Bruker NMR Probes

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

- High-Resolution liquid probes
- Microimaging probes

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
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™ 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.

ê **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.

Probe Families

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

2. 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**oo**f** Insert (for 3H Probes)

CPO: **C**ross **Po**larization

Operation Limitations

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 oC
Standard 5.0mm probes: -150 to +180 oC
Standard >=8.0mm probes: -130 to +150 oC

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

Limitations due to the shim system / magnet:
Maximum shim system temperature: + 80 oC
Magnet flange / bore temperature: 0 to +50 oC
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 oC
Plastic spinners. -50 to +50 oC