



• NMR Probes

Variable Temperature Control for NMR Probes
User Manual

Version 002



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1 About

1.1 This Manual

This manual is intended to be a reference guide for operators and service technicians. It provides detailed information about the user level maintenance and service and overall use of the Bruker device.

The figures shown in this manual are designed to be general and informative and may not represent the specific Bruker model, component or software/firmware version you are working with. Options and accessories may or may not be illustrated in each figure.

Carefully read all relevant chapters before working on the device!

This manual describes parts and procedures relevant to the device version it is delivered with. For older hardware, please refer to the manual supplied at the time.

1.2 Policy Statement

It is the policy of Bruker to improve products as new techniques and components become available. Bruker reserves the right to change specifications at any time.

Every effort has been made to avoid errors in text and figure presentation in this publication. In order to produce useful and appropriate documentation, we welcome your comments on this publication. Support engineers are advised to regularly check with Bruker for updated information.

Bruker is committed to providing customers with inventive, high quality products and services that are environmentally sound.

1.3 Symbols and Conventions

Safety instructions in this manual are marked with symbols. The safety instructions are introduced using indicative words which express the extent of the hazard.

In order to avoid accidents, personal injury or damage to property, always observe safety instructions and proceed with care.



WARNING

This combination of symbol and signal word indicates a potentially hazardous situation which could result in death or serious injury unless avoided.



⚠ CAUTION

This combination of symbol and signal word indicates a possibly hazardous situation which could result in minor or slight injury unless avoided.

NOTICE

This combination of symbol and signal word indicates a possibly hazardous situation which could result in damage to property or the environment unless avoided.

i This symbol highlights useful tips and recommendations as well as information designed to ensure efficient and smooth operation.

1.4 Disclaimer

This guide should only be used for its intended purpose as described in this manual. Use of the manual for any purpose other than that for which it is intended is taken only at the users own risk and invalidates any and all manufacturer warranties. Some parameter values, especially temperature levels suggested in this manual may not be suitable for all systems and could cause damage to the unit. Therefore only persons trained in the operation of the Bruker AVANCE NMR systems should operate the unit.

2 Introduction

Goal of this manual

This manual has been set up in context with the introduction of a completely revised temperature control equipment for Bruker NMR spectrometers.

With the introduction of the new BSVT electronics and pneumatics, also a generation of new chillers as BCU I (probe cooling 0°C), BCU II (probe cooling -40°C), LN2 heat exchanger and LN2 evaporator (down to -150°C) has been established, using the new and very efficient Johnston VT gas coupling system for room temperature probes, efficiently preventing icing effects.

This is a significant step forward in modern probe temperature control and the new concept replaces the former components based on the BVT series.

This manual has been written primarily for our service engineers to give a clear picture of how to set up a temperature control system to meet the customer's requirements. This document is not a customer User Manual and it is solely for internal use only. It is also our intention not to go into deep details and to only relate to technical descriptions specifically required (e.g. BSVT description).

Why is probe temperature control needed?

a) Operation at room temperature

This is the most common mode of operation in NMR. But depending on experiments, spectral shifts generated from changes in sample temperature are not desired and often disturbing. This may occur during longer experiment times (day, night) where even small changes of the laboratory ambient temperature may lead to minor drifts of spectral components (e.g. sucrose, DDS). For this reason, also for room temperature measurements the sample may be stabilized by using a chiller and the system regulates the sample temperature to for example exactly 23°C.

b) Operation at elevated temperatures

For such experiments, internal probe heating is sufficient and precisely regulated with the new BSVT, which also controls (regulates) the selected VT flow rates and other auxiliary flows. Chillers are not required as long as the operating temperature is sufficiently above the room temperature.

c) Operation at temperatures below room temperature

These experiments require chillers and often there are problems due to wrong, old equipment or insufficient knowledge. Especially for very low temperature experiments this manual should be used as a guide. It should also answer questions as how long can a low temperature experiment be performed, what is the risk of magnet quench, can the probe be damaged, what should be taken into account when warming up the system etc.

Introduction to different temperature control methods

In this manual also an overview about different temperature control methods is given, especially because probe temperature control also experienced some evolution within the past decades. We understand it as quite important to have knowledge about this and to be able to explain to customers what the physical properties - what the limitations are and what the system behavior is. Also some extracts from Bruker's requirements for probe temperature control shall be presented.

System configuration and application examples

A typical system set up, along with the required system settings and recommendations, will be described for all of the temperature ranges covered. Details of components, such as chillers, are described in other chapters. The reader of this manual shall also be guided through the corresponding TopSpin menus explanations of warnings and error messages will be given. Also included are hints and tips for trouble shooting.

Probe & Electronics departments BBIO-CH

3 Principle of VT Control for NMR

3.1 How the Temperature of a NMR Sample Can Be Controlled

In general, NMR experiments are sensitive to small changes of the sample temperature. On the one hand, it is possible to measure temperature changes of less than 10mK by dedicated NMR experiments. On the other hand, however, even every small deviations of the sample temperature lead to line broadening and shifting of the NMR lines. This often has a negative impact on the quality of the NMR spectra. Consequently, a precise, stable and reliable regulation of the sample temperature is essential.

A principle sketch of the NMR sample temperature control is given in [Figure 3.1](#). Herein, the VT gas flow (air or nitrogen) streams through a pipe into the sample space, then it 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.

Since the temperature sensor is not inside the sample, the sensor will always measure an offset to the sample temperature. This offset depends on the location of the sensor, the flow velocity and the VT gas temperature. The offset increases almost linearly with the VT gas temperature, and consequently it can be corrected by an appropriate calibration.

The user often performs his NMR experiments at a defined temperature different from ambient temperature. The objective being that the tempering system should accurately control the required sample temperature within a controlled period.

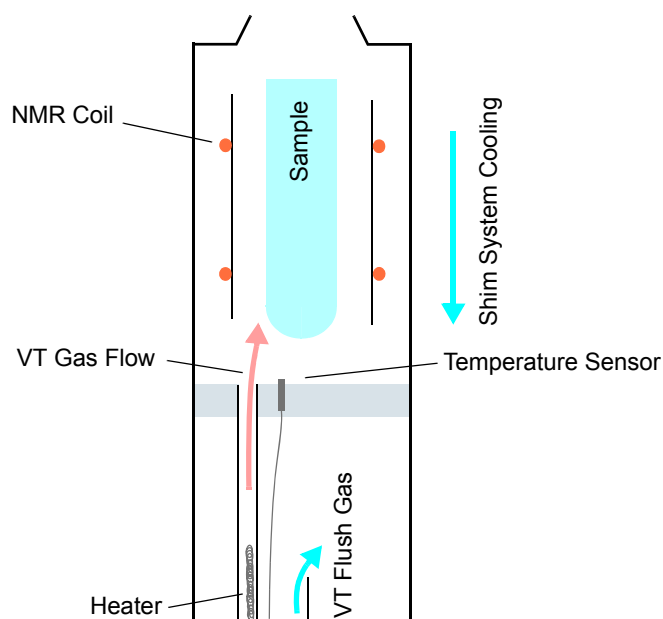


Figure 3.1 Sketch of sample temperature control in a NMR probe

However, the stability of the experiment temperature is subjected to different influences, i.e. the change of the ambient temperature, sample heating caused by RF pulses, joule heating of the NMR coils during RF pulses, flow convection inside the sample, evaporation and condensation of the sample fluid. These effects on the sample temperature are discussed in detail below.

First, the environment of the probe, i.e. the room where the NMR system is operated in, might be subjected to temperature changes caused by the weather or by an air conditioner. These temperature changes can be transmitted to the probe head and consequently the sample. Although this effect may be small, the ambient temperature changes do still influence the sample temperature. Secondly, the sample might be heated by the electrical field of the NMR coils during the experiments. The heating is a function of the electrical losses inside the sample increasing with the salt concentration of the sample fluid.

The next effect is the joule heating of the NMR coils. The electrical resistance of the coils will have a heating effect depending on the RF power applied to them. The generated heat might warm up the VT gas streaming along the coils and finally heat up the sample. Furthermore, the sample heating or cooling effect from the gas flow may not be uniform since it loses heat when it flows along the sample tube. This leads to an undesired temperature gradient over the sample that can cause line broadening. This gradient increases with the VT gas temperature and can even lead to flow convection in the sample, mainly for large sample diameters (10mm and larger). Convection is a severe problem, since it causes temporally instabilities of the NMR measurements.

Another temperature induced effect is the evaporation and condensation of the sample fluid. When the VT gas temperature is close to the boiling point of the sample fluid, a fraction of the sample evaporates. The gaseous sample sometimes condensates at colder regions of the sample tube and forms droplets that can trickle back into the sample fluid. This process destabilizes the NMR measurements.

These mentioned temperature changes have to be handled by the temperature control system that regulate the sample temperature within given bounds.

When the probe head is operated at high temperature, it is important to make sure that the shim system and the magnet bore are not overheated. Therefore, the shim system can be cooled by air flowing from the top of the magnet through holes in the shim system mounting rings. Overheating of the NMR probe can be prevented by flush gas being pumped in the probe via a dedicated pipe. The NMR probes can also be operated at very low temperature. Then it is important also to purge the shim system with dry nitrogen (or dry air), in order to prevent ice forming at the shim system and freezing of the magnet bore. In addition, flush gas should be used to prevent the network components of the probe form ice.

In the following chapters, the temperature control system consisting of the probe, the heating and cooling equipment, and the temperature regulation system will be discussed.

3.2 VT Control for RT Probes

Sample Temperature Control Methods

As described in ["Introduction" on page 9](#), the sample of a probe must be stabilized in temperature in a way, that the convection in the sample fluid is minimal and that any temperature gradient across the active sample volume is reduced as close to zero as possible.

Room temperature probe design has improved over time and today's probes are optimised for maximum heating/cooling efficiency and minimum temperature gradient effects. Probe temperature control evolution steps are known as the very early Dual Flow (direct flow along the sample), FlowTurn (turning flow along the sample for lowest temperature gradients) and today's Extended FlowTurn (turning flow along the sample combined with an integrated bypass to increase the heating/cooling efficiency with higher flows).

Extended FlowTurn (E-FLT)

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).

The extended FlowTurn was introduced in 2010 together with the new Johnston VT gas coupling system, giving higher efficiency to the probes while temperature control. The E-FLT also enables higher flow rates, which is important to achieve the temperature extremes, especially at low temperatures. The Extended FlowTurn will achieve a temperature of -150°C in combination with a LN2 heat exchanger at flow rates of about 1200 l/h.

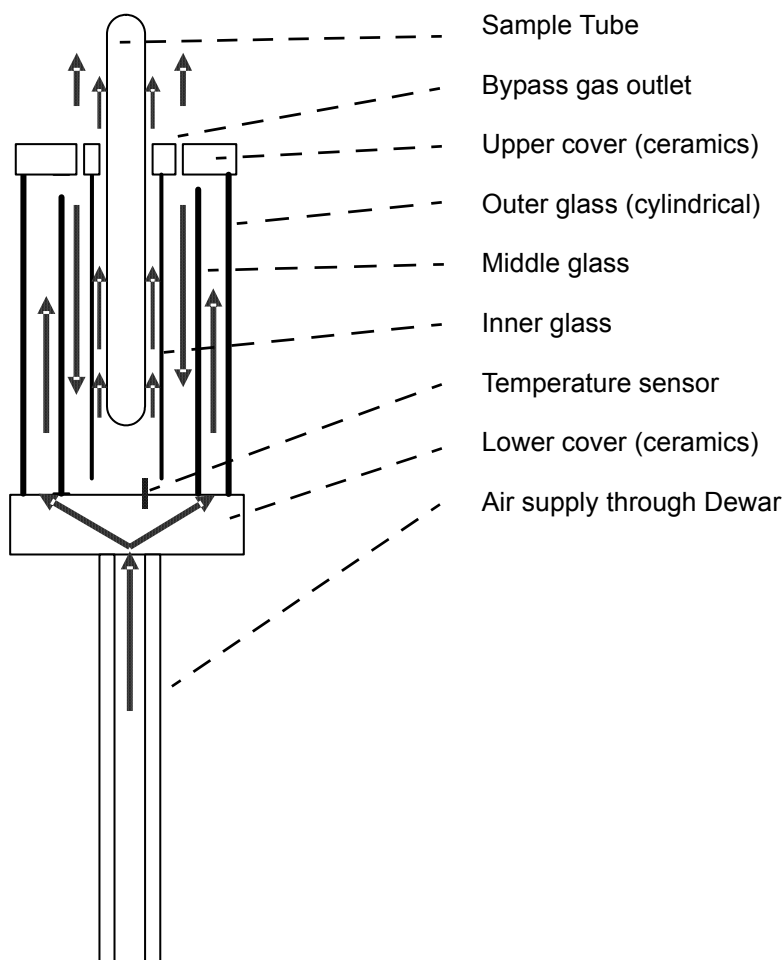


Figure 3.2 Schematic drawing of Extended FlowTurn principle

Description

The VT gas enters the probe through the VT coupling and flows along the well isolated Dewar transfer line within the probe until it reaches the entry to the coil- and sample space. There the flow splits respectively, where it enters and circulates into the outer flow chamber and the middle cylindrical glass. At the upper end, where the flow turns into counter-flow, some part of the VT gas exits the outer flow chamber. This allows higher flow rates and increases the heating/cooling at the outer surface (border) of the coil space. It's there where energy is needed, not inside, since the sample has a small volume and low heat capacity compared to thermal masses as the probe's gradient. After the flow has turned, it produces an opposite temperature gradient at the outer side of the inner glass. After a second turn at the lower end, the remaining gas flows along the sample tube upwards and exits the probe. So this principle allows fast heating/cooling of the sample by keeping a good temperature gradient and is today's standard design along with the new (well isolating) Johnston coupler. Achievable temperature range is -150°C to $+150^{\circ}\text{C}$.

FlowTurn

Probes built after 2003 are equipped with the conventional flow turn. Its 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.

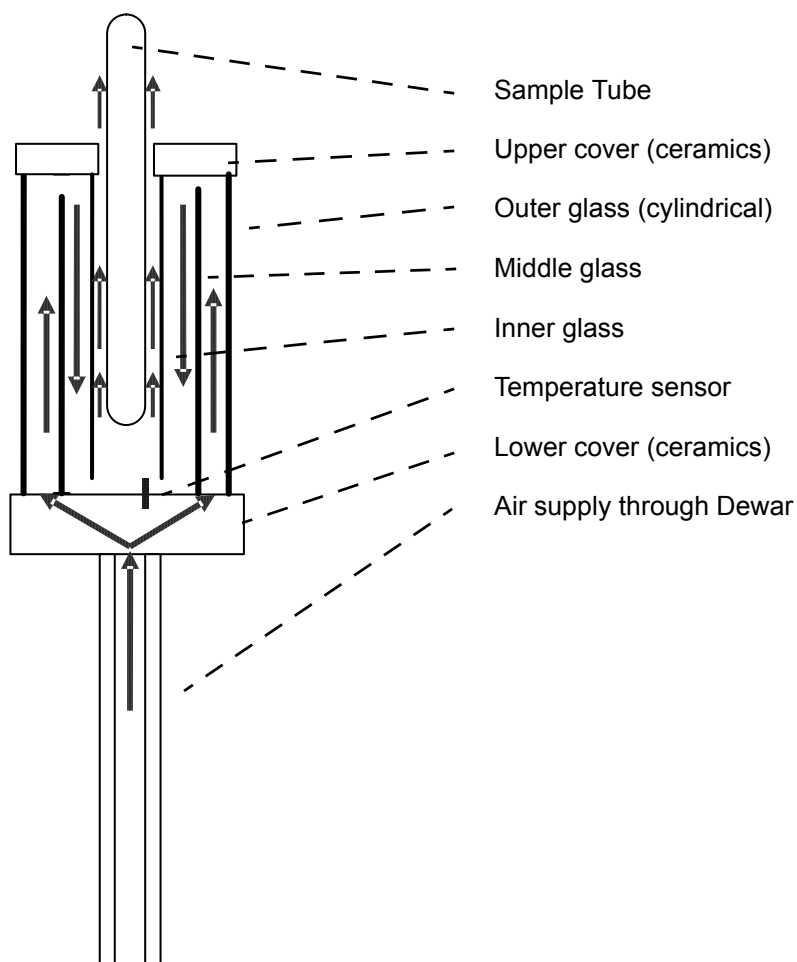


Figure 3.3 Schematic drawing of FlowTurn principle

Description

Except for the missing bypass air outlets, the function is the same as the Extended FlowTurn. However, because the inner glass is very close to the sample tube, only low flow rates are permitted. Consequently, the heating/cooling performance is reduced. These probes are not optimized for extreme low temperature experiments.

Dual Flow

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.

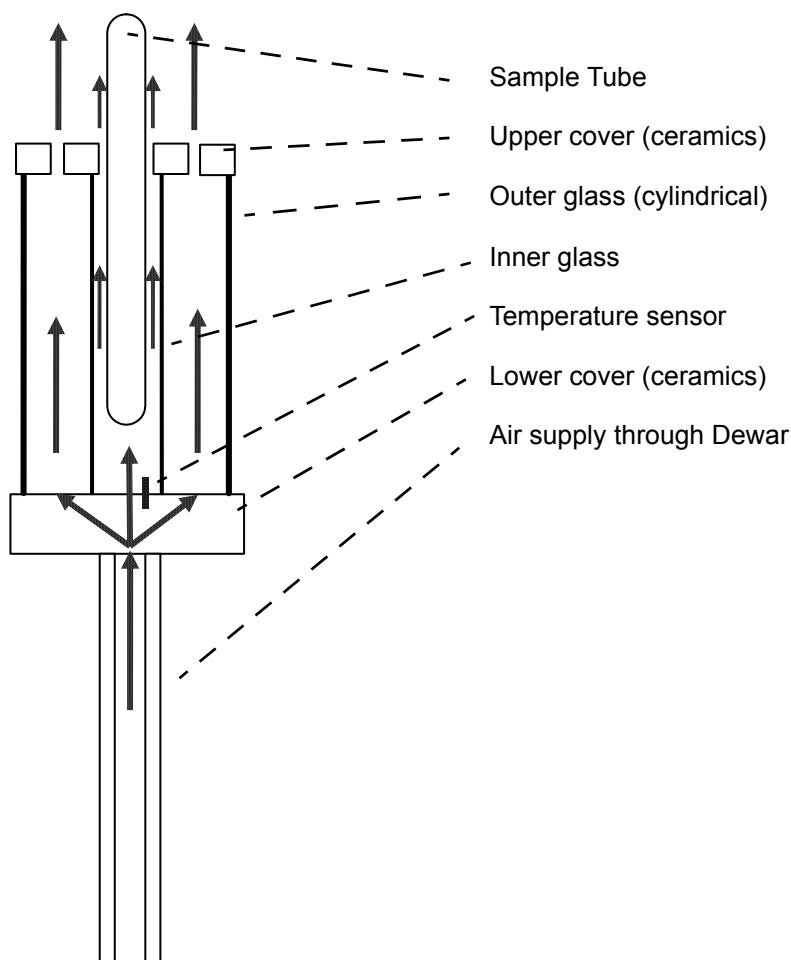


Figure 3.4 Schematic drawing of the Dual Flow principle

Description

The Dual Flow splits the dewared VT gas (through Dewar) into two parts. One part is passing between the inner glass and the sample tube and directly tempers the sample, the other part is passing as a coaxial flow between the outer and the inner glass and exits the probe. This outer flow mainly gives the heating/cooling power to the coil space, the inner gas flow performs the sample temperature control. This principle is not so good for controlling temperature gradients, but allows very high flow rates up to 2000 l/h and heats/cools efficiently. A weak point of this principle always was the balance between inner and outer flow against the not very satisfactory temperature gradients.

Different Probe Classes versus Temperature Ranges

Sample Diameter [mm]	Probe Type	Classification	Temperature Range
10 mm	BBO	Standard	-150..+150°C
	SEF, SEX	HT	-150..+600°C
	SEX	ET	-5..+200°C
5 mm	BB(F)O, DUL, BBI, TXI, QXI, TBI	Standard	-150..+150°C
	DUL, SEF, SEI, SEL, SEX	HT	-150..+600°C
	DUL, SEX	LTA	-180..+150°C
1.7 mm	TXI	Standard	-50..+80°C
1.0 mm	TXI	Standard	-50..+80°C
Note	Probes with XYZ Grad.		≤ +80°C

Table 3.1 Different probe classes

3.2.1 Auxiliary Functions and Recommended Operating Conditions

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 or adjusted settings made 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.

From this reason, 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. See recommended operating conditions at the end of this chapter

Sample T [°C]	-150 ... -80	-80 ... 0	0 ... 80	80 ... 120	120 ... 150	150 ... 180
Flush gas[l/min]	5...10	5	0	5	5	5

Table 3.2 Flush gas, quick guide

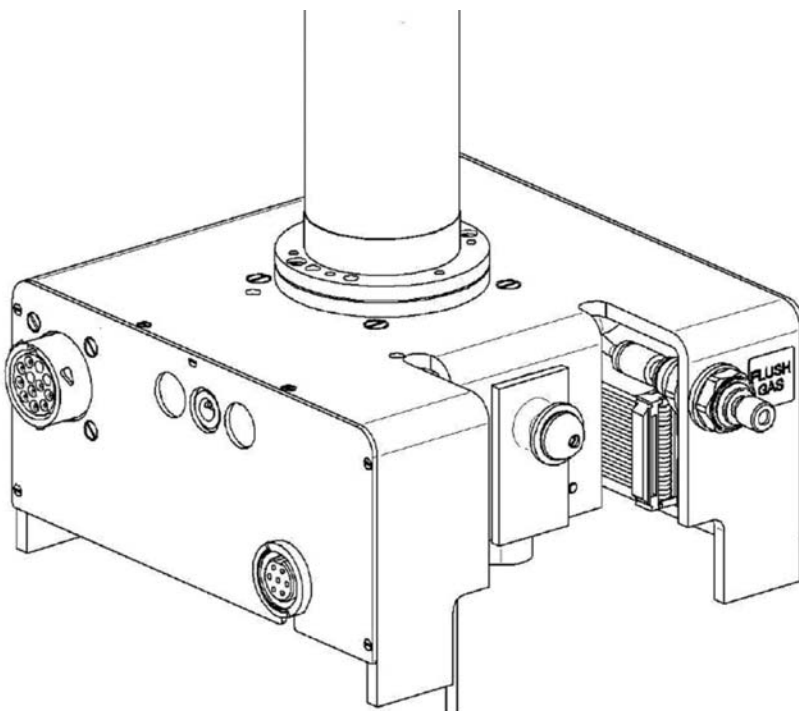


Figure 3.5 Probe socket with former ball seal VT connector and flush gas

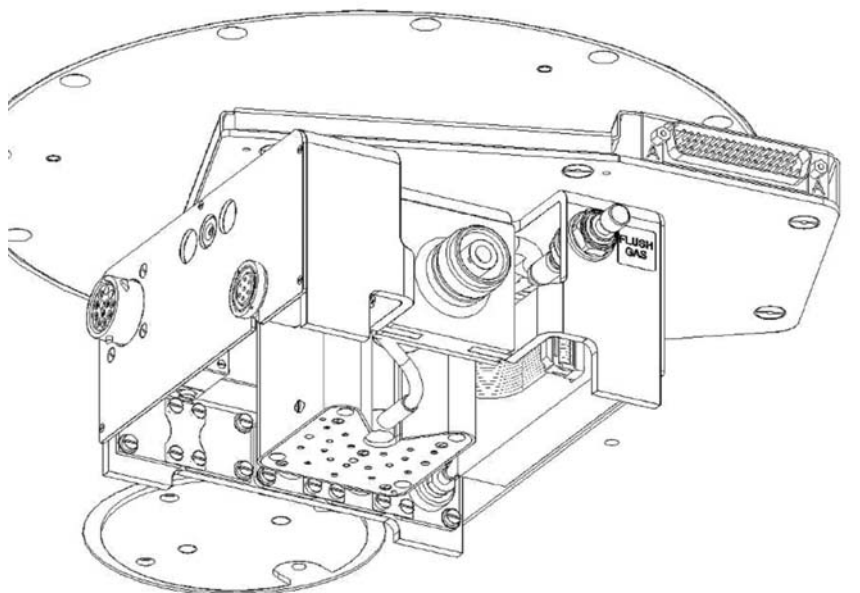


Figure 3.6 Probe socket with new Johnston VT connector and flush gas (below magnet)

Shim and Magnet Protection

Shim protection is essential for experiments at low and high temperatures. There are two main issues to be aware of:

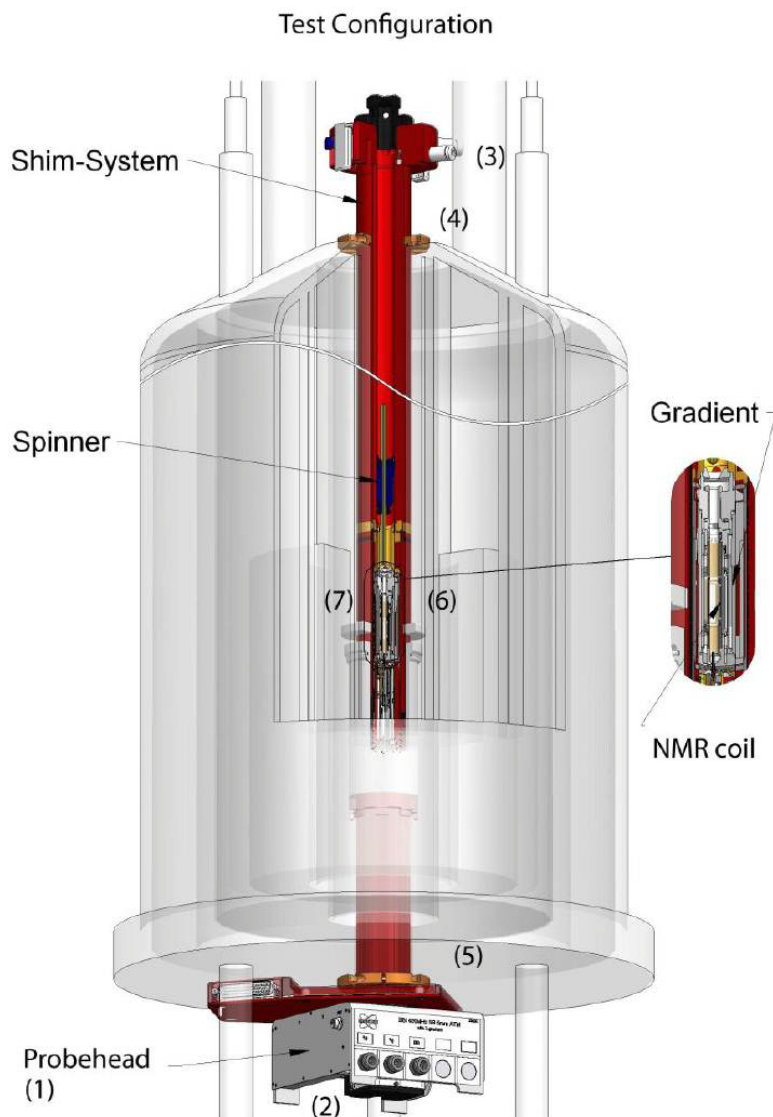
- Maximum shim coil temperature
- Minimum temperatures allowed at the upper and lower magnet flanges

Shim Coil Temperature

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 up to higher than +80°C. This is mentioned on every RT probe customer specification sheet. For this reason, shim cooling (shim 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. This is not a problem at all, since no damage occurs to the coil layers. TopSpin warns and remembers to activate shim gas when the coil temperature falls below +5°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 herewith no risk of magnet quench, occurring as a result of the dewar O-rings freezing. Please see recommended flow rates below or in the recommended operating conditions at the end of this chapter (["Recommended operating conditions for probe temperature control" on page 27](#)).

Typical temperature profile of the magnet flanges and shim coil during a long term experiment with a RT probe at -150°C during 3 days is shown next.



- 1 Probe
- 2 Probe socket and flush gas outlet
- 3 BST shim gas connector
- 4 Upper magnet flange
- 5 Lower magnet flange
- 6 Magnetic center
- 7 Location of shim coil

Figure 3.7 Overview shim system, probe and magnet

Low Temperature Experiment

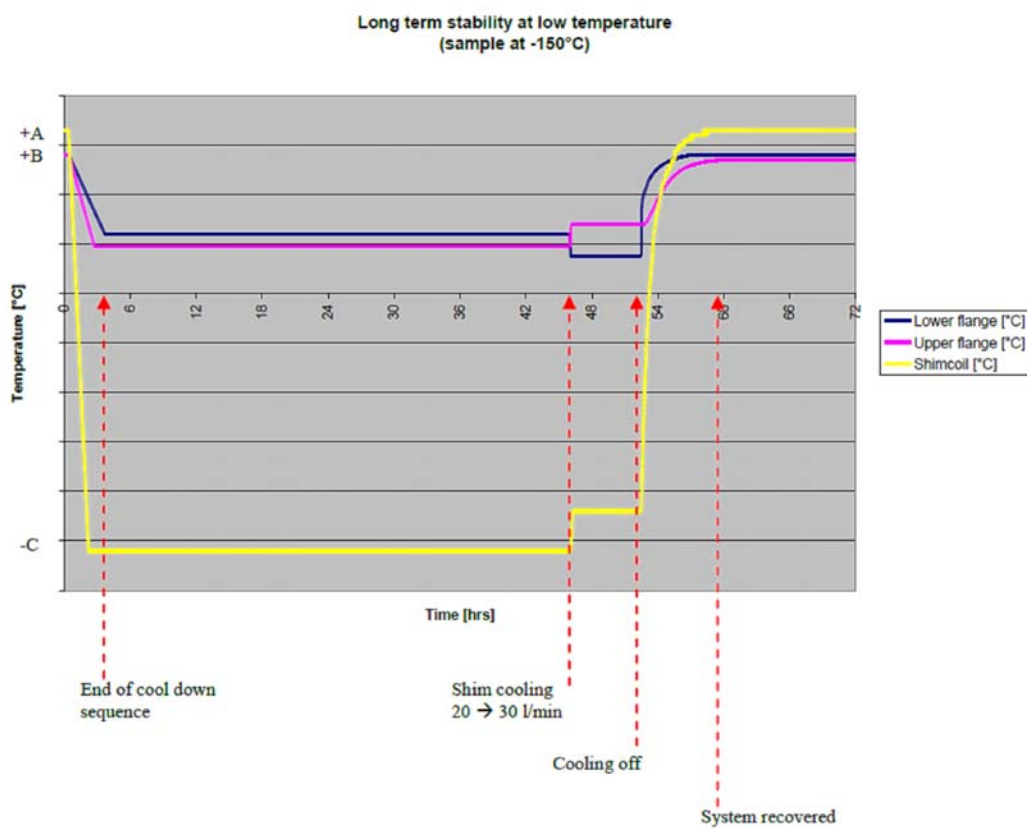


Figure 3.8 Example of magnets (critical) flange temperatures

The above temperature log shows the observation of the magnets (critical) flange temperatures as well as the temperature of the shim coil during a 72 hrs experiment.

Temperature scale 10°C per division, zero degrees at middle axis, shim coil temperature at -52°C.

This temperature log shows that the shimcoil is allowed to go to quite low temperatures without damaging and the flange temperatures stay around 8..12°C depending on shim gas flow. This diagram should help to understand and answer many questions often asked from practical applications during low temperature experiments. Note: shim gas flow of 20 l/min is mandatory (!)

High Temperature Experiment

Just check that shim coil does not exceed +80°C and activate shim gas if TopSpin requests.

Sample T [°C]	-150 ... -80	-80 ... 0	0 ... 80	80 ... 120	120 ... 150	150 ... 180
Shim gas [l/min]	20	20	0	0 ... 20	20 ... 30	40 ... 60

Table 3.3 Shim gas, quick guide

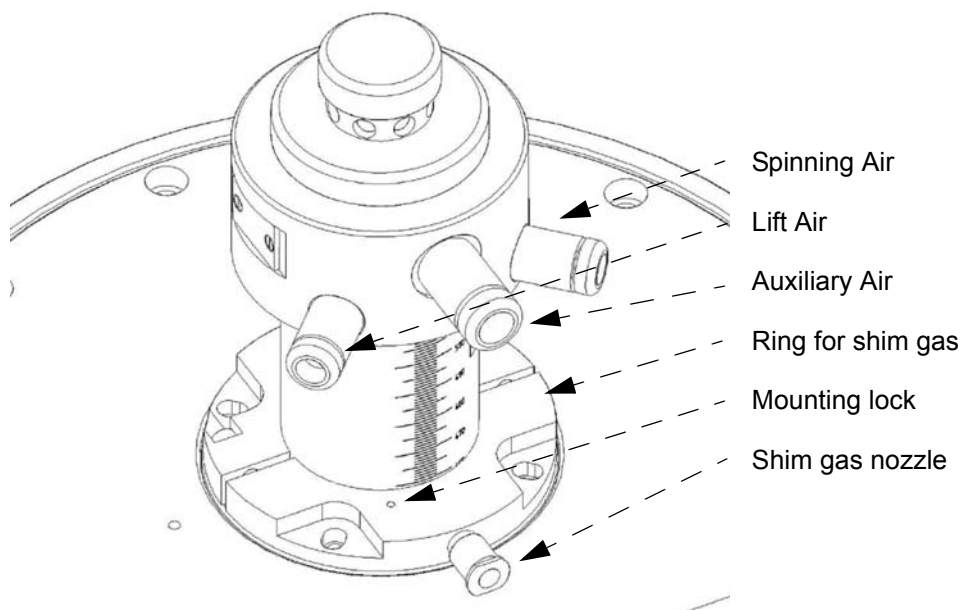


Figure 3.9 Shim gas connector at BST top end

Brief Description of the BST Upper Part

On the top end there are three nozzles, the right one as displayed above is for the spinning air, the left one to supply the lift and the middle one is an auxiliary gas supply (see BASH), in most cases not used.

On the lower end, just above the magnet flange, there is the mounting ring which enables the shim gas to enter the space between the magnet bore and the BST & Shim Coil. This mounting ring contains a radial O-ring seal touching the cylindrical surface of the BST and further has an axial sealing O-ring touching the magnet flange. Herewith, the shim gas can enter the nozzle, flows downwards and exits at the bottom end of the magnet.

Often problems occur when this ring is not properly mounted, so it's inner O-ring holder must properly fit with the mounting lock as displayed above.

All air and gas supplies are delivered by the BSVT, see corresponding technical manuals. For older systems, refer to the BVT manuals.

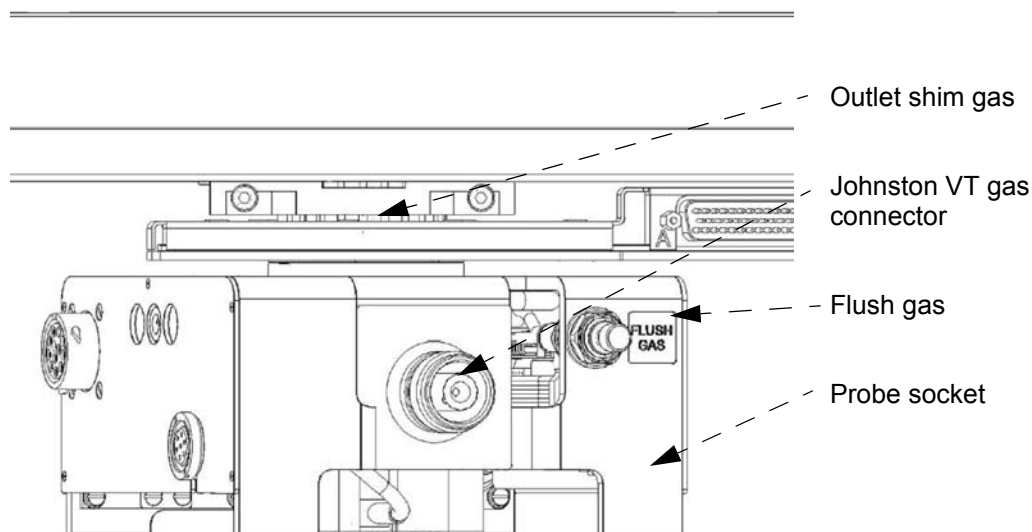


Figure 3.10 Shim gas outlet at magnet bottom

The outlet of all shim gas is peripheral – the gas exits along the circumference of the shim coil. It is important to have dry air, otherwise the inside of the magnet bore will form a lot of ice and all air flow will become restricted.

However, even with the recommended dry air specification (dew point better than -50°C) and flow rate (20 l/min), some icing at the shim gas exit will be observed and this depends strongly on the laboratory's humidity. High humidity leads to more icing. This effect is not a problem and this thin film of ice crystals cannot block the outlet, the dry gas is always keeping the exit slots open. It is also not a risk for the magnet flange, there is no strong thermal capacity within this coating. As shown in the temperature Log above, the temperature of the magnet flanges do not go lower than $+8^{\circ}\text{C}$ under the most extreme conditions (RT probe at -150°C for long time).

Recommend Standard Bore Spinners



Figure 3.11 Z42516 Standard POM Spinner, 0°C to +80°C

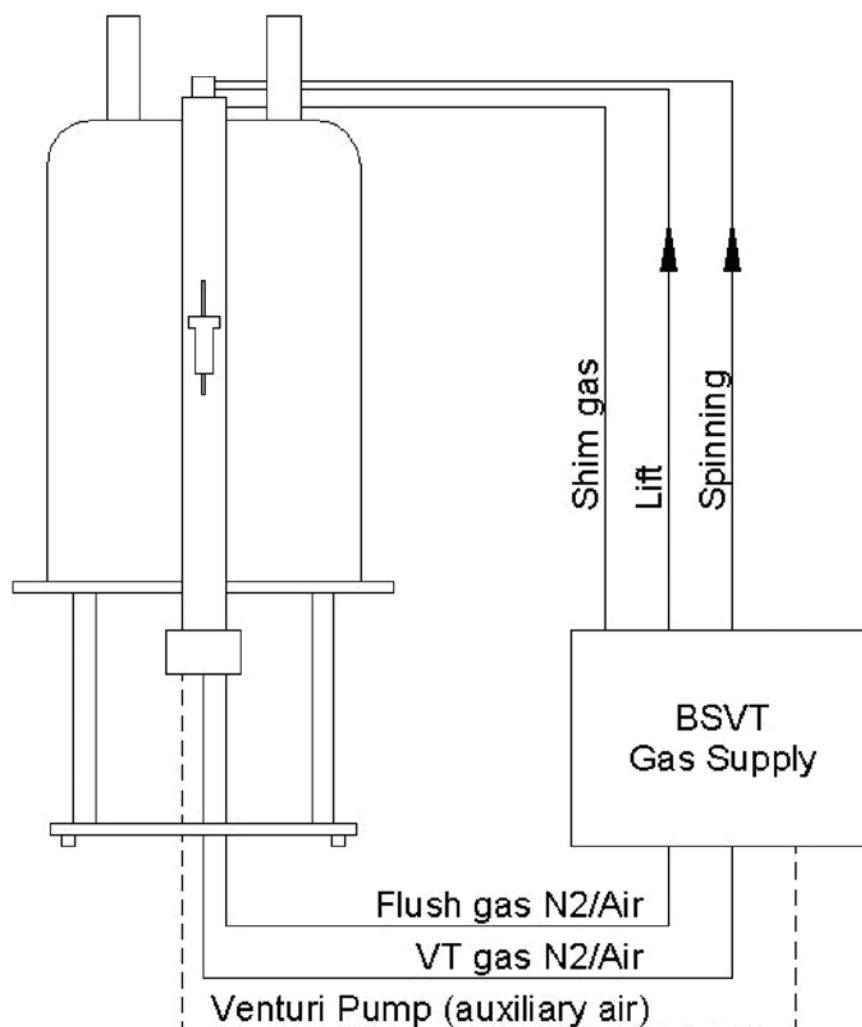


Figure 3.12 H00177 Kel-F Spinner for elevated Temperatures, +80°C to +120°C



Figure 3.13 H00804 Ceramics Spinner for high and low Temperatures, +120°C to +180°C and 0°C to -150°C

System Configuration for Various Temperature Ranges



- 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 ET 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

Figure 3.14 Standard RT & elevated temperatures

Principle of VT Control for NMR

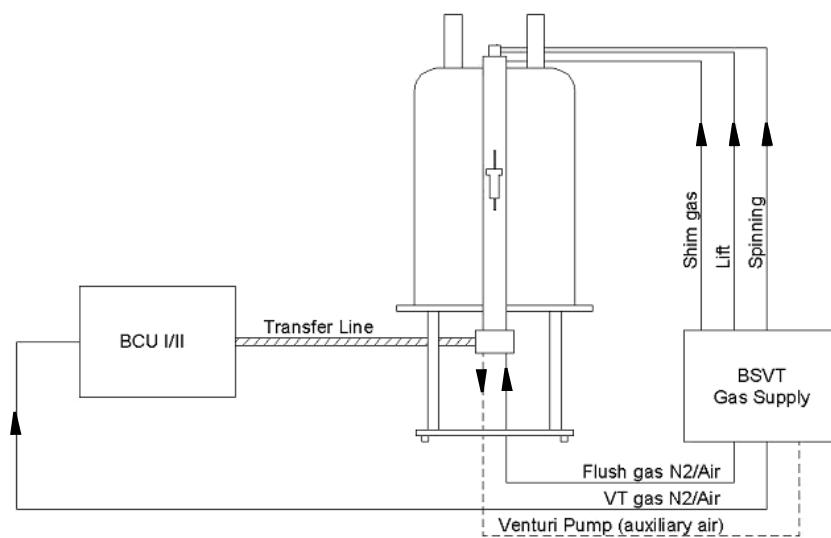


Figure 3.15 Configuration for RT applications down to 0°C and -40°C

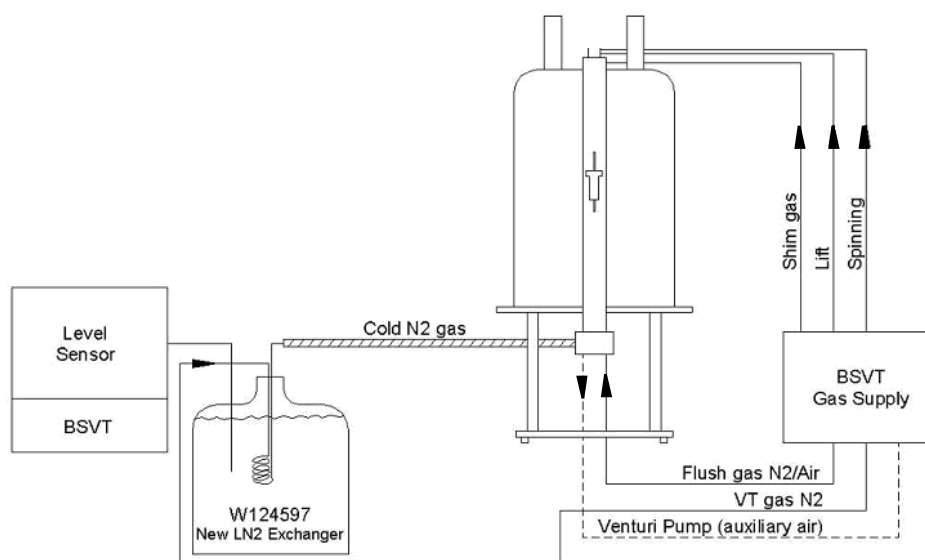


Figure 3.16 Configuration for low temperatures down to -150°C with LN2 Heat Exchanger

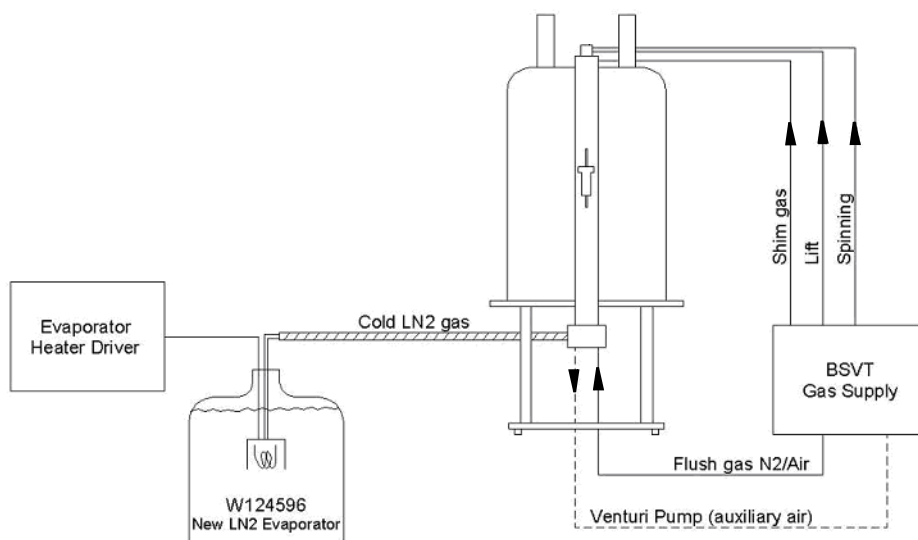


Figure 3.17 Configuration for low Temperatures down to -150°C with LN2 Evaporator

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 exchanger or LN2 evaporator	BCU II (-40°C) or LN2 heat exchanger/ LN2 evaporator for lower temp	BCU I for $T < T_{\text{RT}}$			

Table 3.4 Recommended operating conditions for probe temperature control

3.3 VT Control for Cryo Probes (CRP + CPP)

3.3.1 Sample Temperature Control

Direct Flow – Principle of Heat Exchange:

The flow principle realized in CryoProbes (He CryoProbes and Prodigies) is shown in the [Figure 3.18](#) below. The sample is located centred in the “warm” centre tube (black coloured) which is surrounded by the cold coil(s) (blue-coloured). Although the CryoProbe has an excellent thermal insulation between the sample space and the cold RF coils, a small thermal radiation is constantly cooling the sample. To compensate for radiation losses of the “warm” centre tube and sample due to “unshielded” cold coils, a sufficient VT gas flow is mandatory to keep the sample at a constant temperature and to prevent forming of an undesirable temperature gradient through the sample. Unlike conventional probes, the VT gas flow should be as high as possible for a CryoProbe. It is only restricted by the possibility that the VT gas may lift the sample.

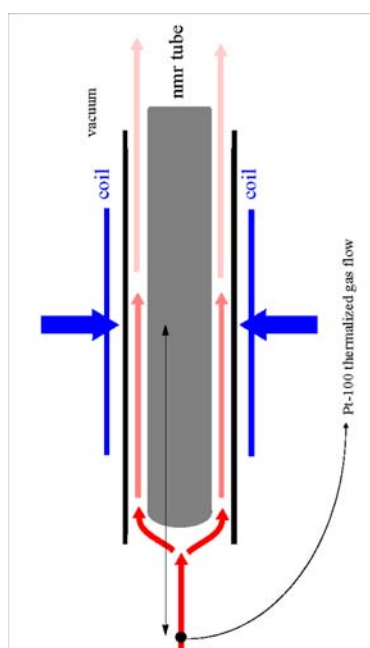


Figure 3.18 Principle of heat exchange for Cryo Probes

The temperature within the sample is controlled via a PT100 resistor which is located in the VT gas flow below the sample. The real sample temperature may differ slightly from that of the temperature sensor. The difference can be up to 10 K, depending on the set point temperature and VT flow rate. Topspin allows a linear correction of such differences. For temperature calibration please refer to corresponding section in this manual.

3.3.2 Cryo Probes for Different VT Ranges

For CryoProbes 3 VT ranges are available: Standard temperature range (ST), low temperature range (LT) and elevated temperature range (ET).

ST: 0.....+80°C

LT: -40....+80°C

ET: 0.....+135°C

Up to now all 5mm CryoProbes are available in all three VT ranges. 10mm CryoProbes are designed for ST and ET range. Micro CryoProbes (1.7mm) are available only for ST range. Sensitivity of a CryoProbe is not compromised by the different VT range options.

Besides the standard temperature range all other ranges are probe options that require additional equipment.

Temperatures around and below room temperature can only be achieved using a VT-gas cooling device like BCU 1 or BCU 2.

- Shim Protection
- Spinners
- Effects on system at the temperature extremes (T&M, shims, shim coil temperature, magnet flange temperatures)
- Recommended equipment such as chillers and other devices
- Sample temperature vs environment temperature stability

3.3.3 Temperature Dynamics

Bruker recommends the following heating rates for CryoProbes:

Probe Type	Target Temperature	Heating Rate
standard probes (ST)	0 °C...+80 °C	5 K per minute, 10 min settling time at final temperature
low and elevated temperature versions (LT/ET)	-40 °C...+135 °C	

Table 3.5 Recommended flow rates Cryo Probes

3.3.4 Recommended Flow Rates

The recommended gas flow rates for proper operation are shown below. To achieve sample temperatures close to the specified extremes or at the extremes, high flow rates may be needed.

Probe Type	Target Temperature	Requested Flow [l/h] ^a
10mm	0°.....+135°C	670-800
5mm	-40°/0°.....+80°/135°C	670-800
1.7mm	0°.....+80°C	400

Table 3.6 Recommended flow rates Cryo Probes

a. litres @ 1 bar 0°C

3.3.5 Auxiliary Functions and Recommended Operating Conditions

Sample Protection

As already pointed out in the last chapter a VT gas flow is mandatory to keep the sample at constant temperature. In the case VT gas fails due to an electric power failure or a breakdown of the compressed air, the sample still inserted in the cold probe will be cooled down rapidly. In most of the cases the sample will freeze and will be destroyed as a result. In cases where the sample protection option is installed, the VT gas flow is maintained with gas at ambient temperature from a backup gas reservoir. How long the sample temperature can be sustained depends on the size and pressure of the backup cylinder. Sample Protection is an option of the CryoPlatform and will provide continuous VT gas operation in the event of a VT gas interruption and, if necessary, will lift the sample out of the cold environment of the CryoProbe to prevent freezing of the sample (for further instructions see CryoProbe Manual).

4 Equipment for VT Control

4.1 Chillers

Overview

For carrying out NMR experiments with sample temperatures T_S below room temperatures T_{RT} , a VT gas chiller is required. BRUKER offers four different chillers:

- SmartCooler™ BCU I -40/50
- SmartCooler™ BCU II -80/60
- LN2 heat exchanger (W124597)
- LN2 evaporator (W124596)

The SmartCoolers™ BCU I and BCU II produce the cooling energy with electrical power via compressor refrigerators. The BCU I cools with one compressor refrigerator and the BCU II with two cascaded compressor refrigerators.

The LN2 heat exchanger and the LN2 evaporator chillers take their cooling energy from LN2.

Here is an overview of the recommended chillers required on the temperature ranges of the sample temperature T_S and the probe:

Sample temp. T_S [°C]	-150 ... -80	-80 ... -40	-40 ... 0	0 ... T_{RT}
Chiller	LN2 heat exchanger / evaporator		BCU II	BCU I
Cooling energy	LN2		Compressor refrigerator	

Table 4.1 Recommended chillers for 5mm RT probes (extended flow turn)

Sample temp. T_S [°C]	-40 ... 0 (CRP LT only!)	0 ... T_{RT}
Sample temp. T_S [°C]	-40 ... 0 (CRP LT only!)	0 ... T_{RT}
Chiller	BCU II	BCU I
Cooling energy	Compressor refrigerator	

Table 4.2 Recommended chillers for 5mm CryoProbes™

A new VT gas coupler system for chillers and RT probes was introduced. The former VT gas coupler system (ball joint type) of the RT probes and the corresponding chillers (e.g. BCU05) had problems with leakage and icing.



Figure 4.1 Former VT gas coupler of RT probes (ball seal, male)



Figure 4.2 Former VT gas coupler of coolers (ball seal, female)

With the new VT gas coupler system (Johnston type), these problems were eliminated. All four chillers (SmartCooler™ BCU I and II, W124597 and W124596) are equipped with the new VT gas coupler.

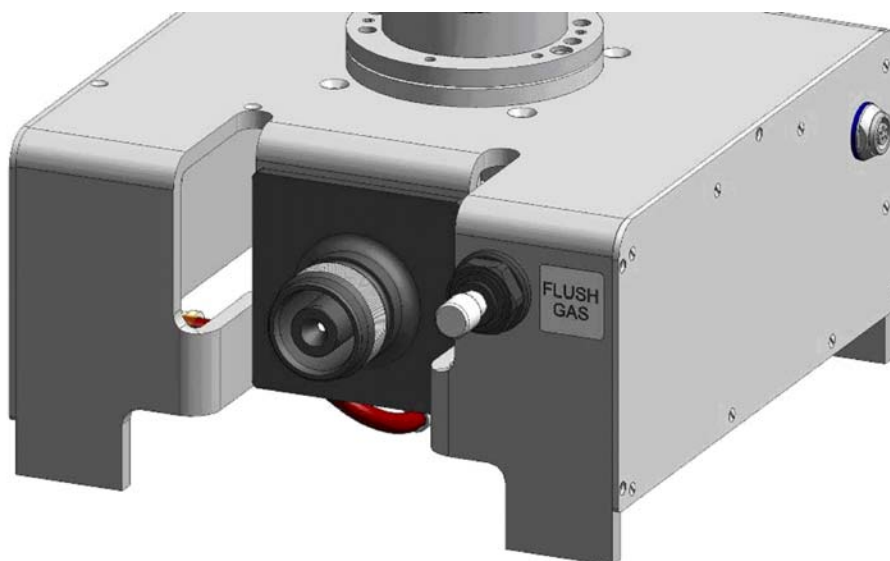


Figure 4.3 New Johnston VT gas coupler for RT probes, female



Figure 4.4 New Johnston VT gas coupler (SmartCooler™, W124597 and W124596) - male

To combine these two different VT gas coupler systems (e.g. Johnston type, chiller) with ball joint type (RT probe) adapters are available. For RT probes with a former VT gas coupler use the adapter Z121532. For a CryoProbe use the adapter BSCU-ADAPTER CRYOPROBE KPL. Z119688.



Figure 4.5 Former VT gas coupler (RT probe)



Figure 4.6 Adapter Z121532 (BCU to former ball joint type VT gas coupler)



Figure 4.7 Adapter Z119688 (BCU to CryoProbe)

4.1.1 BCU I

The SmartCooler™ BCU I -40/50 (see [Figure 4.8](#)) is the successor model of the BCU05 (see also [Table 4.3](#)). The lowest temperature of the cooled VT gas is with a BCU I is below -40°C up to 50 nl/min. The SmartCooler™ BCU I and also the BCU05 produce the cooling energy via one compressor refrigerator with electrical power. This SmartCooler™ is recommended for NMR experiments with a sample temperature T_S between 0°C and T_{RT} .



Figure 4.8 SmartCooler™ BCU I – successor model of the BCU 05

The BCU I is available with a transfer line length of 2m, 3m, 4m, 5m or 8m. The magnet type defines which BCU I has to be ordered. Below is a list of the relative BCU I transfer line lengths associated with the most common magnets.

- **2m:** 400US PLUS/R SB (Nitrogen free Z107439); all Ascend™ (all frequencies!); all USplus SB LH; all USplus SB ULH; all US SB ULH; 300 – 500 US SB LH; 300 & 400 US WB LH; USplus WB LH; 92137 (500/54/AS Magnex); 91843 (500/52 Magnex); 40797 (500/51 AS Oxford); 47410 (AS500/54)
- **3m:** 600 & 700 US SB LH; 500 US WB LH, 800 & 850 US2 Plus SB (Compact)
- **4m:** 600 US WB LH; 800 US SB; 800 – 950 US2 SB; Z29715 (500/52LL); M10910 (900 MHz/54 US2); 29563 (750/52 Magnex); O019 (500/52LL), O048 (400/54 Oxford)
- **5m:** 900 US SB; Z29704 (600/52 LL)
- **8m:** 1000 US SB

For BRUKER magnets the following rules are valid to determine the minimal transfer line length of a BCU I:

- *Ascend™ (all frequencies) and all other magnets $\leq 700\text{MHz}$:* Place the BCU I outside the 5 Gauss line and not less than 1m from the axis of the magnet.
- *Magnets $\geq 750\text{MHz}$:* Place the BCU I outside the 50 Gauss line and not less than 2.7m from the axis of the magnet.



	Smart Cooler™ BCU I	BCU 05
Sample temp. T_S [°C] – PH depend.	0 ... T_{RT}	5 ... T_{RT}
Min. VT gas outlet temperature [°C]	-40	-25...-30
Max. VT gas flow [nl/min], [nl/h]	50, 3000	33, 2000
Dew point temp. T_{DP} VT gas [°C]	< -50	< -50
VT gas coupler type	Johnston	ball joint
Cool down time [min]	~ 15	~ 30
Transfer line length [m]	2, 3, 4, 5, 8	3, 4
Dimensions [mm]	480 x 351 x 425	550 x 500 x 490
Weight [kg]	30	35
Power supply	220V - 240V ~ 50Hz or 100V ~ 50Hz / 100V - 115V ~ 60Hz	230V ~ 50/60Hz 450VA
Steady state / starting current	2.1A / -- @220V - 240V ~ 50Hz or 4.7A / -- @100V ~ 50Hz / 100 - 115V ~ 60Hz	-
Remote control	ON/OFF with BS(V)T and 3 operational modes ("Flush/0"; "1", "2") with BSVT	ON/OFF control from B(S)VT
Monitoring	YES (with BSVT only!)	NO

Table 4.3 Comparison of BCU I and BCU05

⚠ WARNING

The BCU I is magnetic and presents a potential hazard in the vicinity of a magnet (e.g. magnetic stray field of the NMR magnet):

- ▶ **Ascend™ (all frequencies) and all other magnets ≤ 700MHz:** Place the BCU I **outside** the **5 Gauss** line and **not less** than 1m from the axis of the magnet.
- ▶ **Magnets ≥ 750MHz:** Place the BCU I **outside** the **50 Gauss** line and **not less** than 2.7m from the axis of the magnet.

Beside the magnet type also the country or rather the voltage of the power socket is important to select the correct BCU I. The BCU I is available for the following electrical power:

USA / JP / CA: 100V ~ 50Hz / 100V - 115V ~ 60Hz 1~

Other: 220V - 240V ~ 50Hz 1~



CAUTION

Wrong power supply voltage will damage the BCU I:

- ▶ Be sure that the voltage indicated on the label of the BCU I corresponds to the power supply voltage of the power socket

The BCU I VT inlet gas (max. 6bar) has to be dry and should have a dew point temperature T_{DP} below -50°C . This required specification prevents freezing and clogging of the BCU I. In case the VT gas source on site does not fulfill this specification a Bruker membrane dryer (1808577) is available. In addition be aware that all tubing ($\varnothing 8\text{mm}$) consists of TPS. This material has a very low water permeability.



CAUTION

VT gas overpressure will damage the BCU I:

- ▶ Use VT gas with a pressure of max. 6bar

NOTICE

Freezing and clogging of the BCU I with VT gas:

- ▶ To prevent freezing and clogging of the BCU I use dry clean air or nitrogen VT gas at room temperature with a dew point below -50°C
- ▶ For the BCU I & BCU II Bruker recommends the dryer 1808577
- ▶ For all tubing (particularly after the gas dryer!) use only hoses from TPS (low water permeability)
- ▶ Install the gas dryer before the B(S)VT!

The BCU I is equipped with a rotary switch (see [Figure 4.9](#)). With this rotary switch four operation modes are adjustable:

- **"Remote"**: The BCU I is controlled by the BVT or BSVT. The cable from the D-Sub 15 socket of the BCU I to the BVT or BSVT must be plugged in!
- **"Flush / 0"**: This switch setting is used when VT gas at room temperature has to be fed to the NMR probe or the VT gas tubing of the BCU I has to be dried and purged (**Note**: Purge with dry clean air or nitrogen gas at room temperature with a dew point below -50°C). In this setting the compressor does not run! The BCU I is in a manual mode* and cannot be controlled with the BVT or BSVT.
- **"1"**: In this switch setting, the VT gas is only lightly cooled with reference to room temperature T_{RT} ($T = T_{RT} - 10 \dots 20^{\circ}\text{C}$). The BCU I is in a manual mode* and cannot be controlled with the BVT or BSVT.

- **"2"**: In this switch setting, the VT gas is cooled to the lowest specified temperature. The BCU I is in a manual mode* and cannot be controlled with the BVT or BSVT.

*) **Note:** In manual mode the BCU I can be operated stand alone without a communication cable to the B(S)VT.

The BCU I is controlled by the BSVT (not BVT!) and with the rotary switch set to position "Remote", the three operational modes ("Flush / 0", "1" and "2") can be selected via the TopSpin™:

- **"Flush / 0"**: This remote mode is used when VT gas at room temperature has to be fed to the NMR probe or the VT gas tubing of the BCU I has to be dried and purged (**Note:** Purge with dry clean air or nitrogen gas at room temperature with a dew point below -50°C). In this setting the compressor does not run!
- **"1"**: In this remote mode, the VT gas is only lightly cooled with reference to room temperature T_{RT} ($T = T_{RT} - 10...20^{\circ}C$).
- **"2"**: In this remote mode, the VT gas is cooled to the lowest specified temperature.



Figure 4.9 Rotary switch and the two LEDs of the SmartCooler™ BCU I

The BCU I has two LEDs: A "Status" and a "Remote" LED. The "Status" and the "Remote" LEDs indicate the states of the BCU I (see also [Table 4.4](#)).

Status		Remote	
Green flashing:	Cooling down	Green flashing:	Connecting (B(S)VT
Green on:	Ready, stable temperature	Green on:	Connected to BVT or BSVT
Red flashing:	WARNING, overheating ^a	Red flashing:	Connection to BVT or BSVT failed
Red on:	ERROR ^b	Off ^c	No communication with B(S)VT

Table 4.4 LED code chart of the SmartCooler™ BCU I

a. e.g. blocked dust filter

b. Clean dust filter or call service

c. The communication cable between BCU I an B(S)VT is not connected

Equipment for VT Control

For the SmartCooler™ (BCU I / BCU II) the following accessories are available. Some of them are always included in the BCU I and BCU II package.

	Name	P/N	Remark
	Support Clamp For Cooling Unit	Z53660	Always included
	BSCU support	Z119100	Included with SmartCooler™ (transfer line > 2m)
	Probe adapter for probes equipped with ball joint	Z121532	Always included
	BSCU adapter for CryoProbe	Z119688	Required for Cryo- Probes
	MAS Box	Z120576	Required with BVT3x00 and MAS
	Membrane dryer	1808577	Required in case the VT gas does not meet the dew point temperature specs

Table 4.5 Accessories for the SmartCooler™ (BCU I and BCU II)

4.1.2 BCU II

The SmartCooler™ BCU II -80/60 (see [Figure 4.10](#)) is the successor model of the BCU-Xtreme™ (see also [Table 4.6](#)). The lowest temperature of the cooled VT gas is with a BCU II < -80°C up to 60 nl/min. The SmartCooler™ BCU II and also the BCU-Xtreme™ BCU II produce the cooling energy via two cascaded compressor refrigerators with electrical power. This SmartCooler™ is recommended for NMR experiments with a sample temperature T_S between -40°C and T_{RT} .



Figure 4.10 SmartCooler™ BCU II – successor model of the BCU-Xtreme™

The BCU II is available with a transfer line length of 3m, 5m or 8m. The magnet type defines which BCU II has to be ordered. Below is a list of the relative BCU II transfer line lengths associated with the most common magnets.

- **3m:** 400US PLUS/R SB (Nitrogen free Z107439); all Ascend™ (all frequencies!); all USplus SB LH; all USplus SB ULH; all US SB ULH; 300 – 500 US SB LH; 300 & 400 US WB LH; USplus WB LH; 92137 (500/54/AS Magnex); 91843 (500/52 Magnex); 40797 (500/51 AS Oxford); 47410 (AS500/54); 600 & 700 US SB LH; 500 US WB LH, 800 & 850 US2 Plus SB (Compact)
- **5m:** 600 US WB LH; 800 US SB; 800 – 950 US2 SB; Z29715 (500/52LL); M10910 (900 MHz/54 US2); 29563 (750/52 Magnex); O019 (500/52LL), O048 (400/54 Oxford); 900 US SB; Z29704 (600/52 LL)
- **8m:** 1000 US SB

For BRUKER magnets the following rules are valid to determine the minimal transfer line length of a BCU II:

- *Ascend™ (all frequencies) and all other magnets ≤ 700MHz:* Place the BCU I outside the 5 Gauss line and not less than 1m from the axis of the magnet.
- *Magnets ≥ 750MHz:* Place the BCU I outside the 50 Gauss line and not less than 2.7m from the axis of the magnet.

WARNING

The BCU II is magnetic and presents a potential hazard in the vicinity of a magnet (e.g. magnetic stray field of the NMR magnet):



- ▶ **Ascend™ (all frequencies) and all other magnets ≤ 700MHz:** Place the BCU II **outside** the **5 Gauss** line and **not less** than 1m from the axis of the magnet.
- ▶ **Magnets ≥ 750MHz:** Place the BCU II **outside** the **50 Gauss** line and **not less** than 2.7m from the axis of the magnet.



Beside the magnet type also the country or rather the voltage of the power socket is important to select the correct BCU II. The BCU II is available for the following electrical power:

USA / JP / CA: 100V ~ 50Hz / 100V - 115V ~ 60Hz 1~

Other: 220V - 240V ~ 50Hz 1~

CAUTION



Wrong power supply voltage will damage the BCU II:

- ▶ Be sure that the voltage indicated on the label of the BCU I corresponds to the power supply voltage of the power socket

The BCU II VT inlet gas (max. 6bar) has to be dry and should have a dew point temperature TDP below -80°C. This required specification prevents freezing and clogging of the BCU II. Where the VT gas source on site does not fulfill this specification a Bruker membrane dryer (1808577) is available. In addition be aware that all tubing (Ø8mm) consists of TPS. This material has very low water permeability.

CAUTION



VT gas overpressure will damage the BCU II:

- ▶ Use VT gas with a pressure of max. 6bar

NOTICE

Freezing and clogging of the BCU II with VT gas:

- ▶ To prevent freezing and clogging of the BCU II use dry clean air or nitrogen VT gas at room temperature with a dew point below -50°C
- ▶ For the BCU I & BCU II Bruker recommends the dryer 1808577
- ▶ For all tubing (particularly after the gas dryer!) use only hoses from TPS (low water permeability)
- ▶ Install the gas dryer before the B(S)VT!

	Smart Cooler™ BCU II	BCU-Xtreme™
Sample temp. T_S [°C] – PH depend.	-40 ... T_{RT}	-40 ... T_{RT}
Min. VT gas outlet temperature [°C]	-80	-80
Max. VT gas flow [nl/min], [nl/h]	60, 3600	50, 3000
Dew point temp. T_{DP} VT gas [°C]	< -80	< -80
VT gas coupler type	Johnston	ball joint
Cool down time [min]	~20	~30
Transfer line length [m]	3, 5, 8	4
Dimensions [mm]	580 x 420 x 570	740 x 586 x 547
Weight [kg]	70	74
Power supply	220V - 240V ~ 50Hz or 100V ~ 50Hz / 100V - 115V ~ 60Hz	230V ~ 50/60Hz
Steady state / starting current	8A / ???@220 - 240V ~ 50Hz or 13A / ???@100V~ 50Hz/100 – 115V~ 60Hz	10A / 20A
Remote control	ON/OFF with BS(V)T and 4 operational modes ("Flush/0"; "1", "2", "3") with BSVT	ON/OFF with B(S)VT
Monitoring	YES (with BSVT only!)	NO

Table 4.6 Comparison of BCU II and BCU-Xtreme™

The BCU II is equipped with a rotary switch (see [Figure 4.11](#)). With this rotary switch four operation modes are possible:

- **"Remote"**: The BCU II is controlled by the BVT or BSVT. The cable from the D-Sub 15 socket of the BCU II to the BVT or BSVT must be plugged in!
- **"Flush / 0"**: This switch setting is used when VT gas at room temperature has to be fed to the NMR probe or the VT gas tubing of the BCU II has to be dried and purged (**Note**: Purge with dry clean air or nitrogen gas at room temperature with a dew

point below -80°C). In this setting the compressor does not run! The BCU II is in a manual mode* and cannot be controlled with the BVT or BSVT.

- **"1"**: In this switch setting, the VT gas is cooled only slightly with reference to room temperature T_{RT} ($T = T_{\text{RT}} - 10 \dots 20^{\circ}\text{C}$). The BCU II is in a manual mode* and cannot be controlled with the BVT or BSVT.
- **"2"**: In this switch setting, the VT gas is cooled to about -40°C *. In this mode the BCU II simulates a BCU I (Min. VT gas outlet temperature of a BCU I: $< -40^{\circ}\text{C}$).
- **"3"**: In this switch setting, the VT gas is cooled to the lowest specified temperature. The BCU II is in a manual mode* and cannot be controlled with the BVT or BSVT.

*) **Note**: In manual mode the BCU II can be operated stand alone without a communication cable to the B(S)VT.

Is the BCU II controlled by the BSVT (not BVT!) and the switch setting of the rotary switch on position "Remote" the four operational modes ("Flush / 0", "1", "2" and "3") can be selected via the TopSpin™.

- **"Flush / 0"**: This remote mode is used when VT gas at room temperature has to be fed to the NMR probe or the VT gas tubing of the BCU II has to be dried and purged (**Note**: Purge with dry clean air or nitrogen gas at room temperature with a dew point below -80°C). In this setting the compressor does not run!
- **"1"**: In this remote mode, the VT gas is cooled only slightly with reference to room temperature T_{RT} ($T = T_{\text{RT}} - 10 \dots 20^{\circ}\text{C}$).
- **"2"**: In this remote mode, the VT gas is cooled to about -40°C . In this mode the BCU II simulates a BCU I (Min. VT gas outlet temperature of a BCU I: $< -40^{\circ}\text{C}$).
- **"3"**: In this remote mode, the VT gas is cooled to the lowest specified temperature.

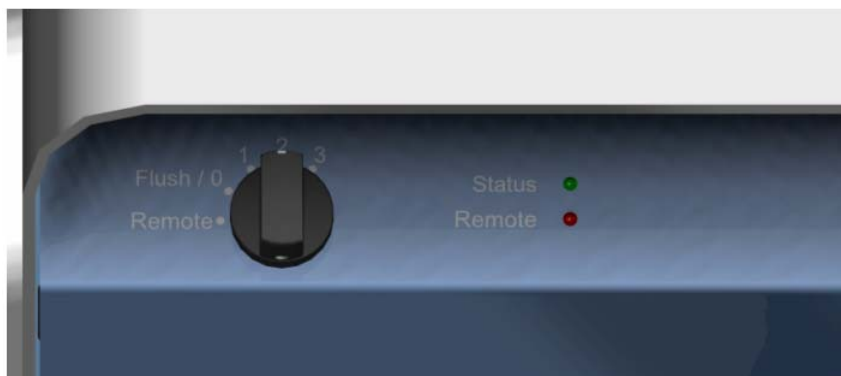


Figure 4.11 Rotary switch and the two LEDs of the SmartCooler™ BCU II

The BCU II has two LEDs (see [Figure 4.11](#)): A "Status" and a "Remote" LED. The "Status" and the "Remote" LEDs indicate the states of the BCU II (see also [Table 4.7](#)).

Status		Remote	
Green flashing:	Cooling down	Green flashing:	Connecting (B(S)VT
Green on:	Ready, stable temperature	Green on:	Connected to BVT or BSVT
Red flashing:	WARNING, overheating ^a	Red flashing:	Connection to BVT or BSVT failed
Red on:	ERROR ^b	Off ^c	No communication with B(S)VT

Table 4.7 LED code chart of the SmartCooler™ BCU II

a. e.g. blocked dust filter

b. Clean dust filter or call service

c. The communication cable between BCU II and B(S)VT is not connected

For the SmartCooler™ accessories are available (see table "[Accessories for the SmartCooler™ \(BCU I and BCU II\)](#)" on page 38). Some of them are always included in the BCU II package.

4.1.3 LN2 Heat 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) close to the temperature of the LN2 (77K). A heat exchanger has to be used for low temperature measurements with sample temperatures T_S between -40°C and -150°C (see also [Table 4.1](#)).

A new multi coil heat exchanger kit W124597 ([Figure 4.12](#)) with Johnston coupler and SS transfer line (insulated with a permanent vacuum) had been introduced. It replaces the heat exchanger kit PU W1101326, kit SS W1101328 and the kit multicoil SS W1101454. Several mechanical parts of the exchange have been changed. The inlet SS tubing going to the heat exchanger coils is now slightly thermally insulated to produce a more constant outlet gas temperature less depending on the LN2 level of the dewar vessel. The three different exchanger coils delivered with the heat exchanger kit have a different numbers of turns: 1.5 turns (coil 1), 4 turns (coil 2) and 6 turns (coil 3).

To get best low temperature performance with the new sample temperature control of all 5 mm RT probes using a N_2 heat exchanger, the new type of N_2 heat exchanger W124597 has to be used! This new type of N_2 heat exchanger has a more stable flow of cold gas and therefore improves the sample temperature control. Use coil 2 (4 turns) or coil 1 (1.5 turns)!

The exchanger has to be used only with dry nitrogen gas to prevent internal icing and blockage of the exchanger.



Figure 4.12 Heat exchanger kit with Johnston coupling and different inserts

4.1.4 Special Equipment

LN2 Evaporator



Figure 4.13 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), W124592 is not recommended, instead W124596 shall be used.

4.2 Console Electronics

Console electronics provide

- variable electric power for the probe heater and – where required – accessory devices (e. g. LN2 evaporator, transfer line heater)
- connection to the temperature sensors and measurement electronics
- adjustable and stabilized gas flow
- monitoring of the heater power, heater temperature and gas flow
- digitally controlled temperature regulation
- control of chillers (compressor based chillers like BCU I or BCU II, LN2 based chillers such as LN2 exchanger or LN2 evaporator) and other accessory devices for extended temperature applications

Operation of the **Bruker (Smart) Variable Temperature** equipment is fully integrated in TopSpin™ where the software ensures optimum interaction between other system parts (e.g. probe characteristics, sample transport, etc.).

Currently two generations of electronics are in use and both are fully compatible with all probe interfaces:

- **BSVT (Bruker Smart Variable Temperature System)** series of electronics is located within the BSMS/2 system and has been delivered with systems since mid 2010. It is highly integrated and uses latest generation of electronic circuitry and valves.
- **BVT (Bruker Variable Temperature Unit)** series of electronics is a standalone system in terms of communication and gas flow control and had been a standard equipment of many generations of Bruker spectrometers. These units are no longer delivered with new systems.

4.2.1 BSVT Series

Overview

This VT system is provided for all NMR applications. The so called BSVT (Bruker Smart Variable Temperature System) replaces all former standalone BVT3000 (Standard, MAS, BEST) and BSMS integrated BVT3200 variants. By using a modular BSMS/2 integrated concept all BVT variants and also former pneumatic units like PNK3, PNK3S and PNK5 as well as the SLCB/2 and SLCB/3 boards are replaced by the new Sensor & Pneumatics Board (SPB) and the Variable Power Supply Board (VPSB). The adaptation of the various probes and temperature control accessory interfaces is realized with smartVT interfaces (also named VTAdapters).

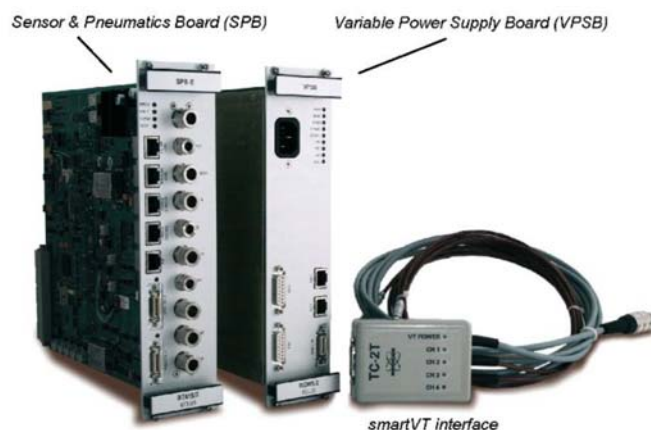


Figure 4.14 BSVT electronics

The BSVT system consists of the following hardware:

- Sensor & Pneumatics Board (SPB or SPB-E)
- Variable Power Supply Boards (VPSB)
- VTA (several styles)

These units are controlled by the BSMS/2 ELCB and are therefore fully integrated into the well known Ethernet™ based communication concept including the web-based service access.

An open architecture provides support of a broad range of equipment

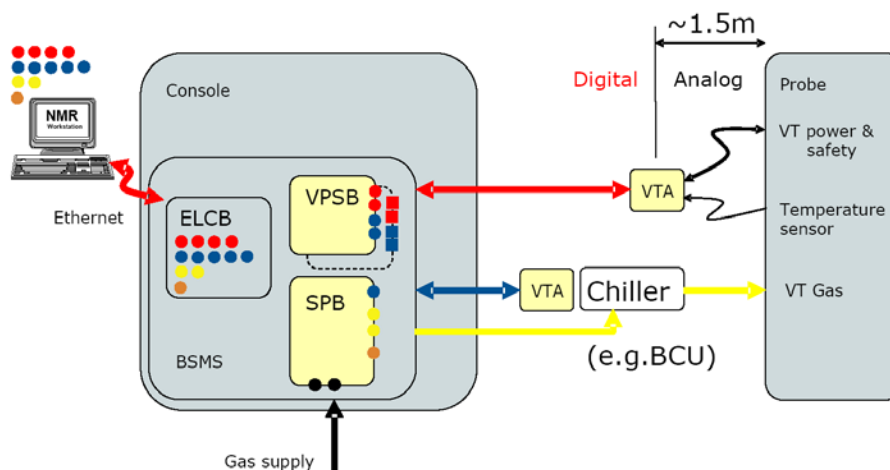


Figure 4.15 BSVT architecture

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.

The VT Adapters for probe head temperature control

- adapts the specific sensor, convert the sensor signal to a digital temperature reading and transmit this value to the BSMS/2 ELCB.
- measures the environmental temperature, use this value to compensate the room temperature dependencies of the specific sensor and transmit the room temperature value to the BSMS, so that also the room temperature can be logged.

Heater power and heater safety sensor signals are fed through the VTA for three reasons:

- the heater power and the corresponding regulation sensor are bundled, thus preventing erroneous usage of a spare sensor for regulation
- the heater current can be filtered close to the probe, suppressing RF noise picked up by the long cable running from the console to the magnet

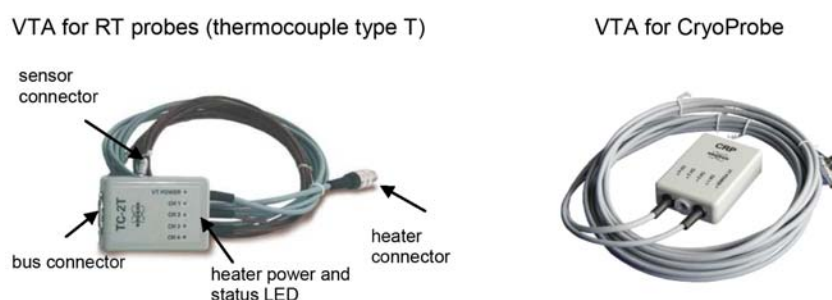


Figure 4.16 Examples of VTA

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 (Z120728BSMS/2 VTA TC-2E, dual sensor)
- thermistors type PT100 (Z116923 BSMS/2 VTA CRP)
- thermistors type PT100 (Z119239 BSMS/2 VTA AUX-2P, temperature reading only)
- 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.

To connect chillers like **BCU-05** or **BCU-X** there is a dedicated VTA available. This accessory adapter can also be connected through a thinner cable that carries only digital signals and low-voltage power supply.

- Z116925 BSMS/2 VTA BCU

To connect low temperature equipment like liquid nitrogen heat exchanger or evaporator that needs heating power, there is also a specific VTA available. This adapter requires connection over a cable that carries digital signals *and* heater power supply.

- Z119238 BSMS/2 VTA LN2

Necessary electrical connections and gas flows are all available on the console back panel:

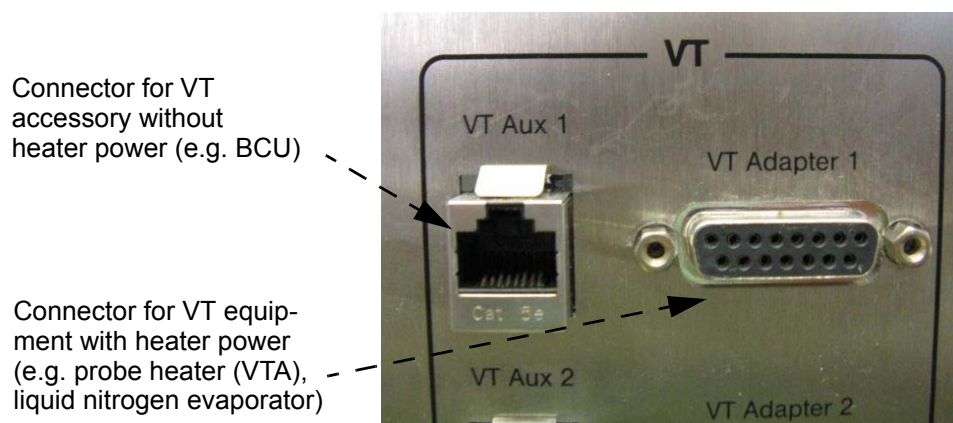


Figure 4.17 Rear panel connectors for VT accessory

VT Gas

VT gas can be set according to the recommendations of the probe used. The flow is *stabilized* and *monitored* using the built-in flow meter and can be accessed via TopSpin™ or BSMS Service Web. The built-in *mass flow* meter is factory calibrated with reference conditions at 0°C and 1013.25 hPa .

Equipment for VT Control

Back panel example:
 AVANCE III CONSOLE
 NANO BAY

Back panel example:
 Example: AVANCE III
 CONSOLE MICROBAY
 or ONEBAY

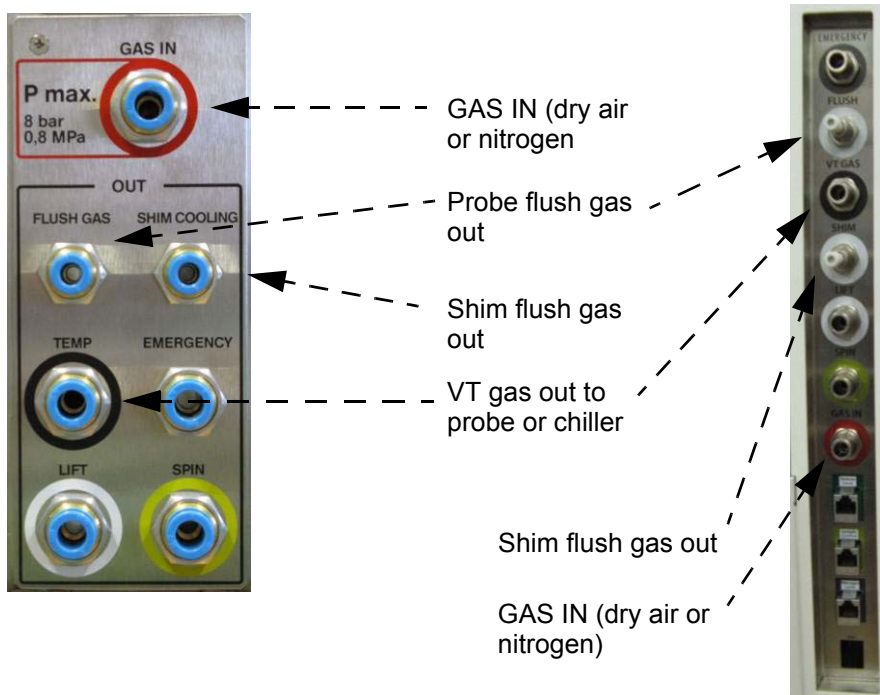


Figure 4.18 Rear panel VT gas connectors

For more information and detailed description please refer to the Technical Manual *BSMS/2 Systems with ELCB* Version 006 or newer, Bruker-P/N Z108028.

4.2.2 BVT Series

The variable temperature units of the BVT series for sample temperature regulation are equipped with a micro controller interface for remote control by the host computer over a RS232 link. The BVT3000 is manufactured in a separate housing whereas the BVT3200(A) is mounted into the console BSMS/2 chassis.

Beside the standard product BVT3000 special versions were available for BEST, MAS or EPR applications.

The units include:

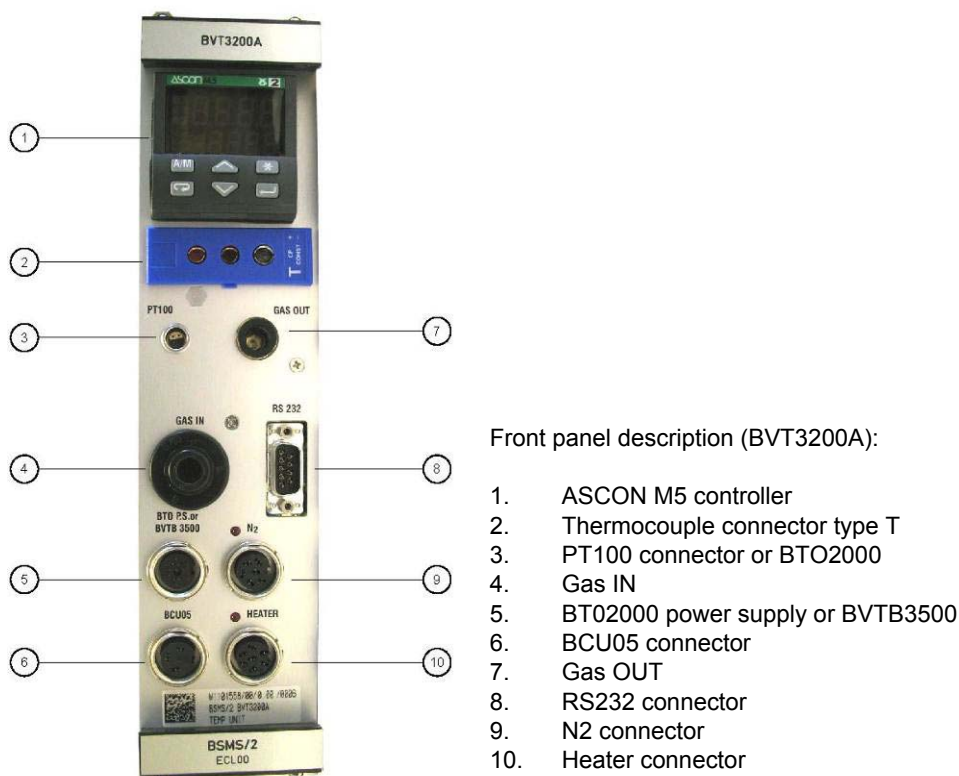
- A main board with a micro controller
- A temperature controller (EUROTHERM or ASCON)
- A main transformer (BVT3000 variants) or Switched Mode Power Supply (for BSVT3200A) that supplies the micro controller and its electronics and the galvanic isolated power stage of the probe heater
- The gas flow circuitry (pressure regulator and a block of four valves for gas flow control)
- An optional printed circuit for liquid nitrogen heater evaporator or liquid nitrogen heat exchanger control



Front panel description (BVT3000):

1. Auxiliary sensors (option)
2. Gas out
3. Gas inlet
4. Pt100 or BTO2000 connector
5. Power switch
6. RS232 connector
7. Heater connector
8. BCU05 connector
9. N2 connector
10. Gas flow indicator
11. BTO2000 power supply or BVTB 3500 connector
12. Thermocouple connector type (T type for NMR ; K type for EPR)
13. Eurotherm controller (2416)

Figure 4.19 BVT3000, front view



Front panel description (BVT3200A):

1. ASCON M5 controller
2. Thermocouple connector type T
3. PT100 connector or BTO2000
4. Gas IN
5. BT02000 power supply or BVTB3500
6. BCU05 connector
7. Gas OUT
8. RS232 connector
9. N2 connector
10. Heater connector

Figure 4.20 BVT3200A, front view

The BVT series of VT units is no longer delivered with new systems.

Main differences between BVT and BSVT series:

	BVT series	BSVT series
Sensor interface	analogue, limited type of sensors	digital, flexible sensor interfaces
VT gas setting	fixed steps	variable, flow regulated
VT gas monitoring	flow detector (on or off)	built-in mass flow meter
Control interface	RS-232	integrated into Ethernet-based communication
Power supply concept	stand-alone (BVT3000 only)	integrated into console

Table 4.8 Differences between BVT and BSVT series

For more information and detailed description please refer to the User Manual *Variable Temperature Unit (VTU)* Bruker-P/N Z31482 available from next Bruker office.

4.2.3 Special Equipment

For very high or low temperature work extra heating or water cooling units are available:

- W1101096 BVTB3500 POWER BOOSTER
- W1208962 BVTE3900 PROBE HEAT EX.50/60HZ

BVTB3500

The BVTB3500 is a power booster unit for both, BSVT or BVT series of temperature control systems. It allows increasing the heating power up to 500 watts. This product is useful for high temperature experiment in NMR or ESR.

The unit is fully controlled by a driving temperature control unit (BSVT/BVT). The BVTB provides the power to the heater at a level set from a control command signal issued by the BSVT or BVT system. It can drive a heater resistance from 6 to 12 ohms.

BVTE3900

The BVTE3900 (P/N W1208962) is a cooling system for high temperature NMR probes. It works in conjunction with BSVT or BVT systems that support *thermocouple type E* and allows operation at very high temperatures (above 150 °C).

It can be used as well for standard or even low temperature.

The system has a cooling circuit, power electronics and safety appliances.

The cooling system comprises a cooling liquid tank and a circulating pump and monitoring circuit to prevent probe overheating.

The power electronics which mainly consists of a power booster is driven by the BSVT or BVT system. The BVTE3900 is fully controlled by the driving temperature control unit. The power electronics provides the power to the heater according a command signal issued by the BSVT or BVT system.

A special version of the BVTE3900 called BVTE3900 LTE (P/N: W1209799) is available. It is intended only for low temperature applications. This system avoids freezing of the probe housing. It works with both BSVT and BVT series of console electronics.

Note

BVTB3500 and BVTE3900 have been designed together with the BVT series of VT units. To allow digital control of the BVTB3500 or BVTE3900 in conjunction with the BSVT system, a separate VTA is necessary (Z119720 BSMS/2 VTA BVTB).

Further Reading

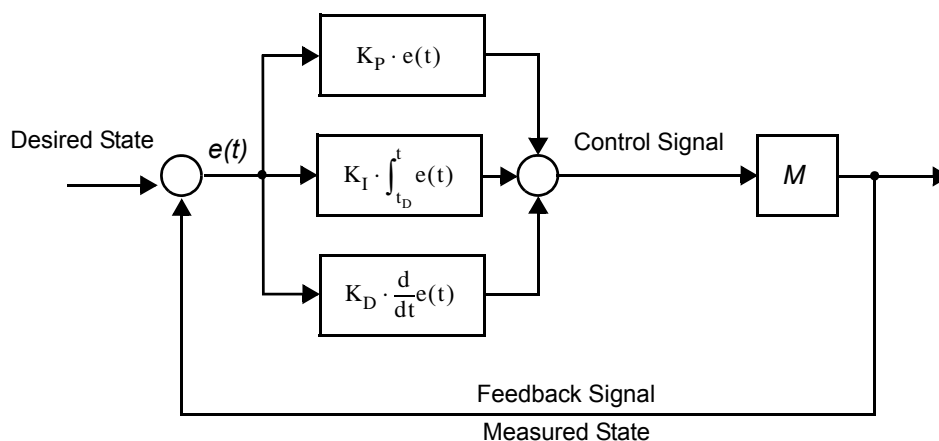
- User Manual *Variable Temperature Unit (VTU)* Bruker-P/N Z31482
- Technical Manual *BSMS/2 Systems with ELCB* Version 006 or newer, Bruker-P/N Z108028

5 Configuration and Applications

5.1 Self-Tune and Temperature Calibration

How to Perform a Self-Tune before Starting an Experiment

The user configurable, stable temperature that is necessary for NMR measurements is stabilized by a PID control system. This system measures in regular intervals the temperature of the sensor in the probe (measured state) and evaluates the deviation from the target temperature (desired state). In addition, this deviation is integrated in an accumulator, and its derivation is evaluated by using an appropriate filter. Accordingly the three weighted terms (proportional, integral and derivative), the heater power is adjusted (control signal).



By tuning the three parameters in the PID controller algorithm, the controller can provide control action designed for specific process requirements (e. g. depending on target temperature, gas flow, VT gas chilling outside the probe).

In both VT controller series (BSVT and BVT), the PID tuning is performed according to the **Ziegler–Nichols** method.

5.1.1 Selftuning a BSVT

Selftuning of a BSVT is a fully automated process (push button). If there are more than one channels involved in temperature control then all channels can be tuned simultaneously. In addition, it is possible, to tune only one channel in a multi channel system, e. g. it is possible to tune only the transfer line heating of a Flow NMR system (and leave the probe heater coefficients untouched) or vice versa.

Selftuning Tab in the VTU Display

There is a specific tab in the VTU display (TopSpin GUI for operation of BSVT based systems), providing all the selftune related actions:

1. Starting the selftune execution of a single channel or the complete VT control system
2. Retrieving the PID settings that have been evaluated by the selftune from the VT controller and storing the settings to the disk
3. Viewing the PID settings resulting from selftune
4. Restoring a PID setting on the VT control system (re-activate a PID setting that has been evaluated in the past and been stored on the workstation).

Selftuning Steps with BSVT

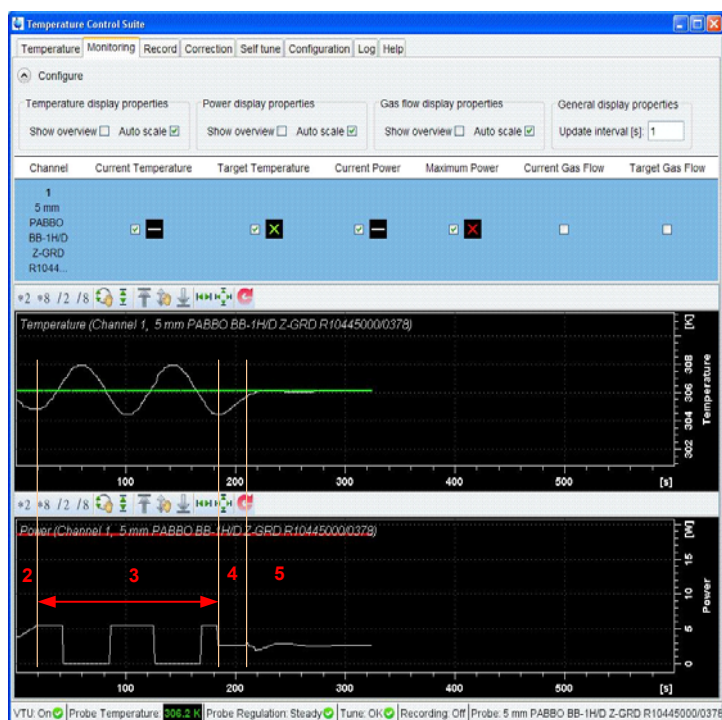


Figure 5.1 VTU Display, Monitoring Window

1. Selftuning can be started when all units involved in the VT control are recognized, initialized and operating. It does not matter if the VT control is in “standby” (off) or “operating (on) state.
2. First, the appropriate heater power is evaluated. If the VT control was operating under similar conditions before then this step is usually complete in a short time.
3. The complete VT system with PID controller, connected probe and accessories (e. g. chiller) is stimulated to oscillate. After a few periods, the appropriate PID coefficients can be evaluated.
4. The VT power is held at a constant value until the temperature has recovered from the oscillation peak.
5. The PID controller is initialized and started with the new PID coefficients.

Considerations

- The **heater power limit** should be set **high enough** in order to have head room for stimulating the oscillations. It is recommended to set the heater power limit to the maximum possible value that still allows safe operation.
- If a **compressor chiller** is used (e. g. BCU I, BCU II, BCU-05, BCU-X), the chiller should have reached the **thermal equilibrium**, before the selftune is started.
- Based on the PID coefficients evaluated by a selftune run, there are several possible derived controller settings:
 - **Standard:** Directly evaluated from selftune result.
 - **Slope limited:** (using standard PID coefficients): The temperature change per time can be limited to a specified value (e. g. to 5 K/min.)
 - **Slow:** Derived PID coefficients (from standard settings) that have more defensive characteristics.
 - **Fast:** Derived PID coefficients (from standard settings) that have more aggressive characteristics – tendency to slight overshoot.

Loading and Restoring Selftune Settings

- Selftune settings are valid for a specific range of operation settings (e. g. target temperature, VT gas flow, chiller strength). There is a **Tune status** indicating whether the selftune settings are “**OK**” or “**Misfit**” for the current operation setting.
- Selftune settings can be read (**Get**) from the PID controller (hardware) and stored to disk.
- It is also possible to load one of these PID settings back onto the PID controller (**Restore to channel ..**).
- In the example below, selftune was made with a target temperature of 306.2 K, a VT flow of 400 lph and a connected BCU type chiller (e. g. BCU I).
- If the Tune status indicates a “Misfit” then either a selftune has to be executed or an appropriate selftune setting has to be restored.

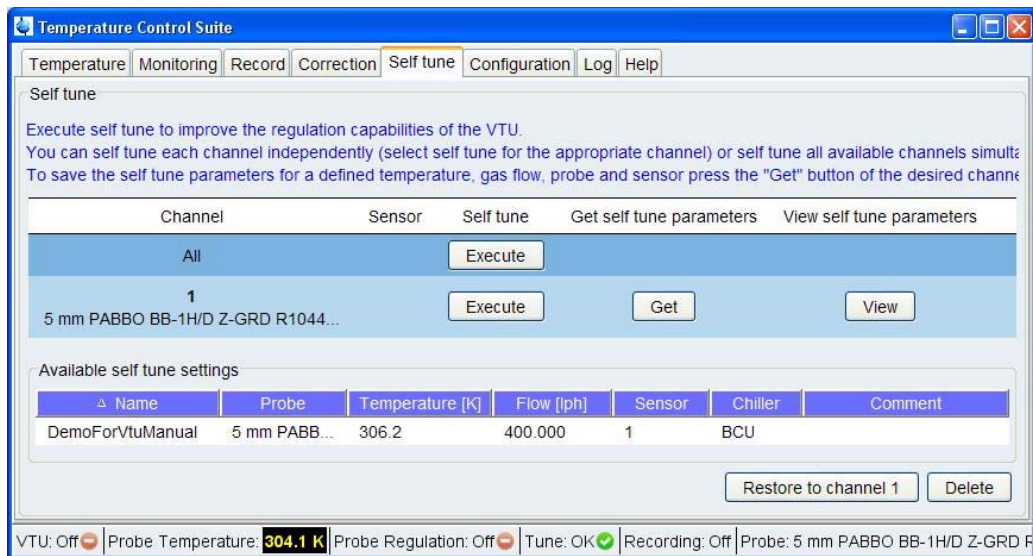


Figure 5.2 VTU Display, Self Tune Window

5.1.2 Selftuning a BVT

BVT systems (e. g. BVT 3000, BVT3200 / 3200A) are always operated with the “edte” software. This software provides also a Selftune operation, which is similar to the BSVT Selftune. The related information is provided in the “edte” manual (“The EDTE Software”, z31464) on the BASH, in chapter 2.

5.1.3 Temperature Calibration

The aim of temperature calibration of a NMR system is the comparability of NMR data (e.g. chemical shifts) measured at the same target temperature at different systems. Due to the construction of the probehead the temperature of the temperature sensor and the sample temperature differ. The difference depends on the type of probe and further on the VT gas flow, the target temperature, the temperature of the environment as well as the cooling unit in use (e.g. BCU I or none). In general you can use any kind of calibration substance with two signals in the proton spectrum and a known relation of the chemical shift difference of those signals and the temperature. In the literature you can find a number of calibration methods using different substances for different temperature ranges. We describe in this section a recommended and proofed standard procedure.

Samples

For room temperature (temperatures between 282 and 320K) by default the 99.8% Methanol-d4 (D, for probes with very high sensitivity) is used for temperature calibration (Findeisen et al., Magn. Reson. Chem. 2007; 45: 175-178).

Temperatures between 300 and 380K are calibrated using an 80% glycol in DMSO-d6 sample (G). For temperatures down to 180K it is possible to use 4% methanol-d4 in methanol (M) or pure methanol (MP).

The *calctemp* au-program (see also edte manual) for calculating the sample temperature from chemical shift differences contains with five different options: (G, GP, M, MP and D).



Figure 5.3 calctemp window

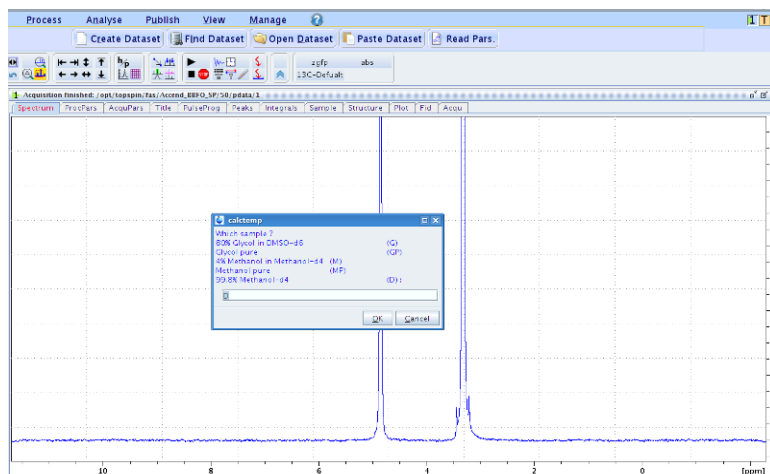
Workflow

In the following a manual workflow for temperature calibration using the TopSpin software (TopSpin 2.1 and newer, see also edte manual) is shown.

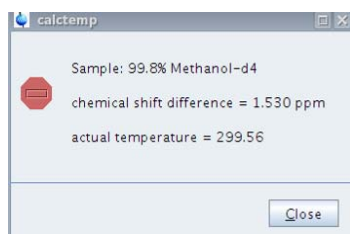
1. Setup your spectrometer (Tuning/matching, proper shimming).
2. Check the correct mounting of your cooling unit (BCU I).
3. Set the air flow you are planning to work with during your experiments (Temperature control suite).
4. Define the temperature range want to work at (e.g. for 303K):
5. Set the temperature to 298K measure a proton spectrum (parameterset ZG, ns 1), change to 308K (wait until stable conditions – might take several minutes) and measure another proton spectrum.

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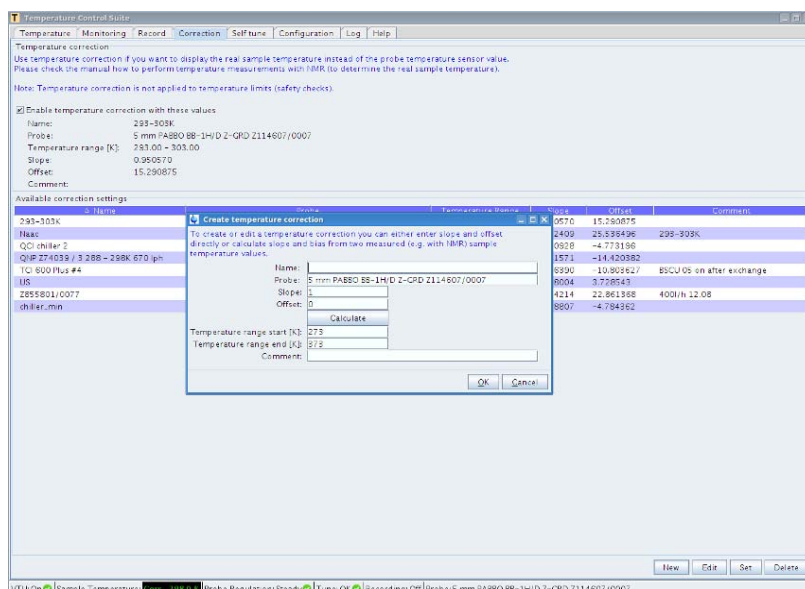
6. Process both spectra using EM and lb=5 (*efp*) and perform a phase as well as a baseline correction.
7. Type *calctemp* into the command line (or *calctemp D* for 99.8% Methanol-d4) for every experiment and calculate the sample temperature:



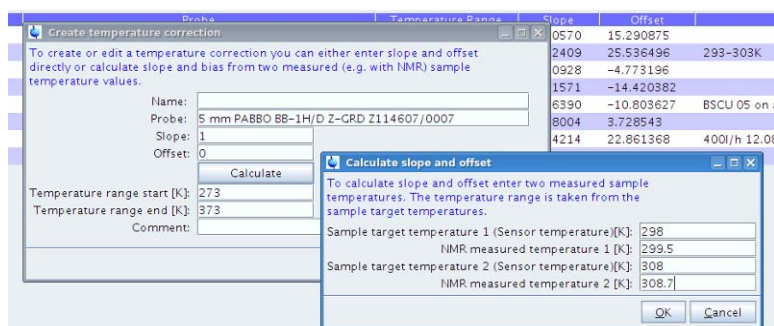
8. The script will calculate the real temperature inside your sample.



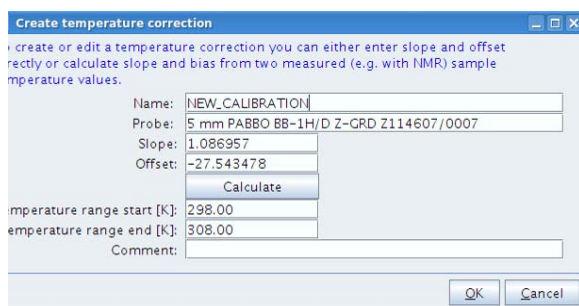
9. Open the Correction folder inside the Temperature Control Suite and click on *New*. The appearing window will ask you to create a new temperature correction. The correction used is linear between 2 points one has to define.



- Click the *Calculate* button and enter the calculated temperature values together with the actual set target temperatures.

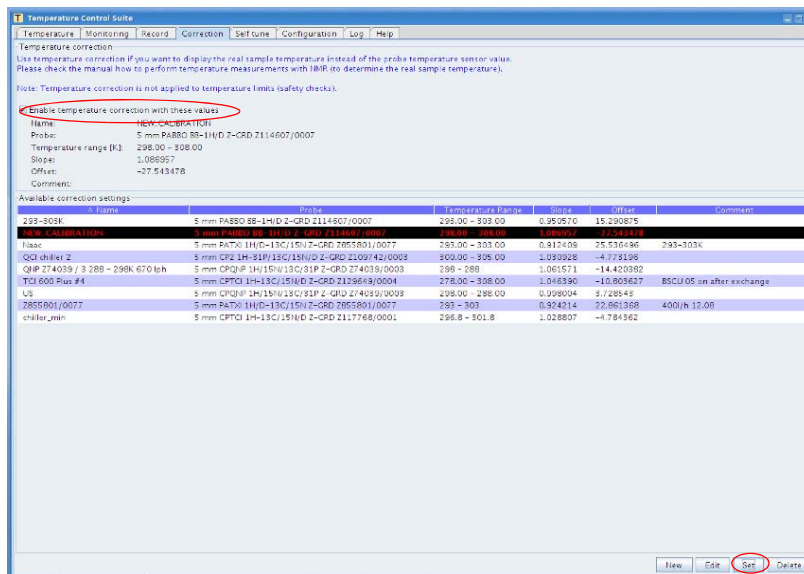


- Press *Ok*. The program calculates slope and offset.
- Name the correction file and press *Ok* again.

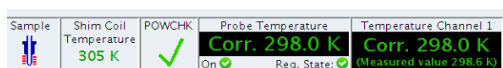
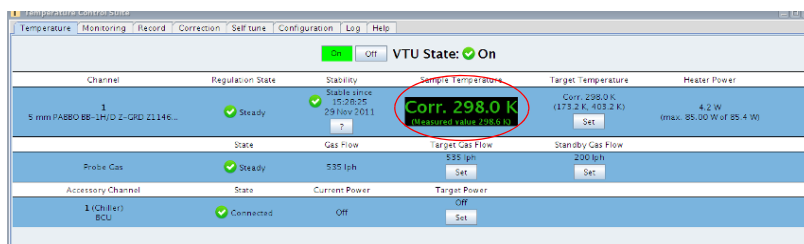


- Finally the new calibration will appear in the list, you have to press *Set* and enable the selected correction.

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14. You can finally see that a correction is used in the Sample Temperature field (Corr. 298.0K) within the Temperature Control Suite and in the TopSpin status line.



15. Check the correction by setting a temperature within the calibration range a measuring the sample temperature (as explained before) using the actual temperature correction. If the correction is correct the sensor temperature and the sample temperature should not differ more than 0.05 K.

5.2 System Requirements for Temperature Control

Before operating at a particular temperature for your application the following questions have to be answered:

- What cooling equipment is available? (BCU I, BCU II, LN₂ heat exchanger/evaporator, none)
- Is the probehead suitable for the temperature range required? Check and define the temperature range of the probe (check in *edhead*, or your probe specifications).



Figure 5.4 Temperature Parameters of the probe in the edhead window

- Do I need to use flush gas?
- Is shim gas required?

Room temperature probes have the possibility to attach flush gas, where CryoProbes do not. Depending on what **Sensor and Pneumatic Board** (SPB or the – **Extended** version: SPB-E) your system is equipped with, the flush and shim gas can be automatically adjusted according to the set temperature (see [Figure 5.5](#) and [Figure 5.7](#)). The settings can be changed in the BSMS service web ([Figure 5.6](#)). Without SPB in the console you have to directly use the N₂ supply together with a needle valve and adjust the shim and flush gas flow manually. In case of the SPB the shim gas is either on or off (BSMS service web). If it is set to “on” a fixed gas flow (1800l/h, not regulated) is delivered. The flush gas has the two settings “auto” on “off” (BSMS service web). If it is set to “auto” a defined gas flow is used depending on the set temperature (T_{set} , see [Figure 5.5](#)).

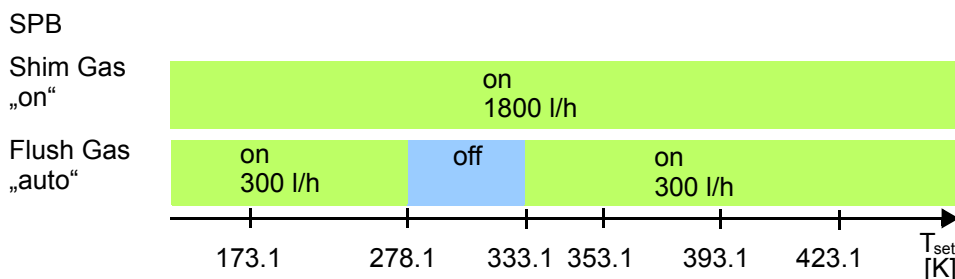


Figure 5.5 Example of default settings for shim and flush gas with SPB.

For the SPB-E more sophisticated settings are possible (BSMS service web, [Figure 5.6](#)):

Gas	Setting
Flush gas:	Auto
	Medium
	High
Shim gas:	Auto
	Medium
	High
	Extreme

Table 5.1 Auxiliary gas flows with SPB-E

BSMS Service Web
Auxiliary Gas Flow

Shim Gas Flow Control

Shim Gasflow Mode:

Shim Gasflow Status:

Shim Gas Flow:

Shim Gas Regulator Time:

Shim Gas Valve

Shim Gas Valve PWM:

Flush Gas Flow Control

Flush Gasflow Mode:

Flush Gasflow Status:

Flush Gas Flow:

Flush Gas Regulator Time:

Flush Gas Valves

Flush Gas Valve PWM:

Flush gas cut-off valve:

Flush Gas Auto Flow Control

	Gas flow definitions		Temperature thresholds
High Flow	<input type="text" value="600"/> l/h	below	<input type="text" value="173.1"/> K
Medium flow	<input type="text" value="300"/> l/h	below	<input type="text" value="278.1"/> K
Flow off	<input type="text" value="0 l/h"/>	below	<input type="text" value="333.1"/> K
Medium flow	see above	else	

Shim Gas Auto Flow Control

	Gas flow definitions		Temperature thresholds
Medium Flow	<input type="text" value="1200"/> l/h	below	<input type="text" value="278.1"/> K
Flow off	<input type="text" value="0 l/h"/>	below	<input type="text" value="353.1"/> K
Medium flow	see above	below	<input type="text" value="393.1"/> K
High Flow	<input type="text" value="2400"/> l/h	below	<input type="text" value="423.1"/> K
Extreme Flow	<input type="text" value="3600"/> l/h	else	

[| Calibration](#) | [Variable Temperature](#) | [Magnet Monitoring](#) | [Sample Handling](#) | [Shim](#) | [Lock](#) | [Grad](#)
[ation](#) | [VT PID Settings](#) | [Gas Flow](#) | [VT Chiller Control](#) | [VT Measurement Control](#) | [Auxiliary Gas F](#)

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Figure 5.6 Auxiliary gas settings in the BSMS service Web (SPB-E)

Hence you can adapt both flows together with the set temperature to meet user requirements. In any case the flow is regulated in contrast to the SPB. The shim and flush gas can also be adjusted according to the target temperature of your sample (BSMS service web) using a defined scheme (“auto”, [Figure 5.6](#)).

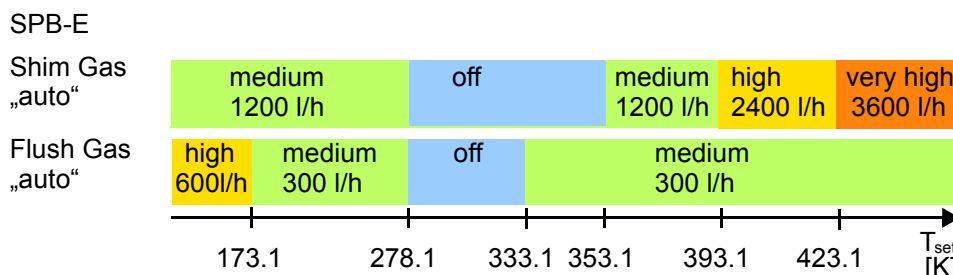


Figure 5.7 Example of default settings for shim and flush gas with SPB-E.

- What are the recommended (optimal) flow settings for this probe? ([Table 5.2](#), set in TopSpin, Temperature Control Suite)
- What is the correct spinner to work with? (see below [Table 5.2](#))

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
Spinner	Ceramics	Ceramics	POM	Kel-F	Ceramics	Ceramics

Table 5.2 Recommended VT gas flows and spinners at different temperatures^a

a. 5mm room temperature probes with extended flow turn

- Which (maximum) heater power should be used (set in TopSpin, Temperature Control Suite)? The maximum heater power is calculated within the system and shown in the Temperature Control Suite window (see below). For optimal temperature regulation one should set the heater value close or equal to the maximum value ([Figure 5.8](#)).

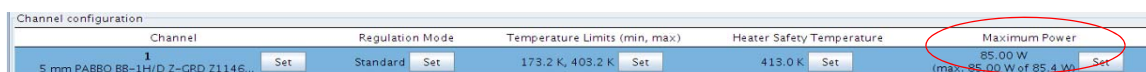


Figure 5.8 Maximum power settings in Watt within the Temperature Control Suite (Configuration) 5mm probes with extended flow turn are defined as standard in the later chapters.

Safety Requirements

During the temperature control of the sample also the temperature of the surrounding material of the probehead will change (warm up, cool down) due to the high or low temperature setting. For safety reasons it is important to make sure that:

1. The temperature of the shim system should always be $-80^{\circ}\text{C} < T < 80^{\circ}\text{C}$.
2. The temperature of the magnet flange (O-Ring!) has to be $3^{\circ}\text{C} < T < 80^{\circ}\text{C}$, especially for long term experiments.
3. The flush the shim system and the probe (shim system temperature control & flush gas) during low temperature experiments (sample temperature $T < 0^{\circ}\text{C}$) to prevent icing and condensed water.

Note:

1. Gas type for “flush gas”: Always use the same type of gas as for the VT gas, but from a separate source (dry clean air or nitrogen at room temperature with a dew point below -50°C).
2. Gas type for “shim system temperature control”: use dry clean air or nitrogen at room temperature with a dew point below -50°C
3. At high temperatures “flush gas” and “shim system temperature control” do have a cooling effect. At low temperatures, “flush gas” and “shim system temperature control” dry the probe inside and the shim system and prevent icing and condensed water.

5.3 Operating RT Probes at Temperatures 0°C up 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).

Before Starting

1. Make sure that the equipment is mounted properly
2. Check temperature range of the probe (edhead)
3. Attaching and setting the flush gas: not necessary
4. Attaching and setting the shim gas: not necessary
5. Recommended (optimal) flow settings for the probe: 535l/h set in TopSpin, Temperature Control Suite)
6. Recommended spinner: POM, Kel-F

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, see also section ["Operating RT Probes at Temperatures down to -40°C" on page 73](#) or ["RT Probe Temperatures down to -150°C" on page 74](#)).

To go up to 80°C (significantly more than room temperature) you don't necessarily need additional equipment (e.g. BCU I). In the following we will focus on the two cases with and without BCU I.

Typical Workflow with Cooling Unit (BCU I)

After checking the first points (see above) and correct mounting of the cooling unit (see section ["BCU I" on page 34](#)) you are ready to start.

To operate from the Temperature Control Suite (TopSpin 3.0 and newer) you have to set the BCU I to *remote status* (see also [Figure 4.9](#) for different operation modes).

1. Start TopSpin and open the Temperature Control Suite (edte)

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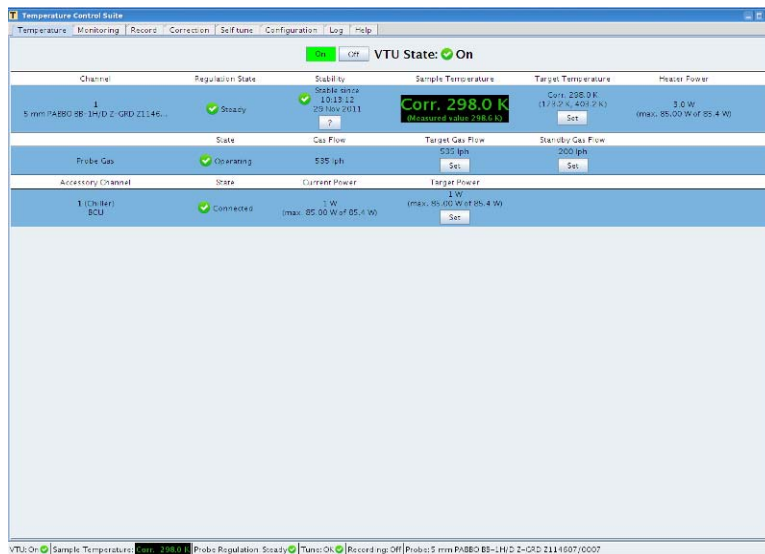


Figure 5.9 Temperature Control Suite

2. Check the configuration settings within the Temperature control suite (Temperature Limits etc.). In Channel 1 you can find the attached probe. The regulation mode is set to standard by default (change if desired). The temperature limits have to be valid for that probe. The heater safety temperature should be set to a slightly bigger value than the temperature limits. If you receive an error message in the beginning of a heating process that the safety temperature was exceeded change the regulation mode to *slope limited* using a defined ramp (see "[Display / Setup of the VT Temperature Limits by TopSpin](#)" on page 83). If the problem remains use a smaller temperature step. The heater power should be set to maximum.

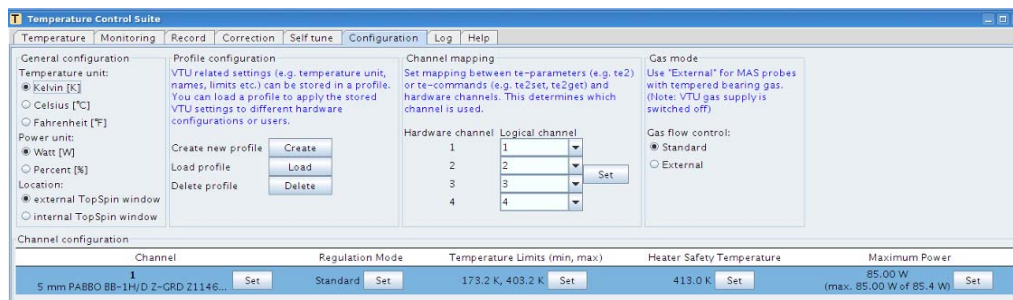


Figure 5.10 Temperature Control Suite /Configuration: settings for regulation mode, temperature limits, heater safety temperature, maximum power

3. Set the BCU I mode (Remote, 1 or 2) depending on your target temperature

Range	BCU I mode
0°C- RT	2 (Maximum chilling)
RT - 40°C	1 (Medium chilling)
up to 80°C	0 (Off - flush, no chilling)

Table 5.3 BCU I modes

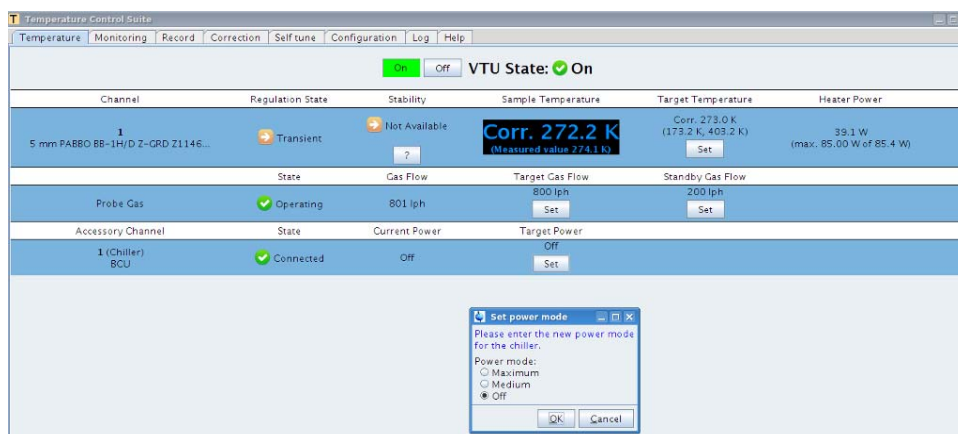


Figure 5.11 Different power mode settings for the BCU I (BCU I on remote)

4. Set target temperature

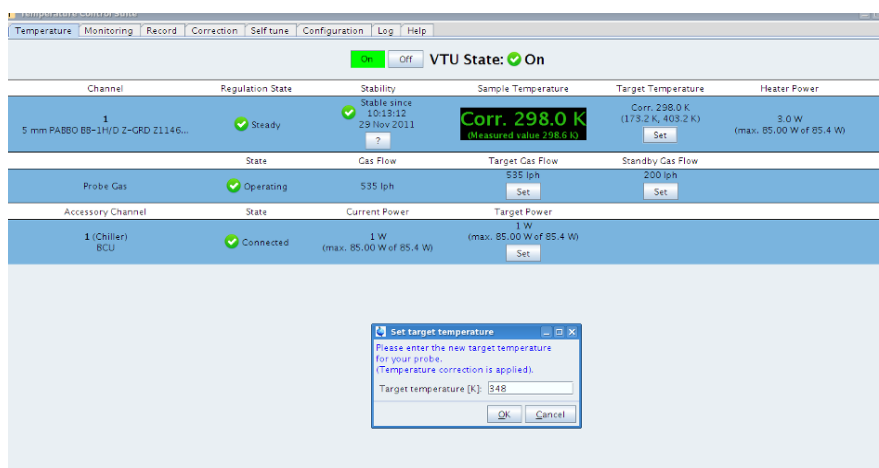


Figure 5.12 Set target temperature

- Wait until the temperature sensor reaches the set temperature (within a few minutes even for a temperature step of about 50 K using regulation mode *standard* for RT probes). Wait for stable conditions of the whole system (e.g. check the heater power, lock level) and inside the sample (different from the temperature sensor status of the probe, see [Figure 5.13](#)) before you start to continue working (e.g. before you start tuning/matching and shimming).

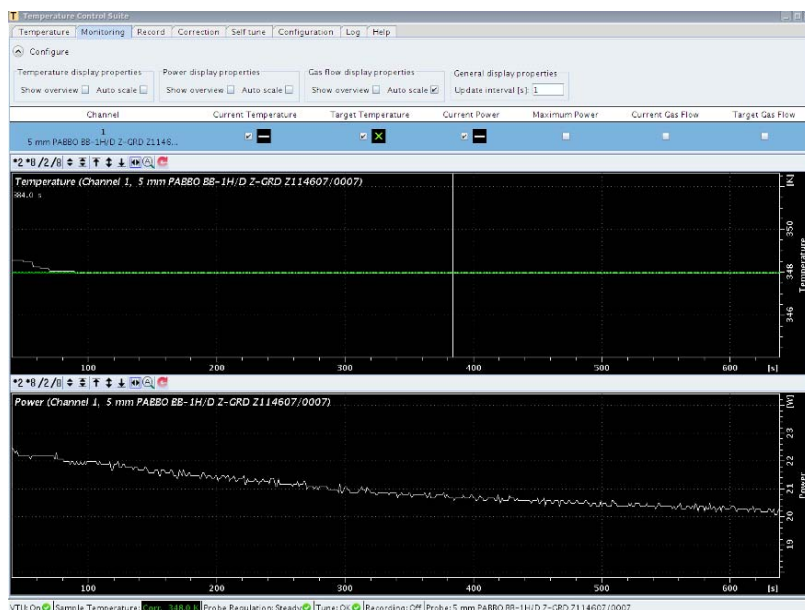


Figure 5.13 Heater power drift (lower part) after probehead sensor appears to be stable (upper part).

6. *Selftune* at target temperature (save PIDs, *only before first setup*)

After entering the new target temperature a warning might appear if the PID parameters are not appropriate for the new temperature (Figure 5.14).



Figure 5.14 Warning message if there are no valid PID parameters for the desired temperature.

Just close that message and proceed with *selftune* (for details see also "[Selftuning a BSVT](#)" on page 56).

7. Check the Shim coil temperature whilst operating at the elevated temperature which is shown as an icon in the TopSpin status bar (if activated before in the status bar preferences), which is **not that critical** for this temperature range.



Figure 5.15 Shim coil temperature in the TopSpin status line

Workflow without BCU I

1. Open the Temperature Control Suite (edte)
2. Check the configuration settings within the Temperature control suite

Range	Operation
0°C to approx. 2 K above Room Temperature (RT)	not possible: only with BCU I or low temperature equipment (see there)
approx. 2 K above RT to + 40°C	use the N ₂ gas/supply
up to +80°C	use the N ₂ gas/supply

Table 5.4 Operation hints when working without a BCU (chiller)

3. Set target temperature
4. The time required to reach target temperature and be able to continue working will be a few minutes (up to 30 minutes).
5. *selftune* (see ["Selftuning a BSVT" on page 56](#))
6. In principle the same operations are carried out as the explanation above (with BCU I), the parameters (heater power) or the time needed to reach the temperature and stable conditions might differ. For temperatures up to 80°C it should be similar to using a BCU I (since BCU I is set in the flush mode).

After Working

There are no specific precautions that need to be taken operating within this temperature range, other than being aware that as you get closer to the edges of the temperature range, heating up, cooling down or returning to room temperature, sufficient time will be needed to regulate (see sections operating at higher or lower temperatures).

5.4 Operating RT Probes at Temperatures up to +150°C

You can work with a BCU I in the flush mode or just the N₂ supply attached to the probe-head.

Before Starting

1. Make sure that the equipment is mounted properly
2. Check temperature range of the probe (edhead)
3. Attaching and setting the flush gas: 300l/h
4. Attaching and setting the shim gas: 1200-1800l/min
5. Recommended (optimal) flow settings for the probe: 535l/h (set in Temperature Control Suite)
6. Recommended spinner: Ceramics

Workflow

The workflow is similar as described above for temperatures up to 80°C (with BCU I).

1. Open the Temperature Control Suite (edte)
2. Check the configuration settings within the Temperature control suite
3. Set BCU I mode to 0 (Off - flush, no chilling)
4. Set target temperature
5. The time required to reach target temperature and will be a few minutes. It can take up to 1 or 2 hours until you are able to continue working.
6. *selftune* (see "[Selftuning a BSVT](#)" on page 56)
7. Checking the Shim coil temperature during operating at those temperatures (up to 150°C) is **critical** ([Figure 5.16](#)). The shim coil temperature should **not exceed 353K**.

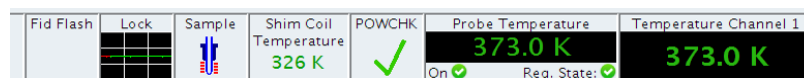


Figure 5.16 Shim coil temperature after 30 minutes working at 373K.

After Working

Change the gas flow back to the suggested value for the new target temperature. Cooling down from 150°C to room temperature takes several minutes for the probe sensor to become stable again. But this does not reflect the time needed for the whole system (shim system etc.) to be at stable conditions again. This will probably take between 1 and 2 hours. You can check this by following the Shim coil temperature and also the heater power monitored in the Temperature Control Suite (see above). Even the sensor temperature appears to be stable the heater power continues to increase, since the heat impact from the whole system is decreasing. When the heater power reaches a stable level you can continue working (shimming). Starting before TopShim might give an error

message or even fail due to the convection stream inside the sample.

5.5 Operating RT Probes at Temperatures down to -40°C

The recommended cooling equipment for that temperature range is the BCU II. In principle it is also possible to use a N₂ heat exchanger.

Before Starting

1. Make sure that the equipment is mounted properly.
2. Check temperature range of the probe (edhead)
3. Attaching and setting the flush gas: 300l/h
4. Attaching and setting the shim gas: 1200l/min
5. Recommended (optimal) flow settings for the probe: 1000l/h (set in Temperature Control Suite)
6. Recommended spinner: Ceramics

Workflow

The workflow is similar to that described above.

1. Open the Temperature Control Suite (edte)
2. Check the configuration settings within the Temperature control suite
3. Set the BCU II mode (Remote, 1, 2 or 3) depending on your target temperature

Range	BCU II Mode
RT down to 0°C	1 (on, chilling)
0°C to -20°C	2 (medium chilling), also 3 is usable
-20°C to -40°C	3 (strong chilling)

Table 5.5 BCU II modes

4. Set target temperature
5. The time required to reach target temperature and be able to continue working will be a few minutes (up to 30 minutes).
6. *selftune* (see ["Selftuning a BSVT" on page 56](#))
7. Check the Shim coil temperature during operation (less critical, [Figure 5.17](#)).

The Shim coil temperature will probably not drop significantly below 5°C otherwise you will be notified. The message is just for your information.

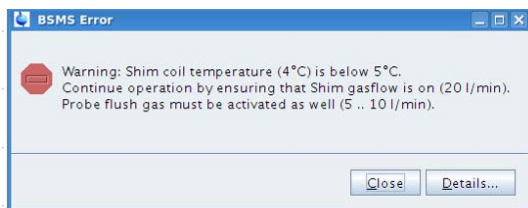


Figure 5.17 BSMS error message if the Shim coil temperature drops below 278 K.

After Working

For changing back to room temperature, set back the BCU II mode to mode 1 (reduced cooling power) and change the target temperature. Adjust the gas flow to the suggested value. Like for high temperatures one has to keep in mind, while going back to room temperature that it takes about 30 minutes until the shim coil temperature is stable enough to successfully perform TopShim.

5.6 RT Probe Temperatures down to -150°C

The recommended cooling equipment for that temperature range is a N₂ heat exchanger.

Before Starting

1. Make sure that the equipment is mounted properly.
2. Check temperature range of the probe (edhead)
3. Attaching and setting the flush gas: 300l/h -600 l/min
4. Attaching and setting the shim gas: 1200 l/min
5. Recommended (optimal) flow settings for the probe: 1200l/h (set in Temperature Control Suite)
6. Recommended spinner: Ceramics

Workflow

The workflow is similar to that described above.

1. Open the Temperature Control Suite (edte)
2. Check the configuration settings within the Temperature control suite (option: use regulation mode: slope limited)
3. Set the BCU I mode to 0 (flush) and attach the flex line to the N₂ dewar or directly attach the N₂ supply to the N₂ dewar.
4. Set target temperature
5. Wait before you continue working until the Shim coil temperature is stable. Since the temperature difference to room temperature is maybe more the 100 K this can take more than 30 minutes up to 1 or 2 hours.

6. Perform *selftune* if no PID parameters are available (see above).
7. Check the Shim coil temperature during operation (critical, [Figure 5.18](#)).

The temperature of the shim coils (Shim coil temperature) will drop far below 0°C depending on the target temperature. The shim coil temperature should **not drop below 193K**.

Fid Flash	Lock	Sample	Shim Coil Temperature	POWCHK	Probe Temperature	Temperature Channel 1
			263 K		Corr. 193.1 K On Reg. State:	Corr. 193.1 K (Measured value 198.8 K)

Figure 5.18 Shim coil temperature after 30 minutes operating at 193K.

After Working

Remove the low temperature device and attach the BCU I to the probe (mode 1 or 2). Set the gas flow back to the recommended value of your target temperature. Like mentioned before it takes some time (up 1 or 2 hours) to get back to operational conditions at room temperature (tuning/maching and shimming), even though the temperature sensor of the probe reaches the target temperature within a couple of minutes.

5.7 Operating Cryo Probes at Temperatures up to +135°C

You can work with a BCU I in the flush mode or just the N₂ supply attached to the probe-head.

Before Starting

1. Make sure that the equipment is mounted properly.
2. Check temperature range of the probe (edhead)
3. Attaching and setting the shim gas: 1200l/min
4. Recommended (optimal) flow settings for the probe: 670l/h
5. (set in Temperature Control Suite)
6. Recommended spinner: Ceramics

Workflow

The workflow is similar like described above for temperatures up to 80°C at RT probes (with BCU I).

1. Open the CryoPanel (type *cryopanel* in command line)
Set Probe Type in the Settings menu to elevated temperature (Figure 5.19).

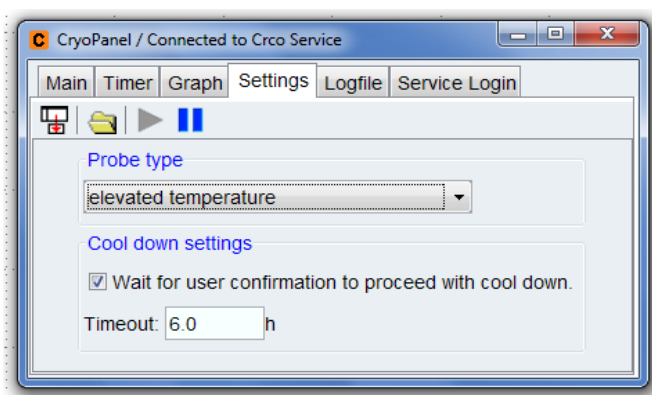


Figure 5.19 CryoPanel settings

Open Temperature Control Suite (edte)

2. Check the configuration settings within the Temperature control suite
3. Set Regulation Mode (slope limited; **max. 10 K in 3 min**, chapter 2.3.3)
4. Set BCU I mode to 0 (Off - flush, no chilling)
5. Set target temperature
6. In general the sensor temperature arrives at the target is stable within a few minutes. Also shimming is normally possible shortly after the sensor appears to be stable, since the whole system is not picking up that much heat (compared to a room temperature probe). You might experience shim problems at elevated temperatures due to convection inside your sample (e.g. also due to melting of the sample mate-

rial in polymer applications), especially using larger sample diameters (10mm Cryo-probe).

7. *selftune* (see ["Selftuning a BSVT" on page 56](#))
8. Checking the Shim coil temperature during operating at those temperatures (up to 135°C) is **less critical** than for RT probes. It just increases by a few Kelvin and will not exceed 353K.

Note

Since there is probably no temperature calibration done at the target temperature you better set the target temperature about 10 K below the maximum (135°C, in the beginning). Otherwise an internal temperature value of the CryoProbe might exceed the maximum and cause an emergency warm-up of the probe.

After Working

Returning from high temperatures to room temperature is possible within a couple of minutes. You can continue working shortly after the sensor temperature is stable.

5.8 Operating Cryo Probes at Temperatures down to -40°C

The recommended cooling equipment for that temperature range is the BCU II. In principle it is also possible to use a N₂ heat exchanger.

Before Starting

1. Make sure that the equipment is mounted properly.
2. Check temperature range of the probe (edhead)
3. Attaching and setting the shim gas: 1200l/min
4. Recommended (optimal) flow settings for the probe: 670l/h(set in Temperature Control Suite)
5. Recommended spinner: Ceramics

Workflow

The workflow is similar to that described above.

1. Open the CryoPanel (type *cryopanel* in command line)
Set Probe Type in the Settings menu to low temperature ([Figure 5.20](#)).



Figure 5.20 CryoPanel settings

2. Open Temperature Control Suite
3. Check the configuration settings within the Temperature control suite
4. Set Regulation Mode (slope limited; **max. 10 K in 3 min**, chapter 2.3.3).
5. Set BCU II mode (Remote, 1, 2, 3) depending on the target temperature (see chapter 4.1.2).
6. Set target temperature
The time required to reach target temperature and be able to continue working will be a few minutes.
7. *selftune* (see "[Selftuning a BSVT](#)" on page 56)
8. Check the Shim coil temperature during operation (**less critical**). The Shim coil temperature will probably not drop significantly below 5°C.

Note

Since there is probably no temperature calibration done at the target temperature you should stay with your set temperature about 10 K above the minimum (-40°C, in the beginning). Otherwise an internal temperature value of the CryoProbe might drop below the minimum and cause an emergency warm-up of the probe.

After Working

Set back the BCU II to mode 1 (reduced cooling power) and change the target temperature. Like for high temperatures on CryoProbes the systems will be ready for work a few minutes after starting the heating up procedure.

5.9 Frequent Asked Questions (FAQ)

- It is not possible to properly shim after changing the temperature even the sensor temperature is stable? TopShim gives an error message.

The temperature measured by the probe sensor is for constructions reasons not entirely consistent with what happens inside the sample. If you applied a quite significant temperature change (more than 20 K) it is not only taking a few minutes more after the probe sensor appears to be stable also the surrounding (e.g. the shim system) of the probe-head (especially valid for RT probes) is picking up some of the heat (or “cold”). This becomes even more an issue for temperatures above 80°C or below -80°C where you have significant heating or cooling. In those cases you can check the Shim coil temperature or follow the heater power in the Temperature Control Suite. It only makes sense to continue working (tuning/matching or shimming) if those values become stable. This might take from minutes to even 1 to 2 hours if you come back from these extreme temperatures towards room temperature (RT).

Since the system is not stable, there are temperature gradients present in the system you will also experience inside your sample. To check if a temperature gradient or convection is present you can turn on the spin and rerun TopShim (or run TopShim with the option *convcomp*, see also the TopShim manual).

After reaching the set temperature the temperature is fluctuating? (see [Figure 5.21](#))

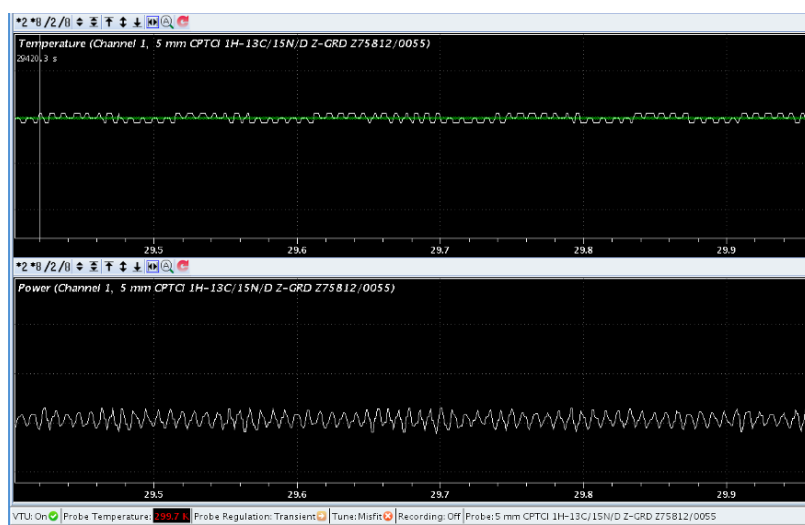


Figure 5.21 Fluctuating temperature (top) and heater power (bottom) due to misfit of PID parameters for temperature regulation.

In this case your temperature regulation parameters (PID parameters) don't match with the new temperature (PID misfit). You have to run *selftune* at this temperature.

- Is there a difference working with different sample diameters (1-10 mm)?

For the extended temperature range (say less than 0°C, above 80°C) there are mainly 5 and 10 mm probes available. As far as you have to stay (because of your probe) within 0 - 80°C there is no difference in temperature handling than for 5 mm. The general workflow is in principle comparable between 5 and 10 mm probes (that's why there is no particular section on this). Differences might arise from the larger sample volume of a 10 mm tube at high temperatures (polymer applications). There are probably more severe convections streams inside such a tube compared to a 5 mm tube and this can cause

more difficulties especially with shimming (use *topshim convcomp*). As far as the gas flow through the probe (flow turn) and the volume of the sample is larger (heat impact), the time frame for heating up or cooling down might become different from a 5mm probe.

- Can I set the heater power to the maximum allowed value?

Yes, you should set the heater power to the maximum allowed value (optimal for temperature regulation), as long as this does not cause problems with your sample.

- Do I have to apply temperature steps of e.g. 10 K (or use a ramp) while heating up the probe by e.g. 100 K?

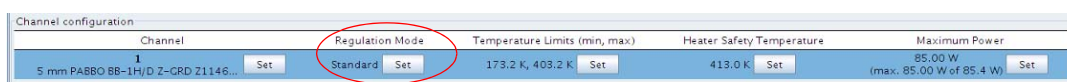
For RT probes you could in principle just set the new target temperature and start. It will not harm your probe. In this case you will reach the desired temperature probably within 1 or 2 minutes (Figure 5.22). On the other hand you have to wait anyway because the whole system is picking up heat and needs time to reach stable conditions. This is normally the most time consuming step. Hence it makes sense to use a temperature ramp (Regulation mode: *slope limited*). For CryoProbes a ramp (see chapter 3.3.3) is recommended (10 K in 3 minutes) while changing the temperature. Since the environment is not that influenced by heating/cooling the probe the whole stabilization process should be faster than for a conventional RT probe.



Figure 5.22 Example of a temperature jump of 80 K, the sensor temperature is depicted on the top, the heater power on the bottom.

- The time required to reach the target temperature is too long?

The first thing to check in such a case is the correct mounting of the equipment providing the gas flow (leakage) to make sure that gas flow really goes into the probe without loss. Are you using the recommended gas flow and the heater power is set to the maximum? If yes, changing the regulation mode to e.g. “fast” might help to speed up the heating process.



6 System and Sample Safety

6.1 BSVT Limits

In the BSVT there are three temperature thresholds providing safety for the probe and the sample:

- **Maximum safety temperature**, checked while monitoring the heater temperature
- **Minimum operating temperature**, and **maximum operating temperature**, checked while monitoring the regulator sensor
- If there is a second regulation sensor then there is also a related maximum supplementary temperature (e. g. for monitoring the chilling gas exhaust temperature).

All three temperature limits can be configured by software.

6.1.1 Maximum Safety Temperature

Monitoring the heater temperature by a separate, redundant sensor prevents the heater from overheating e. g. if the VT gas is missing and the heat is no longer transported towards the sample and the corresponding regulation sensor.

The maximum heater or safety temperature depends on the probe design, and also other settings for the VT control are probe dependent (e. g. operating temperature range, recommended VT gas flow). Therefore, the probe PICS has been extended by these BSVT related parameters, and new probes will provide this information in the future. When initializing a new probe by „edhead“, these values are automatically transferred to the BSVT system (**TopSpin 3.1 PL4 or higher required**).

For probes without BSVT information, these values have to be defined manually in the „edhead“ configuration window (Edit Probe Parameters):

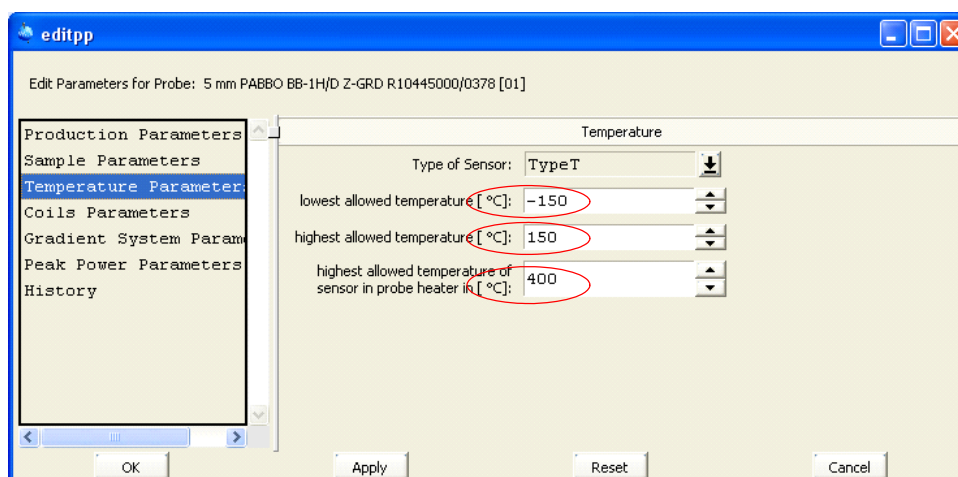


Figure 6.1 Setup of the limits for the safety sensor (TopSpin 3.1 PL4 or higher)

Typical values for the maximum safety / heater temperature are	
Standard room temperature probes (-150°C - +150°C)	400°C
Standard CRP (0°C - +80°C)*	60°C (default) / 90°C (adjusted)**
Low temperature LT-CRP (-40°C - +80°C)*	60°C (default) / 90°C (adjusted)**
Elevated temperature ET-CRP (0°C - +135°C)*	160°C
Former standard CRP (0°C - +60°C)*	60°C (default) / 70°C (adjusted)**

Table 6.1 Typical values for the maximum safety / heater temperature

Notes:

* The CRP type has to be defined also in the Cryo-Tool / Cryo-Panel for the temperature monitoring by the Cryo Platform. If the limits are exceeded then the Cryo Platform shuts down the variable temperature control system (by setting a control signal) and warms up the Cryo Probe.

** The indicated default values of the Cryo Platform can be adjusted / modified by the UniTool (Sample Security Menu...):

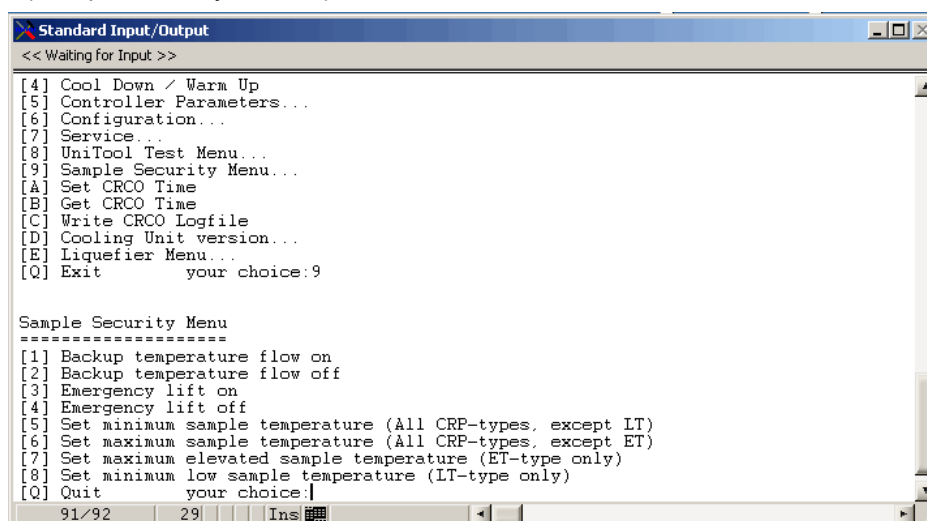


Figure 6.2 Setup of the limits for the CryoProbe by the Unitool

Important:

The same heater temperature PT100 resistor is measured concurrently by the Cryo Platform and the BSVT. However, due to the long 2 wire cable to the Cryo Platform, there is a possible deviation between the two resulting temperature measurements. It is therefore recommended to set the BSVT maximum heater temperature 5°C below the limit defined in the Cryo Platform - this helps avoiding accidental warm ups of the Cryo Probe.

6.1.2 Display / Setup of the VT Temperature Limits by TopSpin

The tab „VT configuration“ in the VT panel of TopSpin provides access to the currently active limits and ranges, and also the Regulation Mode can be modified here:

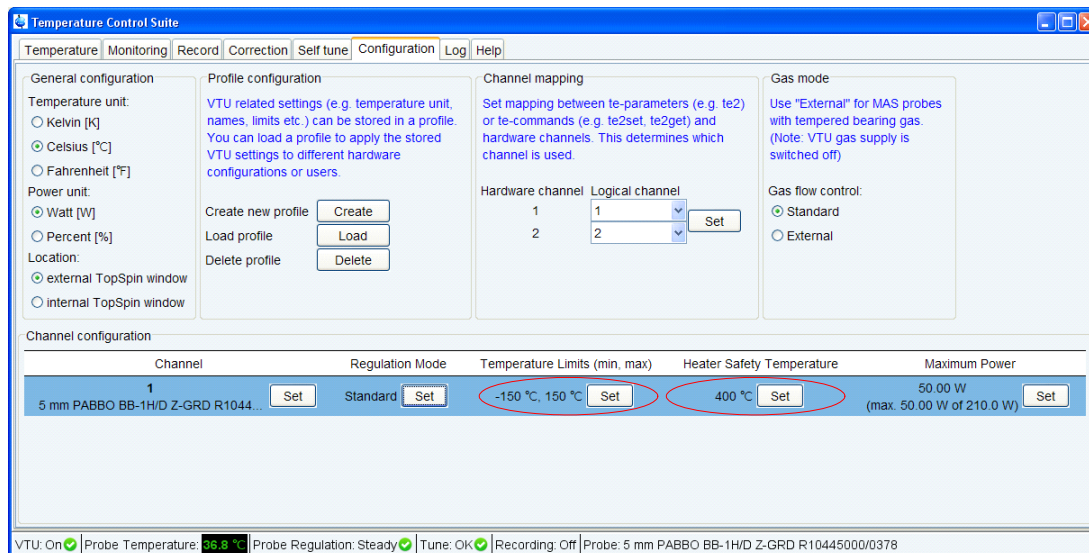


Figure 6.3 Setup / display of limits by the VT Control Suite / TopSpin

The operating temperature range (**Temperature Limits**) has to be set according to the tightest common constraints:

- operating temperature range of the probe
- melting and boiling points of the solvent
- sample temperature range

If the operating temperature is out of the allowed range or if the heater temperature exceeds the maximum safety limit (**Heater Safety Temperature**) then the BSVT is switched off and goes into “standby” state (no active heating or chilling anymore).

The maximum temperature (for VT control) must not exceed the maximum heater / safety temperature. If the aimed VT temperature is close to the maximum limits (e. g. when operating a Cryo Probe) then a **slow regulation mode** is recommended (**slow** or **slope limited**). Otherwise the VT control may try to reach a new target temperature by strong heating and heats up the probe heater above the maximum allowed heater / safety temperature (which would end up in shutting down the BSVT).

The default for **slope limited** VT control operation is **5 °C per minute** and can be adjusted on the BSMS service web in a range between 1 and 100 °C per minute.

6.1.3 Minimum Operating Temperature and Sample Protection

Even if the BSVT is not active / operating, the temperature in the probe and / or in the sample may drop e. g. if a Cryo Probe is in use. Therefore, the sensor temperature is monitored also while the BSVT is in “standby” state, and if the measured temperature drops below the **minimum operating temperature** (+ 5 °C margin) then the BSVT goes into the “**anti freeze protection**” state. In this state, the BSVT is operated with target temperature = 20 °C (room temperature), which should be possible with minimal heater power (and minimal risk).

If the temperature continues dropping, e. g. if heating is not possible due to a missing sensor signal, then the sample lift is activated and the sample is transported to the top of the BST (“**sample protection**” state). The sample is re-inserted as soon as the measured probe temperature is above the minimum limit (+ 5 °C margin).

6.1.4 VT Power Limits

After connection of a new device to an active BSVT channel, the load resistance of the heater (e. g. probe heater, LN2 evaporator heater) is measured by the BSVT, and the maximum power that can be provided by the maximum voltage of 48 V is evaluated.

Examples: If the load resistance is 12 Ω then a maximum power of 192 W is available, whereas for a load resistance of 48 Ω there is at most 48 W available.

A tighter restriction for the maximum power by the user is possible – the user can define a **maximum power** value that is never exceeded by the BSVT. If this limit is too low then the aimed target temperature (and the “stable” state) might never be reached. Also the step response behavior can be influenced by power limitation. Running temperature steps with the PID coefficients evaluated by the selftune typically result in optimal step responses, while power limitation during the transient period might slow down the step response of the BSVT.

Note: In the BVT temperature control unit (BVT 3000, BVT3200 / 3200A), the maximum power value was also used as excitation power for the selftune process. It was therefore important, to chose appropriate maximum power values in order to obtain good selftune results for these former BVT systems.

6.1.5 Temperature Limits for the Shim System

There is a PT100 sensor in the Shim System providing an approximate measurement of the Shim System temperature (the actual temperature may deviate up to +/- 5°C, since the PT100 is operated in a two wire connection over the Shim cables).

The Shim System temperature must not exceed 80 °C, otherwise there is a risk that the Shim System will be damaged. There is a warning issued when the temperature reaches 75 °C, and an error message, when 80 °C are exceeded.

As soon as the Shim System temperature drops below +5°C there is a warning issued, reminding the user to install and run the Shim gas. The following shim gas flows are necessary:

Range	Shim Gas Flow Rate
-150 to +5 °C	Approx. 20 l/min. Shim gas required (prevents water condensation and icing)
+5 to +80 °C	No Shim gas required
+80 to +150 °C	20 to 30 l/min. Shim gas required for Shim System cooling
+150 to +180 °C	40 to 60 l/min. Shim gas required for enhanced Shim System cooling

Table 6.2 Shim gas flow rate

i For operation above +180 °C it is necessary to install a SPB-E, which provides the required Shim gas flow of up to 60 l/min.

A Appendix

A.1 Warning Signs

CAUTION

This combination of symbol and signal word indicates a possibly hazardous situation which could result in minor or slight injury unless avoided.	8
VT gas overpressure will damage the BCU I	36
VT gas overpressure will damage the BCU II	40
Wrong power supply voltage will damage the BCU I	36
Wrong power supply voltage will damage the BCU II	40

NOTICE

Freezing and clogging of the BCU I	36
This combination of symbol and signal word indicates a possibly hazardous situation which could result in damage to property or the environment unless avoided.	8

NOTICE

Freezing and clogging of the BCU II with VT gas	41
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WARNING

magnetic stray field and BCU I	35, 40
This combination of symbol and signal word indicates a potentially hazardous situation which could result in death or serious injury unless avoided.	7

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A.4 Glossar

BCU.....	Bruker Cooling Unit
BST.....	Bruker Sample Transport
BSVT	Bruker Smart Variable Temperature System
BTO	Bruker Thermal Oven
BVT.....	Bruker Variable Temperature unit
BVTB3500	Bruker VT Power Booster
E-FLT.....	Extended FlowTurn
Flush Gas	Auxiliary gas for flushing probe body
LN2.....	Liquid Nitrogen
PID.....	Proportional, integral and derivative - control system
RT.....	Room Temperature
SPB	Sensor & Pneumatics Board
VPSB.....	Variable Power Supply Board
VTA.....	VT Adapter for various temperature sensors and probe heaters

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