Probes

User Manual
Version 002

BRUKER

This manual was written by

D. Marek, R. Triebe, M. Warden

Fällanden, Switzerland
Revised for BASH 2.0 by U. Roos - December 1996
P/N: Z31339
DWG-Nr: 1048002

Contents

Contents

Introduction & Scope 1

General 1.1

This manual applies for the two types of BRUKER NMR probes, which are listed below.

- High-Resolution liquid probes
- Microimaging probes

These probes are classified with a short description in chapter ***3*** (***"Probe Families"***). For all other probes a separate manual is applicable.

This manual provides information about all aspects of operation and maintenance which are of interest for the user. In each chapter, the common properties which apply to all probes are given, followed by information specific to each different type of probe.

Requirements for optimum Performance 2

General 2.1

This chapter describes the conditions which should be met in order to achieve optimum performance of the probe. Only conditions which can be directly influenced by the user are discussed. In striving for top performance the user will be required to optimize the efficiency of the transmit / receive circuitry, the sensitivity of the probe, and the homogeneity of the static magnetic field. The optimum conditions depend on the type of experiment planned and the sample used. Therefore only general guidelines can be given. The precise implementation of these guidelines and the corresponding handling of the probe are detailed in chapter ***5*** (***"Handling and use"***).

Efficiency of the Transmit/Receive Circuit 2.2

Optimum efficiency of the transmit/receive circuit is obtained by tuning and matching the circuit by means of variable capacitors. Maximum efficiency conditions have to be met both for receiving and for transmitting. In the transmitting phase the maximum amount of RF-power from the transmitter has to be absorbed by the spin system in the sample. In the receiving phase the maximum signal to noise ratio (S/N) has to be achieved.

Matching 2.2.1

Optimum power transfer (matching) from the transmitter to the circuit located inside the probe is obtained when the probe circuit impedance is matched to 50 Ω at the frequency of interest. In this ideal case all the power from the transmitter is transferred to the probe without any power being reflected. This power is used to excite the RF-field within the transmit/receive coil. In the receiving phase the perfectly matched circuit will also optimize the S/N of the reception.

Tuning 2.2.2

Optimum tuning of the circuit to the spin system is obtained when the resonance frequency of the RF-coil matches the frequency of the precessing spins which are being observed.

Influence of Samples 2.2.3

In general, the presence of the sample within the probe will significantly change both the matching and the tuning. Dielectric samples may cause significant changes in tuning. Conducting samples will cause a change both in matching and tuning. For the reasons stated above the probe should be matched and tuned with the sample inserted.

Sensitivity of the Probe 2.3

Many experiments require the best signal to noise (S/N) ratio obtainable with the spectrometer. Optimizing the S/N is performed by reducing the noise and increasing the signal. The S/N ratio can only be increased by increasing the signal strength without letting the noise increase at the same rate.

The generation of noise power results in fluctuating voltage which may completely mask the presence of weak NMR-signals. In some cases weak NMR-Signals may be buried in the noise and could be missed unless special attention is paid to increasing the S/N ratio. If a spectrum is recorded and no signal is perceived, this does not necessarily mean there is no signal present. All one can claim is that the signal amplitude is less than the noise amplitude.

Noise Reduction 2.3.1

The noise which has various sources can be divided roughly into noise originating from the sample and noise stemming from the various electronic circuits. The total noise power is the sum of noise power contributions from all sources.

The electronic components are maintained at a given temperature and therefore their noise voltage cannot be reduced by the user.

Increasing the Signal 2.3.2

Sample Concentration

The signal strength is directly proportional to the number of spins exited. The number of spins can be increased by increasing the concentration of the sample.

Increasing the volume of sample also increases the signal amplitude but for a certain probe the usable sample volume is already given.

Number of Scans

The S/N ratio can also be increased by recording a number NS of scans and then taking the average value. The S/N ratio increases with the square root of NS, whereas the acquisition time increases with NS in a linear way.

Pulse Length

Proper adjustment of the RF-pulse length is a further requirement for maximum signal strength.

Field Homogeneity

If the static field is not perfectly homogeneous there will be a spread in precession frequencies causing the spins in one portion of the sample to get out of phase with those in other portions. As this de-phasing of spins progresses the FID signal, being the sum of all portions of the sample will be reduced. Thus, a further requirement for optimal S/N is a maximum homogeneity of the static magnetic field. This can be achieved by shimming as discussed in chapter ***2.4*** (***"Spatial Homogeneity of the static magnetic Field"***).

Spatial Homogeneity of the static magnetic Field 2.4

The line shapes of NMR-spectra depend on the sample under observation and on various factors related to the spectrometer.

The static field produced by the superconducting magnet and its cryo-shim system is already extremely homogeneous. In order to obtain the maximum performance required for high resolution NMR further optimization of the field homogeneity is needed. For example in order to resolve a fine structure of the order of $\Delta f=0.5$ Hz with a 500 MHz spectrometer a relative field homogeneity $\Delta B/B_0$ of better than 10^{-9} is required.

Given that ordinary fluids such as water are highly magnetic compared to the materials used in the probe, the sample itself will generate huge field inhomogeneities at its ends. For this reason the sample has to be of sufficient length so that the static field inhomogeneities at the ends are outside the active volume of the RF coil.

Tiny field inhomogeneities may be caused by the probe itself. It is emphasized that extremely weak magnetization may cause line broadening in high resolution experiments. Because each probe has its own distribution of material the resulting field inhomogeneities differ from one probe to the next even for probes of the same type.

Shimming 2.4.1

In order to minimize the static field inhomogeneities beyond the level attainable with the cryo-shims a set of additional RT field coils (shim coils) is located inside the magnet bore. Each shim coil generates an additional static magnet field which has a specific field profile. The current in each shim coil is adjusted so that the sum of all fields from the shim coils corrects the remaining field inhomogeneities. The process of finding the correct current values for all the shim coils is called shimming.

Sample Rotation 2.4.2

The line broadening which is caused be static field inhomogeneities which remain after shimming may be further reduced by rotating the sample. The rotation frequency has to be chosen so that the relaxation of the spins to their equilibrium position is significantly slower than the period of rotation. In this case the spins are subjected to the average field value taken over a sample revolution.

Temporal Stability of the static magnetic Field 2.5

The static magnetic field generated by the cryo-magnet is not time invariable, but has a minor decay in the course of time and is also influenced by the amount of ferromagnetic material in the vicinity of the magnet. Persons carrying any ferromagnetic objects (pens, keys, pocket-knives ...) in the immediate vicinity of the magnet as well as larger objects (such as cars, trucks, trains and elevators) in the surrounding area will lead to changes of the magnetic field which are not acceptable.

To reduce these effects, a lock system is installed. Usually a substance added to the solvent (or the solvent itself) includes a nucleus otherwise not measured. The precession frequency of this substance is monitored by the system using an extra (lock) channel provided on nearly all probes. In general the lock nucleus is 2H.

The static field is fixed to a defined value with a dedicated field coil B_0 which is part of the RT shim system, using a feed-back loop so that the lock nucleus maintains a constant precession frequency.

Probe Families 3

General 3.1

In this chapter, the various probe types which will be discussed in this manual are listed and described briefly.

Considering the range of magnets, sample sizes, tuning ranges, temperature ranges and built-in options, it becomes obvious that far more than one thousand different probe types are in production. There are, however, many features common to all probes which make a classification into well defined groups possible.

This chapter provides an overview of the various types of probes that are available for high resolution as well as for micro-imaging.

This chapter gives a generic classification of the various probe types which is needed for the description of their operation. However, it does not give specific information about the availability or any performance data of any particular probe. To obtain this kind of detailed information, please consult the current BRUKER marketing documentation or contact your nearest BRUKER sales representative. It should also be noted that there is also a large number of probes available with channels tuned to special nuclei or having other custom-made options, which cannot be listed in this overview.

Classification 3.1.1

For the purpose of this manual, the probes will be classified according to the type (High Resolution / Microimaging), the shim system type (WB, SB), the sample size, the type of connections, the interfaces and type of operation, and finally, the specific electrical properties.

Shim System Types 3.1.2

Probes are being manufactured to fit one of two kinds of shim systems:

SB:

This is the **S**tandard-**B**ore shim system with an outer diameter of 50 mm. It is designed to be mounted in SB magnets but can also be mounted in WB magnet systems. The shim system has an inner diameter of 40 mm. Thus the probes fitting this type of shim system have an outer diameter of slightly less than 40 mm.

WB:

This is the **W**ide-**B**ore shim system with an outer diameter of 88 mm. It is designed to be mounted in WB magnets but can also be mounted in SWB magnet systems. The shim system has an inner diameter of 73 mm. Thus the probes fitting this type of shim system have an outer diameter of slightly less than 73 mm.

The shim systems and the probes are manufactured in a number of different lengths, according to the vertical dimensions of the magnet.

The probes are manufactured to fit various magnets with widely varying field strengths. They are classified according to the appropriate 1H frequency.

High Resolution Probes 3.2

The High Resolution probes described in this manual are probes for top-loading insertion of liquid samples which can be spun if required. This manual does not cover any other types of high resolution probes, such as the LC (Liquid Chromatography flow-cell type of probes). For these probes, refer to the appropriate manuals.

Sample Diameter 3.2.1

The sample diameters also vary between the various probes, allowing the customer to match the probe to the amount of sample available. A general rule is to use the largest probe / sample tube combination which can be sufficiently filled with the available sample substance, according to Chapter ***5.3.1*** (***"High Resolution liquid Samples"***). This gives the best results with respect to the achievable signal to noise ratio. Depending on the experiment other criteria also apply, which limit the useful choice of sample diameters such as achievable field homogeneity, pulse width, to name only a few. For specific information, refer to the specifications of the probes of interest.

In general, the following sample diameters are available:

For SB probes:	2.5, 5, 8*, 10 mm
For WB probes:	5, 8*, 10, 15, 20, 25 mm

* Note: Probes for 8mm sample diameter are available as inverse types only.

Sample VT system 3.2.2

All high resolution probes feature a VT (Variable Temperature) system. This allows the sample to be measured in a wide temperature range. The three basic components of this system are the following:

1. **VT gas duct:** This leads the VT gas (which can be air or nitrogen, nitrogen being preferred because of its cleanliness, dryness, and non-corrosiveness as well as defined constitution) from the source which can be just a hose connected to the VT unit (e.g. B-VT3000), or a more sophisticated system, delivering pre-cooled gas like the B-CU 05. For low-temperature applications, there is an accessory available that delivers nitrogen gas close to 77K to the probe.
   The connection for the VT gas consists of a half-sphere type coupling. The gas source is connected with a simple clamp. The coupling may be either an integral part of the Dewar (see below), or can be a separate metal part connected to the Dewar.
   Inside the probe, the VT gas passes through a glass Dewar and is delivered to

the sample space at the top of the probe. The VT gas passes around the sample, keeping it at the desired temperature. There are two types of flow systems available, the first being the Direct Flow System, where the gas flow passes directly around the sample (used on HT and LT probes, as well as on a number of special probes). The more sophisticated Dual Flow^{TM} system splits the gas stream in two parts, the inner one heating the sample directly, while the outer one heats the coils, making sure that there is no lateral heat loss. After passing by the coils, the outer gas stream passes along the upper portion of the sample, minimizing heat losses through the upper part of the sample. In this way, very low thermal gradients within the sample can be achieved.
The VT gas is vented from the probe through the sample opening on top of the probe, as well as through additional holes located around the collar on top of the probe. The gas then flows through the shim upper part and is then vented to the atmosphere.
In high temperature probes and in the low temperature probe type A the exhaust gas is extracted through the probe.

2. **Thermometry:** The temperature of the gas stream is measured immediately below the sample. The signal from the temperature sensor is fed to the VT unit for measuring and control purposes. There are several versions of the thermometer available, depending on the particular probe:
   - Insertable system thermocouple.
   This is the commonly used configuration with high resolution probes. Temperature is measured by a type T thermocouple (for HT and LT Type A probes a type E thermocouple). The system thermocouple can be inserted into all probes not having a built-in thermocouple. The receptacle is a 4 mm diameter cylindrical part with a central hole of 1.5 mm, protruding from the base of the probe.
   Note: The height of the receptacle is factory-set. Do not attempt to readjust it. This would change the position of the thermometer tip. If misadjusted, the performance of the VT system would be degraded, or the sample may hit the thermometer, resulting in a malfunction of the spinning system and possibly causing damage to the probe and / or the sample.
   - Built-in thermocouple.
   Certain probes have a non-removable thermocouple built-in. In this case, a LEMO-type connector is used to connect the cable.
   - Built-in B-TO2000.
   This is an add-on unit which consists of a thermocouple, integrated with an electronics system, which provides a reference junction as well as a preamplification of the resulting signal. It can be used instead of a regular plug-in thermocouple. It has two connectors, one for power supply, and the other for the signal output. For detailed information, refer to the B-TO2000 manual.
   - Built-in PT-100
   In very special cases, a PT-100 sensor is used for temperature measurement. It is connected to a LEMO-type connector.

3. **Heater:** The gas is heated to the required temperature with a resistive heater. This heater is located within the VT gas duct of the probe in order to minimize the response time of the heater / thermometer control system. There are several types of heaters in use:
   - Insertable system heater.
   This is the commonly used configuration with the high resolution probes. The system heater can be inserted in all probes not having a built-in heater. For insertion see chapter ***5.2*** (***"Inserting the probe in the magnet"***). The heater assembly consists of a shielded heater coil, and a thermocouple unit. The purpose of this thermocouple is to prevent overheating of the heater by shutting it off via the VT unit. This could become necessary in case of a major malfunc-

tion of the VT unit or should the heater be left outside the probe, or should the gas flow not reach the heater, for any reason whatsoever.
- Built-in heater
A number of probes have a heater built-in. In this case, the heater has to be connected directly to the heater cable.

RF properties 3.2.3

There is a vast number of different types of probes, which are distinguished by the types of nuclei for observation and decoupling, their number, combination, and tuning properties. These basic types will be listed and shortly described below. A 2H lock channel is common to all probes, with very few exceptions. The lock channel is factory tuned and matched. If a certain channel is pre-defined for decoupling this does not mean that measurements cannot be performed on this channel, but that the S/N ratio will not be optimal (since the probe has been optimized for S/N on the detection channel).

Fixed-Channel Probes 3.2.4

In these probes, a single nucleus or a number of specific, distinct nuclei are available for simultaneous operation, be it detection or decoupling. The channels are separately tuned and matched by two tuning elements per channel. The following probes fall into this category:

^{1}H Selective (SEL)

The inner NMR coil is tuned to observe 1H.

^{19}F Selective (SEF)

The inner NMR coil is tuned to observe 19F. There is a decoupling channel for 1H. The probe is optimized for low 19F background.

Other nucleus (Selective Probes) (SEX)

The inner NMR coil is tuned to observe a specific nucleus (e.g. 13C, 31P, 11B, 29Si, 27Al). There is a decoupling channel for 1H. The probe is optimized for low background on the observe channel.

There are two special versions of this type of probe:

-Observation of 2H. This probe has a decoupling channel on 1H, and the lock operates on 19F. For the 19F lock, a special hardware accessory for the HPPR is necessary.

-Observation of 3H. This probe has a decoupling channel on 1H. The speciality of this probe is a leak-proof insert. However, the temperature range of this type of insert is restricted.

Dual Probes (DUL)

This probe is for observing 13C. The probe features a 1H decoupling channel, which is designed such that it can be used for decoupling as well as for observation.

Dual Probes (DUX)

This probe is for observing a given nucleus (e.g. 31P). The probe features a 1H decoupling channel, which is designed such that it can be used for decoupling as well as for observation.

Selective Inverse Probes (SEI)

On these probes, the inner NMR coil is tuned to observe 1H. The outer NMR coil is tuned for decoupling on one fixed X nucleus.

Triple Resonance Inverse (TXI)

The inner NMR coil is tuned to observe 1H and the outer NMR coil is double tuned for simultaneous decoupling of two nuclei (e.g. 13C and 15N).

Triple Resonance Observe (TXO)

The inner NMR coil is double tuned to two nuclei (e.g. 13C and 15N), and the outer NMR coil is tuned for decoupling of 1H.

Quattro Resonance Inverse (QXI)

The inner NMR coil is double tuned to 1H and a further nucleus (e.g. 31P). The outer NMR coil is double tuned for simultaneous decoupling of two nuclei (e.g. on 13C and 15N). All four channels can be operated simultaneously.

Broad-Band Probes 3.2.5

One channel (The BB or Broad Band channel) on these probes is tuned and matched by two sets of sliders, which allow a continuous coverage of a very large frequency band with the BB channel. Typically, the tuning range extends from 31P all the way down to 109Ag.

Broadband Observe (BBO)

The inner NMR coil is provided with a BB tuning system and can be used in the BB range. The outer NMR coil is tuned for 1H decoupling and possibly observation, depending on the probe type and application.

Broadband Inverse (BBI)

The inner NMR coil is tuned to observe 1H. The outer NMR coil is provided with a BB tuning system and can be used to decouple any nucleus in the BB range.

Triple Resonance Broadband Inverse (TBI)

The inner NMR coil is double tuned to observe 1H and a further nucleus (e.g. 13C) for decoupling. The outer NMR coil is provided with a BB tuning system and can be used to simultaneously decouple any nucleus in the BB range.

Triple Resonance Broadband Observe (TBO)

The inner NMR coil is provided with a BB tuning system and can be used to observe any nucleus in the BB range. The outer NMR coil is double tuned to 1H and a further nucleus (e.g. 13C) for simultaneous decoupling.

Switchable Probes 3.2.6

Quattro Nucleus Probe (QNP)

Three pre-defined nuclei, usually 31P, 13C, and 15N, or 19F, 31P, and 13C, are tuned on the inner NMR coil and can be selected either automatically or manually one at a time. The outer NMR coil is tuned for 1H decoupling or detection.

An additional software-driven pneumatic unit can provide fully automatic switching between the three nuclei. However, the probe is not suited for rapid and frequent switching.

This probe can be advantageously configured with an automatic sample changer.

CIDNP Probes 3.2.7

CIDNP: (Chemically Induced Dynamic Nuclear Polarization)

The substance being investigated can be irradiated with light, via a quartz optical fibre, during the experiment. This probe is configured with one (SEL) or two channels (DUL / DUX) probe type.

High Temperature Probes 3.2.8

These probes are used to perform high resolution NMR measurements at extreme sample temperatures.

To protect the RF circuits from overheating, a sophisticated water cooling circuit is an integral part of this probe. It restricts the high temperatures to the sample area while the other elements are cooled to about 40 ^{o}C.

For high-temperature operation of this probe, a VT unit with appropriate power (BVT3000 or higher, with the BVT3500 Power booster) and a water and vacuum pump unit (B-MT 05) are required.

This probe is available in SEL or DUL versions.

For details about this probe, refer to the „High Temperature Probehead“ and „High Temperature Accessory“ manuals.

Low Temperature Probes 3.2.9

These probes make it possible to study samples down to -180 ^{o}C.

The following types are available:

Type A (LTA)

This type is especially designed for long-term low temperature experiments. The complete probe body and the high-frequency circuitry are warmed up by a water circuit. The insert is of the direct-flow type.

The probe is available as SEL or DUL only.

Type B (LTB)

This design is very flexible and allows a number of probe type versions. The following types are currently available:

SEL, DUL, BBI, BBO, TXI.

The temperature range is more limited. The insert is of the Dual-Flow type. In this type of probe, only the base of the probe is warmed by a water circuit.

However, two additional heating flanges must be used to prevent the top and bottom magnet Dewar flanges from icing.

For low-temperature operation of this probe, a water pump unit is required. The water flows through the probe base as well as through both heating flanges.

Options for High-Resolution Probes 3.3

Gradients 3.3.1

General

Many of the high resolution probes may be equipped with a Field Gradient Accessory (GRASP). This option allows B0 gradients to be applied during the course of experiments. The theory and use of gradients is described elsewhere.

The gradients are all of the actively-shielded type, i.e. the stray magnetic field generated by the gradient coils is shielded by a second coil which is located around the gradient coil. The field produced by the second coil almost exactly cancels any stray fields outside the gradient which would otherwise generate eddy currents in the magnet structure surrounding the probe and would severely degrade the recovery performance after the application of pulsed gradients.

Gradient types

For the operation of the probes equipped with a GRASP accessory, it is important to distinguish between the following two types of gradients:

Z-Gradient

This type of gradient applies a B0 field whose z-component varies along the Z axis in a linear way.

XYZ-Gradient

This type of gradient consists of three independent gradient sets built into a single package which is integrated into the probe. It has three channels, designated as X, Y, and Z. The B_Z component of each gradient has a linear dependence along the respective axis. This gradient set is equipped with an internal PT-100 thermal sensor, with which the gradient temperature can be monitored.

Q-Switch 3.3.2

Probes with this option have a circuit built-in, which allows the electrical quality factor of a RF channel of the probe to be switched between two states. The detailed description of this system is given in the appropriate manual.

Micro-Imaging Probes 3.4

The micro-imaging probes are designed for NMR imaging and localised spectroscopy of small objects, which fit into the limited sample space available in the SB and WB magnets.

These probes feature high-performance water-cooled gradient systems, together with a variety of exchangeable RF subsystems. The basic design of the micro-imaging probes varies between the two magnet types:

SB Microimaging Probes 3.4.1

This kind of probe consists of a body with exchangeable coil inserts for various sample dimensions. The water-cooled gradient system is plugged into the top of the probe.

Inserts are available for the following sample types:

There are two possible sample types to be used with micro-imaging probes:

1. Top loading (vertical sample) inserts. The samples are inserted in standard NMR tubes (or tubes with a flat bottom) through the BST (Bruker Sample Transport). Special non-spinning spinners are used which always end up in the same position in the probe. This allows for a reproducible sample position with respect to the X and Y axis.
   Inserts for top-loading samples are available for sample diameters of 5, 8 and 10 mm.
2. Direct loading (horizontal sample) Inserts. These Inserts have solenoid coils and are available for samples with diameters of 2 or 4mm. The sample is inserted and removed whilst the probe is outside the magnet.

Inserts are available in the following RF configurations.

There is no lock channel provided in SB-imaging probes. Only SEL (5, 8 or 10 mm) or SEX (5 or 8 mm) Inserts are available for the current probe body.

Due to the limited range of the used trimmers, the lowest frequency on the observe channel must be greater than 50 MHz.

Tuning system

The channels are separately tuned and matched by two tuning elements per channel.

VT system

The VT system is of the direct-flow type. There is an opening along the axis of the probe, which takes the VT heater element. The VT heater element is an integrated, compact part, which consists of the following:

1. Heater
2. Heater protection thermocouple
3. Sample thermometer (Thermocouple)

The probe also features a ball-type connector for the VT air.

Gradient system:

The gradient is of the self-shielded type. It has three gradient coils, X, Y, and Z respectively. The gradient is equipped with a water cooling system and a PT-100 thermal sensor to monitor the temperature. The gradient interfaces to the probe with 8 electrical connectors, two for each gradient, and two for the PT-100 sensor. The gradient is further connected to the body with 2 couplings for the water cooling circuit. The gradient is mechanically attached to the body with a union nut.

At the base of the probe, the electrical connections are terminated in a „Burndy“ connector, which is identical to the one used for the high resolution probes. The base also features two connectors for the water cooling circuit. There are no EMI filters or fuses built into the probe or the gradient itself.

WB Microimaging Probes — *3.4.2*

This probe consists of two major subassemblies:

An RF body with exchangeable coil inserts for various sample geometries.

A gradient subassembly, which consists of the gradient itself, and of a gradient holder tube.

Due to this modular system a variety of RF bodies are available that can be operated with one and the same gradient subassembly.

WB Microimaging Gradient Subassembly — *3.4.3*

The gradient subassembly is semi-permanently attached to the shim system like a standard WB probe. For sample and insert change, the RF body is taken out of the gradient system without removing the gradient system from the magnet.

➪ ***Note: Due to the length of the gradient system, which extends excessively above the magnetic centre, the standard WB turbine, together with the shim upper part, has to be unmounted from the shim system and lifted up by about 5 cm to allow for a proper positioning of the gradient subassembly inside the shim system.***

Gradient system:

The gradient is of the self-shielded type. It has three gradient coils, X, Y, and Z respectively. The gradient is equipped with a water cooling system and a PT-100 thermal sensor to monitor the temperature. The gradient interfaces to the probe with 8 electrical connectors, two for each gradient, and two for the PT-100 sensor.

The gradient is further connected to the body with 2 couplings for the water cooling circuit. The gradient is mechanically attached to the body with a union nut.

At the base of the probe, the electrical connections are terminated in a „Burndy“ connector, which is identical to the one used for the high resolution probes. The base also features two connectors for the water cooling circuit. There are no EMI filters or fuses built into the probe or the gradient itself.

WB Microimaging Universal RF Body 3.4.4

The different inserts are electrically coupled to the RF-body by adjustable capacitors. These capacitors are also used to tune and match the RF circuits of the Insert.

Inserts are available for the following sample types:

There are two possible sample types to be used with micro-imaging probes:

1. Top loading (vertical sample) inserts. The samples are inserted in standard NMR tubes (or tubes with a flat bottom) through the shim upper part. Inserts for top-loading samples are available for sample diameters of 5, 10, 15, 20 and 25mm.
2. Direct loading (horizontal sample) Inserts. These Inserts have solenoid coils and are available for samples with diameters of 2 or 4mm. The sample is inserted and extracted whilst the RF-body is outside the magnet.

Inserts are available in the following RF configurations.

It is possible to have up to three RF channels. Due to the limited space performance deteriorates with an increase in the number of RF channels in an insert.

Single coil inserts are available for sample diameters of 5, 10, 15, 20 and 25 mm. Dual coil inserts are available for sample diameters of 5, 10, 15 and 20mm

RF connections

Due to the versatility in internal paths for the various channels, the BNC RF connectors are only labelled channel 1, channel 2, and channel 3. The correspondence to the various channels may vary from insert to insert and is defined by the documentation accompanying the inserts.

Tuning system

The tuning and matching of the various channels is achieved by two rows of combined slide / turn- action elements. This allows to quickly make coarse changes, while still being able to fine-tune any channel. A special bayonet-like tool is attached to the probe. The allocation of the sliders to the respective channel depend on the insert and are documented with the product.

VT system

The VT system is of the direct-flow type. There is an opening along the axis of the probe, which takes the VT heater element. The VT heater element is an integrated, compact part, which consists of the following:

1. Heater

2. Heater protection thermocouple

3. Sample thermometer (Thermocouple)

The probe also features a ball-type connector for the VT air.

WB Microimaging Dedicated RF Body 3.4.5

For complex probe types, there are also dedicated RF bodies, which can be used only for one sample geometry, but offer complex RF functions. Due to the wide possibilities of these dedicated RF bodies no general description is possible. For further documentation see the detailed documentation delivered with the body.

Probe Code & Nomenclature 3.5

Probes are clearly identified with a code that is defined as follows:

The Code is made up of the following segments:

PH T FM O-D-L-S- X1 X2

PH:Short for Probe(head)

T:Basic probe type (from the list defined below)

F:Basic NMR frequency in MHz (for 1H)

M:Magnet type (SB,WB, SW=SWB)

O:Nuclei on the inner coil separated by a slash and sorted by decreasing frequency (these are usually the observe channels).

D:Nuclei on the outer coil separated by a slash and sorted by decreasing frequency (these are usually decoupling channels).

L:Lock Nucleus

S:Sample diameter

X1:Extra option from the list below (if present)

X2: Extra option from the list below (if present)

When defining Nuclei use only the abbreviation. If there are multiple NMR active nuclei indicate them with the mass number. For standard Broadband use BB and for lowrange Broadband use BBLR.

Examples:

1.Standard dual probe:PH DUL 300WB C-H-D-05

2.Inverse probe:PH SEI 500SB H-C-D-05

3.Multinuclear low range:PH BBO 600SB BBLR-H-D-10

4.QNP probe wit Z Gradient:PH QNP 400SB F/P/C-H-D-05 Z-GRAD

Probe Type Abbreviations 3.5.1

SEL: **Sel**ective IH Probe
SEI: **Se**lective X Nucleus Decoupling, 1H **I**nverse Probe
SEF: **Se**lective 19**F**, 1H Decoupling Probe
SEX: **Se**lective **X** Nucleus, 1H Decoupling Probe
DUL: **Du**a**l** 13C, 1H Probe
DUX: **Du**al **X** Nucleus, 1H Probe
BBI: **B**rad**b**and Decoupling **I**nverse 1H Probe
BBO: **B**rad**b**and **O**bserve, 1H Decoupling Probe
TXO: **T**riple resonance **X**1+X2 Nucleus **O**bserve, 1H Decoupling Probe
or **T**riple resonance **X**1 Nucleus +1H **O**bserve, X2 Decoupling Probe
TXD: **T**riple resonance **X** Nucleus Observe, 1H+X **D**ecoupling Probe
TXI: **T**riple resonance **X**1+ X2 Nucleus Decoupling **I**nverse 1H Probe
TBO: **T**riple resonance **B**roadband **O**bserve, 1H+X Decoupling Probe
TBI: **T**riple resonance **B**roadband +X Decoupling, **I**nverse 1H Probe
QNP: **Q**uattro **N**ucleus (Dual (X1 or X2 or X3) Nucleus and 1H) **P**robe
QXI: **Q**uattro resonance **X**1+X2+X3 Decoupling **I**nverse 1H Probe
MIC: **Mic**roimaging Probe

Option Type Abbreviations 3.5.2

CIDNP: **C**hemically **I**nduced **D**ynamic **N**uclear **P**olarization
HT: **H**igh **T**emperature Probe
LTA: **L**ow **T**emperature Probe Type **A**
LTB: **L**ow **T**emperature Probe Type **B**
Z-GRAD: **Z Grad**ient
XYZ-G: **XYZ G**radient
BRK: **B**io **R**ea**c**tor Probe
IVO: **I**n **V**IV**O** Probe
IVR: **I**n **V**IVO **R**eactor Probe
FLOW: Continuous **Flow** Probe
PERFO: **Perf**used **O**rgans
LEAKPRF: **Leak-pr**oof Insert (for 3H Probes)
CPO: **C**ross **Po**larization

Operating Limitations 4

General 4.1

Take care not to exceed the limitations defined in this chapter and on all documents and tags attached to the probe itself. All information and limitations documented in any form accompanying the delivery of the probe take precedence over any information contained in this document.

There are several categories of operating limitations pertaining to the probes. For each category a subchapter below lists the corresponding limitations. If multiple limitations apply to your probe, the most restrictive limitation is applicable.

Sample type and geometry 4.2

NEVER attempt to use sample tubes greater than the nominal sample diameter of the probe.

NEVER set the sample depth lower than the maximum permissible sample depth, as described in the following table (also documented on the sample depth gauge).

Probe Type	Max Permissible sample depth below MC
2.5mm SB probes	12mm
5mm SB probes, and SB type probes with WB adapter rings	20mm
8mm SB probes, and SB type probes with WB adapter rings	20mm
10mm SB probes, and SB type probes with WB adapter rings	20mm
15mm WB probes	25mm
20mm WB probes	25mm
25mm WB probes	38mm
30mm WB probes	38mm

Sample temperature 4.3

Limitations due to probe types:	
Standard 2.5mm probes:	-120 to +150 ^{o}C
Standard 5.0mm probes:	-150 to +180 ^{o}C
Standard >=8.0mm probes:	-130 to +150 ^{o}C

Microimaging probes:	-50 to +100 ^{o}C
XYZ-Gradient probes:	-50 to +80 ^{o}C
Z-Gradient / 10mm probes:	-50 to +80 ^{o}C
Z Gradient probes for samples <=8mm:	-150 to +180 ^{o}C
HT (High Temperature) probes:	-150 to +600 ^{o}C
LT (Low Temperature) probes Type A:	-180 to +180 ^{o}C
LT (Low Temperature) probes Type B:	-120 to +180 ^{o}C

Limitations due to the shim system / magnet:

Maximum shim system temperature:	+ 80 ^{o}C
Magnet flange / bore temperature:	0 to +50 ^{o}C

The sample temperature range given for the probes (except HT and LT versions) are for short term operation only, and it must be verified that the shim system and magnet limiting temperatures are not exceeded.

Limitations due to spinner type:

The limitation given for the spinners are for the spinner temperature. If you are using a probe with VT gas exhaust via the upper end of the probe, it must be assumed that at least part of the spinner is at to the VT temperature. For probes that have a VT gas exhaust via a vacuum pump the spinner remains rougly at room temperature. For all VT temperatures exceeding the limit of the plastic spinners it is recommended to use the ceramic spinner. In LT / HT probes the ceramic spinner can be used up to the limit of these probes.

Ceramic spinners:	-180 to +180 ^{o}C
Plastic spinners.	-50 to +50 ^{o}C

VT gas flow rate *4.3.1*

The maximum gas flow rates are defined as follows for SB BST:

Non Spinning:

SB ceramic spinners:	1300 l/h
SB white plastic spinners:	1100 l/h
SB blue plastic spinners:	700 l/h

Spinning at 20 Hz:

SB ceramic spinners:	1000 l/h
SB white plastic spinners:	800 l/h
SB blue plastic spinners:	600 l/h

Note that an accessory exists for the BST, that allows for higher VT gas flow rates.

RF Power 4.4

Gradient current 4.5

The currents in gradients are limited by a maximum amount of average heat power that can be input, as well as an aboslute maximum current. If you need maximum current gradient pulses, use only short pulses (<10ms) and respect the given duty cycle given for this current, otherwise the gradient can overheat. If the gradient pulses are clustered within the pulse sequence, then the total sum of pulse time should not exceed 10ms at the maximum current.

Maximum current @ a maximum duty cycle of:

If there is a XYZ-Gradient in the probe:	30A@1:250
If there is a Z-Gradient in a 10mm probe	20A@1:200
If there is a Z Gradient in a probe for samples <=8mm	30A@1:150

Maximum RMS average current:

If there is a XYZ-Gradient in the probe:	2.0A
If there is a Z-Gradient in a 10mm probe	1.5A
If there is a Z Gradient in a probe for samples <=8mm	2.5A

Handling and use 5

Storage 5.1

The probe must be stored in a clean, dry and non-corrosive atmosphere. It is recommended that the probe is stored in the storage box in which it was delivered. Do not store probes in a vertical position without covering the sample opening, otherwise dust might accumulate in the sample area.

Minimum requirements are:

Temperature range:	-20 deg ... +50 deg C
Humidity:	max 95% (non condensing)

Special care must be taken that the storage location is clean and contains no magnetic particles or any sticky material. A probe contaminated in this way will either have too large a background signal, or will no longer be shimmable.

⇨ ***Contamination will seriously degrade the performance of the probe, see chapter 5.9 ("Cleaning").***

Inserting the probe in the magnet 5.2

General 5.2.1

The probe is installed in the magnet using the following steps.

- Select the probe you want to use. Verify that the probe has the diameter (SB=Standard Bore, WB=Wide Bore) and basic 1H frequency corresponding to the magnet and installed shim system you intend to use.
- Wipe the outer shell of the probe with a lint-free material in order to prevent contamination of the magnet bore with magnetic material.
- Verify that the previous probe has been taken out according to chapter ***5.8*** (***"Removal of the probe from the Magnet"***)
- If the probe is a microimaging probe where the sample is mounted directly in the probe, insert the sample according to chapter ***5.3*** (***"Sample Insertion"***)
- Gently insert the probe in the shim system (the protruding object mounted in the centre of the magnet) from below. Take care that the probe axis is aligned with the magnet axis during insertion. Insert the probe slowly, such that the eddy currents do not cause large restraining forces. Strong forces should never be applied during the insertion (and removal).

- Rotate the probe until the mounting points (screw or hooks) are in position and then fix the probe with the mounting system to the shim system. It is recommended to use the same orientation of the probe for all experiments, otherwise you will need to generate shim files for each orientation.

  Standard Bore probes:
  There are six possible mounting orientations. Choose the most convenient position for all connections (cables, VT gas and cooling / heating lines).
  Fix the probe to the shim system using the two mounting screws (these are the two outer gold plated knurled screws diametrically opposite each other).
  If access to these screws is restricted, use the red screwdriver in the accessory kit of the magnet. Tighten these screws alternately without using excessive torque. A high torque does not improve measurements but may damage the probe.

  Wide Bore probes with quick mounting system:
  Verify that the quick mounting unit is open (pull the mounting lever away from the cable connection of the shim system).
  There are two possible mounting orientations. Choose the most convenient position for all connections (cables, VT gas and
  cooling / heating lines).
  Fix the probe by pushing the fixation lever of the shim system towards the cable of the shim system.

  Wide Bore probes without quick mounting system:
  Mounting is the same as for SB probes, but there are only 2 possible mounting positions.

- Connect the RF-cables and filters according to the „HPPR Manual“. Be careful to connect the cables to the correct connector on the probe. The connectors are all identical on a probe and a high power pulse on a wrong channel can destroy the probe.
- If the probe is equipped with a gradient system, connect the cable of the gradient amplifier to the connector on the probe (see the gradient amplifier manual for more details).
- If the probe is equipped with a Q-switch, connect the RF channel selector to the probe (see „RF channel selector manual“ for more details).
- Connect the VT (Variable Temperature) gas tube to the probe, and secure it with the clamp.
- Connect / Insert the VT heater.

  If the probe is equipped with an internal heater connect it with the cable.

  If the probe is not equipped with an internal heater carefully insert the heater of the system into the transfer Dewar and fasten it using the bayonet locking.

- Connect / Insert the VT thermal sensor

  For probes using an external thermocouple, insert the thermocouple completely into the receptacle until a light click is perceived.

  If the probe is equipped with an internal thermocouple connect it with the cable.

  For probes equipped with a PT-100 sensor, connect the PT-100 cable.

  For probes equipped with the B-TO 2000, connect the signal and the power cable to the appropriate connectors on the B-TO 2000 box. If the B-TO

2000 is not permanently attached to the probe, remove it from the probe it is currently attached to, and mount it to the probe you are installing. When removing or inserting the thermocouple of the B-TO 2000 into the probe, take **extreme** care not to break the thermocouple. For all operations with the B-TO 2000 unit, proceed according to the „B-TO 2000 user manual“.

➪ ***WARNING: A Failure to install the complete VT equipment will NOT be detected by the system and may result in an instantaneous full-power operation of the heater. This is a very hazardous situation and should be avoided under all circumstances due to possible fire, personal injury and other consequential damages.***

- If the probe is a HT (High Temperature) or an LT (Low Temperature) probe connect the probe to the B-MT 05 (a temperature control unit) for cooling and warming the probe (see the Manual of the B-MT 05 for details).
- If the probe is a QNP probe and you also want to use the QNP drive unit, install the QNP drive unit according to the „QNP manual“.

Probes with a bulky Base 5.2.2

Depending on the geometry of the magnet stand it may be necessary to turn and / or tilt these probes in the initial phase of insertion into the magnet. Never use excessive force.

SB Micro-Imaging 5.2.3

General

This subchapter describes how the probe is prepared for insertion into the magnet. After this preparation, the other insertion steps are as usual for a SB probe, see chapter ***5.2.1*** (***"General"***). If the insert in the probe is of the top loading type and is to be used in the following measurements, no further preparation is necessary.

If the insert of the probe has to be changed, or the sample is mounted within the probe the following has to be done:

Removal of the gradient from the probe

- Blow the cooling water out of the gradient cooling system of the probe.
- Unscrew the union nut holding the gradient.
- Lift off the gradient from the probe. Be careful not to tilt the gradient when lifting it off the probe, otherwise the insert can be broken.

Removal of the Insert

This step is only necessary if the insert has to be changed. If only the sample is being changed skip this step.

1. Top loading inserts
   Unplug the insert from the probe.
2. Solenoid inserts
   Remove the two fixing screws of the solenoid insert.
   Unplug the insert from the probe.

If you are replacing it with a top loading insert, replace the long screws with the shorter screws belonging to the system (Don‘t take any other screw, the screws belonging to the system are specially non-magnetic).

Mounting of the Insert

This step is only necessary if the insert has to be changed, If only the sample is being changed skip this step.

1. Top loading inserts
   Plug the insert onto the probe in the correct orientation.
2. Solenoid inserts
   If a top loading insert was mounted on the probe, remove the two screws at the location needed to mount the new insert.
   Plug the insert onto the probe in the correct orientation.
   Secure the insert with the two fixing screws that belong to the insert.

Sample placement in the Insert

This applies only to a solenoid insert.
Gently place the sample into the coil of the insert, centring the region of interest with respect to the coil. Take care not to distort the coil.

Remounting of the gradient on the probe

- Plug the gradient onto the probe. Be careful not to tilt the gradient when plugging it onto the probe, otherwise the insert can be broken. Verify that the two O-rings of each cooling water coupling are in place and not damaged.
- Tighten the union nut.

WB Micro-Imaging 5.2.4

General

The WB Micro-Imaging system is made of two subsystems, the gradient subassembly and the RF body (see chapter ***3.4.2***, ***"WB Microimaging Probes"***). For sample or insert change only the inner RF body has to be removed and inserted into the magnet.

Inserting the Gradient subsystem into the magnet

- Loosen the shim upper part of the WB system by opening the screws fixing the shim upper part to the shim coil. Also loose the mounting flange of the shim upper part. Take care to only loosen the screws that connect the two halves of the mounting flange as well as the screws on one half of the flange only. If both flange halves have been moved, the shim files for the probes might have to be changed.
- are loosened from the magnet it will be more difficult to shim the probes.
- Pull out the shim upper part for about 10cm and fix it by closing the screws holding the halves of the mounting flange together. Be careful not to tilt the shim upper part when pulling it out. The delicate inner tubes of the magnet may be deformed or damaged, which could lead to a loss of insulation of the magnet.

- Insert the gradient subsystem as described in chapter ***5.2.1*** (***"General"***) for normal WB probes.
- Loosen the screws connecting the two halves of the fixation flange of the shim upper part and gently lower it onto the gradient subsystem.
- Secure the shim upper part in this position by tightening all the screws of the fixation flange that have been loosened in the process of mounting the gradient subassembly.

Exchanging the Insert on the RF body

- Pull down all three sliders in the matching as well as in the tuning units.
- Open the three latches between the insert and the RF body. Use either a small pin to plug into the hole of the latch and then turn to the left, or pull out the right edge of the latch with the finger nail, or a small screwdriver.
- Unplug the insert. It should come off fairly easy, otherwise the latches have not been opened correctly, or some of the tuning or matching sliders have not been lowered into the lower end position.
- Plug the new insert onto the RF body. Align the tip of the alignment pin with the deepest blind hole of the insert.
- Close the latches between the insert and the RF body by pushing them in to the right. Verify that the latches are properly closed, i. e. that they do not stand out above the surface of the insert / RF body assembly. If the latches cannot be closed, then the insert is not properly plugged onto the RF body.

Sample placement in the Insert

Gently place the sample into the coil of the insert, centring the region of interest with respect to the coil. Take care not to distort the coil. For top loading inserts place the sample tube in the insert. Both types of samples are „held“ only by gravitational force, care must be taken during insertion / removal of the RF body, that the sample / sample tube does not drop out.

Inserting the RF body into the gradient subsystem.

Insert the RF body into the gradient subsystem. Be careful to align the mounting pin of the gradient subassembly with the corresponding hole in the RF body. if the mounting pin cannot be inserted into the RF body one can slide it sideways until the position fits the position of the hole in the RF body.

Push up the RF body to the uppermost position and fix it by tightening the mounting screw of the RF body.

Sample Insertion *5.3*

The following steps are necessary to prepare and insert the sample in the probe. The sample tube should be made of a material which does not include the nuclei to be measured. The common tubes are available in Pyrex (standard) and quartz. Note that when samples are changed the probe might have to be reshimmed (if the samples have different magnetic susceptibilities), see ***"Shimming"***. It may also be necessary to re-tune and re-match the probe (the dielectric constant and/or the electrical conductivity of the samples may be different) see ***"RF-Adjustment"***.

Generally the sample diameter should be identical to the nominal sample diameter of the probe. It is possible to use samples of smaller diameter in a probe but the sensitivity will be reduced due to a reduction in the filling factor.

For filling sample tubes, the following recommendations are given:

- Use the suitable concentration of the substance to be investigated.
- Fill the tube to a height of about 2 times the maximum depth the sample may have below MC (Magnetic Centre). The position of the MC with respect to the spinner can be seen on the sample adjustment gauge. More sample will generally not improve the results, but does no harm as long as it does not spill over the top (this must be avoided under all circumstances). Filling the tube up to the spinner will lead to large thermal losses if the experiment is not performed at room temperature and should be avoided for all such experiments.
- If there is not enough substance to fill the sample as described above, there are the following basic options. Each option has disadvantages which are also described.

    Option 1: Dilute the sample with the solvent, such that the recommended filling level is achieved. This will reduce the signal and may increase the number of scans necessary to achieve the required S/N (Signal to Noise) ratio.

    Option 2: Reduce the length of sample below the MC, such that the amount of sample above and below MC are the same. If the overall sample length is too short, shimming the sample will be difficult and below a certain height even impossible. Note that having the same amount of sample above and below MC gives the best shimming results.

    Option 3: Use special tubes with a solid base and a plug insert that are magnetically matched to the sample. This makes small sample heights shimmable by extending the cylinder of equal susceptibility. Currently, versions of these sample tubes are available that are matched to water.

Samples are generally filled according to standard laboratory procedures. The sample tube can be closed on the upper end by melting, using rubber or plastic plugs or with parafilm. If contamination / evaporation is not a problem, it is possible to use the sample tubes without them being sealed.

The sample tube is inserted into a spinner. Spinners are available in two basic sizes with versions for different tube diameters.

Standard Bore spinners are available for sample tube diameters of 2.5mm, 5mm, 8mm, 10mm and 15mm (the 15mm spinners are only for use in WB magnets with WB probes and a WB to SB adaptor for the shim upper part).

Wide Bore spinners are available for sample tube diameters of 5mm, 10mm, 15mm, 20mm and 25mm.

Spinners are available in plastic and in ceramic versions. The ceramic version must be used for experiments in the high and low temperature range (see chapter ***4.3*** (***"Sample temperature"***)).

For each basic spinner type (SB and WB) a sample adjustment gauge is available. To insert the sample in the spinner proceed as follows:

- Adjust the bottom of the gauge to the maximum depth below MC (Magnetic Centre) of the probe to be used.

- Insert the sample tube into the spinner carefully from above. Be careful to align the axis of the spinner and the sample tube. Do not exert excessive bending forces on the sample tube, which could result in damage to the sample tube. Insert the sample tube into the spinner to a depth greater than required (for easier positioning of the sample with the sample adjustment gauge).
- Insert the spinner with the sample tube into the sample adjustment gauge until the collar of the spinner sits on the edge of the tube of the sample adjustment gauge. This reduces the depth of the sample below MC to a permissible value.
- If the height of liquid of the sample above MC is less than the height of liquid below MC pull the sample tube out of the spinner (upwards) until the height of liquid above the MC is equal to the liquid level below MC. Never push the sample lower than the maximum level allowed.

To insert the sample in the magnet do the following:

- Turn the lift gas stream on by pushing the „LIFT" button on the BSMS keyboard (see „BSMS User's Manual" for more details).
- Wait until the gas stream has built up.
- Insert the sample (with the spinner) in the gas stream above the shim upper part and lower it into the shim upper part until the spinner with the sample is carried by the air cushion of the gas flow. In this position the sample can be let go.
- Verify that the „SPINNING" is off, i.e. the LED is not on or blinking.
- Stop the lift gas stream by pushing the „LIFT" button on the BSMS keyboard (see „BSMS User's Manual" for more details).

SB Micro Imaging (Top loading Sample) 5.3.2

Special non spinning spinners are available for SB Micro-Imaging to transport the sample in and out of the magnet. These spinners always rest in the same position with respect to the gradient system, thus positioning the sample in the same position every time it is inserted. The handling of these non spinning spinners is analogous to the handling of normal spinners see chapter ***5.3.1*** (***"High Resolution liquid Samples"***).

Micro Imaging (Sample mounted directly in Probe) 5.3.3

The insertion and removal of samples in the probe are described in chapters ***5.2.3*** and ***5.2.4*** (***"SB Micro-Imaging"*** and ***"WB Micro-Imaging"***), because this type of sample can only be removed from the magnet together with the RF part of the Micro-Imaging probe.

RF-Adjustment 5.4

General 5.4.1

In order to ensure proper impedance matching between the probe and the HPPR, all active channels of the probe which are intended to be used in the experiment, must be tuned and matched for the frequency needed. A mistuned and mismatched probe may result in longer pulse widths and loss in sensitivity of the probe for this measurement. It may harm the circuits of the power amplifier as well as possibly the HPPR. Tuning and matching values are affected by the sample being measured. It is therefore important to tune and match with the sample of interest inserted in the magnet.

As a general rule for tuning and matching proceed as follows:

If the probe has a BB (Broad Band) channel adjust the broad band channel to the settings as defined on the tag attached to the probe for the nuclei intended to be used on this channel.

Prepare the spectrometer for tuning and matching according to the chapter „Preparing for Acquisition“ in the „AVANCE User‘s Guide“.

As a general rule, it is best to tune and match the lower frequencies first, then proceeding to the higher frequencies.

If tuning and matching of the higher frequencies had to be adjusted very much it is advisable to check (and readjust if necessary) the tuning and matching values for all the nuclei again.

For high resolution liquid probes there are three types of tuning and matching mechanisms.

Fixed Channel Type 5.4.2

These channels have two knobs (in certain probes these are replaced by screw driver tips) that are marked with a „T“ and a „M“ on a coloured ring. The coloured ring marks the channel to which these knobs belong. The ring colour is identical with the colour of the ring around the RF connector of the probe.

The tuning knob is turned to shift the resonant frequency of the probe.

The matching knob is turned to adjust the depth of the wobble curve.

However, depending on the probe type, there may be significant cross-coupling between the functions of the tuning and matching elements, so the adjustment procedure may need to be re-iterated a couple of times until the matching condition is achieved.

These knobs must be adjusted such that the wobble curve meets the baseline at the desired frequency.

BB (Broad Band) Channel Type 5.4.3

These channels have two sets of sliders (one set for tuning and one set for matching) that can be adjusted with the tool attached to the probe. The outer left slider is the one with the highest range (coarse adjustment). The outer right slider is for fine adjustment and does not jump from position to position as the others do.

Therefore adjust the tuning and matching sliders from left to right, starting with the nominal setting given by the tag attached to the probe.

Switchable Channels between three Nuclei 5.4.4

Select channel 2 (middle frequency) of the QNP-switch and adjust the tuning and matching knobs (red knobs) analog to the standard tuning / matching channels.

Select channel 3 (higher frequency) of the QNP-switch and adjust the screwdriver tip behind the QNP-switch with the tool attached to the probe to the best possible setting (lowest reflection at the centre of the wobble display), without adjusting the elements already set.

Select channel 1 (lower frequency) of the QNP-switch and adjust the other screwdriver tip with the tool attached to the probe to the best possible setting (lowest reflection at the centre of the wobble display), without adjusting the elements already set.

Select channel 2 (middle frequency) of the QNP-switch for tuning and matching of the other channels.

Microimaging Probes 5.4.5

For SB (Standard Bore) probes tune and match the probe as if the channels were of standard channel type with the two knobs (one tuning and one matching).

For WB (Wide Bore) probes there are two sets of three sliders. The allocation of the sliders to the respective channel depend on the insert and are documented with the product. Each slider can be adjusted roughly by moving the slide up and down with the tool attached to the probe. The tool attaches to the slide from the bottom with a bayonet fitting. Fine adjustment is made by turning the tool when it is hooked up on the bayonet fitting.

Shimming 5.5

The goal of shimming is to homogenize the static magnet field within the active volume (the area between the coils) of the sample. Inhomogeneities are present due to inevitable imperfections in the magnet, distribution of materials in the probe and the susceptibility of the sample itself.

The shim system is a tube containing a set of coils and is mounted inside the magnet bore. The probe is mounted inside the shim system. The shim system is controlled by the BSMS (Bruker Smart Magnet System) which provides the currents to the shim system as set by the operator.

When shimming a probe / sample one is actually optmiizing the current distribution in these coils. As the inhomogeneities are partly due to the probe and the sample, it is necessary to re-shim every time the probe is exchanged or samples with large changes in susceptibility or filling height are exchanged. In order to reduce the amount of shimming it is recommended to save the shim current values in a shimfile for each probe and possibly also type of sample.

For more details on shimming see the following:

- For handling of the BSMS see „BSMS User‘s Guide“
- For system setup for shimming see „AVANCE User‘s Guide“
- For details to the software see the software manual.

For hints and tips on efficient shimming the reader is referred to the literature.

Experiments 5.6

Variable Temperature (VT) operation 5.6.1

The VT system consists of a controlled gas flow heated by a heater and measured by a thermometer. When operating the VT system, a number of precautions must be taken in order to ensure a correct and safe operation of the system.

Be aware that (except for the HT probes) the VT gas exits the probe essentially at the selected sample temperature. Due to this, the shim system and the shim upper part may be heated or cooled significantly. Generally, low temperatures do not harm the shim upper part or the shim system, but there is danger of freezing the O-rings which seal the magnet bore. If this is allowed to happen, a leak may develop which will lead to loss of thermal insulation and, ultimately, to a quench of the magnet. Therefore, special equipment and procedures have to be used in order to prevent the magnet flanges to be cooled or heated out of the limiting bounds. High temperatures harm all parts of the system. Especially, the temperature the shim system must not exceed the allowable temperature.

➪ ***Take special care not to exceed the temperature limitations given for all parts, see chapter 4.3 ("Sample temperature")***

When using the heater, always make sure that there is sufficient gas flow to remove the heat.

In order to take up VT operations take the following steps:

- - SET the gas flow on the VT unit. (A typical value of the gas flow rate would be 500 l/h, but the optimal flow rate may depend on the experiment.).
- - Verify that the heater is inserted in the probe.
- - Verify that the thermometer is inserted / connected properly
- - Verify that the VT gas line is properly connected to the probe

➪ ***The above mentioned steps will NOT be verified by the system and a leaving out of one of these steps can result in an instant full-power operation of the heater, the VT unit trying in vain to achieve the set temperature at the thermometer. This is a very hazardous situation and should be avoided under all circumstances due to possible fire, personal injury and other possible consequential damages.***

RF experiment setup and execution 5.6.2

The spectrometer is now ready to perform experiments. Take care not to exceed the limitations defined in chapter ***4*** (***"Operating Limitations"***) and on all documents and tags attached to the probe itself. All information and limitations documented in any form accompanying the delivery of the probe take precedence over any information contained in this document.

For the setup of experiments and the handling of the spectrometer see the following:

- „AVANCE User‘s Guide“ for general setup and common experiments.
- „The Software Manual“ for details of parameter setup.
- General literature for explanation of experiments as well as details about the less commonly used experiments.

If you have to change samples proceed according to chapter ***5.3*** (***"Sample Insertion"***) and chapter ***5.7*** (***"Sample removal from probe"***). If the samples have different filling heights and different electrical and/or magnetic properties verify that the probe does not have to be re-tuned / re-matched and re-shimmed see chapters ***5.4*** and ***5.5*** (***"RF-Adjustment"***, ***"Shimming"***).

Samples are often spun around their axis in order to improve the spectral line shape. If this is done it is recommended to use high quality sample tubes that are very straight. When spinning, small peaks may occur above and below the central frequency with a frequency offset of integral multiples of the spinning frequency. A commonly used value for the spinning rate is 20Hz. For details on the setup of spinning rates refer to the „BSMS User‘s Manual“. Be aware of the fact that the sample should not rotate if you are performing X or Y gradient experiments.

Sample removal from probe 5.7

High Resolution liquid Samples 5.7.1

To remove the sample from the magnet do the following:

- Verify that the sample is not spinning.
- Turn the lift gas stream on by pushing the „LIFT“ button on the BSMS keyboard (see „BSMS User‘s Manual“ for more details).
- Wait until the gas stream has built up and the spinner with the sample is carried by the air cushion of the gas flow.
- In this position the sample can be taken away. Be careful not to hit the edge of the shim upper part with the sample tube, the sample tube might break, and spill the sample.
- Stop the lift gas stream by pushing the „LIFT“ button on the BSMS keyboard (see „BSMS User‘s Manual“ for more details).

SB Micro Imaging (Top loading Sample) 5.7.2

To remove the sample from the magnet do the following:

- Turn the lift gas stream on by pushing the „LIFT“ button on the BSMS keyboard (see „BSMS User‘s Manual“ for more details).
- Wait until the gas stream has built up and the spinner with the sample is carried by the air cushion of the gas flow.
- In this position the sample can be taken away. Be careful not to hit the edge of the shim upper part with the sample tube, the sample tube might break, and spill the sample.
- Stop the lift gas stream by pushing the „LIFT“ button on the BSMS keyboard (see „BSMS User‘s Manual“ for more details).

Micro Imaging (Sample mounted directly in Probe) 5.7.3

The insertion and removal of samples in the probe are described in chapters ***5.2.3*** and ***5.2.4*** (***"SB Micro-Imaging"*** and ***"WB Micro-Imaging"***), because this type of sample can only be removed from the magnet together with the RF part of the Micro-Imaging probe. Therefore the first step is to remove the probe from the magnet see chapter ***5.8*** (***"Removal of the probe from the Magnet"***).

Removal of the probe from the Magnet 5.8

- Verify that the sample has been removed from the magnet according to the previous chapter.
- Switch off any VT systems.
- Disconnect all cables and tubing mounted to the probe carefully. It is possible to leave the cooling / heating loops of the HT / LT and Micro-Imaging probes connected to the probes. But be careful that when the probe is removed or inserted into the magnet, that no tilting forces are exerted between the magnet and the probe.
- Disconnect the probe from the shim system:

  If the probe is mounted by screws
  Hold the probe and loosen the mounting screws alternately.

  If the probe is mounted by a quick mounting system
  Hold the probe and release it by moving the mounting lever away from the cable exit of the shim system.
- Pull the probe out of the magnet gently. Be aware that eddy currents exist which exert drag forces on the probe. These eddy currents cease very rapidly shortly before the probe exits the magnet. Therefore do not pull too much and always hold the probe while extracting it from the magnet.
- Store the probe according to chapter ***5.1*** (***"Storage"***).

Micro-Imaging *5.8.1*

For SB Micro-Imaging the procedure to remove the probe is identical to the removal of normal SB probes.

For WB Micro-Imaging probes do the following:

- Switch off any VT systems.
- Disconnect all cables from the RF body.
- Loosen the fixation screw of the RF body. and hold the RF body in its position.
- Lift up the safety latch of the mounting pin of the gradient subassembly. Then lower the RF body out of the system. Note that the safety latch has to be fed through the hole for the mounting pin. Pull the RF body completely out of the magnet gently. Be aware that eddy currents exist which exert drag forces on the RF body. These eddy currents cease very rapidly shortly before the RF body exits the magnet. Therefore do not pull too much and always hold the RF body while extracting it from the gradient subassembly.

If the gradient subassembly also has to be removed from the magnet, because experiments with other probes are intended then also perform the following steps.

- Disconnect all cables and tubing mounted to the gradient subassembly carefully. It is possible to leave the cooling system connected to the gradient subassembly. But be careful that when the gradient subassembly is removed or inserted into the magnet, that no tilting forces are exerted between the magnet and the gradient subassembly.
- Disconnect the gradient subassembly from the shim system. Hold the gradient subassembly and release it by moving the mounting lever away from the cable exit of the shim system.
- Pull the gradient subassembly out of the magnet gently. Be aware that eddy currents exist which exert drag forces on the gradient subassembly. These eddy currents cease very rapidly shortly before the gradient subassembly exits the magnet. Therefore do not pull too much and always hold the gradient subassembly while extracting it from the magnet.
- Loosen the shim upper part of the WB system by opening the screws of the mounting flange of the shim upper part. Take care to only loosen the screws that connect the two halves of the mounting flange as well as the screws on one half of the flange only. If both flange halves have been moved, the shim files for the probes might have to be changed.
- Lower the shim upper part onto the shim coil. Be careful not to tilt the shim upper part when pulling it out. The delicate inner tubes of the magnet may be deformed or damaged, which could lead to a loss of insulation of the magnet.
- Secure the shim upper part in this position. First mount the shim upper part to the shim coil with the appropriate screws. Then tighten all the screws of the fixation flange that have been loosened in the process of mounting the gradient subassembly.
- Store the probe according to chapter ***5.1*** (***"Storage"***).

Cleaning *5.9*

No special precautions have been taken in standard BRUKER probes to avoid contamination from a leaking sample tube. BRUKER accepts no responsibility for any damage which may occur when samples are used containing radioactive or other hazardous materials.

⇨ ***In case of an accident with toxic, radioactive, explosive or biologically active substances, the probe and associated equipment must be cleaned in such a way that there is no longer any danger in any form, especially for all uninformed personnel. If a probe has to be cleaned of all remains of a substance for safety reasons, contact your authorized service station for advice and support, as there is no standard cleaning procedure that is applicable for all probe types. Note that in serious cases it may be necessary for the customer to properly dispose of the probe and replace it with a new one.***

⇨ ***Only use water as cleaning fluid.***

The customer may only clean the outer shell of the probe with a lint-free cloth. For all other cleaning operations contact your authorized service station for advice and support. It may be necessary to send in the probe for a cleaning service, if cleaning of the probe becomes necessary due to excessive background signal.

⇨ ***Do not open probes for cleaning any more than described for normal operation.***

⇨ ***Do not use acetone for cleaning.***

Accessories 6

General 6.1

This chapter describes the most common accessories available for the probes described in this manual. It describes their use and defines what general types are available. For more details about the availability and technical specifications contact your local BRUKER sales representative.

Filters 6.2

For some nuclei whose NMR frequencies are close together the cross talk from one channel to the other can be quite significant. In order to suppress influence from the decoupling channel onto the detection channel a filter is inserted in the RF connection line between the HPPR and the probe (see the HPPR Manual chapter on „HPPR Configuration“ for more details).

Spinners and Samples 6.3

Spinners 6.3.1

Spinners can be divided in three classes

- SB general purpose spinners

  Materials:
  Spinners are available in ceramic, white plastic (heavier plastic version) and in blue plastic (lighter plastic version).

  Sample Diameters:
  Sample diameters of 2.5, 5, 8, 10 and 15mm are available. The 15mm sample diameter is only intended for WB probes.

- SB Non rotating spinners for Microimaging

  Sample diameters of 5, 8 and 10mm are available.

- WB general purpose spinners

  Materials:
  Spinners are available in ceramic and white plastic.

  Sample Diameters:
  Sample diameters of 5, 8, 10, 15, 20, 25 and 30mm are available.

Sample depth gauge 6.3.2

There are two sample depth gauges, one for WB and one for SB spinners. These sample depth gauges should be used to adjust the spinner depth according to chapter ***5.3*** (***"Inserting the probe in the magnet"***)

Standard test samples 6.3.3

Standard test samples are available in sets for each probe type, in order to perform the acceptance tests, and to verify system and probe performance periodically. These samples are filled with an appropriate test substance and sealed by melting of the upper end of the tube.

Empty sample tubes 6.3.4

Empty sample tubes are available in all necessary diameters.

Dedicated fuse system for Microimaging Gradients 6.4

This security box ensures that the gradient cannot be damaged with excessive currents. It is connected to the cooling cycle of the gradient as well as being inserted into current supply line of the gradient.

QNP-Drive Unit for QNP Probes 6.5

For magnets with a stand that has no solid plate at the bottom, first install the mounting plate. Install the QNP-Drive according to its manual.

B-TO 2000 as a System Thermometer 6.6

This is an add-on unit which consists of a thermocouple, with an electronics system, which provides a reference junction as well as a preamplification of the resulting signal. It can be used instead of the insertable system thermocouple and is attached to the probe, using special hardware. For detailed information, refer to the B-TO2000 manual.

Handling Precautions 7

General 7.1

The following precautions should be observed to ensure long probe lifetime:

- NEVER operate probe outside the specified temperature range.
- DO NOT drop the probe; it contains fragile glassware.
- Take sample out before changing probes.
- Be aware of the eddy currents which cease when the probe is out of the magnet. The force hindering the probe from being removed from the magnet reduces drastically shortly before the probe exits the magnet.
- DO NOT set sample eject air too high; set it for smooth sample lift action (see BSMS user‘s Manual).
- DO NOT try to disconnect frozen glass joints, wait until they have warmed up.
- NEVER turn on the heater of the VT system without having checked proper installation of the heater AND thermometer AND the gas line to the probe.
- NEVER turn on the heater of the VT system without sufficient gas flow.
- DO NOT forget to switch the heater off prior to disconnecting any part of the VT system from the probe.
- Turn tuning and matching rods gently; If the force increases rapidly, the end of the tuning range has been reached. DO NOT continue to turn in this direction otherwise the probe will be damaged.
- NEVER put high RF power exceeding the rated power of the probe.
- NEVER readjust any tuning elements inside the probe.
- When shipping a probe DO NOT use the storage box for the probe as a shipping container. Make sure that this box is packed inside another box. Bruker is not responsible for damage done to a probe being shipped to us.

User Service and Maintenance 7.2

User Service and Maintenance is restricted to the following operations:

- Cleaning the probe according to the procedure defined in this document above.
- Exchanging removable insert glass (only if advised to do so by BRUKER Service personnel).

- Exchanging transfer Dewars (only if advised to do so by BRUKER Service personnel).

All other service operations require dedicated test equipment and the strict adherence to service guidelines in order to maintain the quality of the product.

Glossary and Abbreviations **8**

Glossary 8.1

Probe

The probe is the exchangeable measurement cell for NMR spectroscopy / Imaging. For measurement the sample tube containing the sample is inserted into the probe. The probe contains the measurement circuitry (RF and field gradients) directly involved in the measurement (excluding all active processing and amplification circuits), as well as environment control equipment (e.g. temperature).

Sample

This is the substance to be investigated. It is usually contained in a glass tube, the sample tube. Often the term „Sample“ refers to the sample substance together with the sample tube.

Sample tube

This is a glass tube containing the sample of interest. Often the sample tube containing the sample is referred to as the „Sample“.

Shim coil

The shim coil is the part of the shim system with the set of coils used to correct the inhomogeneities and stabilize the static magnetic field. For SB systems the turbine is mounted to this part and is therefore also sometimes included when mentioning the shim coil.

Shim system

The term shim system is used in two meanings. Its narrow meaning is to denote the shim coil. Its broader meaning includes the shim upper part as well as the shim coil.

Shim upper part

The part of the shim system used to transport the sample to the probe. For SB systems the product name is Bruker Sample Transport (BST).

General Abbreviations 8.2

BB	Broadband
BSMS	Bruker Smart Magnet System

Glossary and Abbreviations

BST	Bruker Sample Transport
HT	High Temperature
LT	Low Temperature
MC	Magnetic Centre
NMR	Nuclear Magnetic Resonance
PH	Probe(head)
RF	Radio Frequency
RT	Room Temperature
SB	Standard Bore
VT	Variable Temperature
WB	Wide Bore

Interface Data 9

Gradient connector 9.1

This Burndy connector is used for all gradient systems in probes described in this manual.

The pinout of this connector is as follows:

Table 9.1.

Pin	Function	Used in Z-Gradient	Used in XYZ Gradient	Used in Micro-Imaging
A	Z current +	YES	YES	YES
B	Z current -	YES	YES	YES
C	X current +	NO	YES	YES
D	X current -	NO	YES	YES
E	Y current +	NO	YES	YES
F	Y current -	NO	YES	YES
G	Ground	YES	YES	YES
H	Reserved	n.a.	n. a.	n.a.
J	Reserved	n.a.	n.a.	n.a.
K	Reserved	n.a.	n.a.	n.a.
L	PT 100	NO	YES	YES
M	PT 100	NO	YES	YES

Note: All gradients are floating and electrically isolated from each other and from the PT 100 connections. However, in high resolution probes all the leads pass through RF interference filters internal to the connector and the probe. The ground pin G is connected to the probe‘s shell.

Tables