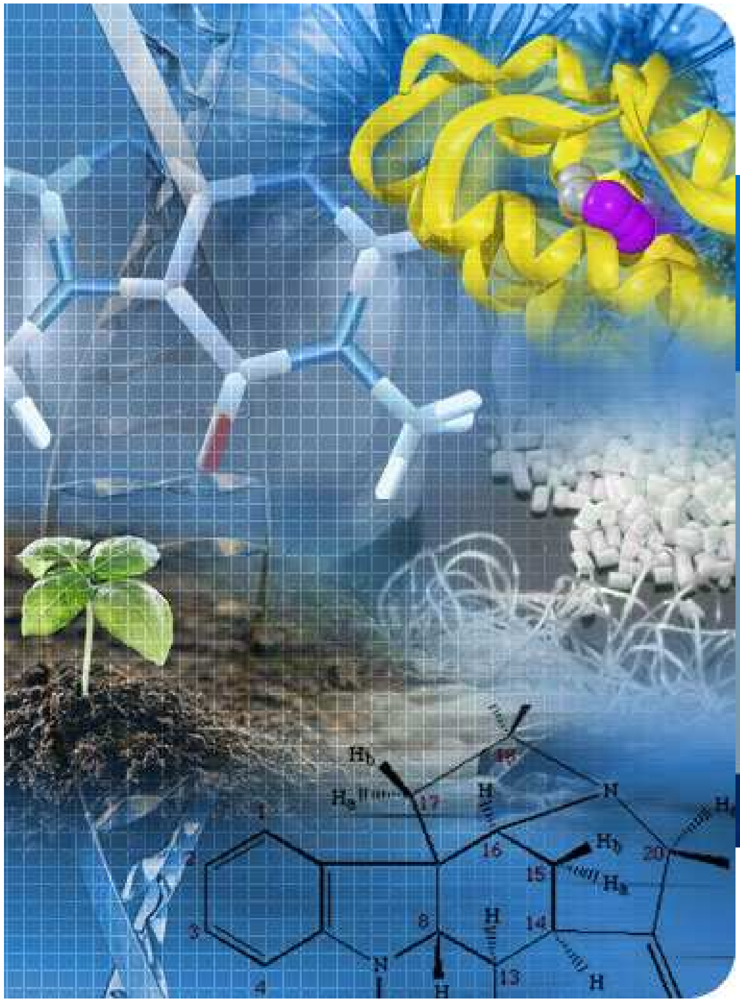


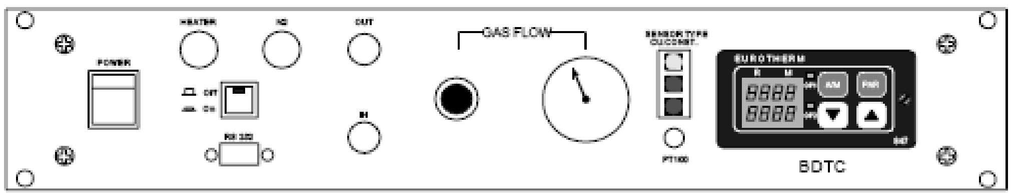
The New Temperature Controller, BSVT

Presented by Mike Brown

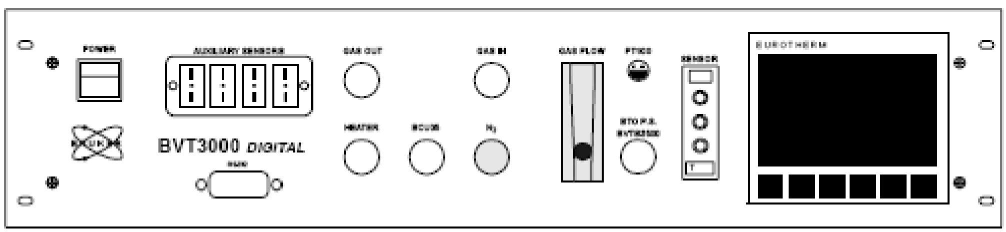
04042012

Old Style Versus new Style

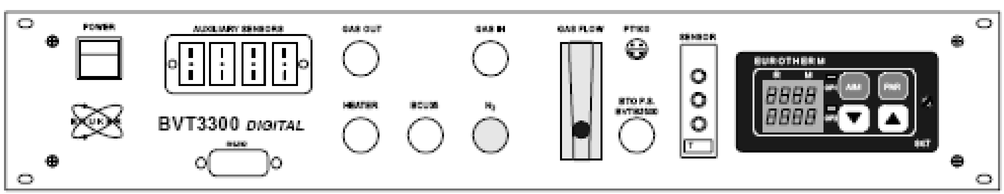
- The old BVT(s) have been replaced



BDTC



BVT-3000



BVT-3300

BVT3500

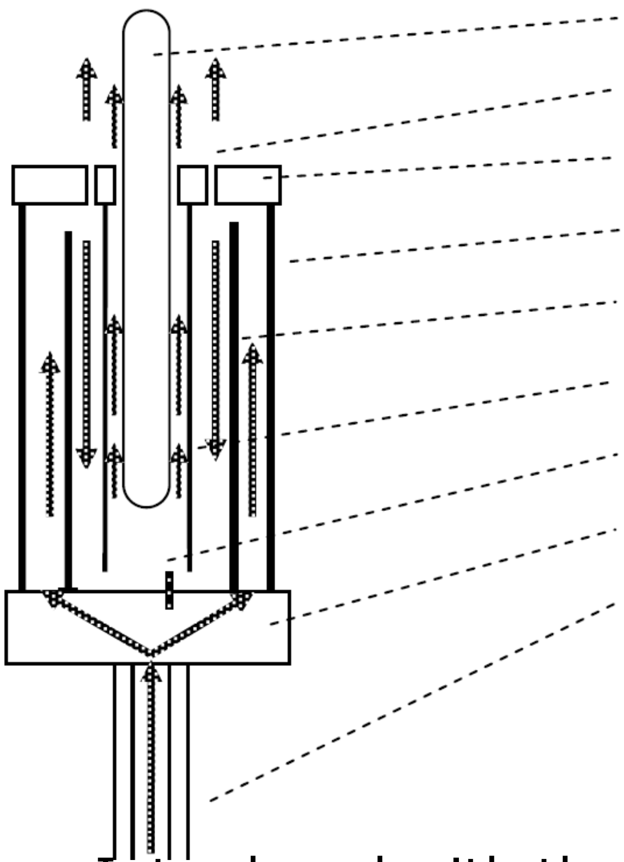
The BVT-3000 and 3300 are still supported in TopSpin 3.1.

The New BSVT

The BSVT consists of:

- Sensor and Pneumatics board (SPB or SPB-E)
- Variable Power Supply (VPSB)
- Variable Temperature adapter (VTA)

Extended Flow Turn Probes (E-FLT)

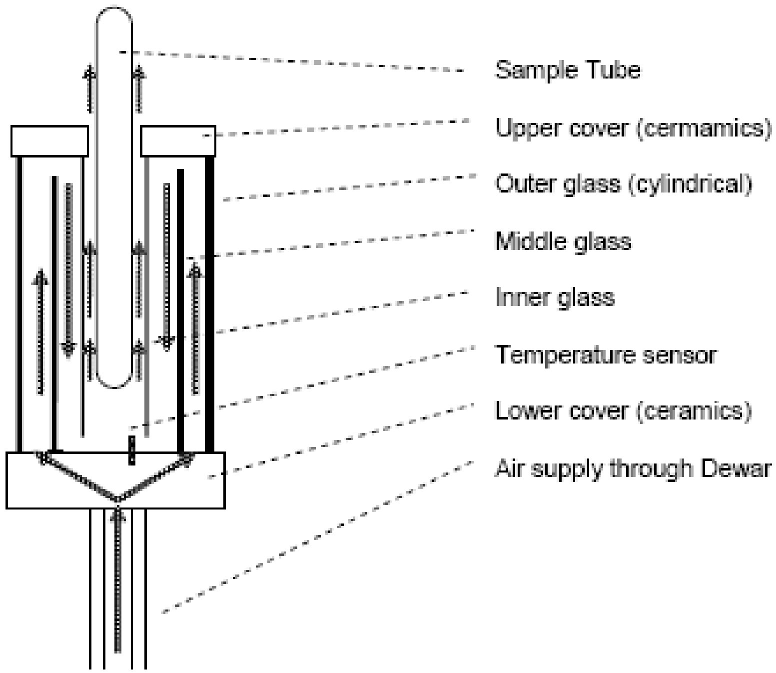


Introduced in 2010

The E-FLT combines the advantages of the FlowTurn (good gradients but lower flow rates) together with the high-flow capabilities of the former Dual Flow (high flow rates but worse gradients). Recommended flow is 535 l/h and can be higher (up to 1200 l/h depending on temperature and spinners).

Introduced with the Johnston VT gas coupling system, giving higher efficiency to the probes.

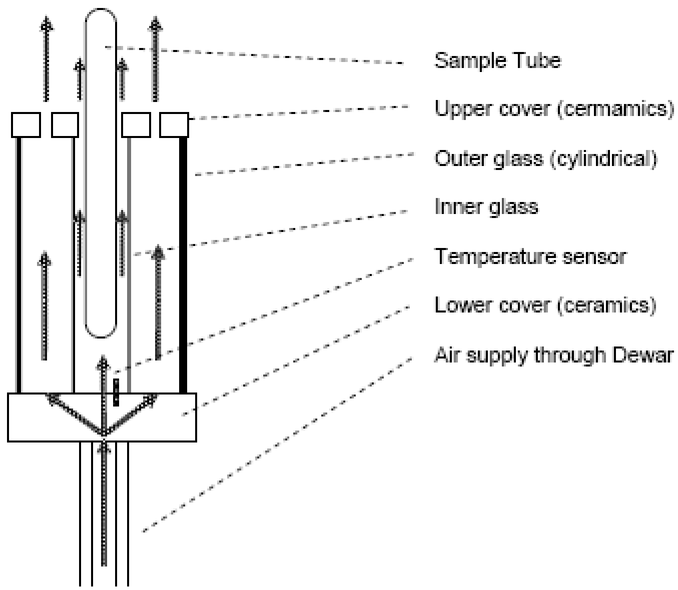
Flow Turn Probe



Probes built after 2003 are equipped with the conventional flow turn. It's function is identical to that of the Extended FlowTurn, but has no bypass flow and such probes do not allow high flow rates. Recommended flow is 400 l/h and should not be higher.

Except the missing bypass air outlets, the function is the same as the Extended FlowTurn. But since the inner glass is very close to the sample tube, only low flow rates are allowed. Subsequently, the heating/cooling performance is lower. Such probes are not optimized for extreme low temperature experiments.

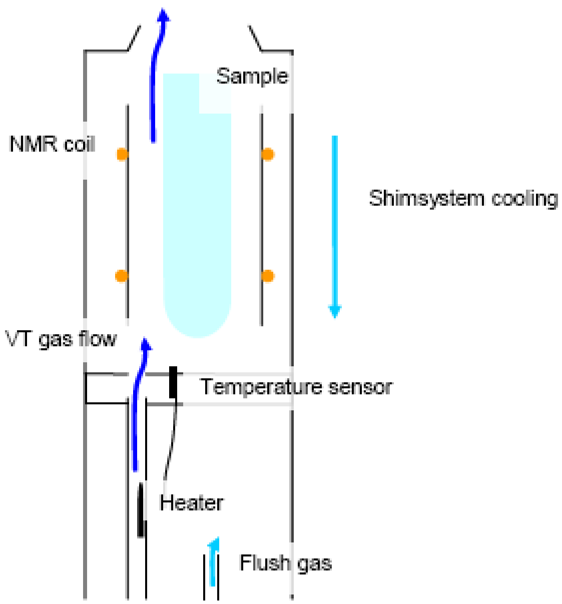
Conventional Dual Flow Probes



Probes built before 2003 are equipped with the classical Dual Flow. This principle allows efficient heating/cooling and high flow rates, since it contains a flow bypass which enables high temperature control power at the outer surface of the coil space, where the losses are.

The major disadvantage of this probe is temperature gradients cannot be optimized. High flow rates up to 20000L/H can be used for low and high temperature work

Principle of VT Control for NMR



- The air or N2 gas flows up through a Dewar in the bottom of the probe where it is heated by the Heater in the Dewar
- The gas then flows around the sample and the NMR coil heating the sample
- The Temperature of the gas heating the sample is measured by the Temperature sensor. The sensor is usually a type T thermocouple and the tip is very close to the sample.
- There is auxiliary gas flow for the Probe Body (or flush gas)
- There is also auxiliary gas flow for cooling (or warming) the shim stack
- These auxiliary gas flows can be controlled by the BSVT, or by needle valves

Problems with Temperature control

- The environment, if the temperature of the room varies then the temperature in the probe will also vary, not by as much, but enough to cause line broadening.
- The sample might heat due to the electrical field created by the RF coils in the probe
- There may be joule heating of the RF coils themselves due to resistance and current flow through them
- The sample heating may not be uniform across the length of the sample, causing a temperature gradient or even convection currents
- There may also be evaporation and condensation of the sample fluid in the sample tube The movement can cause NMR instabilities
- Since the sensor is not in the sample itself there will always be an offset between the BSVT reading and the true temperature of the sample. The offset is dependent upon the gas flow rate and the VT gas temperature.

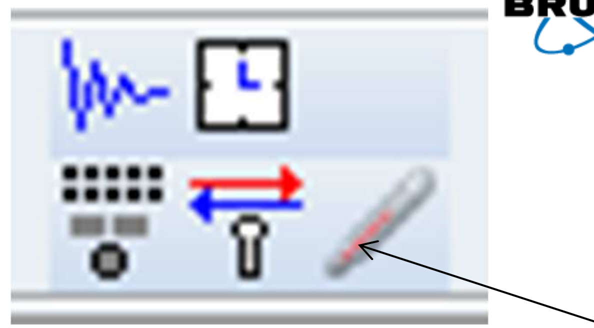
Temperature ranges for different probe classes

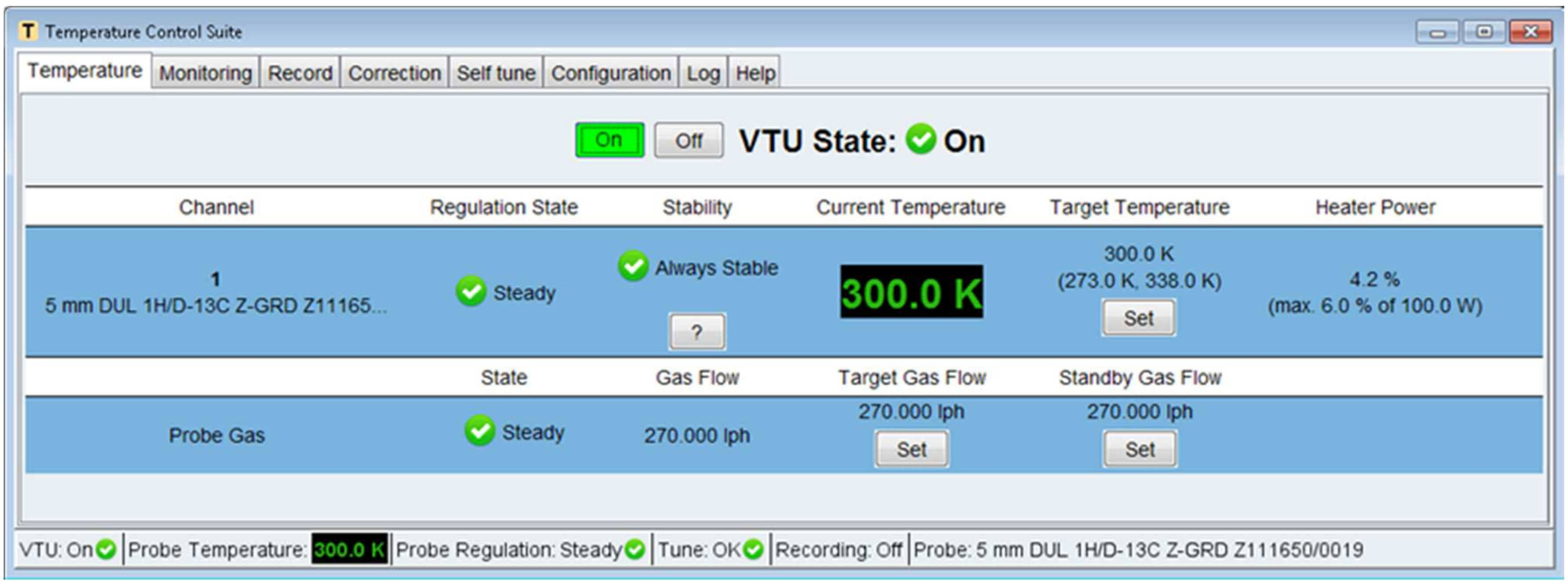
- 5 mm Standard Probes
  - BBO, BBFO, DUL, BBI, TXI, QXI, TBI, -150 -> +150 ^{0} C
- 5mm HT probes
  - DUL, SEF, SEI, SEL, SEX -150 -> +600 ^{0} C
- 5mm LTA
  - DUL, SEX -180 -> +150 ^{0} C
- Probes with an XYZ gradients are restricted to less than +80 ^{0} C

- In any case see your specification sheet for your probe before undergoing any temperature studies.

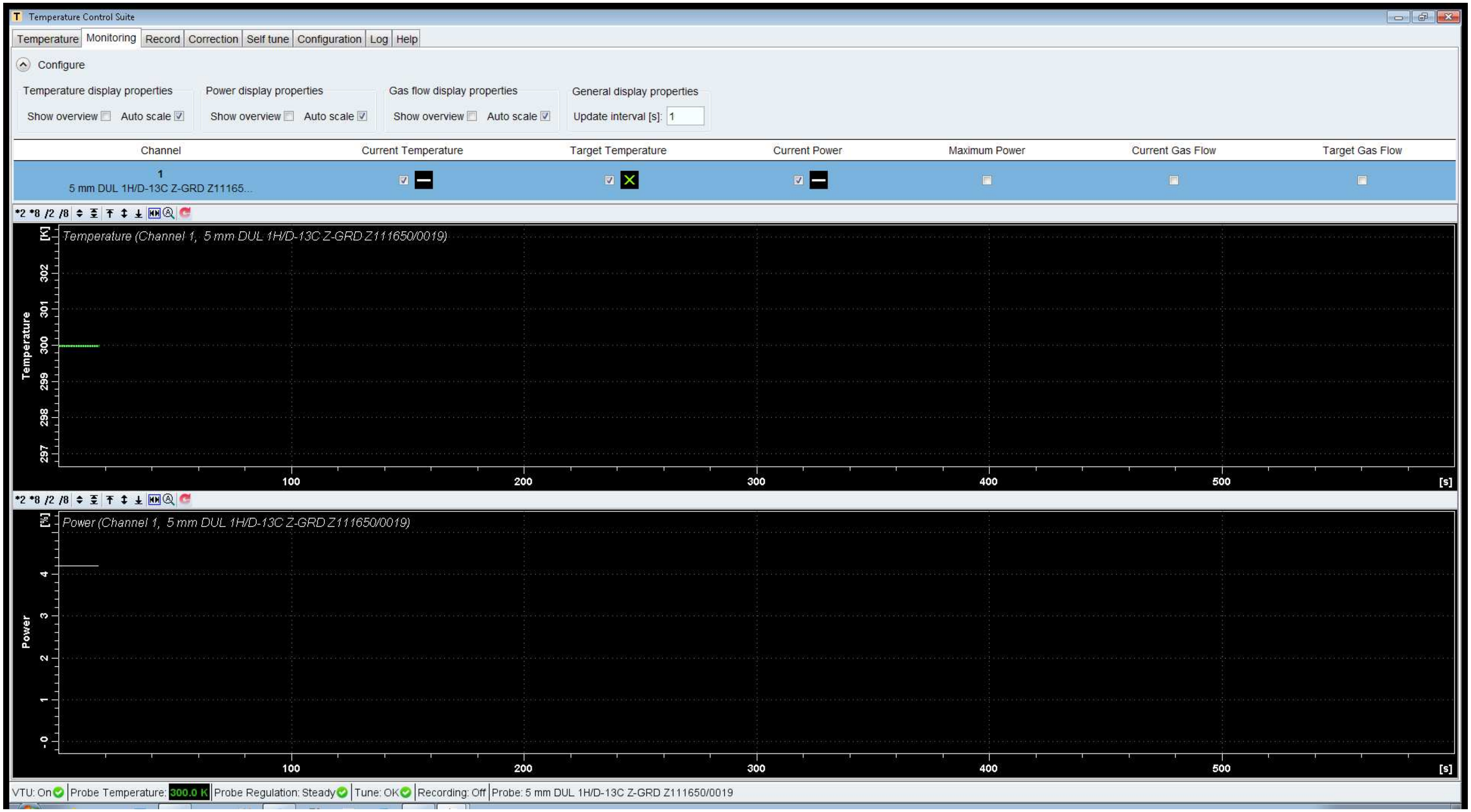
New EDTE

- You can open the new EDTE by clicking the “Thermometer Icon” at the top row

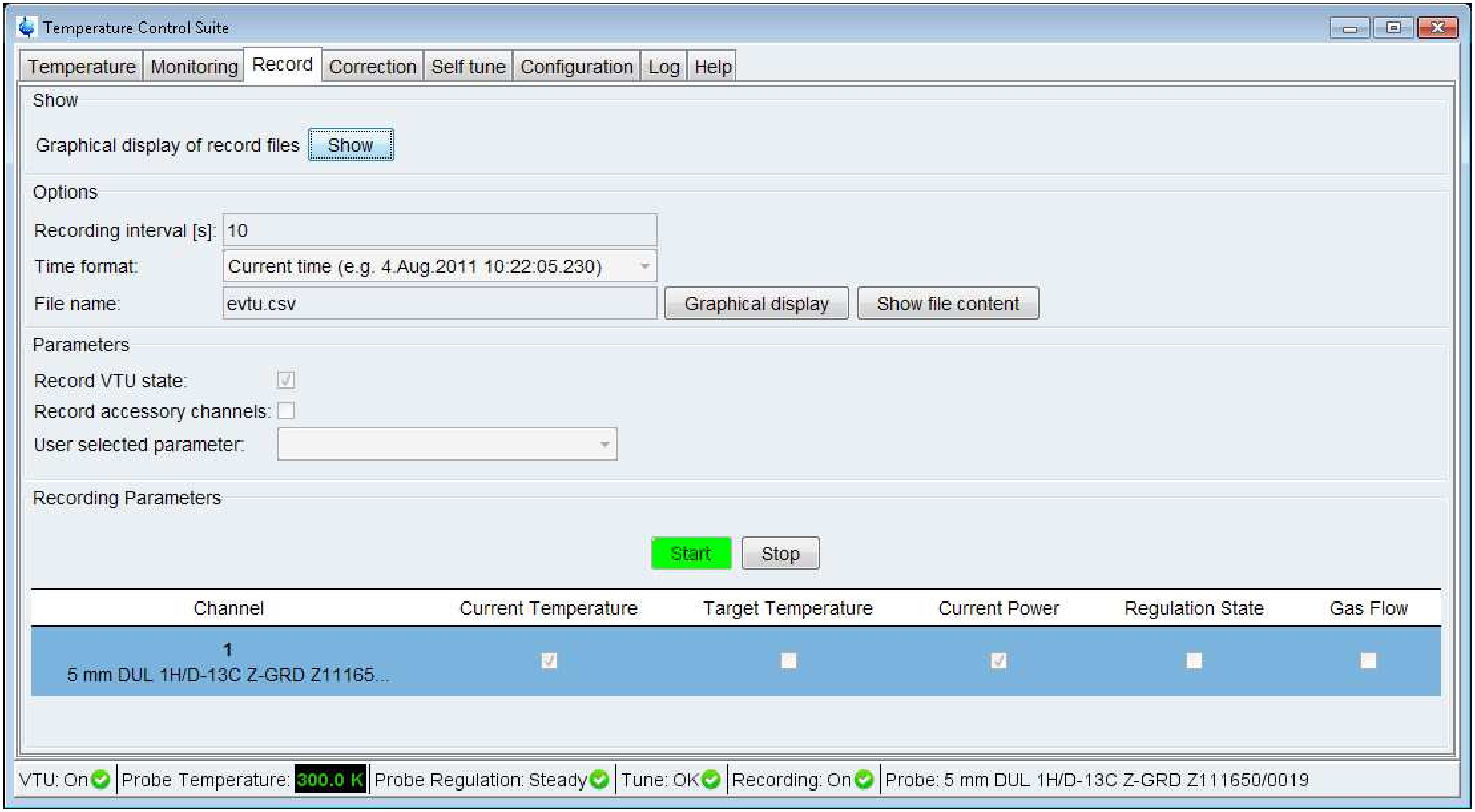




New EDTE

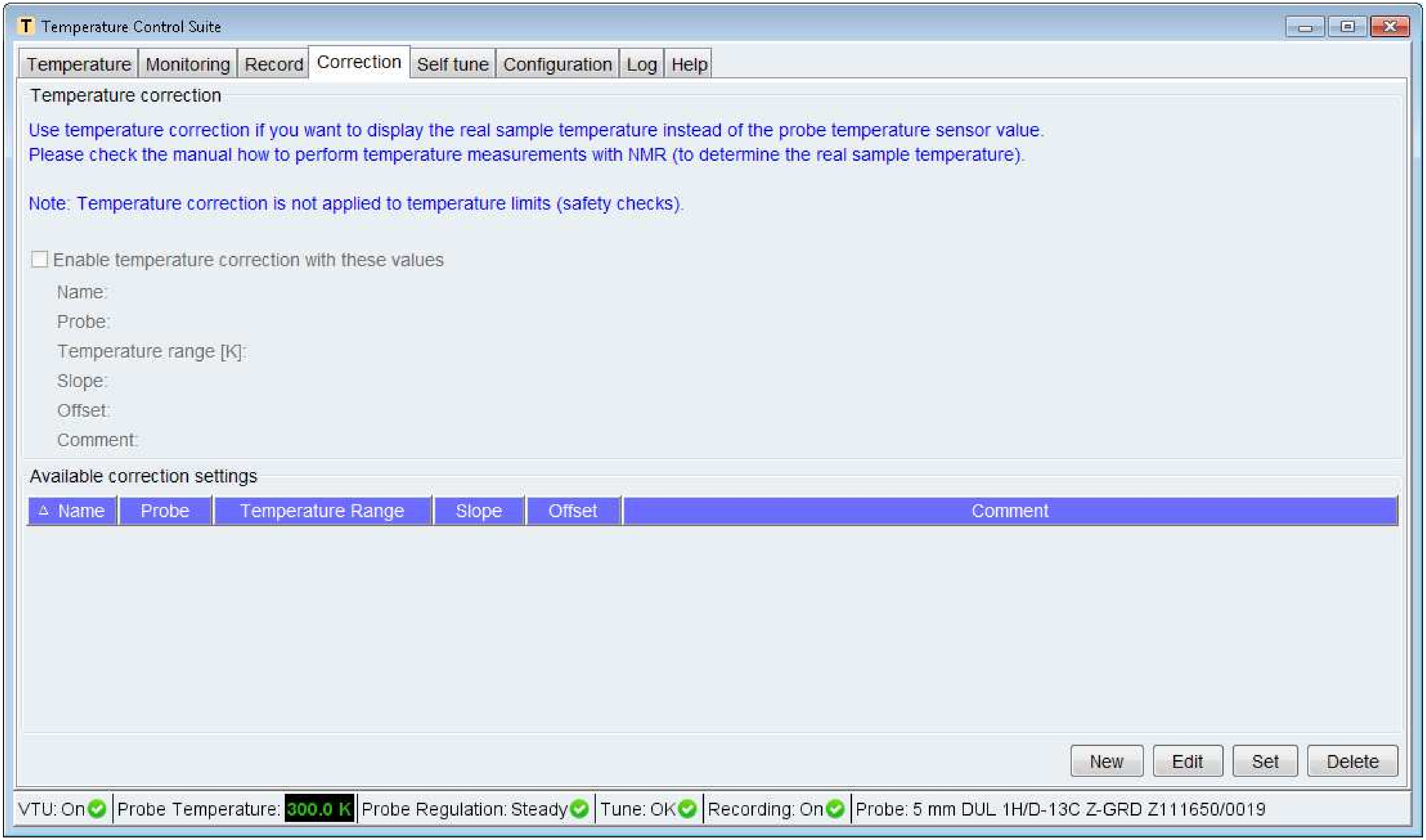

Temperature Control Suite
Temperature
Monitoring
Record
Correction
Self tune
Configuration
Log
Help
Configure
Temperature display properties
Show overview
Auto scale
Power display properties
Show overview
Auto scale
Gas flow display properties
Show overview
Auto scale
General display properties
Update interval [s]: 1
Channel
Current Temperature
Target Temperature
Current Power
Maximum Power
Current Gas Flow
Target Gas Flow
1
5 mm DUL 1H/D-13C Z-GRD Z11165...
Temperature (Channel 1, 5 mm DUL 1H/D-13C Z-GRD Z111650/0019)
Temperature
[K]
302
301
300
299
298
297
100
200
300
400
500
[s]
Power (Channel 1, 5 mm DUL 1H/D-13C Z-GRD Z111650/0019)
Power
[%]
4
3
2
1
0
100
200
300
400
500
[s]
VTU: On
Probe Temperature: 300.0 K
Probe Regulation: Steady
Tune: OK
Recording: Off
Probe: 5 mm DUL 1H/D-13C Z-GRD Z111650/0019

New EDTE

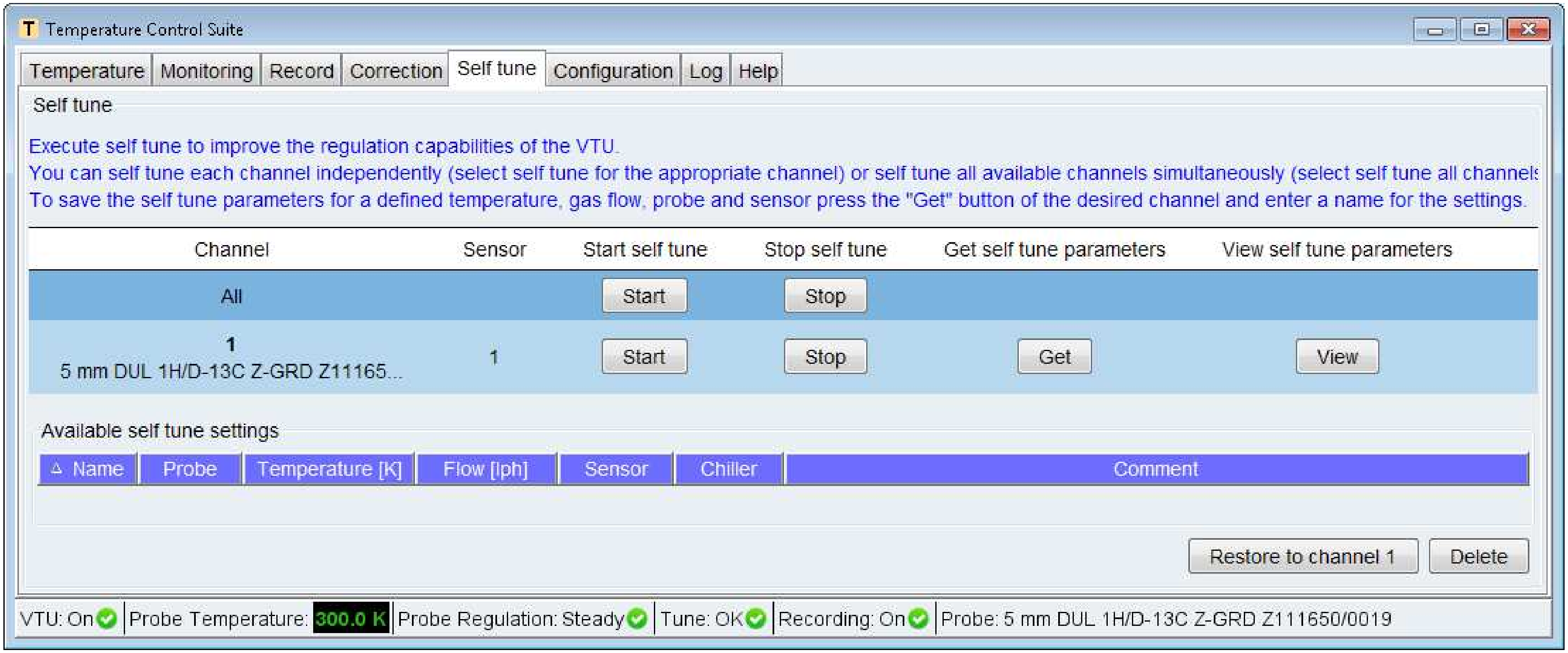


You can record numerous parameters

New EDTE

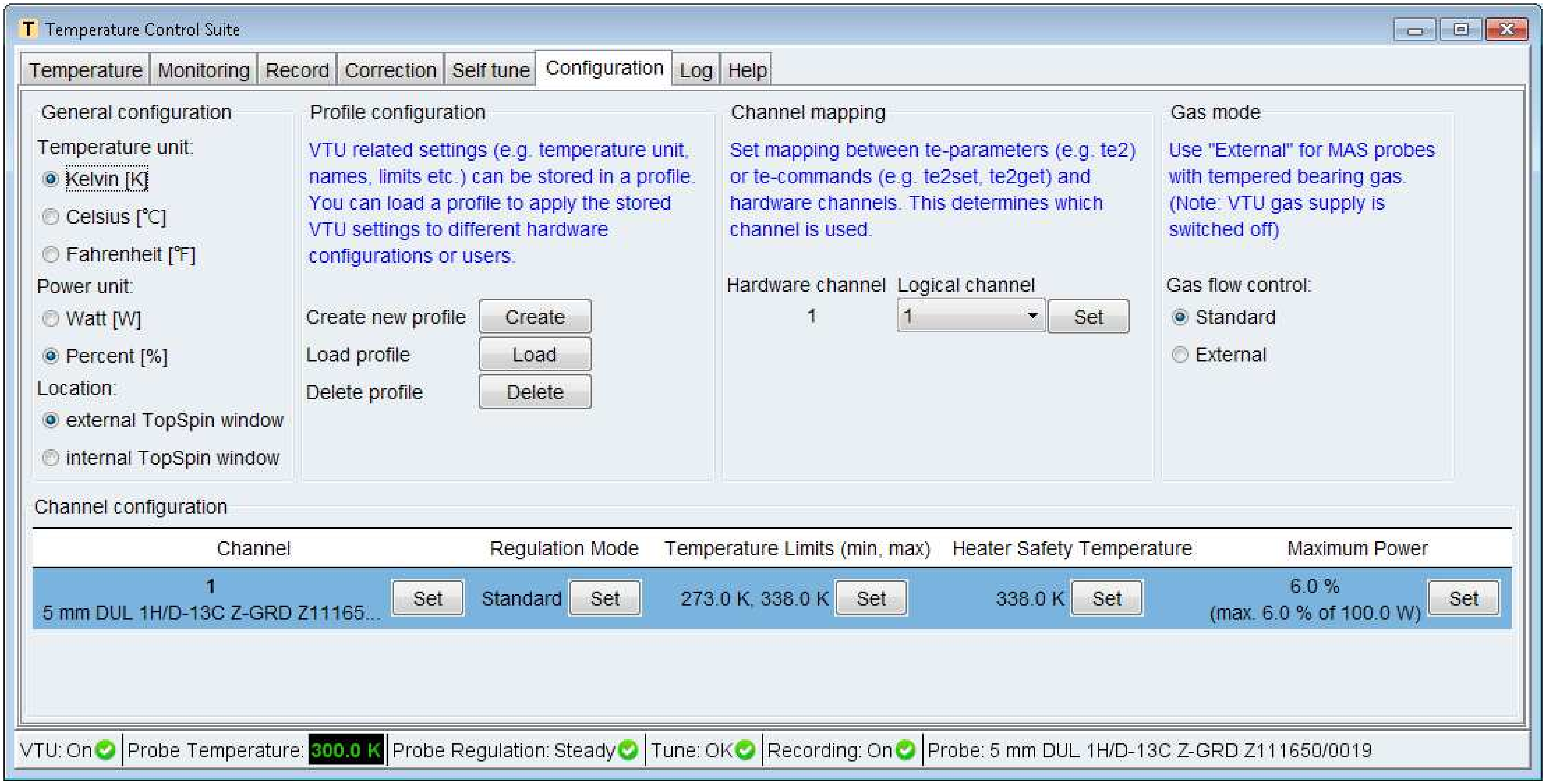

Temperature Control Suite
Temperature
Monitoring
Record
Correction
Self tune
Configuration
Log
Help
Temperature correction
Use temperature correction if you want to display the real sample temperature instead of the probe temperature sensor value.
Please check the manual how to perform temperature measurements 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:
Probe:
Temperature range [K]:
Slope:
Offset:
Comment:
Available correction settings
Name
Probe
Temperature Range
Slope
Offset
Comment
New
Edit
Set
Delete
VTU: On
Probe Temperature: 300.0 K
Probe Regulation: Steady
Tune: OK
Recording: On
Probe: 5 mm DUL 1H/D-13C Z-GRD Z111650/0019

New EDTE



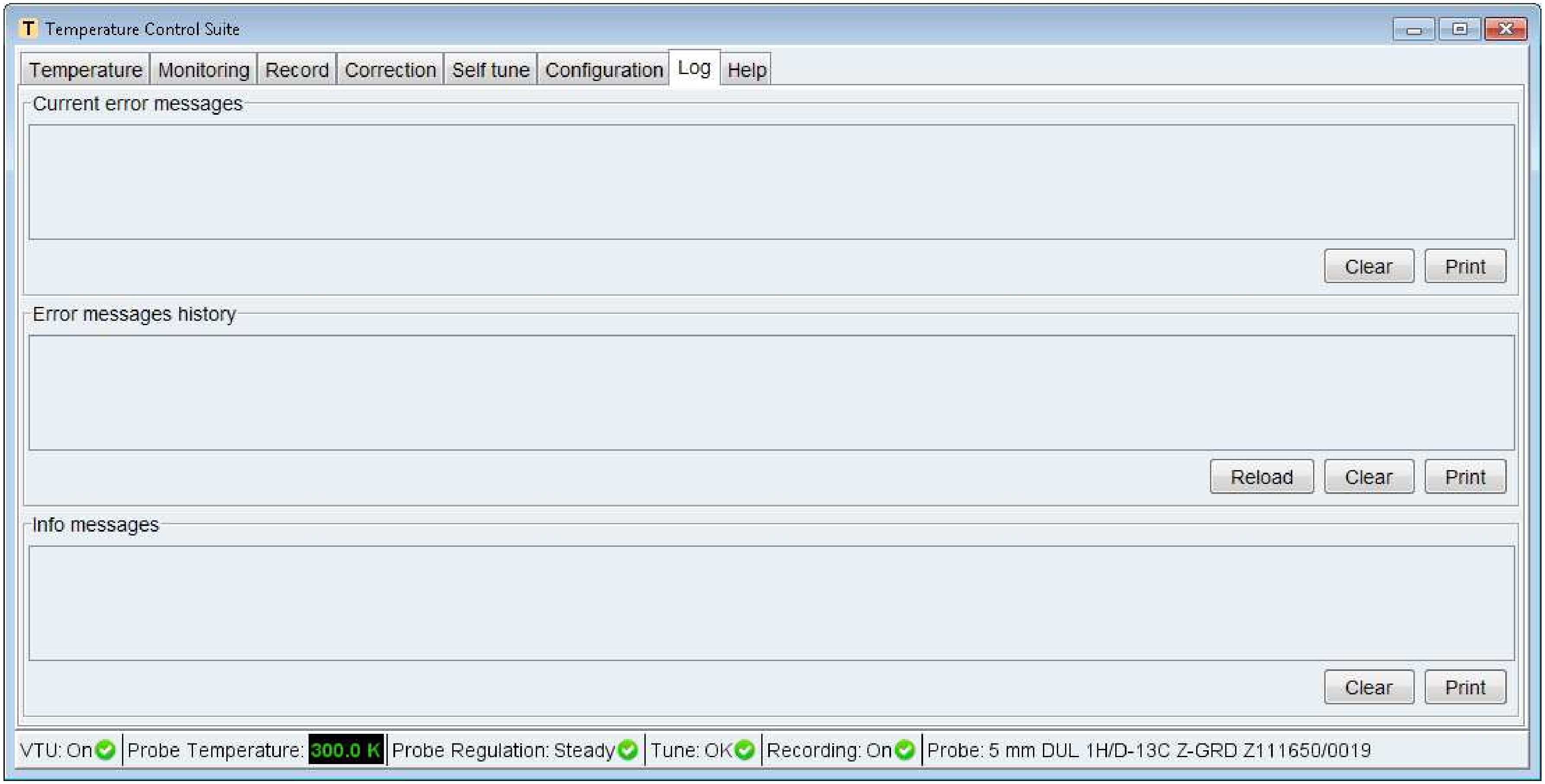
Temperature Control Suite
Temperature
Monitoring
Record
Correction
Self tune
Configuration
Log
Help
Self tune
Execute self tune to improve the regulation capabilities of the VTU.
You can self tune each channel independently (select self tune for the appropriate channel) or self tune all available channels simultaneously (select self tune all channels
To 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 DUL 1H/D-13C Z-GRD Z11165...
1
Start
Stop
Get
View
Available self tune settings
Name
Probe
Temperature [K]
Flow [lph]
Sensor
Chiller
Comment
Restore to channel 1
Delete
VTU: On
Probe Temperature: 300.0 K
Probe Regulation: Steady
Tune: OK
Recording: On
Probe: 5 mm DUL 1H/D-13C Z-GRD Z111650/0019

New EDTE



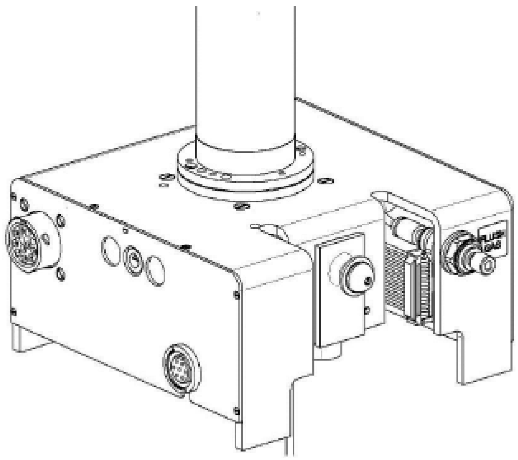
Temperature Control Suite
Temperature
Monitoring
Record
Correction
Self tune
Configuration
Log
Help
General configuration
Temperature unit:
Kelvin [K]
Celsius [°C]
Fahrenheit [°F]
Power unit:
Watt [W]
Percent [%]
Location:
external TopSpin window
internal TopSpin window
Profile configuration
VTU related settings (e.g. temperature unit, names, limits etc.) can be stored in a profile. You can load a profile to apply the stored VTU settings to different hardware configurations or users.
Create new profile
Create
Load profile
Load
Delete profile
Delete
Channel mapping
Set mapping between te-parameters (e.g. te2) or te-commands (e.g. te2set, te2get) and hardware channels. This determines which channel is used.
Hardware channel
Logical channel
1
1
Set
Gas mode
Use "External" for MAS probes with tempered bearing gas. (Note: VTU gas supply is switched off)
Gas flow control:
Standard
External
Channel configuration
Channel
Regulation Mode
Temperature Limits (min, max)
Heater Safety Temperature
Maximum Power
1
5 mm DUL 1H/D-13C Z-GRD Z11165...
Set
Standard
Set
273.0 K, 338.0 K
Set
338.0 K
Set
6.0 %
(max. 6.0 % of 100.0 W)
Set
VTU: On
Probe Temperature: 300.0 K
Probe Regulation: Steady
Tune: OK
Recording: On
Probe: 5 mm DUL 1H/D-13C Z-GRD Z111650/0019

New EDTE

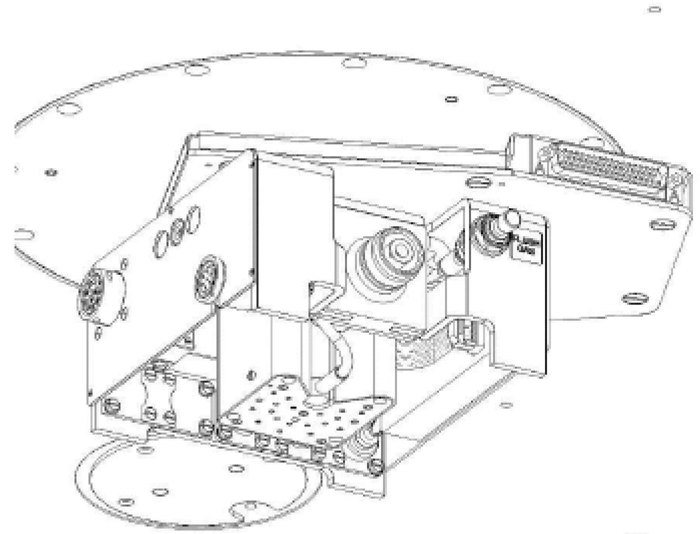


Temperature Control Suite
Temperature
Monitoring
Record
Correction
Self tune
Configuration
Log
Help
Current error messages
Clear
Print
Error messages history
Reload
Clear
Print
Info messages
Clear
Print
VTU: On
Probe Temperature: 300.0 K
Probe Regulation: Steady
Tune: OK
Recording: On
Probe: 5 mm DUL 1H/D-13C Z-GRD Z111650/0019

New BSVT



Probe socket with old style ball seal VT connector and flush gas



Probe socket with new Johnston VT connector and flush gas

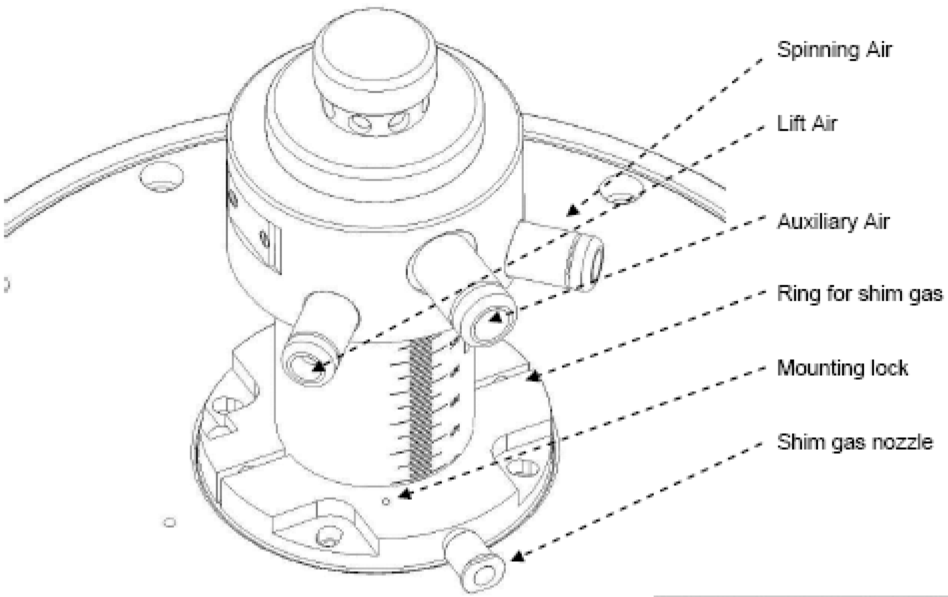
Flush Gas

- When operating probes at high and especially at low temperature, the inner volume of the probe body is thermally stressed.
  - At the upper temperature edge, tuning and matching networks are heated and original tune and match can drift as long as the body temperature is not in equilibrium.
  - At low temperatures, icing is the main problem and can block mechanically moving parts or lead to arcing due to surface conductivity.
- All RT probes have a flush gas connector. The gas supply, as shown next, feeds dry gas (N2 or air – always the same as VT gas) to the upper space where the electrical network is located, directly below the coil space. The gas must have a dew point below -50 C to prevent freezing caused by humidity. The gas then flushes downwards and exits the probe body. Using the flush gas (recommended 5…10 l/min), long term experiments are possible even at the temperature extremes, also at -150° C for Standard RT probes.
- Flush gas is mandatory for operation at low temperature and recommended at the upper temperature limits.
- Flow rate is from 5 to 10 Liters/min

Shim Protection and magnet protection

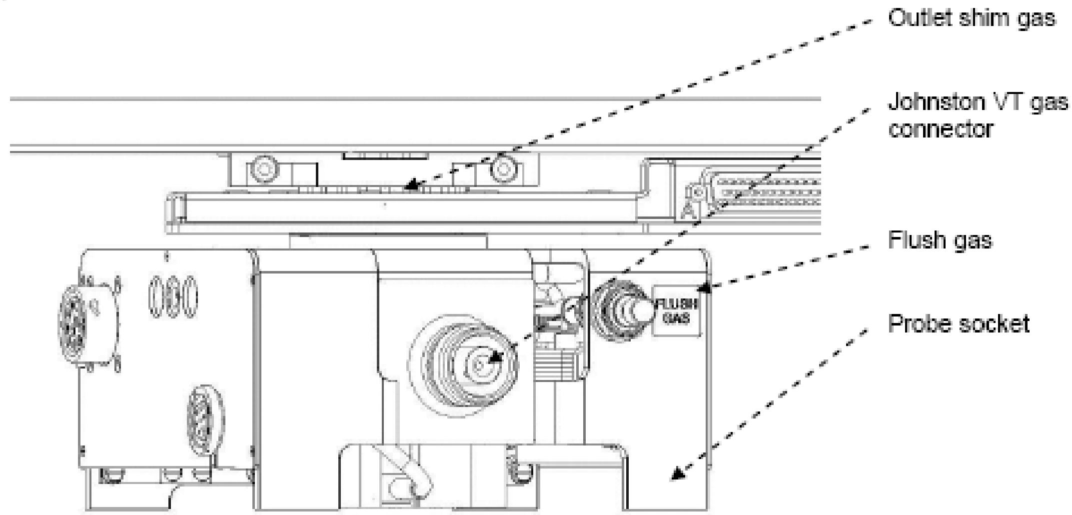
- issues to be aware of:
  - Maximal shim coil temperature
  - Minimum temperatures allowed at the upper and lower magnet flanges
- The shim coil is a package of epoxy and copper flex prints (x-y shims) and conventional copper coils (z shims).
- The epoxy can be damaged if the coil is heated to higher than +80° C.
- Shim stack gas must be activated whenever a warning message is displayed in TopSpin, otherwise the system will shut down. For experiments at low temperature (e.g. -150° C), the shim coil may reach a temperature as low as -52° C.
- Shim gas is in this case not used for “coil heating”, it is used to dry (prevent freezing) the whole shim system and to stabilize the falling temperature within the shim coil and BST.
- With this, the temperature of the magnet flanges cannot fall under the critical temperature of +3° C and with no risk of a magnet quench, occurring as a result of the Dewar O-rings freezing.

Shim Protection and magnet protection



Flow rates for RT Shim stack are from 0 to 60 Liters/min

Flow rates for flush gas are from 5 to 10 Liters/min



Spinners

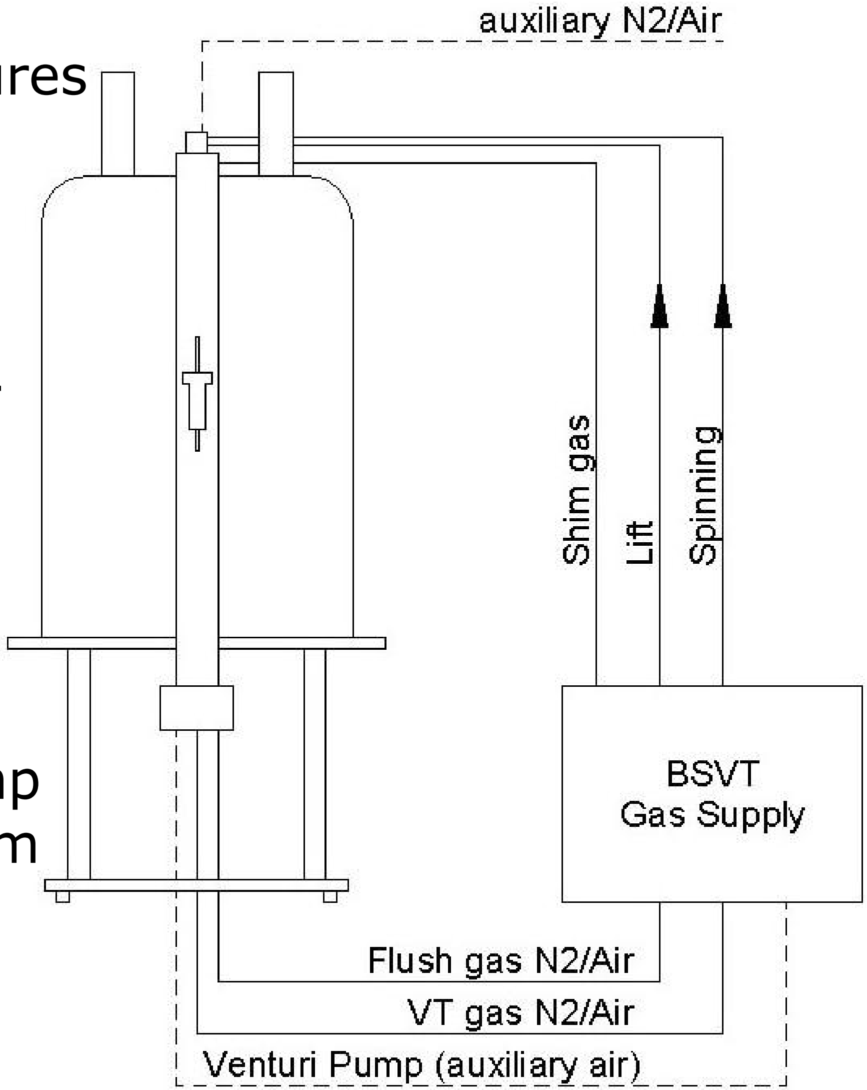
Z42516 Standard POM Spinner 0° C to +80° C

H00177 Kel-F Spinner for elevated Temperatures +80° C to +120° C

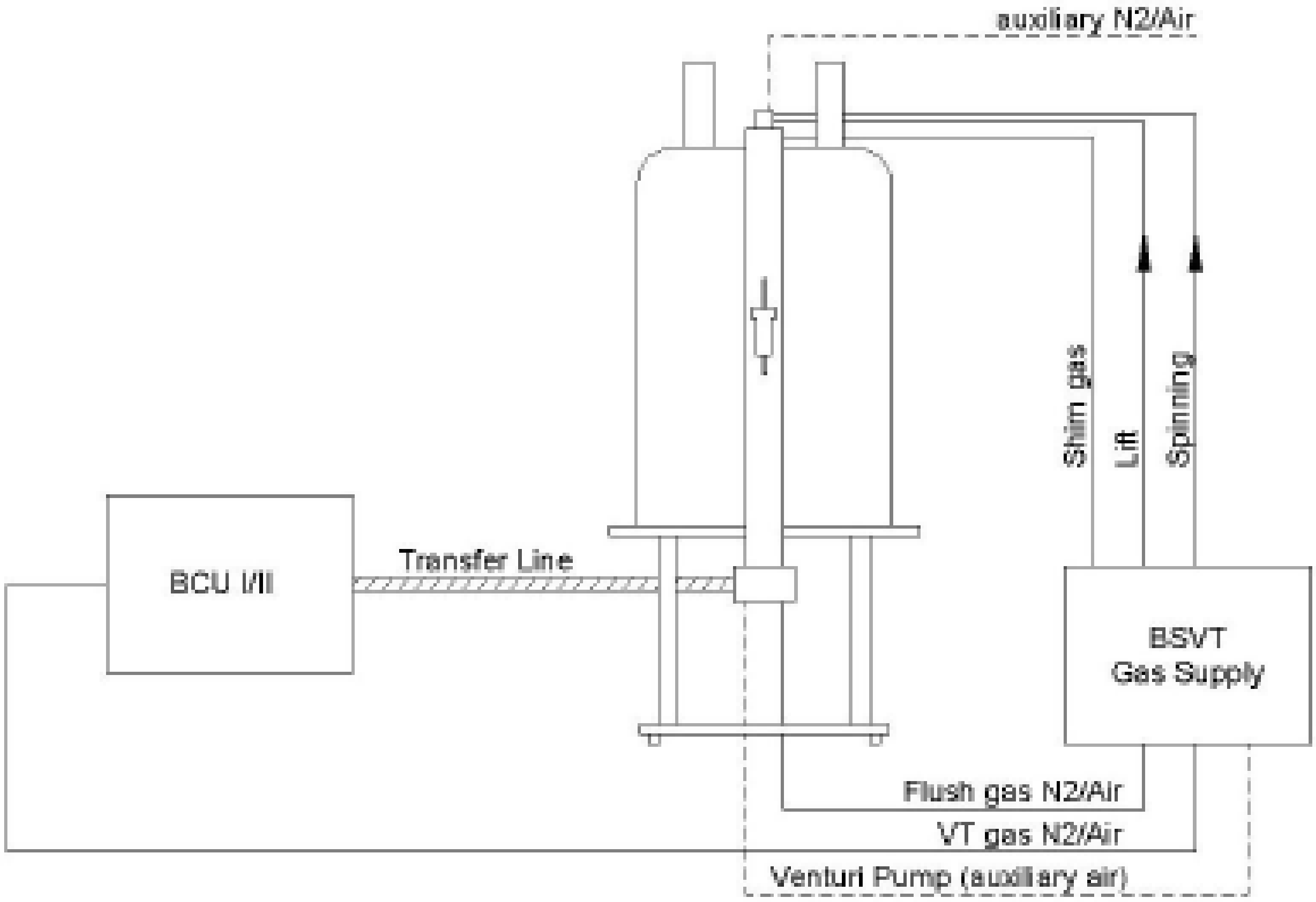
H00804 Ceramics Spinner for high and low Temperatures
+120° C to +180° C and 0° C to -150° C

Configuration for standard RT and elevated temperatures- short term

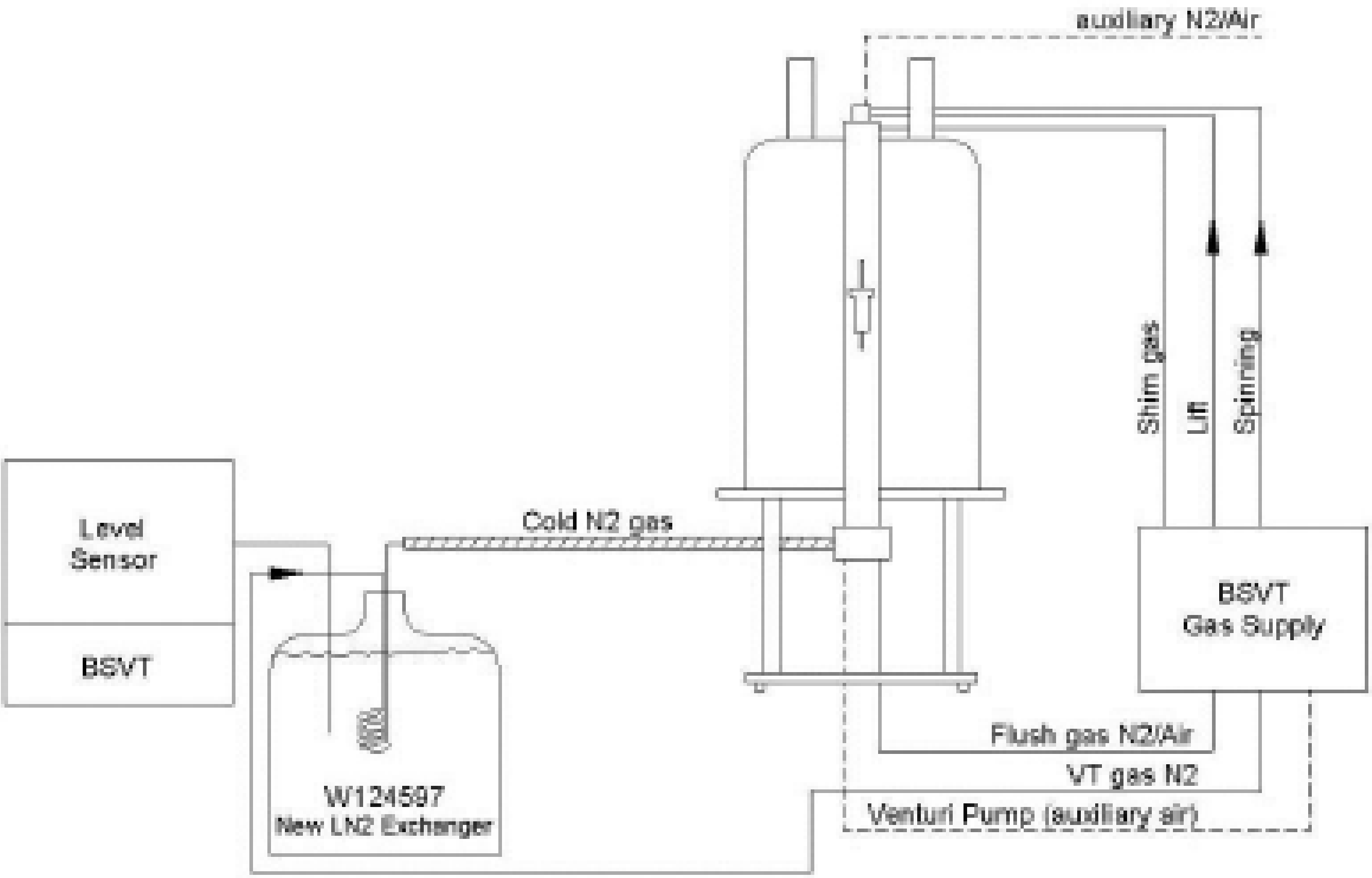
- Standard RT & elevated temperatures VT gas: Use dry air for ≤ 400 MHz and N2 (purity ≥ 95%) for higher frequencies
- Flush gas: Use same as VT gas for probe drying at low temperatures
- Venturi pump: Used to cool FT probes
- Shim gas: Use dry air (for low temp experiments) or normal air for shim cooling
- Lift: Same type as VT gas
- Spinning: Same type as VT gas



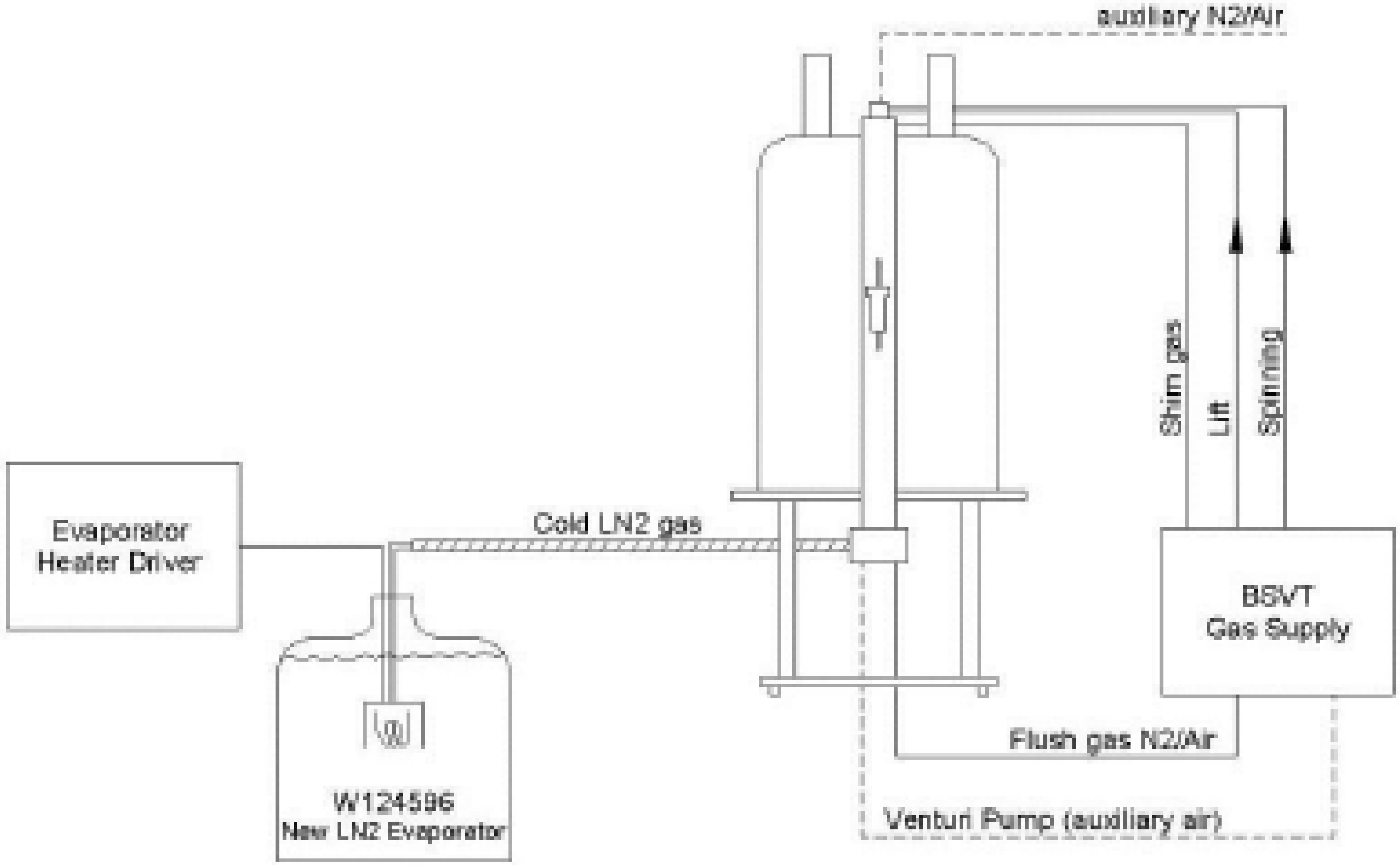
Configuration for RT applications down to 0° C and -40° C



Configuration for low temperatures down to -150° C with LN2 Heat Exchanger



Configuration for low Temperatures down to -150° C with LN2 Evaporator



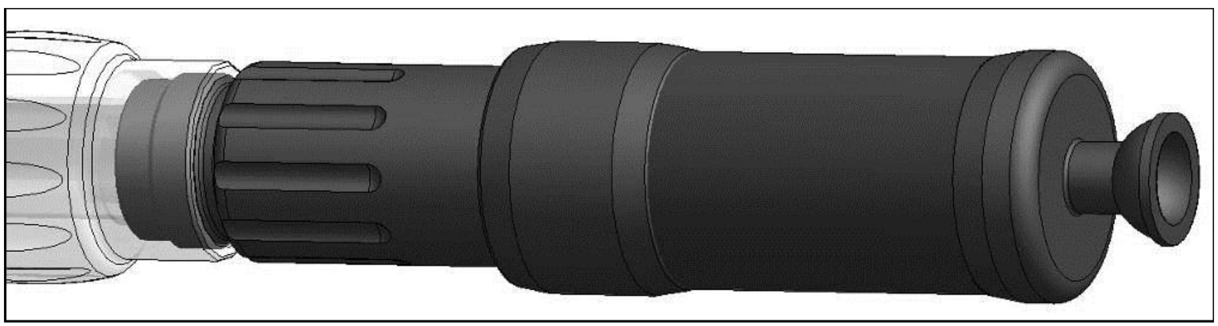
Recommended operating conditions for probe temperature control

Sample T [°C]	-150 … -80	-80 … 0	0 ... 80	80 ... 120	120 ... 150	150 ... 180
VT gas [l/h]	1800 … 1200	1200 … 670	670 ... 535	535 ... 670	535 ... 670	535 ... 800
Recom. VT gas [l/h]	1200	1000	535	535	535	535
Shim gas [l/min]	20	20	0	0 … 20	20 … 30	40 … 60
Flush gas [l/min]	5…10	5	0	5	5	5
Spinner	Ceramics	Ceramics	POM	Kel-F	Ceramics	Ceramics
Chiller	LN2 heat ex-changer or LN2 evaporator	BCU II (-40°C) or LN2 heat exchanger / LN2 evaporator for lower temp	BCU I for $T<T_{RT}$			

New Johnsten VT gas coupler

New Johnsten VT gas coupler for RT probes female

New Johnsten VT gas coupler for RT probes male



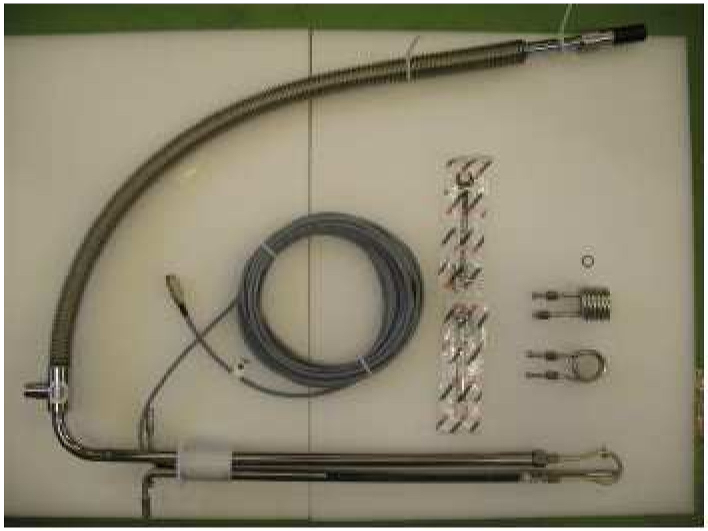
Adapter

New Temperature control Hardware

BCU-1 is the successor to the BCU-05
BCU-1 will go down to -40C

BCU-2 is the successor to the BCU Extreme
BCU-2 will go down to -80C

LN2 Exchanger



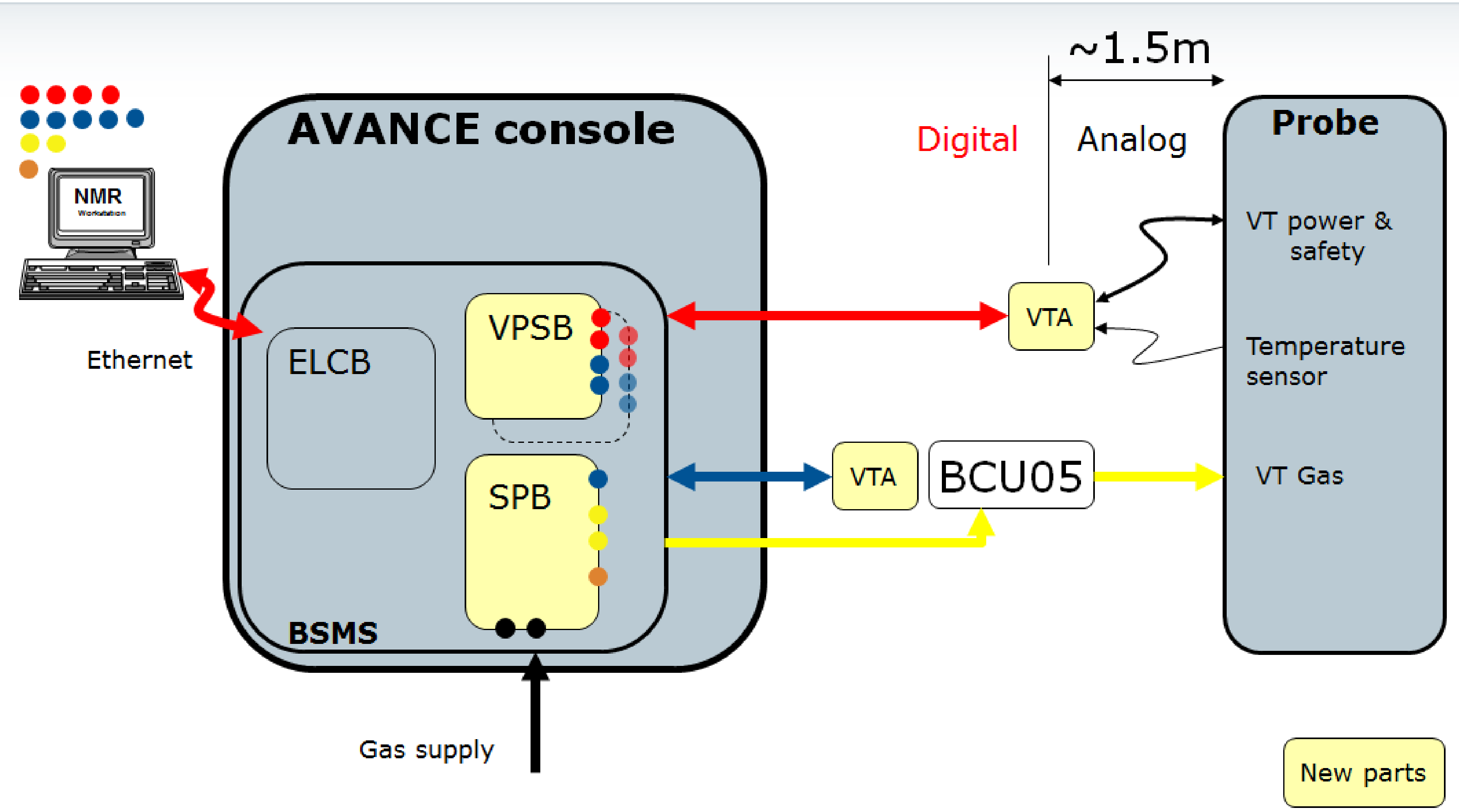
- An LN2 heat exchanger system cools the VT gas coming from a B(S)VT via a heat exchanger immersed in LN2 (Dewar vessel).

- A heat exchanger has to be used for low temperature measurements with sample temperatures between -40° C and -150° C

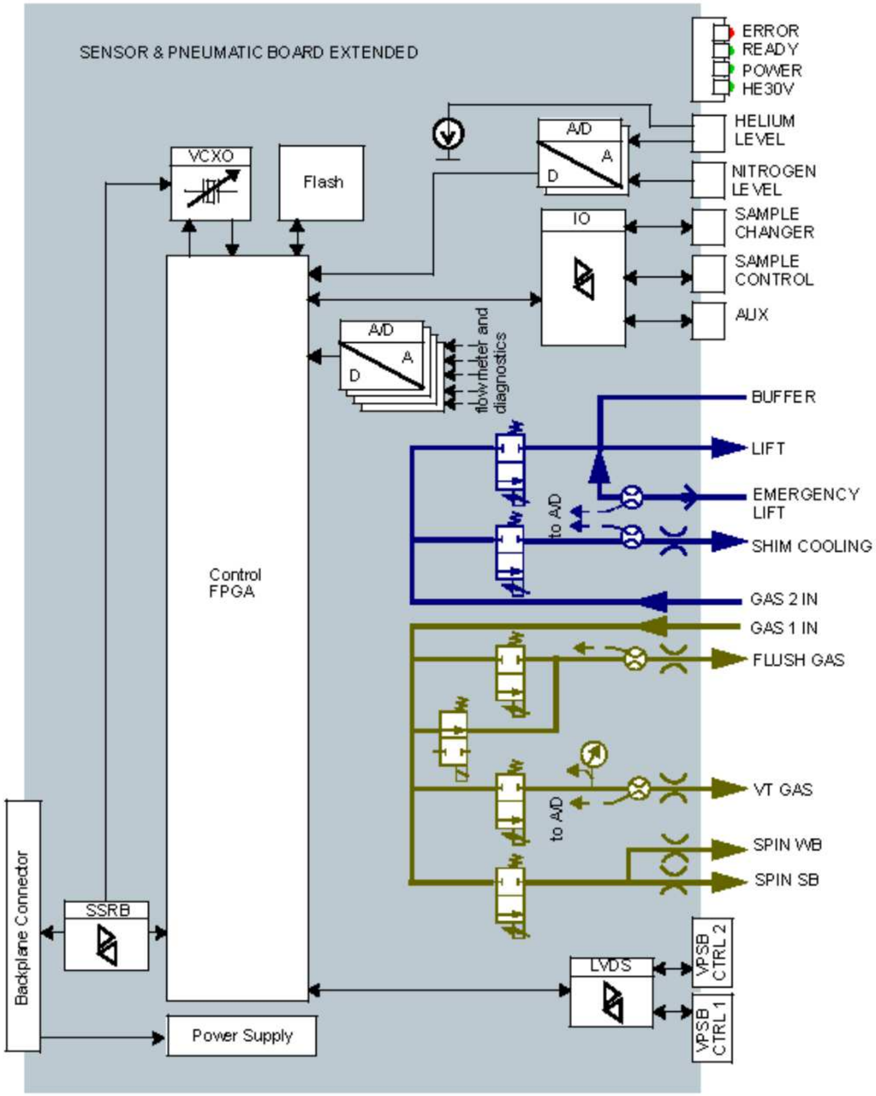
Evaporator Kit with Johnston coupling

The evaporator W124596 produces unsaturated LN2 gas by boiling liquid nitrogen. Special heater drivers are required for operation (see specific product descriptions). For the lowest possible temperatures (e.g. -40° C to -150° C).

The BSVT Temperature controller system



BSVT – Sensor & Pneumatics Board



Example Extended Version

Main characteristics:

- Gas flow sensors
- Pressure sensor
- Power Off mode
- Shim Cooling/Flush Gas
- Emergency Lift input
- Variants

SPB	SB	2000 lph
SPB-E	SB&WB	3000 lph

- Plug & play capability

Benefits:

- Intelligent Spin / Lift / VT control
- Application Integrity
- Full VT system supervision
- Enhanced Probe and Sample Safety

VT interfaces (VTA)

For specific applications a wide variety of temperature sensors and heater interfaces must be supported. Some NMR probes need standard thermocouple sensors type-T, others need PT100 thermistors, some need two sensors etc.

In order to obtain precise and accurate temperature measurements the analogue sensor signal must not be carried over long distances and connector junctions must be minimized. Many users want to work with different NMR probes and a change of the sensor adaptation must be simple

For every temperature sensor and heater adaptation variant or other accessory device a tailored VTA is available but only one type of cable connection is needed for probe to console adaptation. This cable carries wires for digital signals, low-voltage power supply and the heater power.

VT interfaces (VTA)

VTA for RT probes (thermocouple type T)

VTA for CryoProbe

Supported temperature sensors:

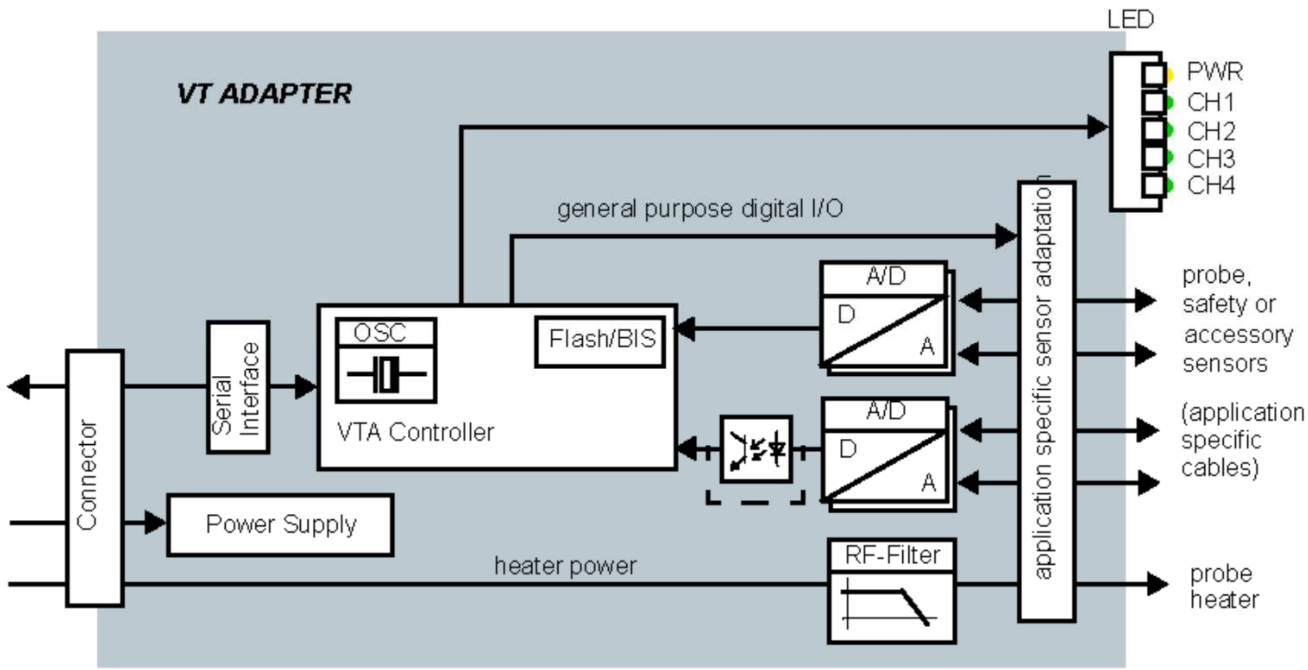
- thermocouple type T (Z119237 BSMS/2 VTA TC-2T with dual sensor and type K safety thermocouple, Z120851 BSMS/2 VTA FLOW-NMR single sensor and PT100 type safety sensor)
- thermocouple type E (Z120728 BSMS/2 VTA TC-2E, dual sensor)
- thermistors type PT100 (Z116923 BSMS/2 VTA CRP)
- thermistors type PT100 (Z119239 BSMS/2 VTA AUX-2P)special sensors like B-TO2000 (Z116924 BSMS/2 VTA BTO)

New VT gas chillers like **BCU-I** and **BCU-II** can be connected ***directly*** to the BSVT system.

VT interfaces (VTA)

Benefits:

- Full integrity with Probe (i.e. warning when missing connection)
- Indication whether power is applied or not
- Automatic identification (BIS) and configuration
- modular concept / VT adapter for any temperature sensing solution
- Plug & play operation



Bruker BioSpin

Operating RT probes at temperatures 0° C to +80° C

The temperature range from 0 to 80° C is considered the standard temperature range from the application point of view. This range is covered by most of the probeheads (RT and CryoProbe).

For temperatures down to 0° C (less than room temperature) a cooling unit (BCU I) is necessary and recommended, otherwise you won't reach the desired temperature. In principle it is also possible to use low temperature equipment. (BCU II or N2 heat exchanger)

To go up to 80° C (significantly more than room temperature) you don't necessarily need additional equipment.

Are there any questions ?

Manuals used:

“NMR Probes, NMR Variable Temperature Control for NMR Probes Technical Manual” Version 001
Bruker Part Number: Z33073

“BSMS/2 Systems with ELCB” Version 005
Bruker Part Number: Z108028

www.bruker-biospin.com