CryoProbe System

User Manual

Version 001

BRUKER

This manual was written by

M. Schick, D. Marek, R. Triebe, D. Oberli

Fällanden, Switzerland

P/N: Z31551
DWG-Nr: 1258001

Contents

Safety

1

Read these safety instructions carefully and make them accessible to everybody working with the CryoProbe System. A CryoProbe can be operated easily and safely provided the correct procedures are obeyed and certain precautions observed.

Terms and symbols 1.1

WARNING:	Disregard of this may lead to personal injury.
CAUTION:	Disregard of this may permanently damage the system.
IMPORTANT:	Disregard of this can lead to malfunctions.
NOTE:	Hint for good operating practice.

Figure 1.1. Hot surface!

The labelled item may be hot. Be careful when touching it!

Figure 1.2. High voltage!

The labelled item houses a dangerous voltage. Do not open it!

Figure 1.3. Dangerous device!

The labelled item presents a potential hazard. Read the manual if you don't know how to handle it!

Figure 1.4. Cold surface!

The labelled item may be cold. Be careful when touching it!

Figure 1.5. Wear protective gloves!

Put on protective gloves before handling the labelled item.

Figure 1.6. Wear protective goggles!

Put on protective goggles before handling the labelled item.

Figure 1.7. Strong attraction by magnet!

The item is magnetic and presents a potential hazard in the vicinity of a magnet. Keep it away from the magnet!

Disclaimer 1.2

BRUKER is not responsible or liable for any injury or damage that occurs as a consequence of non-approved manipulations on the CryoProbe System.

Emergency 1.3

The main switch on the CryoCooling Unit front (see ***"Control indicators on the CryoCooling Unit front" on page 19***) serves as an *EMERGENCY OFF*. It powers down the systems for cryogenic cooling, vacuum, sensors, and helium gas compression. All valves are reset to their default positions. The CryoPreamps inside the CryoProbe, however, are not affected by an *EMERGENCY OFF* because they are supplied from the HPPR. If the system is kept *OFF*, it will slowly warm up due to thermal conduction.

NOTE: Because an *EMERGENCY OFF* shuts down also the supervisor electronics, it should only be used as a last resort.

When powered on again, the CryoController will first analyze the system state and determine a way to reach a stable situation before restarting the He Compressor etc.

See ***"Emergency Off" on page 71*** for details.

Personnel safety 1.4

All persons who work with or in the close vicinity of a CryoProbe System must be informed about its safety issues and emergency procedures.

WARNING: Do not disconnect any tube or cable from a running CryoProbe System unless *WARM* and *UNPLUG* light up on the CryoCooling Unit front panel.

If in doubt: Put on goggles and protective gloves!

WARNING: Do not manipulate connectors, screws, valves or pressure relief valves other than those that are explicitly described in the CryoProbe System manuals for operator use.

Inherent safety

The CryoProbe System is designed for inherent safety. Pressure relief valves, sensors, and error handling in hardware and software have been included to protect operator, equipment, and environment.

CE certification

CryoProbe, CryoCooling Unit and He Compressor are CE certified.

Technically qualified personnel only

Only persons with a basic technical understanding of electricity, pressurized gas systems, and cryogenics should operate and maintain a CryoProbe System. User interface, system messages, and manuals require a good understanding of the English language.

No user-serviceable parts inside

There are no user-serviceable parts inside a CryoProbe, a CryoCooling Unit cabinet, a He Compressor, or any other component of a CryoProbe System. Do not open these devices.

BRUKER warranty expires if the CryoProbe was opened by unauthorized personnel.

WARNING: Two persons are required to lift the heavy panels of the CryoCooling Unit. Be careful with the panels, there might be sharp edges on their inside which could cause injuries.

WARNING: If you have to work with an open CryoCooling Unit cabinet, put on protective goggles and gloves.

Pressurized cold helium gas cycle

The CryoPlatform works with **helium gas** (He) that is **pressurized** up to about 25 bar and cooled to **cryogenic** temperatures around 20 K. All pressurized parts are kept in strong enclosures which are designed to hold back gas jets or ejected particles in case of a rupture. If unprotected skin is exposed to cold He, severe cold burns are possible.

The helium gas volume inside the cryogenic cooling cycle is small and presents virtually no danger of suffocation. However, the He steel-cylinder contains a substantial gas volume, note the warning below (***"Pressurized helium gas supply" on page 10***).

NOTE: If a pressure or vacuum leak appears, the CryoProbe System will be automatically stopped and warmed up to ambient temperature.

Pressurized helium gas supply

WARNING: Move, connect, and operate the He steel-cylinder carefully. Obey all safety precautions pertinent to high pressure gas containers and magnetic objects.

WARNING: The He steel-cylinder and its entire transport path must always be outside the 0.5 mT range of the magnet.

WARNING: Fix the He steel-cylinder reliably to a wall. All local safety regulations for the installation of pressurized gas systems must be obeyed.

The helium pressure hose between the He steel-cylinder and the CryoCooling Unit carries a steel wire that must be fixed to the units at its ends. If crossing of walkways cannot be avoided, the He Hose must be covered or buried. Moreover, the He Hose must be fixed to a wall or to the floor once every meter.

WARNING: If the He Hose is not fixed it can whip around in case of a rupture.

WARNING: If a large quantity of helium gas escapes from the He steel-cylinder during a short period, there is a danger of suffocation, particularly in small rooms. Care for good ventilation and fresh air supply after an accidental release of large quantities of helium gas.

Overpressure release noise

Overpressure in the system is avoided by software control and mechanical safety valves. In case of malfunction of software as well as human interference into valve settings, the release valves can open with an extremely loud bang! The sound protection cabinet will reduce the noise to a safe level, therefore do not operate with an open cabinet.

WARNING: If a service action on an open CryoCooling Unit cabinet cannot be avoided while the He Compressor runs or while the helium gas supply is manipulated, the ears must be protected.

Electrical safety

The CryoCooling Unit’s degree of protection against electrical hazard complies with IEC IP20, i.e. all electrical parts are protected against touching.

WARNING: All electrical connectors must be used as supplied by BRUKER. Do not substitute them by other types.

No hazardous substances

There are virtually no substances in a CryoProbe System that could be hazardous for an NMR user. See the *CryoProbe Installation* manual for materials that need special consideration upon relocation or disposal.

Lifting the CryoProbe

WARNING: Two people are needed to insert and remove the CryoProbe. When kneeling down at the magnet bore, your body posture is not suited to lift the heavy CryoProbe (~12 kg) on your own. For two people it is very easy. Take care not to injure your back!

Magnetic stray field

When working within the 0.5 mT stray field of the magnet, all magnetic parts and tools must be avoided or handled with great care.

CAUTION:

Deposit mechanical watches and cards with a magnetic stripe (e.g. credit cards) outside the 0.5 mT range of the magnet.

Safety of CryoProbe equipment 1.5

CAUTION:

- Do not bend the CryoProbe.

  Do not hold the CryoProbe at its upper tube, always carry it at its body.

- Do not open the CryoProbe.

  There are no user-serviceable parts inside. A CryoProbe cannot be sealed or reassembled without special equipment. Even undoing some screws can destroy factory settings and will in general render the CryoProbe unusable.

- Never force a CryoCoupler into position.
- Do not obstruct the operation of the safety-valves on the top and front faces of the CryoProbe body.
- Do not move a cryogenically cold device.
- Do not try to fix a leak on a cold part because cracking of frozen o-rings, valves etc. may occur.
- Excessive RF power can destroy the CryoProbe or the HPPR CRP. Obey the limitations given on the specific ‘LIMITATIONS - WARNINGS’ sheet. See also ***"RF power limitations" on page 58***.

First aid 1.6

If cold helium gas comes in contact with eyes or skin, immediately flood the affected area with cold or tepid water.

Introduction 2

BRUKER CryoProbes™ offer a dramatic increase in signal-to-noise ratio (S/N) by reducing the operating temperature of the NMR coil assembly and the preamplifier. Their spectroscopic handling is very similar to a conventional probe. While the sample temperature is stabilized at a user-defined value around room temperature, the NMR coil assembly - located a few millimeters from the sample - is cooled with cryogenic helium gas. An automatic closed-cycle cooling system controls all functions and guarantees excellent stability during short and long-term experiments. As a result, the system is easy to handle. CryoProbes open new fields for NMR applications e.g. where low sample concentration or long measurement time are critical.

How to use this manual 2.1

This *CryoProbe System User Manual* describes the CryoProbe System and its daily operation.

If you have a specific question, use

- ***"Contents" on page 3***, or
- ***"Frequently asked questions" on page 93***“

to locate the answer.

Novice users of a CryoProbe System should read ***"Safety" on page 7***.

Further information can be found in the manuals listed in ***"Related documents" on page 89***.

CryoProbe System overview 2.2

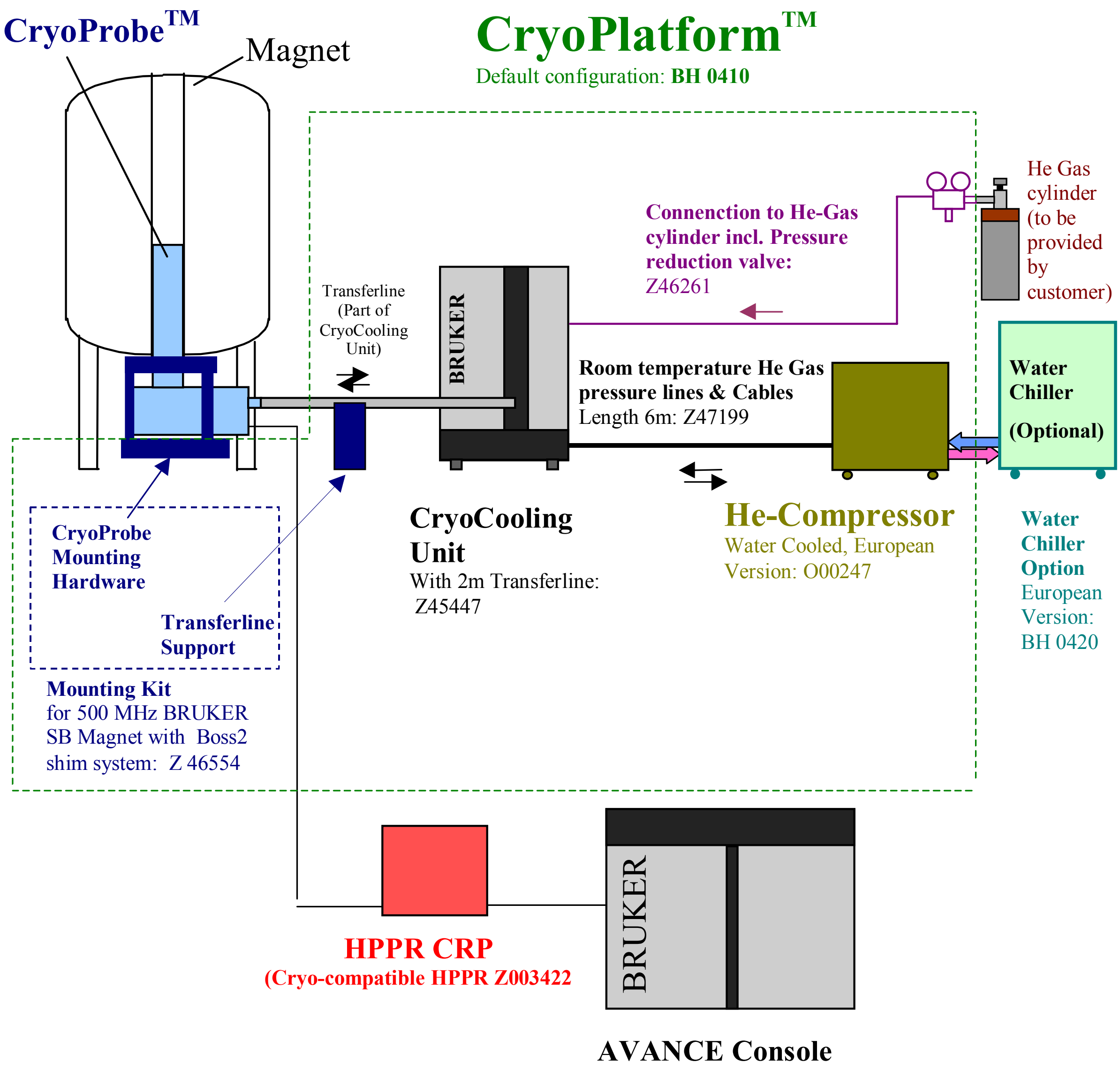
A CryoProbe System consists of several subunits: CryoProbe, CryoPlatform, cryo-compatible HPPR CRP, He steel-cylinder, and optional water chiller (***Figure 2.1.***).

The term ‘CryoPlatform’ summarizes the parts required to operate a CryoProbe such as the CryoCooling Unit, the He Compressor, the Mounting Hardware at the magnet etc. It is compatible with all BRUKER CryoProbes and only one per spectrometer is needed.

Conventions 2.3

SMALL CAPS ITALIC	setting of a hardware switch or button
`Courier small`	contents of a file

Figure 2.1. The CryoProbe™ System



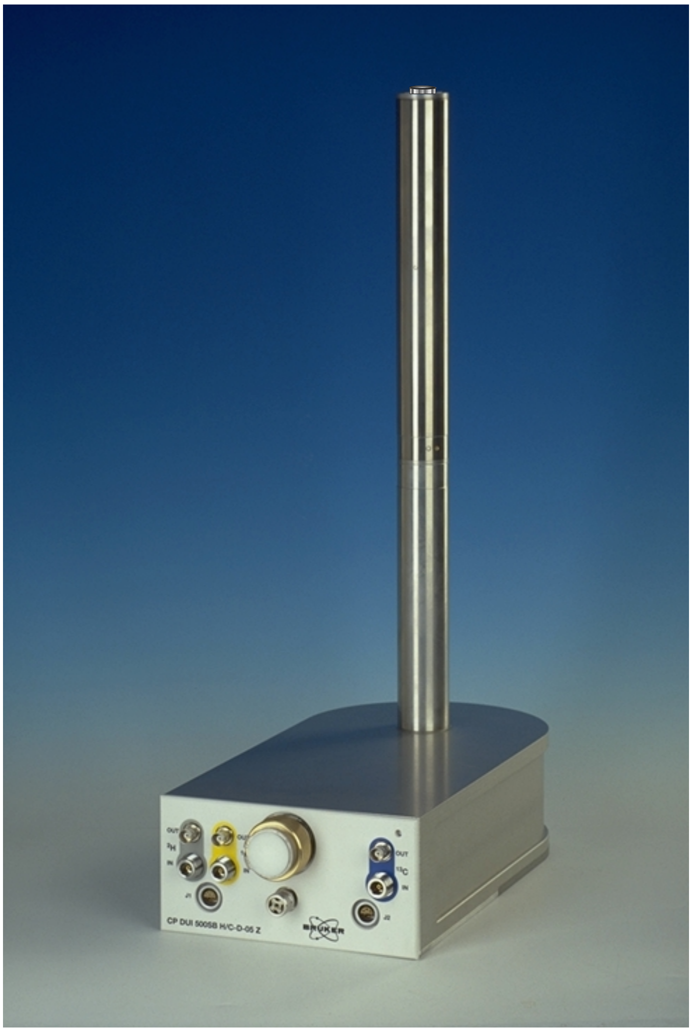
`Courier small italic`	system response
`Courier`	file or directory name
`Courier bold`	Unix™ or Windows™ NT keyboard command
`Courier italic bold`	BRUKER NMR Suite keyboard command
Times bold	Unix or Windows NT object clicked with the mouse
Times italic bold	BRUKER NMR Suite object clicked with the mouse
Times italic	host name, user name etc.
< >	place holder

System description 3

CryoProbe 3.1

The CryoProbe™ is an NMR probe with the essential parts of the RF preamplifier integrated. Both the NMR coil assembly and the CryoPreamp are cooled by cryogenic helium gas (He) to achieve an extremely efficient operation of the NMR coil assembly and to significantly reduce thermal noise. Thus, the overall signal-to-noise ratio is dramatically enhanced.

Figure 3.1. A CryoProbe™



Where applicable, the CryoPreamps, transmit/receive switches, RF filters for the receiver pathways, and control circuits are built into the CryoProbe body.

The CryoProbe is connected to the CryoPlatform with the standardized CryoCoupler and He Transferline for cooling as well as with several sensor cables. It requires special interface cables for the z-gradient and for the VT unit.

Technical data

Dimensions	190 × 365 × 95 mm [w × d × h] (body only, excluding connectors) length including front connectors: 383 mm
Overall height	578 mm (500 MHz), 628 mm (600 MHz)
Weight	~12 kg

Sample temperature control 3.1.1

The sample is kept at a temperature in a range specified on the 'LIMITATIONS - WARNINGS' sheet which comes together with each individual probe. Its temperature is controlled and regulated in the usual way with a continuous gas flow. There is a vacuum insulation between sample and cryogenically cooled NMR coil assembly. Still, due to thermal radiation a slight steady cooling acts on the sample from the surrounding CryoProbe cavity. This cooling, however, is easily compensated by the regular VT gas flow.

Note the ***"Sample temperature control" on page 55***.

Reproducibility 3.1.2

The performance characteristics of a CryoProbe do not change significantly with time. Warm-up/cool-down cycles are of little effect on shim values, tuning & matching, pulse angles, lineshape, or sensitivity. However, some fine tuning of parameters might be necessary comparable to what is known from conventional probes.

Differences to a conventional probe 3.1.3

Although the CryoProbe is in a way a regular high-resolution NMR probe, many of its systems are unique and have not been used in an NMR system before. All users of a CryoProbe System must be aware of its differences to a conventional probe to ensure appropriate use and maximum performance.

Mounting

The CryoProbe includes fragile parts and needs very careful handling. Since several electronic components (e.g. preamplifier) and parts of the cooling system are included in the CryoProbe, the weight is substantially increased as compared to conventional probes. A special Mounting Hardware is attached directly to the magnet bottom to carry the CryoProbe. **Two people** are needed for the actual lifting of the heavy CryoProbe (~12 kg) into the magnet.

Connections

There are more connectors on the CryoProbe than on a conventional probe due to the built-in preamplifiers and the necessary control circuits (see **Figure 3.1.**).

Sample handling, tuning, shimming

There are no differences to a conventional probe with regard to sample handling, sample lift, and sample spinning. Samples with a diameter smaller than specified can be used like in conventional probes.

A CryoProbe is tuned and matched like a conventional probe with actuators from below. Before the usual ***wobb*** feature of XWIN-NMR can be used, the appropriate RF pathway must be enabled with the command ***crpwobb***. CryoProbes must not be tuned or matched when warm!

Established shimming strategies are still valid and no unusual shim settings are to be expected.

RF power

RF peak and average power must not exceed certain limits. Due to the high efficiency of the CryoProbe, the limits are lower than for conventional probes. Power levels for all kinds of RF pulses (hard, soft, shaped, CPD, spin-lock...) may be significantly lower than on conventional probes. In particular, the decoupling power required for a given RF field strength is usually much smaller than in a conventional probe. See the specific LIMITATIONS - WARNINGS sheet, which is delivered with each CryoProbe.

Length and power of RF pulses are calibrated as usual.

Sample temperature and minimum required VT gas flow

The sample is subjected to a rather high flow of VT gas specified on the 'LIMITATIONS - WARNINGS' sheet which comes together with each individual probe. The VT gas flow must be maintained at all times. This compensates for the slight cooling due to thermal radiation from the surrounding CryoProbe.

Subsequent modifications

No modifications, upgrades, or frequency changes are possible for an existing CryoProbe.

Acoustic noise

The CryoPlatform includes several mechanical pumps and compressors which are sources of acoustic noise. A characteristic periodic hiss of the Gifford-McMahon cooler is clearly audible from the CryoCooling Unit and the Flexlines despite their acoustic insulation. The He Compressor and an optional water chiller are also noisy but can conveniently be located in an adjacent room.

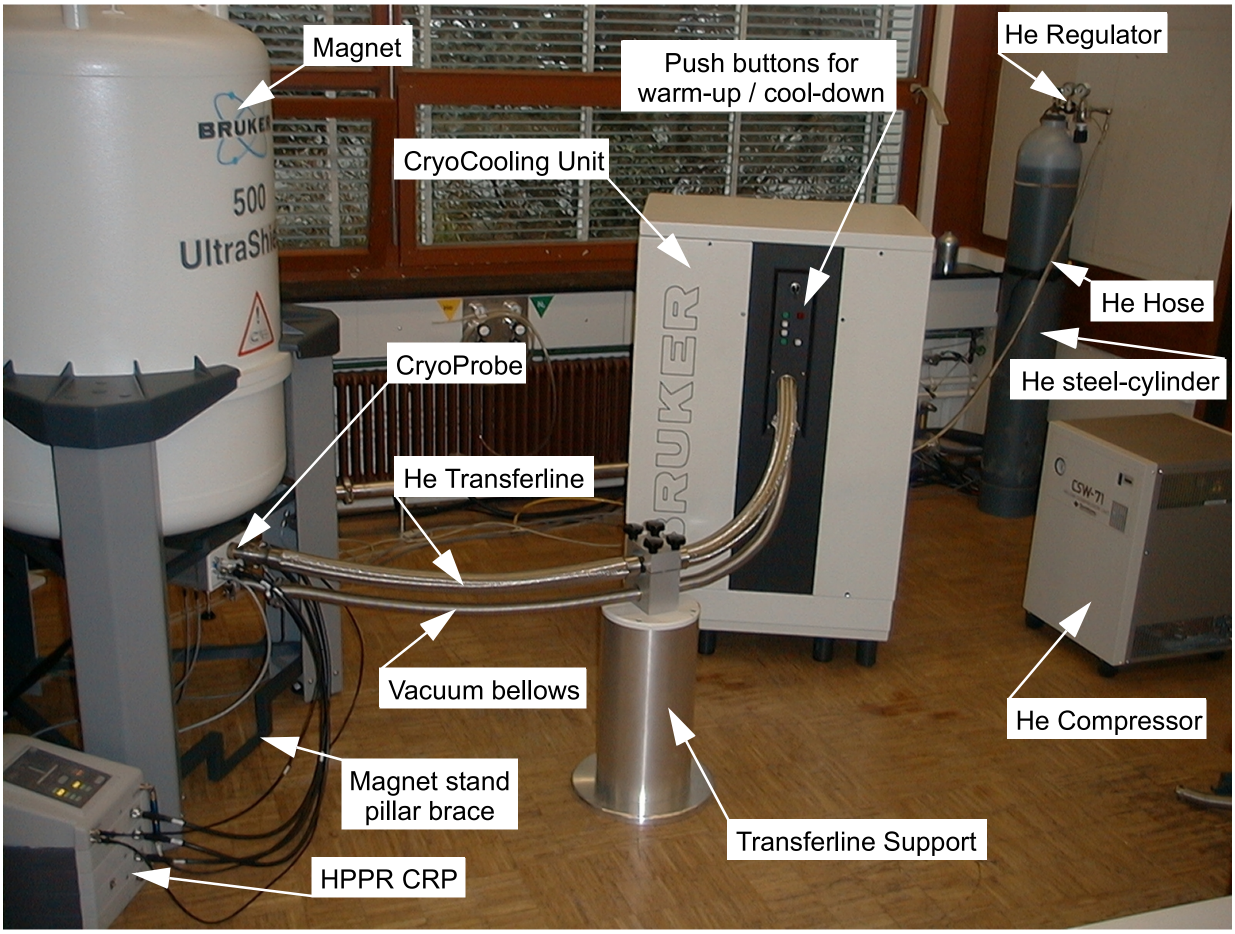
CryoPlatform 3.2

The CryoPlatform™ is required once per spectrometer and supplies the entire infrastructure for the operation of CryoProbes, i.e. the cooling and all control functions. It is a push-button system which performs all operations needed for an entirely automatic cool-down, cold operation, and warm-up of the probe. Although capable of stand-alone operation, the CryoPlatform is fully integrated into the AVANCE spectrometer system.

A CryoPlatform (***Figure 3.2.***) consists of CryoProbe Mounting Hardware at the magnet, a CryoCooling Unit with an integrated He Transferline to the CryoProbe, a Transferline Support, a separate He Compressor with water- or air-cooling, Flexlines between He Compressor and CryoCooling Unit, a He Regulator on a He steel-cylinder, an interface cable for the VT unit, and an optional magnet stand modification for certain magnet types.

Not considered part of the 'CryoPlatform' are the CryoProbe, the cryo-compatible HPPR CRP, the He steel-cylinder, the Gradient Filter Box, and an optional water chiller.

Figure 3.2. A CryoPlatform™



Mounting Hardware 3.2.1

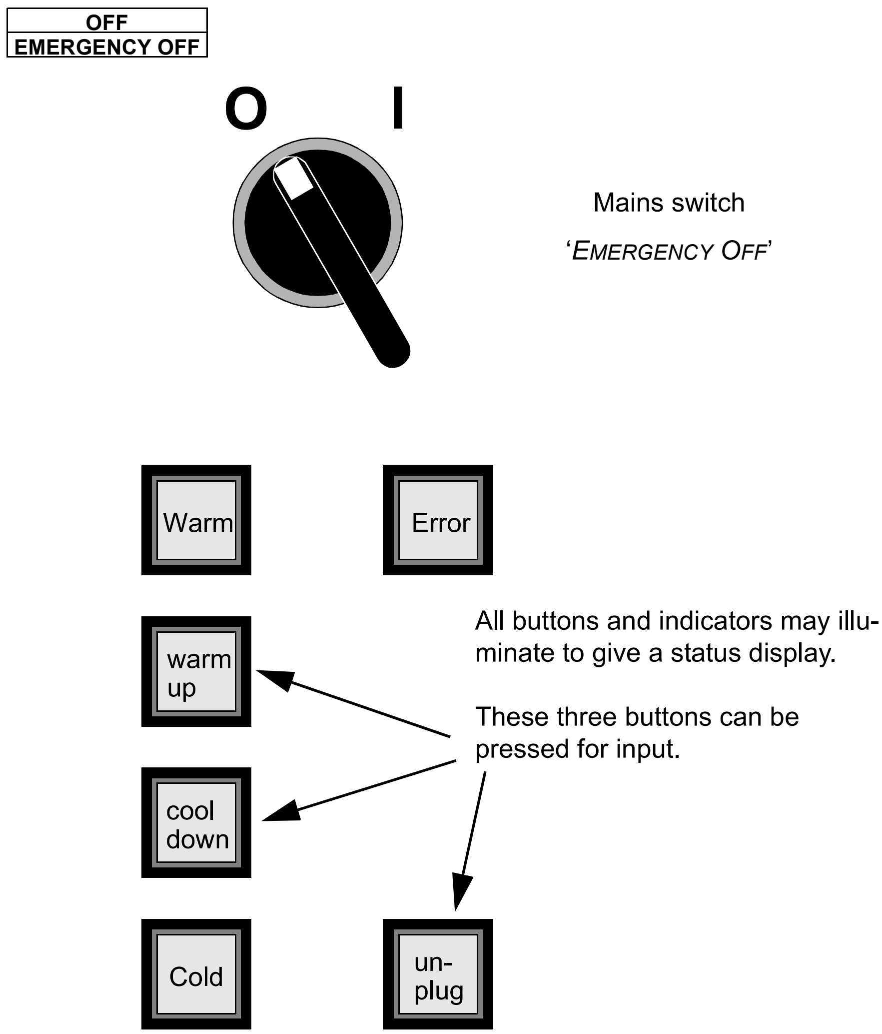
A special fixture must be mounted to the lower RT flange of the magnet bore to carry the weight of the CryoProbe. The Mounting Hardware is attached to the magnet flange with an interface plate which replaces the lower shim system attachment ring. When not used for CryoProbe mounting, this plate does not interfere with conventional probes.

CryoCooling Unit 3.2.2

The most prominent part of the CryoPlatform is the CryoCooling Unit. Inside, a so-called ‘Coldhead’ expands compressed He and thereby cools it to cryogenic temperatures. Cold He is then circulated through the CryoProbe via an insulated He Transferline. Vacuum pumps maintain insulation of the CryoProbe and the CryoCooler. All operations are supervised by the built-in CryoController unit.

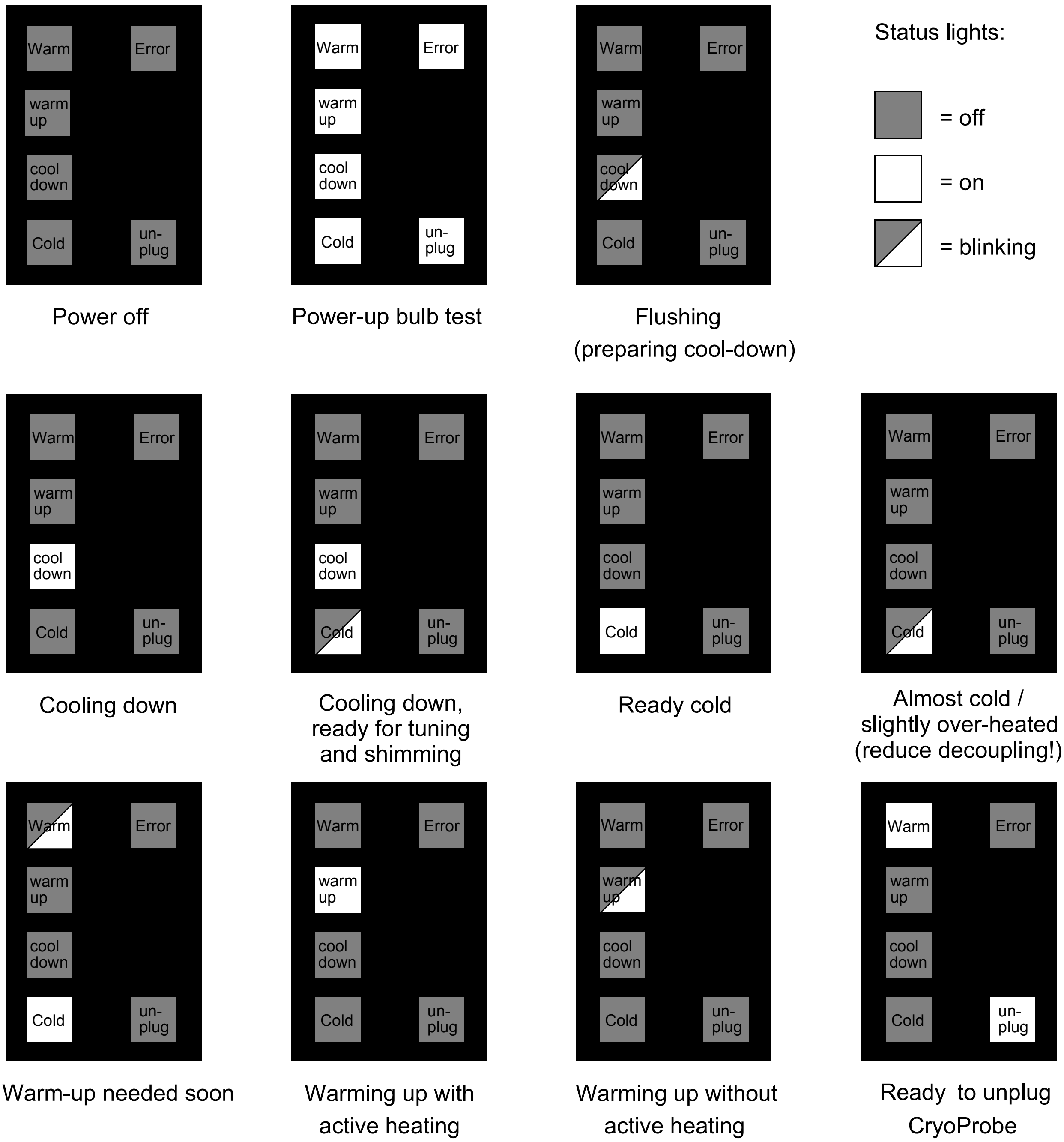
Controls on the CryoCooling Unit

Figure 3.3. Control indicators on the CryoCooling Unit front



Status display by the control buttons

Figure 3.4. Status display by the control buttons



After pressing *UNPLUG*, any sensor cable can be removed from the CryoProbe during ~1 min without causing an error.

ERROR reset

ERROR may light up in any of the above situations (***Figure 3.4.***) in addition to the lights displayed. To reset the *ERROR* on the CryoCooling Unit front panel, press

COOL DOWN if the system is already cold or cooling down. If the CryoProbe System is warm or warming up, press WARM UP to reset an ERROR. In case of an ERROR please consult also the chapter **"Troubleshooting" on page 71**.

LED test

All LEDs light up automatically after the CryoCooling Unit main switch is turned on.

Technical data: CryoCooling Unit

Dimensions	800 × 720 × 1300 mm [w × d × h]
Weight	400 kg
Electricity	AC 230 V (1 phase) 50/60 Hz, 500 W average, 1500 W peak, required external fuse upstream: T 10 A (T = time-lag fuse).
Acoustic noise	max. 61 dB(A) 2 m distant (CryoCooling Unit only)

The CryoCooling Unit complies with overvoltage category II and its degree of protection is IP20.

Coldhead

The actual cooling device inside the CryoCooling Unit is the Coldhead. It is a two-stage Gifford-McMahon cycle cryo-refrigerator that uses compressed He for continuous closed-cycle refrigeration.

CryoController

All processes are performed and supervised autonomously by the CryoController which is located inside the CryoCooling Unit. Thus, the CryoCooling Unit does not rely on any external computer or software for its operation. However, an RS232 interface is present that can be connected to a dedicated PC terminal, running special software. The software functions available on the external computer are solely for the purpose of remote control and monitoring.

He Compressor *3.2.3*

The He Compressor unit provides compressed He and electrical power to the Coldhead inside the CryoCooling Unit. It consists of a compressor pump, a heat exchanger, and an Adsorber.

CAUTION: Never use the He Compressor with a gas other than high purity He. It would be contaminated and the system made unusable with a high cost for cleaning and repair.

Technical data

(see also the He Compressor technical manual for an extended list of specifications, it is delivered with the CryoPlatform)

Dimensions		(w × d × h in mm)
P/N O00245	(3 × 200 V AC, air-cooled)	550 × 550 × 885
P/N O00246	(3 × 200 V AC, water-cooled)	450 × 500 × 684
P/N O00247	(3 × 400 V AC, water-cooled)	450 × 500 × 684

Weight (approx.)

P/N O00245	(3 × 200 V AC, air-cooled)	140 kg
P/N O00246	(3 × 200 V AC, water-cooled)	120 kg
P/N O00247	(3 × 400 V AC, water-cooled)	125 kg

Ambient operating temperature 5 to 28°C

Air humidity (relative) max. 90%

Electricity

line voltage (± 5%)	P/N O00245	AC 200 V, 50/60 Hz, 3 phase,
	P/N O00246	AC 200 V, 50/60 Hz, 3 phase,
	P/N O00247	AC 380 - 415 V @ 50 Hz, 3 phase, AC 460 - 480 V @ 60 Hz, 3 phase.
power consumption	maximum	8.3 kW
	steady state	7.5 kW
internal fuses	P/N O00245	29 A for mains, automatic 3 × 2 A for control circuit 3 × 3 A for fan 3 × 2 A for Coldhead
	P/N O00246	29 A for mains, automatic 3 × 5 A for control circuit and Coldhead
	P/N O00247	13 A for mains, automatic 3 × 3 A for control circuit and Coldhead

Helium gas pressure	static		17.5 bar	(254 psi)
	operating	high	21 bar	(300 psi)
		low	6 bar	(85 psi)
Pressure relief valve setting			27.3 bar	(390 psi)

Cooling water requirement (P/N O00246 and P/N O00247 only)

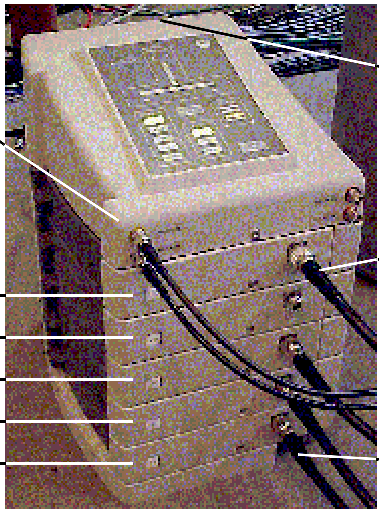
water flow rate:	4 - 10 L/min (240 - 600 L/h)
recommended flow rate:	7 L/min (420 L/h)
typical cooling power:	6.1/7.5 kW at 50/60 Hz
water temperature (at He Compressor inlet):	4 - 28°C
recommended water temperature:	15°C

Acoustic noise ca. 65 dB(A) 2 m distant

Cryo-compatible preamplifier assembly 'HPPR CRP' 3.3

Although a CryoProbe has its own set of cold preamplifiers built-in, some HPPR functions such as RF filters in the transmission path, probe tuning, and selection of the received signal must be handled externally by a modified HPPR CRP assembly (***Figure 3.5.***). Besides being suitable for the operation with CryoProbes, this HPPR CRP can be used with all conventional probes. For technical information on the HPPR CRP and its wiring, see the *CRP RF Electronics Technical Manual*.

Figure 3.5. Cryo-compatible preamplifier assembly 'HPPR CRP'



Technical data

Data for TXI configuration (cover and 5 HPPR modules), an X-BB module is included by default:

Dimensions (w × d × h in mm)	260 × 350 × 420
Weight	25 kg

Water chiller (optional) 3.4

The He Compressor generates about 7.5 kW of heat. If water-cooling has been chosen but no cooling water is available, a water chiller should be used. Fresh water is not recommended because of cost, purity, and environment protection.

Technical data

The following example is valid for the water chiller available from BRUKER in Europe. It is of the 'split type', i.e. it consists of two units: a main unit indoors with pumps for a primary and a secondary cycle linked by a heat exchanger, and a radiator outdoors that disposes the heat from the secondary cycle to the atmosphere. Local suppliers in other parts of the world are likely to offer similar devices.

Dimensions		(w × d × h in mm)
main cabinet (indoors)		690 × 920 × 1490
radiator (outdoors)		1895 × 470 × 832
Weight		
main cabinet (indoors)	(120 kg empty)	250 kg
radiator (outdoors)	(94 kg empty)	120 kg

Electricity	
	AC 400 V, 50 Hz, 3 phase 3.6 kW, 8 A average, 51 A peak
Air temperature for outdoor radiator	-10 to +40°C
Acoustic noise of outdoor radiator 10 m distant	41 dB(A)

Principle of operation *3.5*

Gifford-McMahon cycle refrigerator *3.5.1*

In ***Figure 3.6.***, a Gifford-McMahon cycle refrigerator is shown schematically. It consists of a closed cylinder and a displacer that covers about three quarters of the cylinder volume. The displacer is moved up and down, thus the two spaces above and below the displacer are varied from zero to maximum but the total volume remains constant. A seal on the displacer prevents leakage from one volume to the other.

The two spaces are connected through a regenerative heat-exchanger and linked to a gas supply. The gas supply consists of inlet and outlet valves, a gas compressor, and high and low pressure reservoirs. Its valves are synchronized with the position of the displacer. A heat exchanger downstream of the gas compressor cools the gas to ambient temperature before it enters the Gifford-McMahon cycle refrigerator.

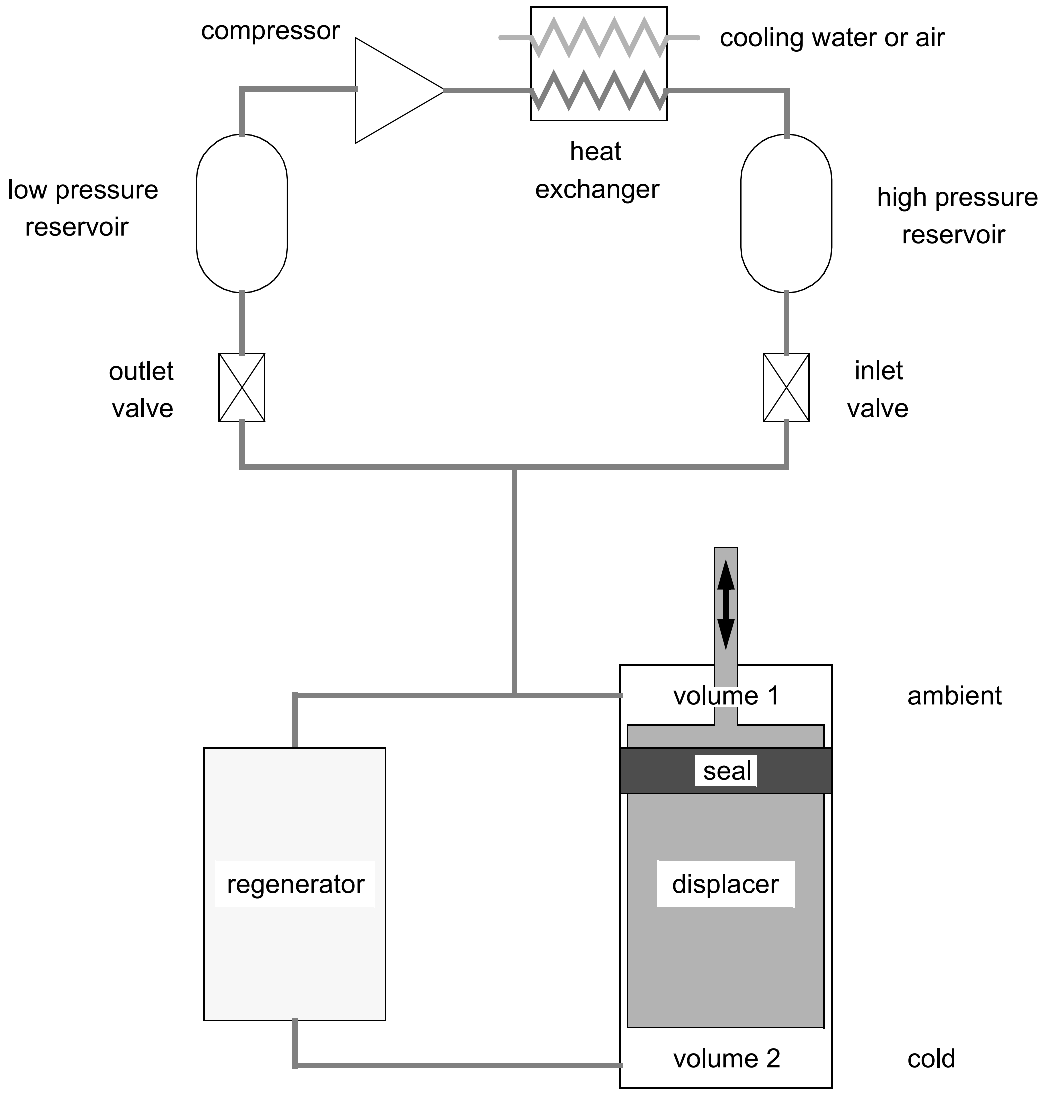
The pressure above and below the displacer is always the same except for small pressure drops across the regenerator when gas is flowing through it. Virtually no work is required to move the displacer in the cylinder. No work is done on the gas and the gas does no work on the displacer. Pressure in the system is increased or decreased only by operation of the inlet or outlet valves.

Inside the regenerator, finely divided metallic material pre-cools the gas when passing downwards to the cold space and heats the gas when returning from the cold space.

The refrigerator operates as follows:

1. Pressure buildup
   With the displacer at the bottom of the cylinder and the outlet valve closed, the inlet valve is opened and the pressure in the system increases. High pressure gas enters through the inlet valve to fill the regenerator and the space above the displacer, volume 1.
2. Intake stroke
   With the inlet valve still open, the displacer is moved from the bottom of the cylinder to the top. This displaces high pressure gas from the space above the displacer, volume 1, through the regenerator to the space below the displacer, volume 2. In passing through the regenerator, the gas cools down and contracts. Additional gas enters the system to maintain the maximum cycle pressure.
3. Pressure release and expansion
   With the displacer at the top of the cylinder, the inlet valve closes and the outlet valve opens to the low pressure reservoir. Gas escapes and the pressure decreases, causing a drop in the gas temperature. The temperature decrease

Figure 3.6. Gifford-McMahon cycle refrigerator



in the bottom cylinder space, volume 2, is the useful refrigeration process of the cycle.

4. Exhaust stroke
   With the outlet valve open, the displacer moves from the top to the bottom of the cylinder, displacing fluid from volume 2, below, to volume 1, above the displacer. As it flows through the regenerator, the fluid is heated by the matrix to near ambient temperature. This process cools the matrix ready for the gas entering in the next cycle.

Software *3.6*

The CryoController can be interfaced to a computer running Windows™ 95/NT4 or later. Monitoring software is delivered with the CryoProbe System. Furthermore, the XWIN-NMR AU programs ***crpon***, ***crpoff***, ***crpwobb***, ***crp2hon***, and ***crp1hxon*** are needed. They are part of XWIN-NMR 2.6 and later. For XWIN-NMR 2.0 or later, an add-on package is available from *ftp.bruker.de*: `/pub/nmr/xwinnmr/utilities/crponoff.tar.gz`.

Standard procedures

4

Most operations are handled in full automation by the CryoController except for those requiring mechanical actions like mounting and removal of the CryoProbe at the magnet or tuning and matching.

LIMITATIONS - WARNINGS sheet 4.1

Each CryoProbe is delivered with a specific ‘LIMITATIONS -WARNINGS’ sheet. Consult this document before

- preparing and inserting a sample,
- setting up the sample temperature and VT gas flow,
- applying any RF power to the CryoProbe (including lock),

to prevent damage.

Handling 4.2

CAUTION: The most **fragile parts** of a CryoProbe are:

- sample **cavity**
  Avoid fast dropping of samples with the sample lift.
  Do not introduce any objects into the cavity (not even a soft cotton bud!).
  Due to manufacturing processes it is not possible to clean the sample cavity with strong solvents.
  See ***"Cleaning the sample cavity" on page 67*** for further information.
- probe **tube** and its joint to the body
  Do not hold the CryoProbe at its tube. Support and carry the CryoProbe only at its body. Do not bend the tube.
- **CryoCoupler**
  Do not force the CryoCoupler into position.
- **connectors**
  Do not bend the connectors for vacuum, RF, sensors, or gas.

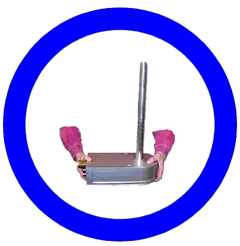


Figure 4.1. Carry a CryoProbe only at its body!

Figure 4.2. Never hold a CryoProbe at its tube!

CAUTION: Do not heat or cool the CryoProbe housing from the outside (e.g. do not try to speed up the evacuation process by heating the CryoProbe with a heat gun).

Mounting *4.3*

Insertion and removal of the CryoProbe can be easily accomplished by the spectrometer operator with the help of a second person.

WARNING: Do not attempt to insert or remove the heavy CryoProbe (~12 kg) at the magnet without the help of a **second person**. Because your body's posture is unfavorable when kneeling down below a magnet, you may easily injure your back when lifting the CryoProbe on your own!

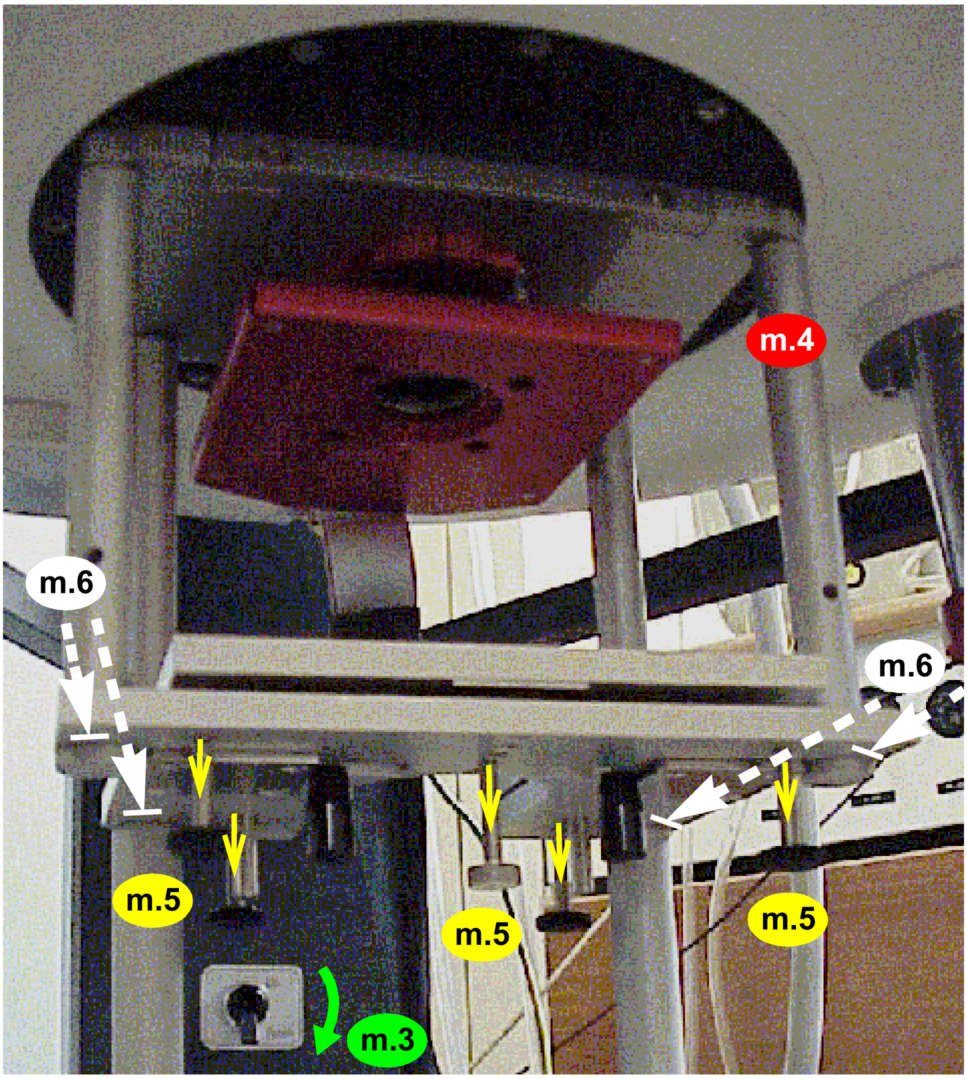
IMPORTANT: Read ***"Handling" on page 27*** if you're going to handle a CryoProbe for the first time.

NOTE: A QNP pneumatic unit is spatially incompatible with a CryoProbe setup and has to be removed during CryoProbe operation.

Table 4.1. Mount the CryoProbe

step	action
m.1	Terminate any NMR experiment and remove sample and probe.
m.2	Set all RF power levels ***p1*** and ***sp*** to 120 dB (recommended).
m.3	**Lower** the magnet air suspension.
m.4	Inspect the Mounting Hardware: All **four guiding rails** must be in place and fixed tightly (two long vertical bars in the rear and two short ones in front).
m.5	Prepare the CryoProbe support plate: all **five screws** should be moved almost out of the plate.
m.6	Verify without a CryoProbe that the support plate will **snap** into the notches on the rails (***Figure 4.3.***). Remove it afterwards again.

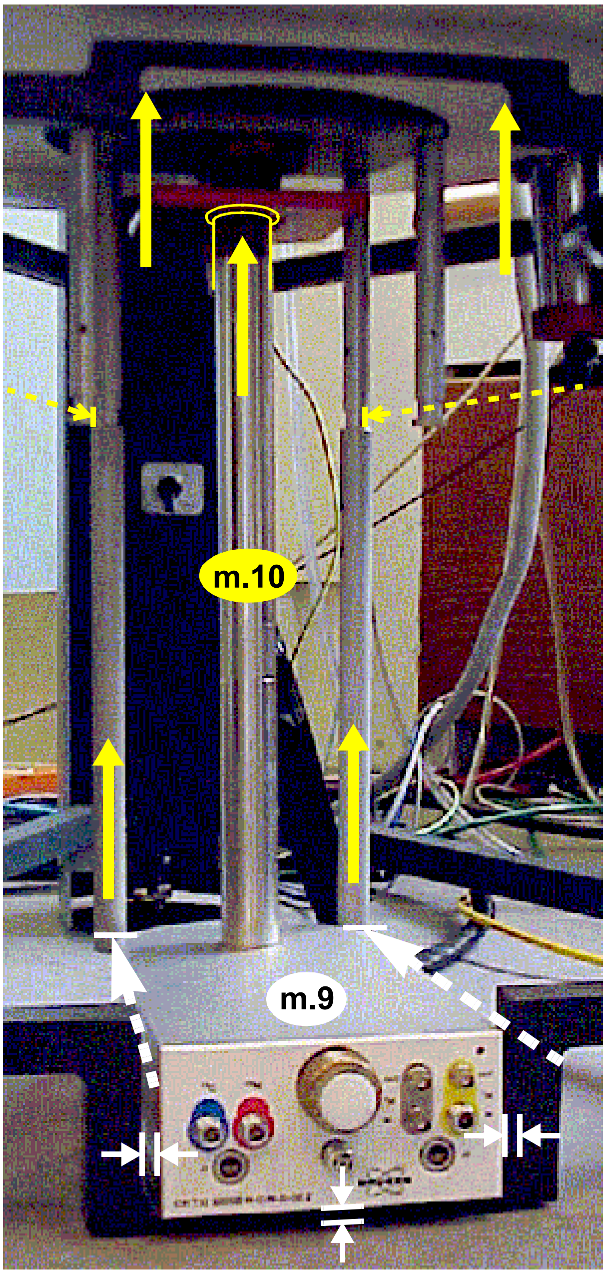
Figure 4.3. Preparing the Mounting Hardware



m.7	Check the **indicator** in the upper right corner of the CryoProbe front. It must not stick out but flush with the front plate. If it sticks out, a problem with the vacuum insulation may have occurred - contact BRUKER! Do not try to move the screw, neither in/out nor by rotation.
m.8	Carry the CryoProbe at its bottom when moving it from its storage place to the magnet. It is fragile but weights about 12 kg. **CAUTION:** Never hold a CryoProbe at its tube!

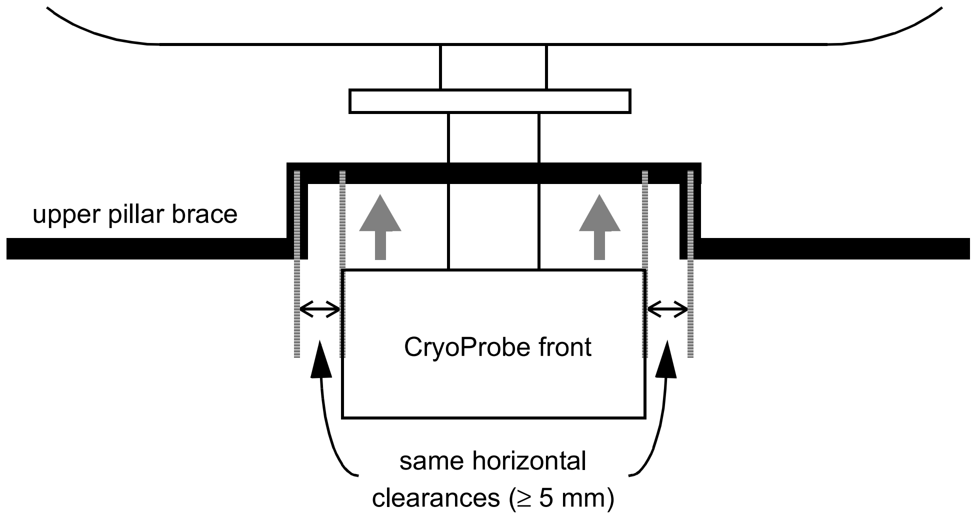
m.9	**WARNING:** While mounting the CryoProbe, the magnet must **not** be suspended on its anti-vibration feet. **WARNING:** Do not attempt to insert or remove the heavy CryoProbe (~12 kg) at the magnet without the help of a **second person**. You may easily injure your back when lifting the CryoProbe on your own! Remove the protective cap from the top of the CryoProbe. Move the CryoProbe carefully from the magnet front to below the bore. Do not let it rub on the floor or on the cranked pillar brace (***Figure 4.4.***). Let its body **touch the two long guiding rails** that extent downward from the Mounting Hardware.
m.10	**Align** the CryoProbe top with the shim system bore. **Push it gently up** into the magnet until the support plate can be snapped into the notched guiding rails. **Snap** the support plate into place. Verify correct seat of all four support points. **IMPORTANT:** While sliding it carefully into the magnet, prevent bending of the CryoProbe tube by keeping it close to the rails - but do not press it against them. Do not use any extra force to push it up, it should slide without resistance.

Figure 4.4. Sliding the CryoProbe into the magnet



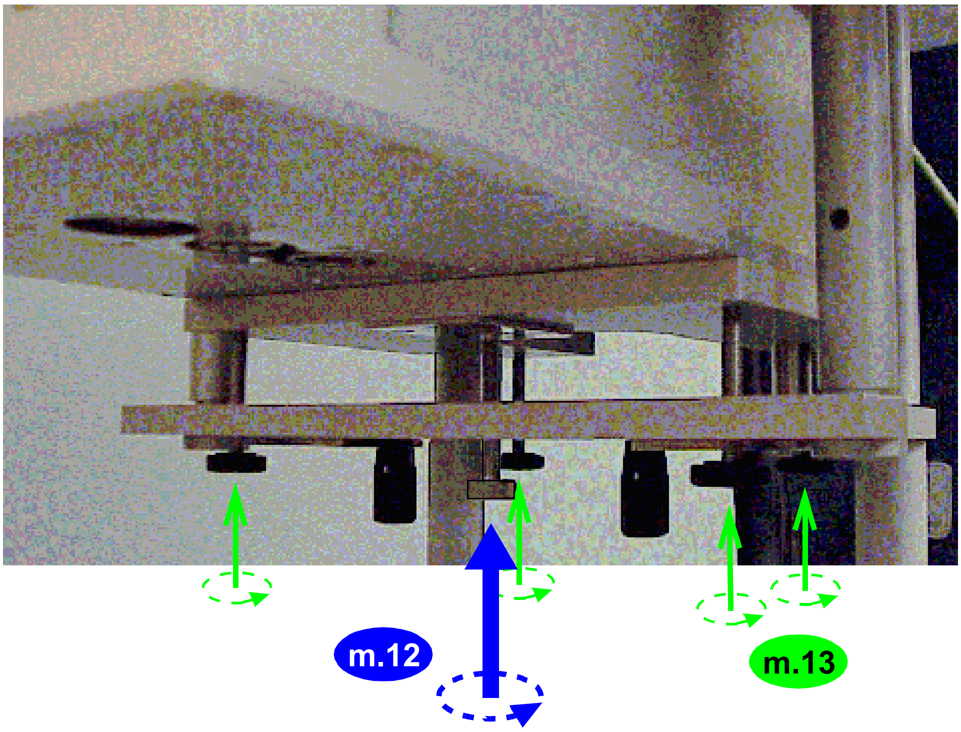
m.11	Check the **orientation** of the CryoProbe in the magnet: Its front must be able to meet the He Transferline. **IMPORTANT:** If cranked pillar braces are present in the magnet stand, the CryoProbe must be centered in their gap at all times (see ***Figure 4.5.***).

Figure 4.5. CryoProbe and optional cranked pillar braces



m.12	Push the CryoProbe up gently by **turning the screw** below its center until the CryoProbe body almost touches the shim system (gap ≈ 0.5 mm). Feel for the end position, but upon reaching it, do not use great force to tighten the screw (***Figure 4.6.***).
m.13	Turn the other **four screws** on the support plate in an alternate fashion such that they support the CryoProbe gently. Take care not to tilt its body by forcing one screw more that the other screws.
m.14	**Lift** the magnet. **CAUTION:** If the CryoProbe is not correctly aligned, its body might touch the optional horizontal pillar braces when the magnet is lifted by about 10 mm onto its anti-vibration stand. The forces involved can easily break the CryoProbe! Before the magnet is lifted, there should be a symmetric **horizontal clearance of** ≥ **5 mm** between the CryoProbe and any fixed obstruction above its body (see ***Figure 4.5.***). Switch on the air suspension of the magnet and watch the CryoProbe body rise. If it is about to be jammed against any fixed part, re-align the CryoProbe or lower the magnet immediately!

Figure 4.6. Final positioning of the CryoProbe



Sample spinning test 4.4

Spinning test

Insert a typical sample into the magnet and check if it can be spun. Remove the sample after the test.

IMPORTANT: No sample must be in the magnet during the CryoProbe cool-down. Insert samples only if the *COLD* button on the CryoCooling Unit is on or at least flashing.

For recommendations on sample tube quality, filling heights, and spinners, refer to the *CryoProbe System User Manual*, ***"Samples and spinners" on page 55***.

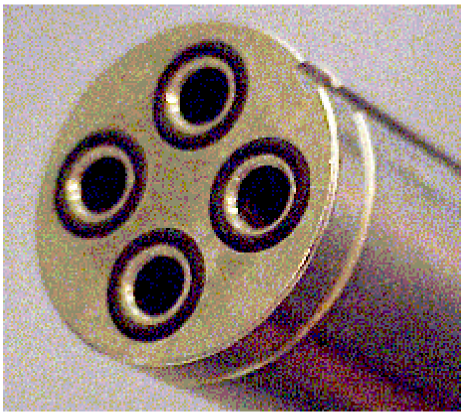
If spinning does not work, see ***"Spinning problems" on page 74***.

Joining the CryoCoupler *4.5*

Table 4.2. Join the CryoCoupler

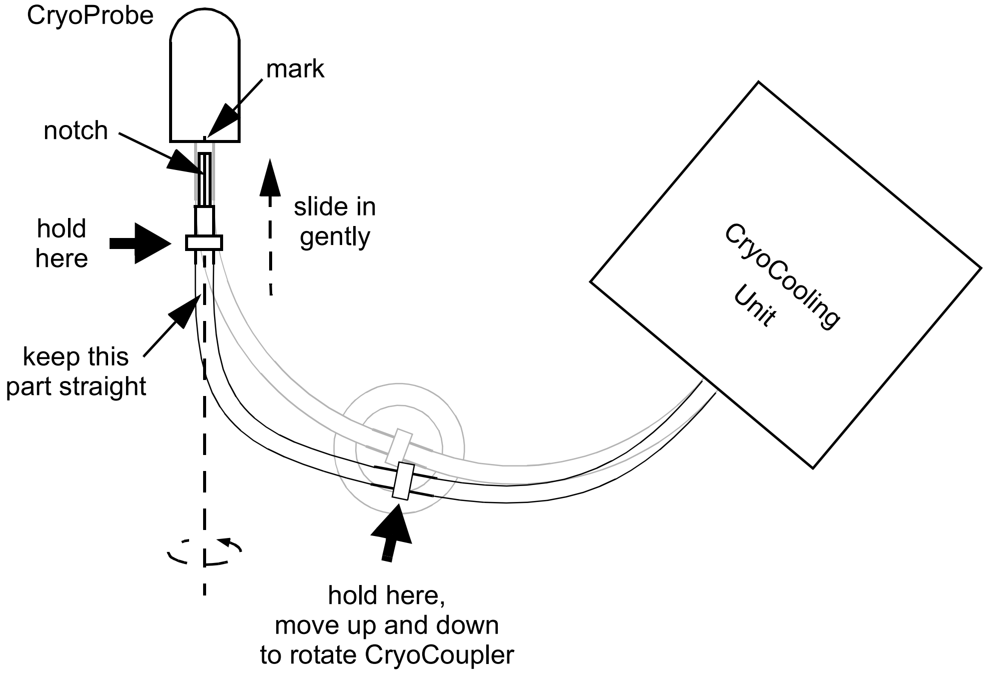
step	action
j.1	**Detach the He Transferline** from the Transferline Support but let it rest on the column.
j.2	Remove the **protective caps** from the CryoCouplers on He Transferline and CryoProbe.
j.3	Check the **four o-rings** on the He Transferline CryoCoupler (***Figure 4.7.***): are they in place, clean and undamaged? If not replace with the o-rings found in the spare parts box delivered with each CryoProbe System. The o-rings are of type viton and size 7.1 by 1.6 mm.

Figure 4.7. O-rings on the CryoCoupler



j.4	Hold the **CryoCoupler** on the He Transferline with **one hand**. Take the **vacuum joint** which is 1 m away from the CryoCoupler into the **other hand** (see ***Figure 4.8.***).

Figure 4.8. Inserting the CryoCoupler



j.5	Align the CryoCoupler such that the **notch** on the He Transferline meets the **bolt** inside the CryoProbe's receptacle. Keep the CryoProbe end of the He Transferline rather **straight**. When **gently moving up and down** the hand that supports the He Transferline, you will feel where the rotational alignment is just right.
j.6	Slide the CryoCoupler **into the CryoProbe**. It should fit smoothly without any significant movement of the suspended magnet. **CAUTION:** If the CryoCoupler gets stuck, do not force it into position. Do not bend the He Transferline to an extreme shape to make it fit - permanent damage on CryoProbe or He Transferline could result! The He Transferline Support may be in the way during the insertion process. If this is the case, remove the CryoCoupler, move the Transferline Support a little aside and repeat the insertion process. When a smooth insertion fails, take the CryoCoupler out and try again. If the insertion still fails, the relative position of magnet and CryoCooling Unit may have changed since the last successful coupling. Then, a corrective twist in the He Transferline is needed, see *Installation Manual: Joining the CryoCoupler.*

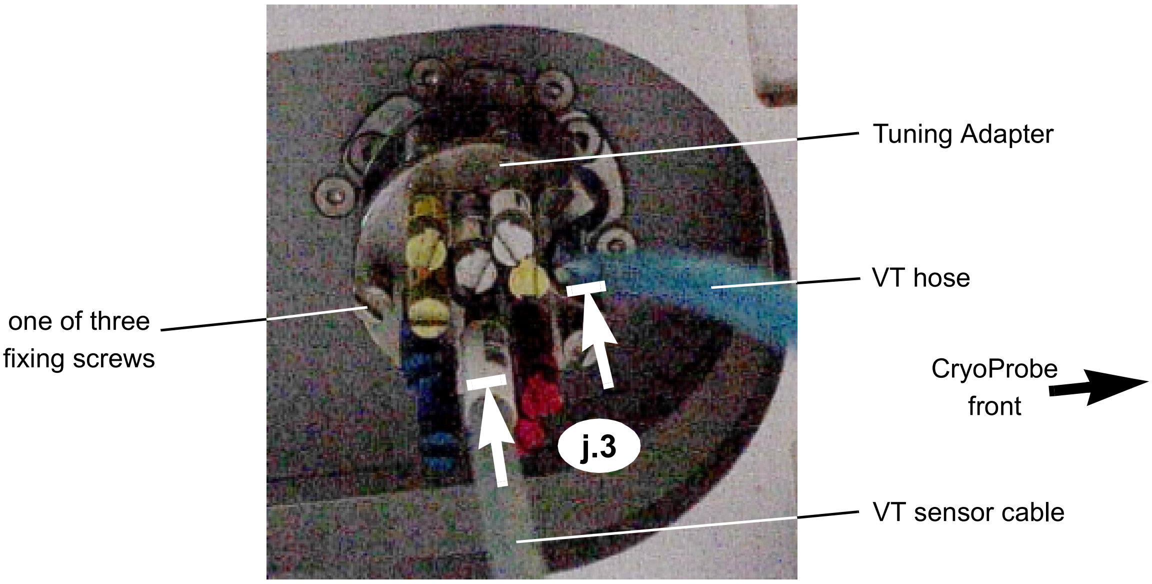
j.7	Use the special open-end **titanium wrench** to fix the CryoCoupler gently to the CryoProbe. Take care not to damage any connectors on the CryoProbe front. Hold the He Transferline end with your other hand to take up the torque applied.
j.8	Position the **Transferline Support** such that the He Transferline falls exactly (to within 1 mm) into its bed.
j.9	**Lock the He Transferline** on the Transferline Support with the four black screws.
j.10	Make sure that the **CryoProbe is still centered** inside the (optional) cranked pillar braces. If not, relocate the Transferline Support accordingly.

Connecting *4.6*

Table 4.3. Connect the CryoProbe

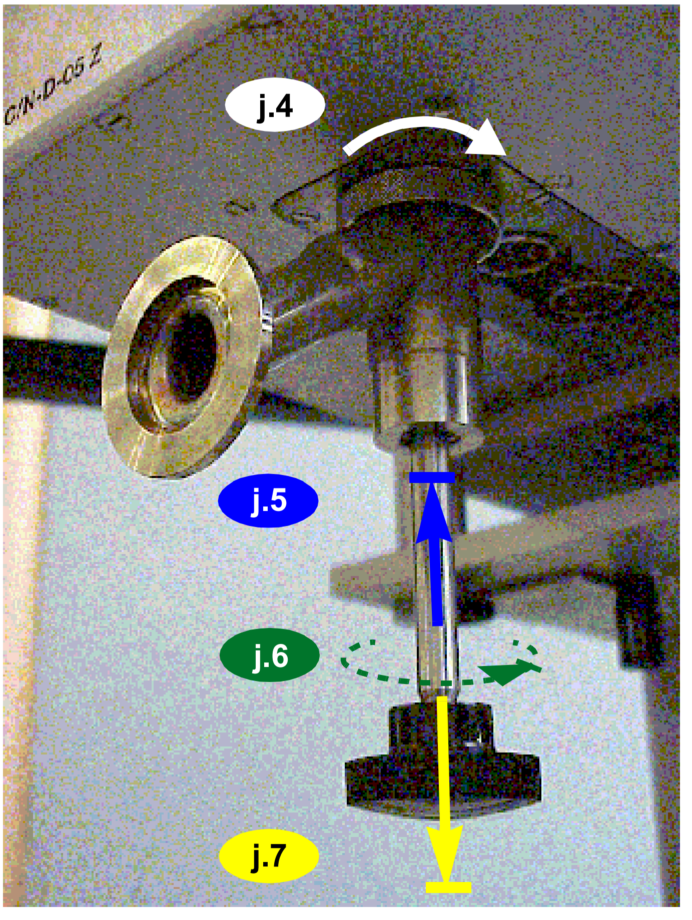
step	action
j.1	Plug the two **sensor cables** from the CryoCooling Unit into the CryoProbe bottom and into J2 on the front, respectively. Connect the CryoPreamp supply cable from the HPPR CRP to J1.
j.2	Connect the **VT sensor cable** and the **VT gas hose** to the probe bottom. Check if all connections for the VT Interface Box are properly made. At the VT unit, e.g. B-VT3000, the Pt100 sensor must be selected. The reading in `edte` should indicate **room temperature**.
j.3	Attach the **Tuning Adapter** (***Figure 4.9.***) to the CryoProbe bottom. Use the blue Tuning Tool to lock its mounting screws. **NOTE:** There are specific Tuning Adapters for each type of CryoProbe.

Figure 4.9. Connecting VT sensor cable and VT hose (bottom view)



j.4	Mount the **Vacuum Adapter** to the CryoProbe bottom such that it points to the front. Connect the **vacuum tube**.
j.5	**Push in** the actuator screw of the Vacuum Adapter (black handle) in as far as it will go (***Figure 4.10.***).
j.6	**Turn** the actuator screw **clockwise** as seen from the bottom. Its thread must grip into the thread of the CryoProbe's vacuum plug. Continue gently until the stop is reached but do not force it into the stop.

Figure 4.10. Open the CryoProbe vacuum plug



j.7	**Draw out** the CryoProbe's actuator screw by at least 5 cm (2 inch) until a stop is reached, thereby taking the vacuum plug out of the Cryo-Probe. **NOTE:** The vacuum system will be activated later. If you doubt that the vacuum plug was fully taken out, you may unmount the Vacuum Adaptor from the CryoProbe and the vacuum tube for a visual inspection. Contact to air will not spoil the CryoProbe's vacuum chamber.
j.8	Connect the **remaining RF and gradient cables** (for details, see *Cryo-Probe RF Electronics Technical Manual*).
j.9	Regulate the **VT gas flow rate** to (at least) the value specified on the LIMITATIONS - WARNINGS sheet.
j.10	Set the **VT heater power limit** as given on the LIMITATIONS - WARNINGS sheet.

j.11	Switch the **VT heater** power *ON*.

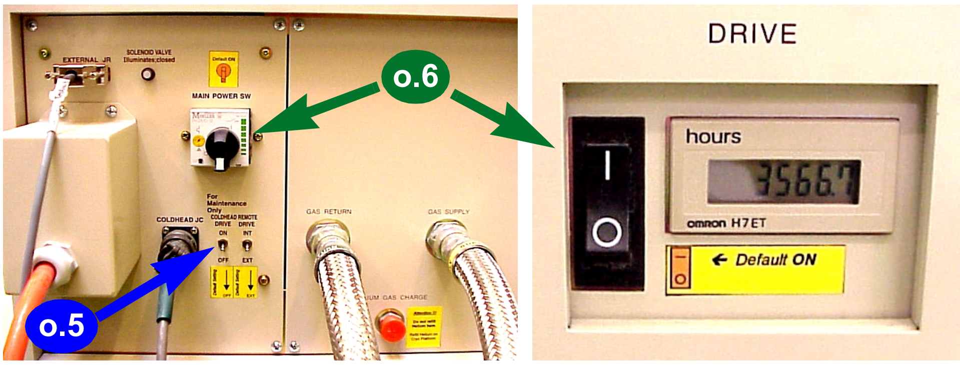
Power-on *4.7*

If the CryoProbe System is *OFF* or has not been used immediately before, go through the following steps to make sure that all components are *ON* and ready.

Table 4.4. Power-on

step	action
o.1	All units of the CryoProbe System must be set up correctly.
o.2	Start the **cooling water** supply to the He Compressor (water-cooled He Compressor only).
o.3	The He steel-cylinder main valve must be open and the primary pressure gauge on the He steel-cylinder should read **40-200 bar**.
o.4	The secondary pressure gauge should read **22-25 bar**, adjust the He Regulator accordingly. Initially, the secondary pressure may rise slowly but only during the first few minutes. Check the pressure after about 20 min.
o.5	At the **He Compressor** front, ***DRIVE* must be *OFF*** in the beginning. On the backside, *COLDHEAD DRIVE* must always be *OFF* and *REMOTE DRIVE* in *EXT* position. **CAUTION:** If *COLDHEAD DRIVE* and *REMOTE DRIVE* are in the wrong positions, the Coldhead inside the CryoCooling Unit can be damaged.
o.6	Switch the **He Compressor** *MAIN POWER SW* at its backside ***ON***, then ***DRIVE*** at its front *ON*. The unit will go into **stand-by** mode. It is started later by the CryoController.
o.7	Turn *ON* the **CryoCooling Unit** main switch at its front. The CryoController will initialize the CryoPlatform. After a few moments the **green *WARM*** and the **white *UNPLUG* must be on**. If *ERROR* lights up, try to reset it by pushing *WARM UP* and check if all sensor and supply connections are ok.

Figure 4.11. Switching on the He Compressor



VT setup 4.8

> **IMPORTANT: Unlike conventional probes, the sample temperature inside a CryoProbe must be actively maintained with the gas flow and the heater switched on even if room temperature is desired.**

See ***"Sample temperature control" on page 55*** for details on the sample temperature setup.

> **CAUTION: Failure to establish a VT gas flow prior to cool-down will lead to extremely low temperatures in the sample cavity, freeze the sample and possibly damage the CryoProbe.**

Both N_2 gas and dry air can be used as VT gases. For a CryoProbe, there is no difference between them with respect to shimming or RF performance.

CAUTION: Do not initiate a cool-down if no regulated VT gas flows. Do not insert a sample if the CryoProbe is not at its operating temperature or if the temperature in its sample cavity does not stabilize at the desired value.

Table 4.5. VT setup

step	action
v.1	**Connect the VT gas** inlet of the CryoProbe either directly to the VT unit or to a BCU05 gas cooler but do not switch the BCU05 *ON* yet. **CAUTION:** When cold operation has been started, **the VT gas supply must not be interrupted** before the CryoProbe has been warmed-up again. Thus, a BCU05 gas cooler can only be inserted while the Cryo-Probe is warm.
v.2	**Set** the VT gas **flow rate** and VT **heater power** limit according to the values given on the specific LIMITATIONS - WARNINGS sheet of the Cryo-Probe. Do **not** switch the VT heater ***on*** yet. **NOTE:** A high VT gas flow rate is recommended but it must not lift the sample.
v.3	Verify that there is **no sample in the magnet yet.**
v.4	**Configure** the VT unit for the **Pt100** temperature sensor with ***edte***. Make sure the Pt100 sensor cable is connected properly at CryoProbe and VT unit. **NOTE:** An optional **B-TO2000** unit for conventional probes uses the same type of Pt100 cable. The cable is suitable for the CryoProbe and there are no special considerations when using a B-VT3000. A **B-VT2000**, however, cannot be toggled properly from B-TO2000 to Cryo-Probe operation with ***edte*** (although no error occurs); it must be **set explicitly at its EUROTHERM** module. With a B-TO2000, the EUROTHERM would be set neither for a Pt100 (code 0225) nor for a Cu/Const thermo-couple (code 0203) but with the special code 5203. When using a Cryo-Probe, make sure the EUROTHERM is configured for Pt100 with **code 0225**. See the *B-VT2000 Operating Manual* (P/N W1101034) for instructions.
v.5	Set the **sample temperature** to somewhere above room temperature, e.g. ***300 K***.
v.6	Switch the VT heater ***on***.
v.7	**Wait** until the ‘sample’ temperature and the heater power in the ***edte*** window settle.

CAUTION: Do not interrupt the VT unit or its gas flow at any time while the CryoProbe is cold. The sample may cool down to very low temperatures, potentially breaking the sample tube upon freezing or damaging the substance under investigation. Moreover, only a constant dry gas flow will avoid water condensation inside the CryoProbe.

NOTE: Damages of this kind are **not covered by the warranty**. BRUKER is not liable for destroyed samples due to disregard of the instructions given in the CryoProbe documentation.

Sample Safety Option 4.8.1

In case of a VT gas cut, the Sample Safety Option will continue the VT flow with gas at ambient temperature from a backup steel-cylinder. How long the sample temperature can be sustained depends on the size and pressure of the backup bottle.

If a precious sample in a solvent with high melting point is used like water or benzene, and an unattended long-term experiment is planned, e.g. over a weekend, check if the backup gas steel-cylinder could last for the whole time (which is the worst case).

Cool-down 4.9

NOTE: If any detectable malfunction appears, a cool-down will be automatically stopped and the CryoProbe System is automatically warmed-up to ambient temperature.

CAUTION: Do not move a cryogenically cold device. Do not try to fix a leak on a cold part because cracking of frozen o-rings, valves etc. may occur.

Table 4.6. Cool-down

step	action
c.1	Check if the 1) **cooling water** supply is active (if applicable); 2) He steel-cylinder is connected and open. Its primary pressure must be larger than 40 bar and its secondary pressure stable at **22-25 bar**; 3) CryoProbe is properly installed and connected; 4) CryoCooling Unit is *ON* and if ***WARM* and *unplug*** indicate that the unit is ready; 5) *SUPPLY PRESSURE* gauge on the **He Compressor** front reads about **17.5 bar** (= 1.75 MPa or 17.9 kgf/cm^2). If the pressure is higher, the excess He will be released automatically during the cool-down preparations. If the pressure is much lower than 16 bar (= 1.6 MPa or 16.3 kgf/cm^2), see *Refill He* in the *Installation Manual*; 6) **He Compressor** *MAIN POWER SW* at its backside and *DRIVE* at its front are ***ON***.
c.2	Make sure that enough **VT gas** flows through the sample cavity with properly limited heating (see LIMITATIONS - WARNINGS sheet). The **temperature** in the sample cavity must be set and remain stable.
c.3	Verify that there is **no sample in the magnet yet.**
c.4	**Start** the cool-down by pushing the *COOL DOWN* button on the CryoCooling Unit or in the ***CryoTool***. *COOL DOWN* will start to flash, indicating that the system is preparing the cool-down process.

c.5	*Cool down* will keep flashing as long as the ambient temperature preparations (evacuation, flushing cycles and charging with He) are in progress. Several pneumatic **pops** will be audible. At this point, it is recommended to check the secondary pressure at the He Regulator: it should always come back to **22-25 bar**. The primary pressure must remain ≥ 40 bar at all times.
c.6	~15 min later, **CryoCooler and He Compressor** will be **started automatically** to perform the cool-down. *Cool down* changes to steady white during the actual cool-down. The characteristic **periodic hiss** of the CryoCooler will be audible then. **IMPORTANT:** If no periodic hiss is audible and an error message appears on the monitoring computer contact Bruker.

While the cool-down is in progress for about 2 h, there is plenty of time to do the ***"HPPR CRP configuration" on page 44***.

c.7	**If *Cold*** on the CryoCooling Unit **flashes**, it will take about half an hour more until the final temperature is reached and stabilized. **NOTE:** A cool-down must not take more than 4 h; if it does, the CryoController will interrupt automatically and warm up the system (see ***"Cool-down doesn't reach cold state" on page 77***).
c.8	**Verify** that the **'sample' temperature** reading from inside the cavity is at its preset value and stable. Now a **sample can be inserted**. At this point, the ***"Low temperature limit in sample cavity" on page 45*** should be determined. **NOTE:** For recommended sample depth and filling height, see the 'Limitations - Warnings' sheet for the CryoProbe and ***"Samples and spinners" on page 55***. **IMPORTANT:** Always keep an eye on the sample temperature with `edte` when working with the CryoProbe. If the temperature drops, eject the sample and keep monitoring the 'sample' temperature inside the CryoProbe cavity.
c.9	Set all RF power levels `pl` and `sp` to 120 dB (recommended).
c.10	Enter `crpwobb` in XWIN-NMR and **tune** the CryoProbe on all channels **including ^{2}H**, see ***"Tuning and matching" on page 47***. When done, enter `crpon`.
c.11	**Shimming** can be started while *Cold* is still flashing, see ***"Shimming and Lock-in" on page 48***.
c.12	When ***Cold*** is **continuously on**, the final conditions are stable.

WARNING: Do not disconnect any tube or cable from a running CryoProbe System unless *unplug* lights up on the CryoCooling Unit front panel. Pressurized cryogenic helium gas is circulated between CryoProbe, CryoCooling Unit, and He Compressor. It could cause cold burns on unprotected eyes and skin when tubes are disconnected during operation.

NOTE: The small indicator in the upper right corner of the CryoProbe front plate will move in upon cool-down. Do not try to move this screw.

HPPR CRP configuration 4.10

IMPORTANT: If a conventional probe was used right before you mounted the CryoProbe *and* if your HPPR CRP features more than 5 modules (not counting the cover module), you should check the internal wiring between the HPPR CRP modules and their cover module.

Hardware settings

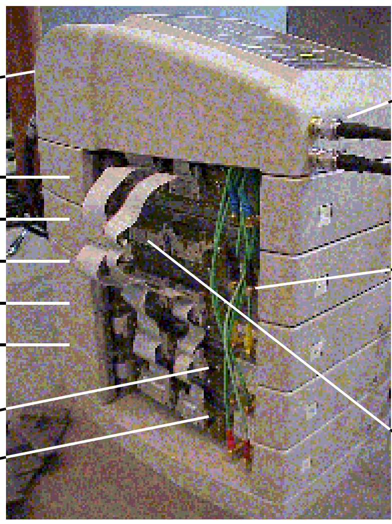
For a CryoProbe TXI with two X-channels, the HPPR 13C CRP module is connected to the HPPR cover module at 'X-BB' and displayed in ***edasp*** as ***X-BB31P_2HS***, whereas the HPPR 15N CRP module is wired to 'UB' and shows up as ***X-BB19F_2HS***. The conventional HPPR X-BB module is not connected when a CryoProbe TXI is used, neither to the ribbon cables nor to the RF lines that lead from the left side to the cover module (***Figure 4.12.***).

To change the HPPR CRP assembly's internal wiring, shut down its power supply by switching OFF the AQR unit inside the spectrometer cabinet. When done, switch on the AQR and run an XWIN-NMR ***cf*** dialogue (NMRsuperuser permissions required). The first X-module in the HPPR CRP should be recognized as ***X-BB31P_2HS*** and the second module of e.g. a CryoProbe TXI as ***X-BB19F_2HS***, irrespective of their actual function and frequency.

RF routing with ***edasp***

When the RF hardware wiring is complete, RF pathways need to be configured in XWIN-NMR with ***edasp***.

Figure 4.12. Internal wiring of a HPPR CRP for a TXI CryoProbe



Selection of CryoPreamps

To make full use of the CryoProbe's signal-to-noise advantage, its built-in 'cold' preamplifiers must be selected for detection with ***crpon***.

NOTE: After a power-on, the 'warm' preamplifier modules of the HPPR assembly are selected by default - yielding the same result as a ***crpoff***.

Depending on the kind of NMR experiment, it may be desired to observe a nucleus for which the CryoProbe has no built-in CryoPreamp but a decoupling channel linked to a conventional 'warm' HPPR preamplifier module.

Example of a special setup

Task: Observe ^{13}C on a CryoProbe TXI in the presence of a high-sensitivity ^{2}H lock.

Setup: In a CryoProbe TXI, the 13C channel is intended for decoupling only and features no CryoPreamp. However, ^{13}C detection is possible when using the warm HPPR 13C CRP preamp module. Send its output to the FT receiver but feed the output of the build-in cold ^{2}H preamp into the lock receiver. This configuration is established with ***crp2hon***.

Possible combinations

See ***Table 4.7.*** for all possible combinations of warm and cold preamps and their corresponding XWIN-NMR commands. The letter 'X' stands for any nucleus but ^{1}H, e.g. ^{13}C, ^{15}N, ^{2}H. Of course, the build-in 'cold' CryoPreamps are to be preferred. There are no waiting periods or the like needed when toggling between cold and warm preamps.

Table 4.7. Preamplifier selection

observe	preamp	lock	situation	XWIN-NMR
^{1}H or X	cold	cold	default	***crpon***
^{1}H or X	warm	cold	no CryoPreamp available for the chosen nucleus; debugging	***crp2hon***
^{1}H or X	cold	warm	debugging	***crp1hxon***
^{1}H or X	warm	warm	using a conventional probe; debugging	***crpoff***
wobble	warm	warm	tuning and matching any channel	***crpwobb***

More details are given in the *CryoProbe RF Electronics Technical Manual*.

Low temperature limit in sample cavity 4.11

The procedure described in this section is needed if measurements at or below room temperature are planned. For the adjustment of the VT heater power limit, the lowest temperature that can actually be reached in the CryoProbe sample cavity, T_{min} , must be known.

CAUTION: Never cool the cavity below the sample temperature lower limit $T_{limit, low}$ as given on the specific CryoProbe LIMITATIONS - WARNINGS sheet, even if the VT gas cooling would permit to do so.

Table 4.8. Determine lowest sample cavity temperature

step	action
t.1	Insert a sample with a **low melting point**, e.g. 'Lineshape' (mp = - 64°C).
t.2	Select the **lowest allowed sample temperature** as given on the specific LIMITATION - WARNINGS sheet of the CryoProbe, e.g. -10°C.
t.3	Set the **VT heater power limit** according to the LIMITATIONS - WARNINGS sheet. Leave the VT heater ***off***.
t.4	Select the desired **VT gas flow rate**. **NOTE:** A high VT gas flow is generally to be preferred for a CryoProbe as long as it does not lift the sample. See the minimum flow rate on the 'LIMITATIONS - WARNINGS' sheet and the explanations in ***"H2O samples" on page 60***.
t.5	When the sample temperature has **stabilized** after a few minutes, write down its value. This is T_{min} which will be used later for the setup of NMR experiments (see ***"Sample temperature control" on page 55***).
t.6	**NOTE:** The following determination of $T_{min, BCU05}$ in the presence of a BCU05, step ***t.6*** to ***t.9***, is time-consuming (~1h) because the gas cooler needs time to reach its operating temperature. If $T_{min, BCU05}$ is not needed now, skip this test and execute it when convenient. If a **BCU05** is present, switch it *ON*.
t.7	Make sure that the **sample temperature** is set to the lowest allowed value as given on the specific LIMITATION - WARNINGS sheet of the CryoProbe, e.g. -10°C.
t.8	Switch the **VT heater** ***on***. **NOTE:** Here, the VT heater is needed only for the protection of the Cryo Probe during the unattended operation of the BCU05. While the sample temperature is above the allowed lower limit (e.g. -10°C), the VT heater power must be 0%. **NOTE:** Observe the sample temperature for a minute and make sure it does not rise in an uncontrolled way. If it rises too far or too fast, reduce the VT heater power limit such that the sample temperature stabilizes. In emergency, eject the sample. Never interrupt the VT gas flow!

t.9	Wait until the sample has **equilibrated** again (~1h) and write down its actual temperature as $T_{min, BCU05}$. **NOTE:** If the sample temperature would fall below -20°C even with the VT heater being active, an automatic security warm-up of the CryoProbe will be started. If this happens, switch *OFF* the BCU05 and increase the VT heater power limit carefully. When $T_{sample} > -20°C$, press *COOL-DOWN* to re-establish cold operation of the CryoProbe. **NOTE:** The VT hose must not be covered with ice at the point where it enters the CryoProbe body. If the CryoProbe body is cooled by the VT gas and becomes wet with condensed water, the hose must be extended. Since the VT gas flow must never be interrupted while the CryoProbe is cold, a warm-up to ambient temperature is needed before the hose can be exchanged.

Tuning and matching — *4.12*

IMPORTANT: A CryoProbe must not be tuned or matched when warm because the properties of the RF circuits are quite different when cold. No resonance may be visible at ambient temperature.

Before attempting to tune and match a CryoProbe, the green *COLD* light should be on or at least flashing. It indicates that the temperature is sufficiently low for a meaningful and persistent adjustment.

Tuning and matching settings are reproducible after a warm-up/cool-down cycle. They are not more susceptible to a change of solvent than in conventional probes.

Wobbling

The wobbling feature is handled by the 'warm' preamplifiers inside the HPPR CRP instead of the built-in CryoPreamps. Therefore, the RF pathway to these preamplifiers must be configured with special XWIN-NMR commands.

Table 4.9. How to wobble the CryoProbe

step	action
w.1	Configure nuclear frequencies and routing in ***edasp*** as usual. For wobbling ^{2}H, one channel must be set to ***2H*** and routed via a suitable X-amplifier to the ***2H*** preamp module.
w.2	To wobble - all channels incl. ^{2}H (recommended), enter ***locnuc off, crpwobb*** and ***wobb***, - all channels except ^{2}H while the lock is operating, enter ***crp2hon,*** and ***wobb***.
w.3	Tune and match with the Tuning Adapter, see next paragraph.
w.4	When tuning and matching is done, enter ***stop, locnuc 2H, ii*** and ***crpon***.

Operating the Tuning Adapter

A CryoProbe is tuned and matched from below like a conventional probe with the actuators of its specific Tuning Adapter (***Figure 4.9.***). Long actuators are for tuning, short ones for matching. Use the blue Tuning Tool to operate the actuators. The common BRUKER color code applies:

yellow: ^{1}H
white: ^{2}H
blue: ^{13}C
red: ^{15}N

CAUTION: Some of the tuning elements are factory-set and have no corresponding actuator in the Tuning Adapter. Do not attempt to change them.

CAUTION: Do not force the actuators beyond their easily recognizable limits (clockwise: stop, counter-clockwise: overrun). Do not challenge the limits of the tuning and matching range if it can be avoided. Do not use tools other that the Tuning Adapter to adjust the tuning or matching.

Use of additional RF filters

All necessary filters are already built-in the CryoProbe and the HPPR CRP. If additional RF filters are considered, see ***"Additional RF filters" on page 58***.

In any case, additional RF filters must be included before tuning or matching is done.

Shimming and Lock-in *4.13*

For lock-in and shimming, the green *COLD* light should be on or at least flashing. When *COLD* lights steadily, experiments can start.

Always obey the power limitations as given on the specific 'LIMITATIONS - WARNINGS' sheet when sending RF power into the CryoProbe (see also ***"RF power limitations" on page 58***).

The usual XWIN-NMR ***`lock`*** procedure can be used for lock-in. In contrast to the 1H and X channels, the lock channel of a CryoProbe may need as much (or even more) RF power as a conventional probe. If in doubt about the lock power, perform an AUTO LOCK POWER calibration with the BSMS keyboard and store the result in the CryoProbe's ***`edlock`*** table.

If no lock wiggles can be found, the shim may be far off the optimum. Insert an H_2O/D_2O sample and shim on the ^{1}H FID in the ***`gs`*** mode until the ^{2}H signal is strong enough for lock-in.

Shimming can be done on the lock level as usual. There are no particular gradient values to expect. Gradient shimming is recommended.

After lock-in and shimming, the CryoProbe is ready for NMR experiments. Consult the ***"Recommended NMR parameters" on page 55***. Pay special attention to the ***"Sample temperature control" on page 55*** and to the ***"RF power limitations" on page 58***!

Warm-up 4.14

WARNING: Do not disconnect any tube or cable from a running CryoProbe System unless *UNPLUG* lights up on the CryoCooling Unit front panel. Pressurized cryogenic helium gas is circulated between CryoProbe, CryoCooling Unit, and He Compressor. It could cause cold burns on unprotected eyes and skin when tubes are disconnected during operation.

Warming-up the CryoProbe is usually not necessary. It can run without interruption for up to 10 000 h (the limit is actually set by the mechanical wear inside the CryoCooler). A warm-up is required only if

- requested by the CryoController (see **"Warm-up needed soon request" on page 63**),
- the CryoProbe should be removed from the magnet,
- the CryoCooling Unit is going to be moved,
- there are no NMR measurements to be done for at least a week.

If in doubt, leave the CryoProbe running in its cold state.

Table 4.10. Warm-up

step	action
w.1	Terminate NMR experiment and **eject** the sample. **CAUTION:** Keep the VT unit operating even without a sample!
w.2	Press the ***WARM UP*** button on the front of the CryoCooling Unit. Its light will be steady on then. The **He Compressor stops** immediately but restarts after ~ 2 min. No periodic hiss is audible during warm-up.
w.3	**Wait** until green light *WARM* comes on. **CAUTION:** Do not enforce a warm-up by any additional means of external or internal heating!

Removal 4.15

Insertion and removal of the heavy CryoProbe (~12 kg) can be easily accomplished by the spectrometer operator with the help of a **second person**.

WARNING: Do not attempt to insert or remove the heavy CryoProbe (~12 kg) at the magnet without the help of a **second person**. Because your body's posture is unfavorable when kneeling down below a magnet, you may easily injure your back when lifting the CryoProbe on your own!

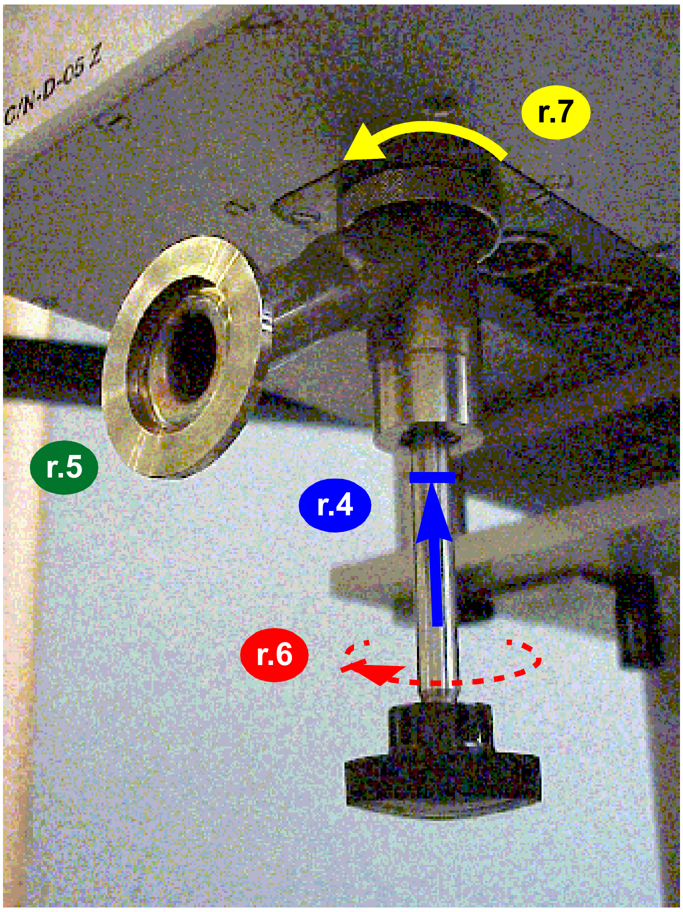
CAUTION: Never attempt to remove a CryoProbe from the magnet when cold. Always wait until it has reached room temperature because the CryoProbe or the He Transferline may be damaged when moved cold.

It is impossible to remove the CryoProbe from the magnet while it is connected to the He Transferline. There are geometrical restrictions and the danger of bending the CryoProbe tube with the large lever of the He Transferline.

Table 4.11. Remove the CryoProbe

step	action
r.1	Make sure that the **warm-up** of the CryoProbe has been completed (see ***"Warm-up" on page 49***). All connections on the probe should stay in place during the warm-up. Leave the probe as it is until it reaches room temperature (max. 2 h).
r.2	When ***WARM* and *UNPLUG*** are lit: Disconnect all cables and tubes from the CryoProbe front and bottom.
r.3	**Detach** the He Transferline from the Transferline Support: undo the four black screws and lift them off. Leave the He Transferline mobile but supported such that no forces can act on the CryoProbe when the magnet air suspension is lowered later on.
r.4	Insert the **Vacuum Plug** into the CryoProbe bottom by pushing up the black handle of the Vacuum adapter. It should move easily at the beginning, but needs a little more force on the last cm when the actual seal is made (***Figure 4.13.***).
r.5	Detach the **vacuum bellows** from the Vacuum Adapter.
r.6	Separate the handle of the Vacuum Adapter from the Vacuum Plug by turning it **counter-clockwise** at least ten turns. It should move out slowly when leaving the thread of the vacuum plug. If in doubt, proceed with the next step. The CryoProbe's vacuum chamber is not damaged when subjected to air.
r.7	Remove the **Vacuum Adapter**.

Figure 4.13. Removing the vacuum connector from the CryoProbe



r.8	Use the special titanium open-end wrench to **unlock** the CryoCoupler. Be careful not to damage any connector on the CryoProbe front.
r.9	**NOTE:** Use the same technique for removing the CryoCoupler as for connecting it (cf. ***"Mounting" on page 28*** and ***Figure 4.8.***). **Support** the He transferline at its original height while drawing the CryoCoupler carefully out of the CryoProbe. Make sure to exert **no torque** on the CryoProbe body. Do not use large forces, the suspended magnet must not move.
r.10	Check if all **4 o-rings** on the He Transferline CryoCoupler are still in place (***Figure 4.7.***).
r.11	Close both the CryoProbe and the He Transferline firmly with their special **caps**.
r.12	Switch *OFF* the **air suspension** of the magnet such that the magnet comes down onto its stand.

r.13	Loosen the **four outer screws** in the Mounting Hardware support plate all the way down.
r.14	Screw down the **pusher screw** in the center and lower the probe by a ~10 mm.
r.15	**WARNING:** For lifting the heavy CryoProbe out of the magnet, **two people** are needed! Support the CryoProbe's **center** with a hand and push it back into the magnet. Do not push from below its front because the resulting lever could break the fragile joint between CryoProbe body and tube. Take out the **support plate** while pressing the two little handles below its bottom.
r.16	Let the CryoProbe slowly slide out of the magnet - it weights about 12 kg! **Keep its body in sliding contact with the two guiding rails.** Make sure not to put any leverage or strain on the CryoProbe. When leaving the shim system, hold the CryoProbe top gently with one hand to avoid hitting anything.
r.17	**CAUTION:** Never carry a CryoProbe at its tube, hold it at its body only. Cover the sample cavity with the **Protection Cap** to prevent water condensation, dust contamination, and intrusion of foreign objects. Put the CryoProbe into its flight case for safe storage in a dry and clean place.
r.18	Immediately close the **magnet** bottom or insert another probe to avoid intrusion of dust or magnetic particles.
r.19	Switch *ON* the **air suspension** of the magnet.

Power-down *4.16*

The CryoProbe System may be shut down after the indicators *WARM* and *UNPLUG* light up and the warm-up terminates. A shut-down does not have a drawback in terms of equilibration times or performance etc. However, the power consumption and the aging of a 'warm' CryoPlatform are small since most devices have been switched off automatically.

Table 4.12. Power-down

step	action
d.1	If the CryoProbe is still in the magnet: terminate any NMR experiment, eject the sample, and warm-up (see ***"Warm-up" on page 49***).
d.2	Switch *OFF* the **CryoCooling Unit** at its front.
d.3	Close the He steel-cylinder **main valve** but do not change the He Regu lator.

d.4	Switch *OFF* the **He Compressor** *MAIN POWER SW* on its back.
d.5	In case of a water-cooled He Compressor: close the **cooling water** supply or switch the water chiller *OFF*. **NOTE:** If the water chiller is remote controlled from the CryoCooling Unit, it will be shut down when the CryoCooling Unit mains is switched off.

IMPORTANT: The He compressor and the Flexlines must remain pressurized to about 15 bar at any time to prevent contamination. When the CryoProbe System is shut down, it automatically vents some parts while others remain pressurized. Its seals are designed to hold the pressure over an extended period. Do not try to release this pressure by any means! Safety-valves will prevent damage if excess pressure builds up accidentally.

See ***“Relocation of a CryoPlatform”*** in the *Installation Manual* if the CryoProbe System shall be moved to another place.

Spectrometer operation with conventional probes *4.17*

The HPPR CRP preamplifier assembly can be operated with conventional probes just like a standard HPPR. For ^{13}C observe experiments with conventional probes, either the 13C CRP or the X-BB module can be used. If the HPPR CRP contains more than 5 preamp modules, the wiring inside the HPPR CRP needs to be checked (see the *CryoProbe RF Electronics Technical Manual* and ***"HPPR CRP configuration" on page 44***).

IMPORTANT: If you have to change the HPPR CRP assembly’s internal wiring, shut down its power supply by switching *OFF* the AQR unit inside the spectrometer cabinet. When done, switch *ON* the AQR and run an XWIN-NMR `cf` dialogue.

Recommended NMR parameters 5

General 5.1

When running a long experiment with extensive decoupling or spin-lock, check the progress of the acquisition during the first few minutes after its start. In case of excessive heating the NMR coil assembly with RF, the system will send error messages and might automatically halt the experiment or even warm-up the CryoProbe if its warnings have been ignored (see also ***"Sample temperature control" on page 55***).

Samples and spinners 5.2

The following BRUKER spinner types were tested so far:

opaque white	recommended
blue	ok
ceramic	not approved

Use only high quality sample tubes with high axial symmetry. Susceptibility-compensated tubes (e.g. Shigemi) are recommended. In the worst case, however, a bad sample tube just cannot be spun.

Observe the limits pertaining to the sample parameters as given on the LIMITATIONS - WARNINGS sheet for each individual CryoProbe such as

max. sample diameter,
sample depth limits,
min. VT gas flow rate,
recommended filling height.

Filling the sample tube above the recommended height will neither increase the signal-to-noise nor improve the shimming, but it will deteriorate the precision of the sample temperature control.

Sample temperature control 5.3

Although the CryoProbe has an excellent thermal insulation between the sample space and the cold RF coils, a small thermal radiation is constantly cooling the sample. A sufficient VT gas flow is crucial to avoid temperature gradients in the sample. The higher the VT gas flow, the smaller the temperature gradient. Unlike conventional probes, a VT gas flow as high as possible is preferred in a CryoProbe, i.e. the VT gas may almost lift the sample. See the ‘LIMITATIONS - WARN-

INGS' sheet of the CryoProbe for the minimum gas flow rate (which is often higher than the *maximum* flow rate that conventional probes can handle).

CAUTION: The VT gas must not be interrupted or switched off at any time while the CryoProbe is in cold operation. Samples with high melting points like water or benzene may freeze within a few seconds.

Both N_2 gas and dry air can be used as VT gases. For a CryoProbe, there is no difference between them with respect to shimming or RF performance.

For measurements at low sample temperatures, a BCU05 gas cooler is required. If the target sample temperature is close to the temperature of the VT gas, the VT Interface Box must be set to *LOW*. In any case, the allowed sample temperature range, the minimum VT gas flow rate, and the maximum VT heater power must be obeyed as given on the CryoProbe-specific 'LIMITATIONS - WARNINGS' sheet. Details are listed below.

Symbols used

T_{target} : target temperature for the sample

$T_{VT\ gas}$: temperature of the VT gas before it enters either the CryoProbe directly or a BCU05 gas cooler

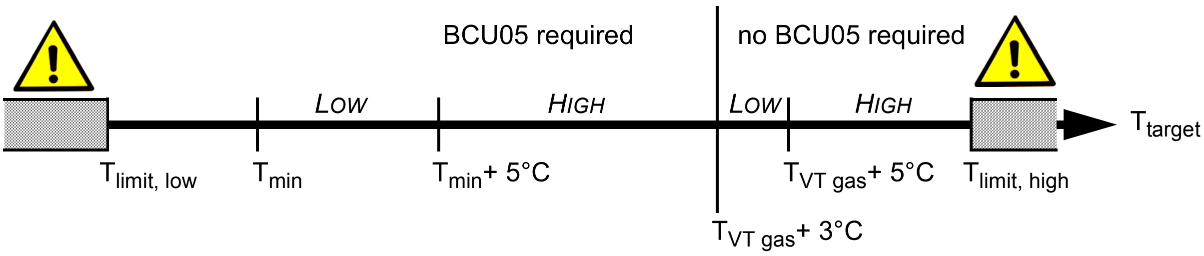
T_{min} : minimum achievable sample temperature in a cold CryoProbe when the VT heater is ***off***

$T_{limit,\ low}$: lower limit of the temperature in the sample cavity, see the specific 'LIMITATIONS - WARNINGS' sheet of the CryoProbe

$T_{limit,\ high}$: upper limit of the temperature in the sample cavity, see the specific 'LIMITATIONS - WARNINGS' sheet of the CryoProbe

.

Figure 5.1. The different ranges of the target temperature



Measurements above $T_{VT\ gas}$+ 3°C — *5.3.1*

An optional VT gas cooler **BCU05 is not required**. Connect the gas flow either directly to VT unit or switch off the BCU05.

Depending on the target temperature (see "***Figure 5.1"***) the selector switch on the VT Interface Box has to be set accordingly to

- *LOW* — For $T_{VT\ gas}$+ 3°C < T_{target} < $T_{VT\ gas}$+ 5°C

• *HIGH* — For $T_{VT\ gas}$+ 5°C < T_{target} < $T_{limit,\ high}$
This is generally the preferred position unless the temperature control appears to be unstable for temperature setpoints close to the incoming VT gas temperature.

CAUTION: Obey the upper limit for the sample temperature $T_{limit,\ high}$ and the maximum VT heater power as given on the 'LIMITATIONS - WARNINGS' sheet. This temperature must not be exceeded - not even for a short time.

NOTE: The VT Interface Box splits the VT unit heater energy and disposes part of it as heat at the box. When the box is in *LOW* position but forced to heat the sample beyond 65°C, a safety circuit will prevent the box from overheating. A *`heater failure`* will be reported by the VT unit and in the ***`edte`*** display.

Measurements below $T_{VT\ gas}$+ 3°C — *5.3.2*

An optional **BCU05 gas cooler is required**.

Depending on the target temperature (see "***Figure 5.1"***) the selector switch on the VT Interface Box has to be set accordingly to

• *LOW* — For T_{min} < T_{target} < T_{min} + 5°C

• *HIGH* — For T_{min} + 5°C < T_{target} < $T_{VT\ gas}$+ 3°C
This is generally the preferred position unless the temperature control appears to be unstable.

CAUTION: Obey the lower limit for the sample temperature $T_{limit,\ low}$ as given on the 'LIMITATIONS - WARNINGS' sheet. This temperature must not be exceeded - not even for a short time. Never use a liquid nitrogen evaporator to provide cold VT gas.

CAUTION: Use a special VT adapter or a hose connection of sufficient length such that, at the point where the hose enters the CryoProbe, there is no freezing or condensation.

Tuning the regulation loop — *5.3.3*

Use the self-tune feature of the B-VT units to optimize the sample temperature regulation loop. Write down the control parameters of the B-VT unit and also the VT heater power required to achieve the desired temperature. Set the heater limit accordingly.

NOTE: A B-VT2000 cannot be self-tuned with ***`edte`*** (although no error appears) but must be set directly at its EUROTHERM unit. See the *B-VT2000 Operating Manual* (P/N W1101034) for instructions.

Wiring — *5.4*

See also the *CryoProbe RF Electronics Technical Manual* for details.

NOTE: A Radiation Damping Control Unit (RDCU) cannot be used because it is designed to be inserted in the single transmission line between preamp and probe. A CryoProbe, however, has built-in CryoPreamps and the connection between NMR coil assembly and preamp is not accessible from outside. Thus, the current versions of the two systems are incompatible.

Preamplifier 5.4.1

NOTE: Whenever there are two connectors per channel of the CryoProbe (transmit and receive), there is a specific cryogenically cooled preamp build-in with superior signal-to-noise. If there is only one connector, the conventional preamp in the HPPR CRP will be used for detection. However, such a channel is not intended for observation and its sensitivity is generally not specified.

An HPPR CRP with more than 5 modules may require manual re-wiring for multinuclear NMR experiments with a conventional probe (see ***"HPPR CRP configuration" on page 44***).

Additional RF filters 5.4.2

All necessary filters are already incorporated in the CryoProbe and the HPPR CRP.

IMPORTANT: If it seems as if additional RF filters are needed there to improve the performance of an experiment, there is probably a malfunction in one of the RF components. In particular, no low frequency stop filters must be introduced into the pulse transmission paths between HPPR CRP and CryoProbe.

RF power limitations 5.5

CAUTION: Before you enable the RF transmitters: Make sure that neither the CryoProbe nor the HPPR modules can be overloaded by RF power.

Always ensure optimum tuning and matching because the unmatched case is even more demanding for the RF electronics.

Keep parameter sets for conventional and CryoProbes well separated.

Peak RF power 5.5.1

Due to the high efficiency of the CryoProbe, the **maximum RF power** may be **significantly less than those on conventional probes**. The highest power levels available in AVANCE spectrometers have the potential to destroy the NMR coil assembly. Use power levels ***p1*** and ***sp*** higher than 10 dB (e.g. 6 dB, 0 dB, -3 dB etc.) with extreme caution!

Before setting an RF power level ***p1*** or ***sp***, look up the **RF power limits**

- of the **CryoProbe** on its specific LIMITATIONS - WARNINGS sheet,
- of the **built-in RF Unit** and the **HPPR CRP modules** in the *CryoProbe RF Electronics Technical Manual*, and

- of the **conventional HPPR modules** in the *HPPR Technical Manual* on the BASH-CD.

For each channel, **the lowest value found must not be exceeded** by the respective amplifier. Usually, there's a table in the spectrometer installation documents that relates amplifier powers in watt to `p1` settings in dB.

If in doubt, start with powers that are **at least 10 dB below** the values common to your conventional probes. Increase them gradually if necessary. Do not exceed the CryoProbe's specific maximum power and the HPPR module's maximum load.

Simultaneous hard pulses 5.5.2

CAUTION: When two RF pulses are applied to an NMR coil at the same time, their peak voltages add. Arcing may occur and permanently damage to the coil.

To prevent arcing, the power of two simultaneous hard pulses[1] must be reduced by 6 dB each. In case of three simultaneous hard pulses, the reduction is 10 dB.

For specific information refer to the 'LIMITATIONS - WARNINGS' sheet.

Average RF power 5.5.3

CAUTION: The decoupling power required by a CryoProbe for a given RF field strength is much smaller than in a conventional probe.

In cases where the average RF power exceeds the cooling capacity of the system, the NMR coil assembly cannot keep its nominal operating temperature. Artifacts, unusual noise or a slight mistune/mismatch could occur if the temperature becomes unstable. In order to warn the operator when the NMR coil assembly warms up by **more than 5 K**, ***COLD*** **will start flashing**. Should this occur, reduce the average RF power until the light is steady green again. If most of the average RF power is due to decoupling during the acquisition, consider a shorter acquisition time.

However, **if the overheating situation is stationary** within the first limit, i.e. just *COLD* flashing but no *ERROR*, the cooling system usually manages to stabilize the coil temperature. It might be **possible to acquire NMR data** of sufficient quality, perhaps at the expense of signal-to-noise ratio, some mismatching, increased t_1-noise etc. Execute a sufficient number of dummy scans.

If this warning is ignored but the NMR coil assembly is heated further, there is a second factory-set temperature limit of **15 K above** the optimum operating temperature at which ***ERROR*** **starts flashing**. Stop the NMR experiment immediately! If the NMR coil assembly temperature exceeds the optimum operating temperature by **20 K**, the CryoController **warms up the CryoProbe automatically** as indicated by a flashing *WARM UP* and *ERROR*.

CAUTION: Should an automatic warm-up be initiated, abort the experiment immediately and make sure that no further RF is going into the CryoProbe.

After the RF-induced heating has been reduced, it is feasible to immediately *COOL DOWN* again and to restart the experiment (at a lower power level and/or shorter

1 A 'hard pulse' is a short pulse at maximum allowed RF power.

decoupling periods!). COOL DOWN will flash three times quickly, acknowledging the cool-down request. Usually, the system should be ready again within 15 minutes.

Receiver setting 5.6

The effective digitizer resolution ***dr*** should be 18 bit or more to keep quantization noise at a negligible level. Use a small sweep width ***swh*** for your particular experiment.

Do not employ an automatic receiver gain adjustment ***rga*** because it typically uses only about 50% of the available ADC range. To find the optimum receiver gain ***rg***, increase the sweep width ***swh*** such that the dynamic range ***dr*** is still the same as in your particular experiment. Increasing ***swh*** assures that there are no strong signals outside the digital filter width. Adjust ***rg*** so that more than 80% of the ADC's effective range 2^{dr-1} is used, e.g. at least 100 000 units on the y-axis in the FID display window ***acqu*** if the effective digitizer resolution is ***dr*** = 18. After having determined the optimum ***rg*** change ***swh*** back to its original value and proceed with your experiment.

For small receiver gain values, i.e. ***rg*** < ~256, the signal-to-noise tends to decrease in proportion to ***rg***. If the CryoProbe is always used with samples that yield a strong NMR signal like an H_2O solution, a permanent reduction of the overall receiver gain may be beneficial because this decreases also the noise added inside the receiver. The modification is a jumper setting in the RX22, ECL07 or higher. As a consequence, ***rg*** values will be higher than before by about 12 dB for all measurements.

CAUTION: Do not try to change the jumper settings on the highly sensitive RX22 board on your own. It is recommended that you have a BRUKER service engineer carry out this modification upon installation of your CryoProbe.

Shimming 5.7

Gradient shimming 5.7.1

Make sure to have *FIELD* adjusted such that the lock wiggles are centered in the lock window to avoid H_0 offsets between gradient shimming and lock-in operation. If an ACUSTAR is used, check its DC offset adjustment by observing the lock level while pressing its Z *RESET* button.

H_2O samples 5.7.2

A temperature gradient inside the sample solution can spoil the resolution even if the shim and the water suppression are excellent. The most prominent temperature gradient is linear along the z-axis. Since the position of the water resonance is temperature-dependent, a linear resonance shift along the z-axis would result. When shimming with D_2O using the lock level, one would balance the temperature shift of the water resonance with the z-shim because this would superimpose the water signals from different regions of the sample and thus increase the lock level. But a misset z-shim affects all resonances and spoils their lineshapes. In case of an apparently ‘optimum’ shim but unsatisfactory resolution, start an acquisition in ***gs*** mode with on-line Fourier transform and touch-up Z, X, Y, ZX, and ZY (other shims are usually of little effect). When changing the VT gas flow rate, the procedure must be repeated.

Solvent suppression *5.8*

There is no preferred solvent suppression technique for use with a CryoProbe. All standard methods like presaturation, WATERGATE, and Excitation Sculpting can be used as usual. Select a method that allows ***rg*** ≥ 100 for which the receiver noise is negligible.

Due to the inherent geometrical restrictions in a cryogenic NMR probe, the RF coils must be located further away from the sample as compared to conventional probes since the vacuum thermal insulation requires some additional space. As a consequence, RF fringe fields extend further above and below the main sample region, which in turn makes the solvent suppression more difficult.

Gradients *5.9*

Gradient experiments may benefit from increasing the gradient strength relative to conventional probes by 10-20 units on the %-scale. Sine shapes with 100 steps resolution are recommended.

Pulse programs *5.10*

There are no special commands or particular sequences required in pulse programs for use with a CryoProbe.

Automation *5.11*

Automation with IconNMR and sample changer can be done as usual.

CAUTION: Make sure that acquisition parameter sets for conventional probes are not confused with those for a CryoProbe! CryoProbes usually operate at significantly lower RF powers and higher VT gas flow rates.

NOTE: At high decoupling power levels the z-shim may change slightly. To preserve optimum resolution do the following:

Table 5.1. How to determine Δz

step	action
w.1	Set the lock gain such that the lock is on the top gridline in your lockdisplay. Run your particular experiment with gs for a couple of minutes and determine the amount Δz you have to change your z-shim to have the lock back to the top gridline.
w.2	Add some dummy scans to your experiment and add Δz to the z-shim.
w.3	Run your experiment.
w.4	Once the experiment is finished, subtract Δz from the z-shim.

Monitoring and Maintenance 6

In this chapter, the various indicators for the CryoProbe System status are introduced. The system should be monitored continuously in order to preserve performance, identify emerging problems early, prevent serious damage, and to trace faults back to their origins.

Actual problems, faults, and solutions are discussed in ***"Troubleshooting" on page 71***.

NOTE: BRUKER warranty expires if the CryoProbe was opened by unauthorized personnel.

Displays & Diagnostics 6.1

See ***"Status display by the control buttons" on page 20*** for an "explanation" of the blink codes.

CryoTool interface 6.1.1

The CryoController inside the CryoCooling Unit operates the CryoPlatform autonomously. Its status reports and error messages can be displayed on a PC under Windows™ 95/NT4 by the CryoTool software. The displaying computer is connected directly to the CryoController using an RS232 interface. Most CryoPlatform variables like temperatures, He pressures, and valve configurations can be monitored in a special NMRsuperuser mode.

The power consumption of the He heater inside the CryoProbe ***Insert-H5*** is a good indicator for the cooling efficiency. Any problem in the cryogenic gas flow system will affect this heater power.

`Warm-up needed soon` request 6.1.2

Minute contaminations of the He cooling loop with air or moisture can hardly be avoided. They will gradually freeze out at the cold parts inside the system, potentially restricting the He flow rate. A slow decrease of cooling power with time results. This situation is automatically detected by the CryoController, and when the blockage exceeds a factory-set level, the `Warm-up needed soon` message appears and *WARM* starts flashing.

However, this does not call for immediate action. When the message appears, there is still 10-20% time of unrestricted operation available ('100%' being the interval between the last initiation of a cool-down and the eventual automatic warm-up). It may take days or even weeks until the actual warm-up condition is reached.

See ***Table 6.1.*** for a workaround that will temporarily solve the problem. If you want to have the problem fixed immediately in a more permanent way, arrange a BRUKER service visit within the next days. Keep the system running in the meantime - if it warms up, the contaminations would thaw and distribute throughout the system which makes the service actions more difficult.

Table 6.1. How to postpone a `Warm-up needed soon` *request*

step	action
n.1	Complete the current experiment or set of experiments.
n.2	Start a ***WARM-UP*** **at the next convenient occasion**.
n.3	Leave it warm for at least 1 h.
n.4	Press ***COOL-DOWN*** again.

In any case, a special procedure has to be performed by a BRUKER engineer to remove the impurities from the system. This will typically take overnight and the following morning.

CryoProbe insulation indicator 6.1.3

In the upper right corner of the front plate, there is a little hole with an indicator inside. This indicator must be flush with the CryoProbe front at room temperature. If the indicator sticks out, a problem with the CryoProbe's insulation may have occurred - contact BRUKER! Do not try to move the screw, neither in/out nor by rotation. When cooling-down the CryoProbe, this indicator will move in.

Periodic checks 6.2

CAUTION: Check your CryoProbe System carefully as recommended at regular intervals. Lack of monitoring and preventive maintenance can result in degraded performance or even in permanent damage.

Monitor the He Compressor's pressure gauge. When idle, the static pressure inside the He Compressor loop is about 17.5 bar.

For the water-cooled He Compressor, check the cooling water flow rate and temperature.

Log-book 6.2.1

All users should record the status of the CryoProbe System at regular intervals, e.g. at the start and stop of each cool-down or warm-up. Use copies of ***Table 6.2.*** on the following page to create a log-book. All observations are important information for BRUKER service engineers when they start to diagnose a problem.

Table 6.2. Operating log for CryoCooling Unit and He Compressor

Date	Time	Elapsed time [h][a]	Elapsed time [h][b]	Main pressure [bar][c]	Process[d]	Remarks

a as displayed on the He Compressor front
b as displayed on the CryoCooling Unit back
c He steel-cylinder primary pressure [bar], must be > 30 bar
d e.g. ‘cool-down’, ‘cold operation’, ‘start HSQC’Page ....

Logfile 6.2.2

Logfiles are recorded automatically by the CryoController and periodically stored on the monitoring computer as `auto<yymmdd>.log` (`yymmdd` being the creation date) in the default directory `D:\app\Bruker\CryoTool\LogFiles`. For the diagnosis of problems, your BRUKER service station will usually ask you to send a copy of the latest logfile via e-mail.

NOTE: The ‘Elapsed Time’ and the ‘Main pressure’ as of ***Table 6.2.*** cannot be recorded in the automatic logfile. Thus, your log-book and the logfile will contain important complementary information.

Replacement of the He steel-cylinder 6.2.3

The He steel-cylinder must be exchanged when its main pressure drops below 30 bar. It is recommended to have a full He steel-cylinder ready by the time the pressure has come down to 40 bar. With a standard size 50 ℓ He steel-cylinder, there will be still 2-3 warm-up/cool-down cycles possible.

If a 50 ℓ He steel-cylinder with an initial pressure of 200 bar lasts for significantly less than 40 warm-up/cool-down cycles or if the CryoProbe System requires about one He recharge/week there is a small He leak somewhere. If the long-time He loss is higher than a preset value, the CryoController will warm-up the system automatically. See ***"He leakage" on page 81***.

NOTE: The He steel-cylinder cannot be exchanged (or e.g. used for the magnet’s LHe refill) while the CryoProbe is in cold operation because the He system would be inevitably contaminated by air. However, since He is mostly consumed when a cool-down is initiated, a cold CryoProbe System can run a long time even with an almost empty He steel-cylinder as long as the He pressure is ≥ 30 bar.

Table 6.3. Replace the He steel-cylinder

step	action
b.1	Make sure that the CryoProbe System is ***WARM*** and in stand-by mode, i.e. ***UNPLUG*** is lit.
b.2	**WARNING:** Wear protective glasses when working on pressurized systems! Do not stand in the direction of any high pressure tube or valve of the He steel-cylinder. Close the **main valve** on the He steel-cylinder but do not change the He Regulator setting.
b.3	Detach the He Hose from the CryoCooling Unit. Because of the self-sealing connector, you have to release the remaining pressure from the He Hose. To do that attach the **Dump Tool** to the He Hose. You will hear a hiss from the escaping helium. Wait until all pressure is released.
b.4	Verify that the secondary pressure has dropped to **zero** and remove the Dump Tool.
b.5	**Detach** the He Regulator from the He steel-cylinder but do not change its valve setting.

b.6	Put the **protective cap** onto the He steel-cylinder.
b.7	**Replace** the He steel-cylinder. **CAUTION:** When moving the steel-cylinders, keep them as far as possible from the magnet but always outside the 0.5 mT range.
b.8	Attach the new He steel-cylinder **securely** to a wall, mount the He Regulator, and fix the safety cable of the He Hose at the He steel-cylinder.
b.9	Open the valve on the He steel-cylinder. The primary pressure should read about **200 bar** and the secondary pressure **22-25 bar** as before.
b.10	He leakage test: Close the He steel-cylinder **main valve** again and wait for about ½ h until the pressure has equilibrated across the line. Read the secondary pressure, wait for about 2 h, and read again. If the pressure has dropped, search for leaks with a He detector or an appropriate liquid (see ***"He leakage" on page 81***).

Software update *6.3*

See ***"Load firmware" on page 37*** and ***"Load additional Xwin-nmr software" on page 52*** in the *CryoProbe System Installation Manual*.

Cleaning *6.4*

Cleaning the sample cavity *6.4.1*

The CryoProbe sample cavity is extremely fragile. Even a tiny scratch inside can spoil the CryoProbe performance and entail a major repair action. Preventive cleaning is not recommended - clean only in case of problems.

CAUTION: Do not put any objects or cleaning devices into the sample cavity! In particular, soft cotton buds must not be introduced under any circumstances - the CryoProbe cavity would almost inevitably be damaged!

If dirt or liquid must be removed from the sample cavity, follow the procedure given in ***Table 6.4.*** below.

Table 6.4. Clean the CryoProbe sample cavity

step	action
k.1	**Remove** the CryoProbe from the magnet and observe all handling precautions.

k.2	Put the CryoProbe **upside down** onto the edge of a level surface, e.g. a table, such that it cannot fall down. Its tube must point down but without touching anything.
k.3	Protect your eyes with **goggles**.
k.4	Connect the **VT gas** to its regular input at the CryoProbe bottom and select a flow rate ≥ **1000 ℓ/h** in `edte`.
k.5	If some debris or liquid is trapped inside the sample cavity, **flush** it out with jets of (1st) **water** and (2nd) **alcohol**. Use a syringe or a wash-bottle which you direct from below into the sample cavity. **CAUTION:** Do not immerse in alcohol for an extended period of time. Do not use strong solvents like acetone! Take extreme care not to touch the inside of the cavity. Do not flush anything but VT gas through the VT gas channel inside the CryoProbe. Do not reverse the direction of VT gas flow.
k.6	Wait until the VT gas stream has **dried** the entire cavity.
k.7	Set the **VT gas flow** rate back to its previous value and detach the VT gas hose from the CryoProbe.

If this procedure does not solve the problem, contact BRUKER.

Panels and housings 6.4.2

The panels and housings of the CryoProbe System components may be cleaned using a non-fluffy cloth dampened in a mild detergent. Do not use abrasive cleaners, scouring powders, organic solvents (e.g. alcohol or acetone), or any harsh chemicals. Wipe the soap residue off with a clean, damp cloth, then dry with a clean dry cloth. Dyes of printed labels might be delicate and require special attention.

If the He Compressor is of the air-cooled type (P/N O00245), its air-cooler fin must be kept clean.

Scheduled maintenance 6.5

There are two counters for the operating hours in the CryoProbe System. One is on the front of the He Compressor, it is incremented only while the He Compressor is actively running. The more important counter is located on the CryoCooling Unit backpanel. It counts the hours of active system operation, i.e. it starts incrementing when *COOL DOWN* is pressed and it stops counting when *WARM* and *UNPLUG* light up.

Exchange of Coldhead in CryoCooler

Needed every 12 months (recommended) or every 10 000 operating hours. The heart of the CryoPlatform is a Gifford-McMahon cycle refrigerator which is driven by the He Compressor. Due to its mechanical principle, this system requires periodic maintenance by a specialist. The maintenance actions will take two days.

NOTE: If more than 10 000 operating hours elapsed on the CryoCooling Unit's counter since the last regular service, some parts may have worn out seriously. An in-depth inspection and service will be required that takes more than two days.

IMPORTANT: If no scheduled maintenance is carried out, individual components will eventually fail due to aging or wear-out, and they may even cause substantial damage to other parts. However, under virtually no circumstances a hazard for the user would be present.

Exchange of Adsorber in He Compressor

Needed every 20 000 h. Since a specialist must visit each 10 000 h anyhow for the Coldhead exchange, Adsorber and Coldhead exchange can be combined.

Troubleshooting 7

See also the *Troubleshooting* chapter in the *Installation Manual*.

Read ***"Safety" on page 7*** before manipulating the CryoProbe System!

WARNING: Do not manipulate tubes, valves, or the like on a working CryoProbe System. The helium gas pressure in the running system can go up to 30 bar (above this, the safety valves will release excess pressure). A significant gas pressure of about 17.5 bar may be present in some system components even when warm, switched off, and disconnected.

CAUTION: Do not open the CryoProbe! Without appropriate tools, the CryoProbe can easily be damaged in an attempt to open it.

There are no repair actions that could be performed by the user.

For troubleshooting, there is usually no advantage in taking the CryoProbe out of the magnet except for those cases where it is obviously necessary, e.g. to remove dirt from the sample cavity.

Emergency procedures 7.1

Emergency Off 7.1.1

Main switch on CryoCooling Unit front

The rotary main switch on the CryoCooling Unit front (see ***"Control indicators on the CryoCooling Unit front" on page 19***) acts as an '***EMERGENCY OFF***' for the whole CryoProbe System. Its major consequence is that all units inside the CryoCooling Unit are disconnected from mains.

When to use

If time permits,

- check the system messages in the ***CryoTool*** on the laptop. Usually, the CryoController handles errors and there may be no need for an *EMERGENCY OFF*.
- consider if pressing *WARM UP* or *COOL DOWN* could resolve the situation.

Imaginable situations for an *'EMERGENCY OFF'* are earthquakes, open gas leaks, intimidating noise from pumps or compressors, a series of pneumatic bangs, or obvious malfunctioning of the CryoController.

Consequences

Irrespective of the actual state of the CryoProbe System, an *EMERGENCY OFF* will take it to a safe and stable state (see ***Table 7.1.*** for details). Cold and vacuum are preserved to allow for a fast resumption of the original task when the system is powered-up again. The pressurized helium gas (He) in the CryoCooling Unit and the Cryo Probe is dumped upon power-off but pressure is maintained inside the He Compressor and the Flexlines. If the system is kept OFF, it will slowly warm up due to passive thermal conduction.

Table 7.1. Consequences of an EMERGENCY OFF

device	state	potential damage	status at power-up	next action after power-up
CryoProbe	DC supply preserved (it is supplied from HPPR CRP); pulse transmission and detection continues but will be unstable due to warm-up; sensors off	none	continue	continue
Sample temperature control	VT gas flow continues; VT heater off	none	VT gas flow continues, VT heater off	operator must re-enable VT heater
CryoCooler	immediate stop; all valves go to safe default positions; He dumped from CryoCooling Unit and CryoProbe; stable	none	default	CryoController decides
He supply	valve between He Hose and CryoCooling Unit closed; stable	none	closed	CryoController decides
Vacuum system	immediate stop; vacuum preserved; stable	none	off	CryoController decides
CryoController	immediate stop	none; logfile interrupted	booting	analyses system state and decides about next action
Laptop or workstation	continue	none	continue	continue
He Compressor	immediate stop; stand-by; He pressure is kept in He Compressor and Flexlines; stable	none	stand-by	CryoController decides
Water chiller (optional)	continue	none	continue	continue
AVANCE cabinet and HPPR CRP	continue	none; no data lost	continue	continue

External emergency button (optional)

On the back panel, there is a socket labeled REMOTE SWITCH which can be connected to an optional remote 'emergency' button. The consequences of interrupting this circuit loop are identical to a mains power OFF. See *Installation Manual* ***"Remote Emergency button" on page 59*** for installation hints.

First diagnosis 7.2

Before you call BRUKER, or if you have no idea how to address the problem, answer the following questions (see also "CryoProbe service" on page 87):

- which errors or warnings appear?
- what are the readings of the pressure gauges etc.?
- which parts seem to be ok/not ok?
- how did the failure occur and what happened before the failure?
- which experiment did you run or intend to do?
- is the sample special, did you do a similar NMR measurement on the sample before either with the CryoProbe, with a conventional probe, or on another spectrometer?

Simple checks:

- try to reproduce a standard spectrum you successfully recorded not long ago with the same sample and identical experimental parameters.
- replace the CryoProbe with a conventional probe and run the experiment again.

Logfile 7.2.1

On the laptop, a logfile is continuously updated with all system messages. It resides in the default directory `D:\app\Bruker\CryoTool\LogFiles`.

Fuses and reset buttons 7.3

CryoCooling Unit

Automatic fuses are located on the back panel along with a melting fuse (5x20 mm, 50 mA).

To reset an *ERROR* on the CryoCooling Unit front panel, press *COOL DOWN* if the system is already cold or cooling down. If the CryoProbe System is warm or warming up, press *WARM UP*'.

He Compressor

See the schematics in the He Compressor operation manual for the location of the fuses. The manual is delivered with the CryoProbe System.

HPPR CRP

There is a red reset button on the rear of the cover module

General failure 7.4

If the displays are erratic, blank, or unresponsive, this suggests a general failure of the CryoController, probably resulting from a problem in the power supply or in the CPU circuit.

1. Check the connections and cables between the units.
2. Reboot the CryoController with the rotary main switch on the CryoCooling Unit front (this is the same as an ***"Emergency Off" on page 71***).
3. Check the fuses.

No mains 7.4.1

Is there a plug in the REMOTE SWITCH socket at the CryoCooling Unit rear panel? The plug short-circuits the two pins of the socket. If the plug is missing or if the short inside is broken, the mains power of the whole CryoCooling Unit is inhibited.

Check ***"Fuses and reset buttons" on page 73***.

Spinning problems 7.4.2

The circumstances of a sample spinning problem restrict the choice of possible causes. Go sequentially through the following list, it is organized by increasing complexity and decreasing likelihood of the fault.

During operation

Is the pneumatic gas supply for sample spinning still ok? Check its input pressure at the rear of the spectrometer cabinet.

Is the VT gas flow rate too high such that it lifts the sample?

Eject the sample and check if it is ok.

Has the vertical alignment of the magnet changed? Check with a water-level.

After insertion of a new sample

Try another sample or another spinner. Check if a high quality sample tube (e.g. a BRUKER standard sample) in a high quality spinner can be spun.

Check the CryoProbe cavity for dirt and clean it if necessary (see ***"Cleaning" on page 67***).

When the CryoProbe had been removed and was mounted again

Is the CryoProbe at the correct position inside the shim system, i.e. is there a gap of ~0.5 mm between the CryoProbe body and the shim system bottom plate? Is the shim system bottom plate fixed firmly? Try another probe: Is the same unusual gap present? Had the shim system been removed some time ago?

If the gap is larger, do the five screws in the Mounting Hardware fail to support the CryoProbe? If they are ok, lower the CryoProbe by a few mm and re-insert it again. If this doesn't help, inspect the CryoProbe top and the shim system for dirt or damage.

If there is no gap, check if the CryoProbe's circumferential top edge touches the blue spinner stator: take the CryoProbe out and put a soft non-magnetic ring onto its top, e.g. an o-ring or a ring made of cardboard. The ring should have a well-defined height and an outer diameter of less than 38 mm. Insert the CryoProbe. Measure the new gap and subtract the height of the ring. The missing length between CryoProbe and spinner stator results. If it is in the order of 1 mm or more, call your BRUKER service office

Indicator on CryoProbe sticks out 7.4.3

In the upper right corner of the front plate, there is a little hole with an indicator inside. This indicator must not stick out but be flush with the CryoProbe front. If the indicator sticks out, a problem with the vacuum insulation may have occurred - contact BRUKER! Do not try to move the screw, neither in/out nor by rotation. When cooling-down the CryoProbe, this indicator will move in.

CryoController failure 7.4.4

Reboot the CryoController via the mains *OFF/ON* on CryoCooling Unit front. See ***"Emergency Off" on page 71*** for the consequences of a mains *OFF/ON*.

The CryoController inside the CryoCooler cabinet has an overheat protection.

Leak in He supply 7.4.5

If the He Regulator or the He Hose between He steel-cylinder and CryoCooling Unit are accidentally damaged, close the He steel-cylinder main valve immediately, then close the He Regulator. If time permits, watch the pressure gauges and let the remaining He gas escape before closing the He Regulator.

WARNING: If a large quantity of helium gas escapes from the He steel-cylinder during a short period, there is a danger of suffocation, particularly in small rooms. Moreover, high local concentrations of helium gas can penetrate the seals of an NMR magnet, thereby spoiling its vacuum insulation and eventually lead to a quench. Care for good ventilation and fresh air supply after an accidental release of large quantities of helium gas.

Cool-down problems 7.5

COOL DOWN *command is ignored* 7.5.1

A cool-down can only be initiated when the whole CryoProbe System is at room temperature. If a passive warm-up has occurred a few hours ago, the system may not be completely warm yet. In this case, the ***CryoTool*** will display *`Waiting for system to passively warm up.`*

Insufficient vacuum 7.5.2

If the CryoController reports `Insufficient vacuum` and stops the vacuum pumps, or if the vacuum pumps sound strange,

- the vacuum bellows may not be properly connected at the CryoProbe or at the Transferline Support,
- the Vacuum Adapter may not be locked tightly to the CryoProbe bottom or its o-ring might be bad,
- the Vacuum Plug may sit inside the CryoProbe instead of being drawn out with the Vacuum Adapter,
- the o-rings in the vacuum bellows connections might be bad,
- if you hear a squeal sound that comes and goes, the turbo vacuum pump may not come to speed.

General vacuum test

Press *COOL DOWN* and wait for diagnostic messages.

He Compressor does not run 7.5.3

The He Compressor is started approx. 15 min after a cool-down was initiated. At the same moment, *COOL DOWN* will stop flashing and remains steady.

Table 7.2. Troubleshooting the He Compressor

Symptom	Possible cause	Elimination
MAIN POWER and *DRIVE* remain *ON* when switched but the He Compressor does not start after He flushing is finished.	1. No mains power. 2. Abnormal room temperature.	1. Check mains wiring, fuses, and circuit breakers. 2. Maintain room temperature at 5° - 35°C.
MAIN POWER remains *ON* but *DRIVE* flips back to *OFF*.	1. Abnormal static He pressure.	1. Adjust He supply pressure.
He Compressor stops after several minutes of operation and *DRIVE* goes *OFF*.	1. Abnormal operating He pressure. 2. air-cooled only: Insufficient cooling of compressor operating room triggers thermal protection. 3. air-cooled only: Air flow for He Compressor is blocked. 4. air-cooled only: Air flow through the He Compressor unit is blocked by dirty fin. 5. air-cooled only: Fan does not operate. 6. water-cooled only: Insufficient or no water cooling triggers the thermal protection.	1. Adjust He supply pressure or charge He as required. 2. Check room temperature and cooling system of compressor operating room. 3. Check the space around the He Compressor. 4. Clean the heat exchanger fin. 5. Check fuses (see ***"Fuses and reset buttons" on page 73***), if OK then replace fan. 6. Confirm that cooling water is flowing at the proper flow rate.

Table 7.2. Troubleshooting the He Compressor (Forts.)

Symptom	Possible cause	Elimination
He Compressor has shut down but the switches remain *ON*.	1. No mains power. 2. Abnormal operating He pressure. 3. Abnormal He pressure due to gas leak. 4. Abnormally high discharge He temperature due to high room temperature. 5. Abnormally high discharge He temperature due to failure of solenoid valve or high pressure switch.	1. Check fuses and circuit breakers, see ***"Fuses and reset buttons" on page 73***. 2. Adjust He supply pressure or charge He as required. 3. Check He pressure, charge He, look for leak (see ***"He leakage" on page 81***). 4. Check room temperature and cooling system of operating room. 5. Restart the unit and check the pressure. If it is abnormal, check solenoid valve or high pressure switch.

Cool-down doesn't reach cold state 7.5.4

The cool-down procedure must have reached the cold state within 4 h, otherwise a warm-up is initiated automatically. After the system has warmed-up completely, press COOL DOWN again.

Did the message `Warm-up needed soon` appear recently? If yes, see ***"Warm-up needed soon request" on page 63***.

Cold operation problems 7.6

COLD flashes 7.6.1

It may happen occasionally that *COLD* starts flashing during an experiment. Just continue the experiment and keep an eye on the temperature readings. The data quality is usually not affected.

Emergency warm-up 7.6.2

There are two kinds of emergency warm-ups:

In an **active** emergency warm-up, He is circulated through the system by the He Compressor. The Coldhead is disabled then and no periodic hiss is audible.

During a **passive** emergency warm-up, all units except for the supervising Cryo-Controller are switched off. The system is warmed up by thermal conduction only which typically takes overnight. Note that a cool-down attempt will be refused until all parts have reached ambient temperature unless the system has been in the passive emergency warm-up only for a very short period of time.

The following events will cause an emergency warm-up:

Active emergency warm-up

- Overheating due to excessive RF power. Stop the NMR experiment, press *COOL DOWN*, and modify the experimental parameters such that the average RF power is reduced (see *User Manual*, ***"Average RF power" on page 59*** for more details).
- Loss of vacuum. Check for a vacuum leak.

Passive emergency warm-up

- Loss of He pressure. Check for a He leak.
- Cooling water supply interrupted for a while (water-cooled He Compressor only). When the cooling water is back, press *COOL DOWN*. If this fails, the temperatures within the system are already too high to re-initiate a *COOL DOWN*. In this case wait until *UNPLUG* lights up. Such a passive emergency warm-up typically takes overnight.
- Fault in the CryoController. An internal watchdog will shutdown the unit within a few seconds if the CryoController doesn't respond anymore. Reboot with a CryoCooling Unit mains *OFF/ON.*
- Overheating of the CryoController when the CryoController fan fails. In this case, an internal overheat protection shuts down the whole unit. After a while, the CryoController can be rebooted (via CryoCooling Unit mains *OFF/ON*).

CryoProbe temperature unstable 7.6.3

Extensive decoupling or trim pulses can heat up the CryoProbe and eventually even cause an emergency warm-up. Try to reduce RF powers, durations, or use alternative pulse sequences if possible.

When calling BRUKER for advice, make sure to have information about the pulse sequence, experimental parameters, and sample properties at hand.

Condensed water or ice on the CryoProbe 7.6.4

Since the CryoProbe housing is somewhat colder than room temperature in normal operation, some water may condense in environments with high air humidity. Under these circumstances, condensed water would also be found at the magnet's gas outlets. Check your air conditioning.

Condensed water may also appear if a VT gas cooler, e.g. a BCU05, is employed with a VT hose that is too short. In this case, ice accumulates at the VT gas entry of the CryoProbe. Switch *OFF* the VT gas cooler, warm-up the CryoProbe to ambient temperature, and extend the VT hose such that there is no ice on the CryoProbe end of the VT hose.

Ice on the CryoProbe housing indicates a problem with the vacuum isolation. In this case, the CryoProbe System will be automatically warmed up by the CryoController. Since on-site repair of a CryoProbe is impossible, it must be sent to BRUKER. Send also the latest logfile (see ***"Logfile" on page 73***) via e-mail because it facilitates problem analysis and thus speeds-up the repair process considerably.

CAUTION: If a CryoProbe inside the magnet has ice on it and automatic warm-up is not already in progress, initiate a warm-up immediately! The magnet bore must be protected against low temperature exposure. A cold magnet vacuum seal may become leaky and the magnet could quench. However, do not try to accelerate the CryoProbe warm-up with e.g. a heat gun!

Sample temperature problems 7.6.5

IMPORTANT: Do not interrupt the VT unit or its gas flow at any time while the CryoProbe is cold. The sample may cool down to cryogenic temperatures, potentially breaking the sample tube or damaging the substance under investigation. VT gas must flow through the CryoProbe at all times even if no sample is present to avoid water condensation.

Sample temperature doesn't reach preset value

- too cold: verify that the VT heater is *ON*. Increase the VT gas flow or the VT heater power limit but make sure the sample is not lifted.
- too warm: increase the VT gas flow (see minimum flow rate on the 'LIMITATIONS - WARNINGS' sheet) but make sure the sample is not lifted.

Sample temperature unstable

- Is the VT gas flow sufficient and stable (see minimum flow rate on the 'LIMITATIONS - WARNINGS' sheet)?
- Is the *HIGH/LOW* setting on the VT Interface Box appropriate for the desired temperature range (see ***"Sample temperature control" on page 55***)?
- If a B-VT3000 is available, use its self-tune feature. Obey the limits given on the 'LIMITATIONS - WARNINGS' sheet.
- Is a B-VT3300 or B-VT3200 employed? These units are not recommended because their temperature regulation is more coarse than that of a B-VT3000, thus small artifacts could occur in temperature-sensitive experiments. The self-tune feature on a B-VT3300 does not work properly with a CryoProbe.

Unusual sounds 7.6.6

Is the characteristic periodic noise from the Coldhead missing although the He Compressor is active? Turn *OFF* the main switch at the CryoCooling Unit front and call BRUKER.

A mechanical blockage, broken or false wiring of the Coldhead power cable between CryoCooling Unit and He Compressor could be the cause. Also, the fuses of the DC 24 V control circuit inside the He Compressor might be blown.

Mains power interrupt 7.6.7

In case of a complete mains power breakdown, all valves in the CryoProbe System will switch back to their default positions such that no over-pressure can develop anywhere in the system. In the absence of active cooling, all cold parts will passively warm up to ambient temperature, typically overnight.

NOTE: After a passive warm-up, a *COOL DOWN* request will be accepted by the CryoController only if the entire system had reached room temperature before.

When the mains power comes back, the CryoController analyses the actual state of the system and decides if the system will be warmed-up and set into stand-by mode or if the cryo-operation conditions are to be re-established. The latter decision will be appreciated especially if a power failure occurs outside the working hours. When the user returns to the NMR lab, the CryoProbe System will be already on its way to re-establish measuring conditions. The NMR acquisition, however, is controlled by the CCU in the spectrometer cabinet and will not be re-initiated automatically for safety reasons.

The system will cool down again if conditions permit. However, this will work only when the temperature has not exceeded a certain value. If the system had become too warm, it will not automatically cool down again but has to be warmed-up. Press *WARM UP* to complete the warm-up cycle in a controlled way, wait until ambient temperature is established, then *COOL DOWN* again.

Mains interrupt on individual units

NOTE: As a troubleshooting tool, *UNPLUG* can be pressed at any time. This permits to disconnect any control cable in the CryoProbe System for about 1 min without causing an *ERROR*.

These are the consequences of a power cut on individual CryoProbe System components during operation:

CryoProbe: An interrupt of the cable between the CryoCooling Unit and the CryoProbe connector J2 would usually cause no damage. The CryoController will send an error and initialize an emergency warm-up. The emergency warm-up can then be terminated and cold operation continued by pressing *COOL DOWN*.

An interrupt of the electronics supply cable between the HPPR CRP and the CryoProbe connector J1 will temporarily disable the cold electronics, leading to an almost vanishing NMR signal. Besides this, however, it will have no impact on the overall cryogenic operation.

CryoCooling Unit: A mains power cut is equivalent to an ***"Emergency Off" on page 71*** and would normally not cause any damage. Upon return of power, the CryoCooling Unit reboots and - depending on its condition - it may even decide to continue with its last task.

VT unit: A B-VT3x00 unit will stop the VT gas flow and the heater upon a mains interrupt. The heater is not switched on automatically when the power returns. Thus, the sample is in danger of freezing (see ***"VT gas interrupt" on page 81***). A UPS is recommended for them.

AVANCE spectrometer: A power cut would usually cause no damage - except for the workstation harddisk which could crash. The mains supply of an AVANCE cabinet is automatically disabled. Before switching on the spectrometer manually after return of power, set the major loads *OFF* (e.g. the amplifiers) to avoid blowing fuses with an initial current spike. The CryoPlatform, however, is normally not disturbed by a spectrometer shutdown.

Water chiller (optional): A water chiller will usually not be damaged by a power cut, and it will resume its task upon return of electricity. However, when the cooling water supply is stopped, the He Compressor halts and a passive emergency warm-up will be initiated by the CryoController.

VT gas interrupt 7.6.8

CAUTION: Never allow the VT gas to be interrupted or switched *OFF* while the CryoProbe is cold! If a VT gas interrupt occurs: **eject the sample immediately!**

A VT gas interrupt may not be evident until an ‘overheat’ error of the VT heater occurs or the lock drops - which can be too late for sample solvents with a high melting point like water or benzene. Even if an automatic ‘emergency warm-up’ of the CryoProbe System were initiated, it would be too slow to prevent these samples from freezing.

Cooling water leakage 7.6.9

This section applies only if a water-cooled He Compressor is used.

If a closed-cycle water chiller is used: determine the cause of leakage before refilling the chiller.

If a water leak cannot be sealed during operation, the CryoProbe must be warmed up.

If the cooling water supply is **interrupted** or drops such that the heat cannot be removed anymore, the He Compressor and the CryoCooling Unit will stop after a while and perform a passive emergency warm-up. Upon return of the cooling water, press *COOL DOWN* to continue cold operation or *WARM UP* to terminate.

He leakage 7.6.10

WARNING: A leak in a pressurized gas system indicates a potential hazard. Before you do anything to fix the problem: Put on protective glasses and gloves! See also ***"Pressurized cold helium gas cycle" on page 9***.

If a He loss exceeds the equivalent of about one recharge/week, the CryoController will stop the system automatically.

He steel-cylinder empty 7.6.11

CAUTION: The He steel-cylinder cannot be exchanged while the CryoProbe is in cold operation because the He system would be inevitably contaminated by air. For the same reason, do not try to recharge He at the He Compressor back while the system is in cold operation.

Warm-up the CryoProbe and proceed according to ***"NOTE: The ‘Elapsed Time’ and the ‘Main pressure’ as of Table 6.2. cannot be recorded in the automatic logfile. Thus, your log-book and the logfile will contain important complementary information." on page 66***.

Pneumatic gas pressure too low 7.6.12

If the pneumatic gas pressure drops to below the minimum specified value, the correct operation of the valves is no longer assured. If an undefined valve configuration appears and some system parameter exceeds its allowed range, the CryoController will automatically warm-up or even shut-down the system.

'Shot' noise 7.6.13

Don't panic - it is just an overpressure relief valve that opens for your safety! There is no need to evacuate the lab. Protect your ears and **turn the main switch on the CryoCooling Unit front *OFF***.

A 'single-shot' occurs if the He flow is somewhat blocked while the He Compressor continues to pump, whereas 'machine-gun' noise is due to a complete blockage. Possible causes are an undefined error in the CryoController (which can be reset by a mains *OFF/ON*), blockage due to frozen contaminations, a broken pneumatic valve, or a misset valve due to insufficient pneumatic gas pressure. Each compartment in the system is protected by a relief valve.

How to proceed after power down

Switch *ON* again after a few seconds.

> **In case the CryoProbe System had been cold** or was almost cooled down, the CryoController may decide to continue cooling and start the He Compressor automatically. If the 'machine-gun' noise comes on again, press *WARM UP*. The system will pause, check its temperature, reconfigure the He flow internally, and start the He Compressor automatically. If the 'machine-gun' noise returns, turn the mains *OFF* and let the system warm up passively overnight. When the system is completely warm, it will accept a new *COOL DOWN* request (but it will refuse to cool down as long as it has not reached room temperature!).

Initiate a new *COOL DOWN*. Report any malfunction during subsequent cool-down preparation, cool-down process, or cold operation to BRUKER. If the problem persists, turn the system *OFF* and let it warm up. Close the He steel-cylinder main valve.

See ***"Emergency Off" on page 71*** for details about switching off the mains.

RF problems 7.7

General checks:

- Is the HPPR CRP properly configured with `crpon` (see ***"HPPR CRP configuration" on page 44***)?
- Are all connectors at the right place and in contact? Especially, is the power cable to the CryoProbe properly connected at the back of the HPPR CRP or HPPR/2 cover?
- All RF filters needed for the CryoProbe are already built-in. Do not use extra filters. If it looks as if extra RF filters would be needed to improve the situation, contact BRUKER.
- Are the ribbon cables at the side of the HPPR CRP assembly properly connected? The HPPR CRP cover module can handle up to two modules for X-nuclei (not counting the 2H module) but not more.

- Are the jumpers at the left side of the HPPR CRP modules in place (see ***Figure 4.12.***)?

- Check ***crpwobb*** and ***wobb*** for all selectable channels. Do you get a tunable dip on your display?

CAUTION: A CryoProbe must not be tuned or matched when warm.

- To check whether an RF problem has its origin in the 'cold' preamplifiers inside the CryoProbe, use the conventional 'warm' preamplifier modules of the HPPR CRP assembly instead. Select the 'warm' modules with ***crpoff*** and wobble the CryoProbe before acquiring data.

-- Does the spectrometer work satisfactory with a conventional probe? Check in particular those functions and measurements which seem not to work properly with the CryoProbe, if possible.

Tuning/Matching not OK 7.7.1

CAUTION: A CryoProbe must not be tuned or matched when warm.

CAUTION: Some of the tuning elements are factory-set and have no corresponding actuator in the Tuning Adapter. Do not attempt to change them.

CAUTION: Do not force the actuators beyond their easily recognizable limits (clockwise: stop, counter-clockwise: overrun). Do not challenge the limits of the tuning and matching range if there is no need. Do not use tools other that the Tuning Adapter and the blue Tuning Tool to adjust the tuning or matching.

Execute ***crpwobb*** and ***wobb***: a tunable dip must appear on the display for each channel. This indicates if most of the signal routing to and from the CryoProbe and most of the CryoProbe itself are working.

If wobbling fails, check if a conventional probe can be wobbled.

Resonance does not react on tuning or matching

The little color-coded knobs in the Tuning Adapter may get loose and turn freely. Detach the Tuning Adapter from the CryoProbe and tighten the small nuts which lock the tiny handle to the threaded stick. Put some superglue on the thread to fix the nuts permanently.

Lock problems 7.7.2

No lock signal

If no lock wiggles appear after ***crpon***, execute ***ii*** or ***lock*** to initialize the communication.

Check if the lock channel can be wobbled: Set ***locnuc off*** in XWIN-NMR, establish the correct ^{2}H frequency and RF routing with ***edasp***, execute ***crpwobb***, and run ***wobb***. If wobbling works, the NMR coil, tuning elements, built-in transmit/receive switch, and the 2H HPPR CRP module must be ok but the built-in cold ^{2}H preamplifier is in doubt.

Lock unstable

Any fluctuations in the pneumatic gas or the VT gas supplies can directly affect the lock level. When using nitrogen, any even minute leaks in the VT gas lines will cause a mixing of nitrogen and air. Due to the paramagnetic susceptibility of air (due to its oxygen content), fluctuation in the shimming and consequently in the lock level will result.

Temperature gradients in the sample can also cause lock level instabilities, in particular with acetone-d_6 lock solvent, if they are large enough to affect the lineshape of the lock signal and thereby its maximum intensity. Thus, a sufficient flow of VT gas is mandatory to provide a homogeneous temperature across the sample. Obey the minimum gas flow rate specified on the LIMITATIONS - WARNINGS sheet.

On the other hand, the lock level is quite sensitive to the VT gas flow rate, in particular if a blue plastic spinner is used. (Blue spinners are not recommended because they are light, white plastic spinners are preferred.) Usually, a VT gas flow rate of ≥ 600 ℓ/h is specified on the LIMITATIONS - WARNINGS sheet but 800 ℓ/h may already lift the sample, resulting in an unstable lock level.

Spurious signals 7.7.3

All RF filters needed for the CryoProbe are already built-in. Do not use extra filters. If it looks as if extra RF filters would be needed to improve the situation, contact BRUKER.

Background signals

Background signals are NMR signals of substances that are located somewhere close to the cavity or any part of a tuned probe circuit. Cryogenically cooled probes are more prone to background signals for two reasons: (1) the high signal-to-noise ratio of the receiving system enhances the background by the same factor as the desired signals; (2) the nuclear magnetization of any material increases in proportion to $1/T$ (Curie law) which results in a considerably stronger NMR response from substances located in the cold parts of the resonant circuit.

Consequently, a background virtually cannot be avoided and is usually much larger than for conventional probes. However, it can be removed in most cases by a simple baseline correction (e.g. ***abs***).

Sharp background signals, however, should not be present. Clean the sample cavity with a special procedure (see ***"Cleaning" on page 67***).

Vibration sidebands

If there are vibration sidebands in the spectrum (which are absent in a reference spectrum taken with a conventional probe),

1. check the lock regulation parameters,
2. verify the proper attachment of the He Transferline to the Transferline Support,
3. check if the CryoProbe is tightly fixed to the magnet,
4. check the proper operation of the magnet anti-vibration dampers.

   CAUTION: Before lifting the magnet, make sure that nothing will obstruct the vertical movement of the CryoProbe. In particular: if cranked magnet pillar braces are mounted, the CryoProbe body must fit their gap (see ***Figure 4.5.***).

If the problem persists, contact BRUKER.

Accidental misoperation 7.8

Too much RF power

Check if the probe can still be wobbled.

CAUTION: A CryoProbe must not be tuned or matched when warm.

RF pulsing into a warm CryoProbe

The built-in protection may have handled this, check if the probe can still be wobbled.

Cables or bellows not connected during NMR experiment

This case might have been noticed by the CryoController which would then set disable the CryoProbe System to protect it. Check sample, wobbling, and error messages.

Sample broken

Eject the sample.

Warm-up and unmount CryoProbe. Clean it (see ***"Cleaning the sample cavity" on page 67***, remove debris from shim system and spinner stator (remove shim system only if really necessary; see ***"Replacing the shim system (optional)" on page 24***). Do not remove the blue spinner stator from the shim system! If in doubt, call BRUKER for inspection and repair. Do not attempt to fix any problems at the CryoProbe yourself.

Dirt inside the sample cavity

See ***"Cleaning the sample cavity" on page 67***.

Are evacuation and cool-down still possible?

BRUKER contact 8

Submit your inquiries about CryoProbe sales and service to your local BRUKER representation. Use the following address only if they cannot help you.

CryoProbe information 8.1

CryoProbe information head offices:

BRUKER AG
Probe Department
Industriestrasse 26
CH-8117 Fällanden
Switzerland

phone: ++41-1-825 91 11
fax: ++41-1-825 96 96
e-mail: cryoprobe.info@bruker.ch
www: http://www.bruker.ch

BRUKER Instruments, Inc.
44 Manning Road
Billerica, MA 01821
U.S.A.

phone: ++1-978-667-9580
fax: ++1-978-667-0985
e-mail: sales@nmr.bruker.com
www: http://www.bruker.com

CryoProbe service 8.2

CryoProbe service head offices:

BRUKER AG
Service Department
Industriestrasse 26
CH-8117 Fällanden
Switzerland

phone: ++41-1-825 91 11
fax: ++41-1-825 96 96
e-mail: cryoprobe.service@bruker.ch
www: http://www.bruker.ch

BRUKER Center
BRUKER Instruments, Inc.
15 Fortune Drive
Billerica, MA 01821
U.S.A.

phone: ++1-978-667-9580, then press 2
fax: ++1-978-667-6168
e-mail: center@nmr.bruker.com
www: http://www.bruker.com

Fault report 8.3

Before calling BRUKER service, try to isolate your problem (see ***"First diagnosis" on page 73***). BRUKER will usually request the following information:

- CryoProbe System order number (e.g. *BH025199*), or
- Spectrometer order number (e.g. *ZH056397*),
- Workstation brand and operating system version (e.g. *SGI Indy with IRIX 6.5* or *HP Vectra VLi with WinNT 4.0*),

- XWIN-NMR version including patchlevel (e.g. *XWIN-NMR 2.5 pl4*). To get this information in UNIX, type `patchlevel`; in WinNT 4 click **Start - Programs - Bruker NMR Suite - NMR Suite x.y - Bruker Utilities - Miscellaneous - xwinversion.cmd** and **patchlevel.cmd**),
- CryoController *Firmware* and *CryoTool* version (see the **Cryo Main** window on the CryoProbe System PC, e.g. *`Firmware: 991112, Cryo Main (Sep 06 1999)`*; if the CryoProbe interface is not active, click **Start - Programs - CryoTool - CryoTool** and select **COM1**),
- Actual hardware configuration when the problem appeared,
- Description of NMR experiment and circumstances under which the problem occurred,
- Description of the problem,
- Which error codes were displayed?
- What did you do so far to analyze or fix the problem?
- Which RF power levels did you use? Simultaneous RF transmission on multiple channels?

Send the latest logfile from the CryoProbe System PC to BRUKER via e-mail. It is called `auto<date>.log` and resides in the directory `D:\app\Bruker\CryoTool\LogFiles`.

Related documents

A

The following documents contain further information.

CryoProbe Site Planning Questionnaire

A questionnaire for potential CryoProbe customers about their NMR laboratory and spectrometer. BRUKER needs this information for tailoring the CryoProbe System to the customer's needs and for preparing its installation.

CryoProbe System Site Planning Guide (P/N Z31524)

User guide for planning the installation of a CryoProbe System. It contains specifications, information about compatibility with existing magnets and spectrometers, and site planning examples.

CryoProbe System Site Preparation Manual (P/N Z31553)

This manual accompanies the *Site Preparation Set* which is delivered before other devices are sent. After being installed by the customer, the set provides the infrastructure for the actual CryoPlatform.

CryoProbe data sheets

RF power limits, sample temperature range etc. specific for the actual CryoProbe.

CryoProbe System Installation Manual (P/N Z31555)

Installation and initial setup of a new or relocated CryoProbe System and its optional devices.

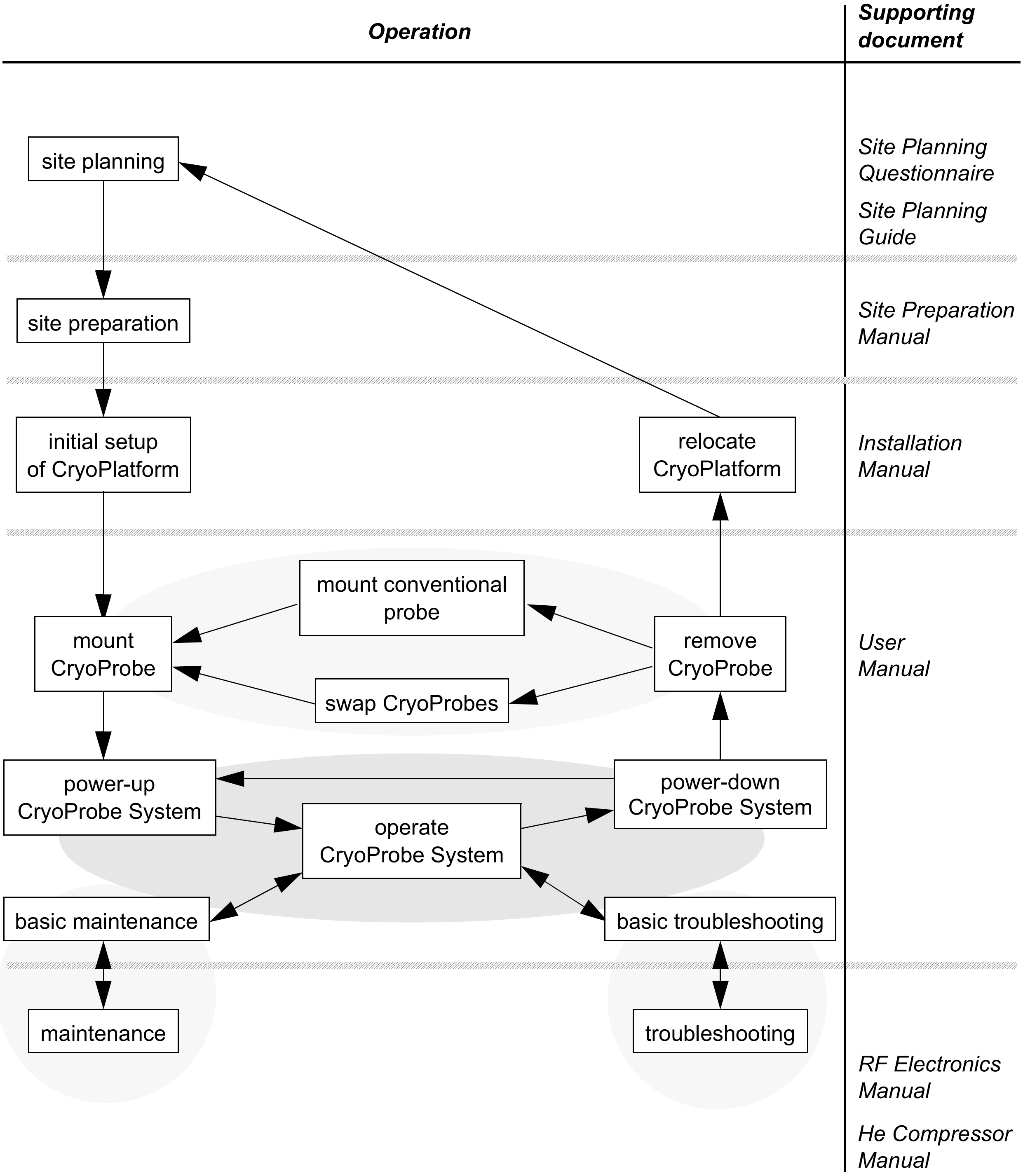
He Compressor technical manual

The operation manual is delivered with the He Compressor.

CRP RF Electronics Technical Manual (P/N Z31474)

Describes the RF wiring between CryoProbe and spectrometer, explains how to configure the HPPR CRP, lists technical data, and contains service information for the preamplifier system.

Figure A.1. When to use which CryoProbe document



Conversion of metric units

B

1 bar ≡ 0.1 MPa

1 bar ≈ 14.5 psi

1 bar ≈ 1.02 kgf/cm^2

1 Pa ≡ 0.01 mbar

1 psi ≡ 68.95 mbar

1 kgf/cm^2 ≈ 0.98 bar

1 kg ≈ 2.2 lb

1 lb ≡ 0.4536 kg

1 mm ≈ 0.04 inch

1 m ≈ 3.28 feet

1 inch ≡ 25.4 mm

1 foot ≡ 0.3048 m

1 Nm ≈ 8.85 lbf-inch

1 lbf-inch ≈ 0.113 Nm

1 ℓ (liter) ≈ 0.264 gallon (U.S.)

1 ℓ (liter) ≈ 0.220 gallon (Brit.)

1 gallon (U.S.) ≈ 3.79 ℓ

1 gallon (Brit.) ≈ 4.55 ℓ

1 kWh ≈ 3.6 MJ

1 kWh ≈ 3412 btu

1 MJ ≈ 0.278 kWh

1 btu ≈ 0.293 Wh

1 mT ≡ 10 Gauss

1 Gauss ≡ 0.1 mT

°C to °F:

$T_{°F} = (T_{°C} \times 1.8) + 32$

°F to °C:

$T_{°C} = (T_{°F} - 32)/1.8$

Table B.1. Conversion between °C and °F temperature scales

°C	-30	-20	-10	0	10	20	30	40	50	60	70	80	90	100	110
°F	-22	-4	14	32	50	68	86	104	122	140	158	176	194	212	230

Frequently asked questions

C

System configuration C.1

Which CryoProbe types are or will be available?

Please ask your local BRUKER representative for the current choice of probes and accessories.

Can the CryoProbe be used with any magnet and spectrometer?

Any standard bore magnet can host a CryoProbe. However, specifications are guaranteed only if the B_0 homogeneity of the magnet suffices (i.e. if it allows to reach specifications with recent conventional BRUKER probes) and if a BOSS-2 shim system is present. One mechanical restriction is given by the clearance below the magnet between shim system and floor that is needed for insertion of the CryoProbe. Another restriction can be vacuum valves, drop-off plates or the like at the magnet dewar bottom. Ask your BRUKER representative for compatibilities.

Since the CryoProbe System must interact with an AVANCE spectrometer, it cannot be connected to other spectrometer types.

How much space is required by the CryoProbe System?

Rough estimate: 6 m^2. If the He Compressor and the optional water chiller are located in an adjacent room, about 4 m^2 suffices. See the ‘CryoProbe Site Planning Guide’ for details.

Can conventional probes still be used on a spectrometer that is equipped with a CryoProbe System?

Yes. A conventional probe can be connected to the spectrometer as usual. However, certain special probes or auxiliary devices might cause geometrical conflicts which can be easily and quickly resolved by removing the guiding rods of the Cryo Probe Mounting Hardware.

Is a VT gas cooler recommended?

The CryoProbe can be operated with and without a VT gas cooler. Currently, only the BCU05 is approved. It is needed for measurements below room temperature and slightly above room temperature (i.e. up to 3 - 5°C higher). A nitrogen evaporator must not be used.

NMR operation C.2

Do any experimental restrictions result from the high Q factor of the CryoProbe?

The Q factor is optimized for each NMR coil assembly to minimize any problems with ring-down times, radiation damping, or excitation bandwidths, still allowing maximum gain in sensitivity.

Is the CryoProbe more sensitive to external disturbances?

Just in proportion to its higher signal-to-noise ratio.

Is it possible to measure water samples?

Yes.

Is it possible to measure samples with a high salt concentration?

Yes, but the usual restrictions apply: the higher electrical conductivity of a salt solution reduces the penetration depth of the RF, spoils the Q factor of the resonance circuit, and introduces additional spectral noise.

Is shimming more difficult?

Manual shimming is not much different from conventional probes. There are no particularly strong shim gradients to be set. Gradient shimming is possible.

Does the user have to modify the pulse programs?

Usually not. Of course, those conventional pulse sequences that are optimized for high signal-to-noise and suppression of spurious signals are still highly recommended.

Which experimental parameters do I have to be careful with to avoid damage to the CryoProbe?

Maximum RF power. In general, a CryoProbe requires significantly less RF power to achieve the same pulse lengths as conventional probes.

Does the CryoProbe change its characteristics during long decoupling or spin-lock periods?

Long decoupling or spin-lock periods tend to warm-up the RF components in any probe. For such experiments, it might be advisable to equilibrate the CryoProbe with dummy scans before data acquisition starts. Significant changes in tuning & matching are not to be expected.

Do experimental parameters like shims and pulse angles change after a warm-up/cool-down cycle? Is the sensitivity preserved?

Parameters like shim, tuning & matching, or pulse angles are constant with minor variations as known from conventional probes. Experience so far indicates that the excellent sensitivity of the CryoProbe does not suffer from repeated warm-up/cool-down cycles if the recommended operation procedures are obeyed.

Is the CryoProbe performance stable and reproducible in the long term?

Experience so far: Yes.

Will the CryoProbe be damaged if the cryocooling is accidentally interrupted during a measurement?

An interrupt in the cryocooling should not break the CryoProbe.

Basic aspects *C.3*

Why does the CryoProbe have such a high signal-to-noise ratio?

Thermal noise is greatly reduced by cooling the NMR coil assembly and the preamplifiers to cryogenic temperatures. Furthermore, the low electrical resistance enhances the Q factor of all resonant circuits in the probe and its filters.

What is inside a CryoProbe?

The CryoProbe contains a tuned NMR coil assembly, a gradient coil, preamplifier electronics, and supervisor electronics inside a vacuum isolated dewar. Cold helium gas is circulated to cool the NMR coil assembly and the preamplifier electronics while the sample is kept at ambient temperature. All the common probe functions needed for RF transmission, tuning, gradient pulses, and VT gas duct are built into the CryoProbe, while the sample lift and spinning are provided as usual by the shim upper part.

What does the NMR coil look like?

The NMR coil assembly generates a transverse B_1-field for the tuned frequencies. Its material, geometry, and associated RF circuits are optimized for each type of probe. Details of the coil assembly design are proprietary knowledge of BRUKER.

What is the helium consumption of the CryoProbe System?

No liquid helium is used whatsoever. Helium gas (He) is needed for the initial fill of the closed-loop system and for flushing the system before each cool-down. During cold operation, the He consumption is negligible. A standard He steel-cylinder (50 ℓ) can last for more than 40 cool-down/warm-up cycles.

Can the user repair any part of the CryoProbe?

No, there are no user-serviceable parts on or inside a CryoProbe. Essential parts of a CryoProbe may easily be broken during opening or closing. Therefore, service actions on the CryoProbe can only be done at the factory. **BRUKER warranty expires if the CryoProbe is opened by unauthorized personnel.**

Glossary

D

Adsorber

Device inside He Compressor that adsorbs oil and other impurities from the circulated helium gas (He).

BBIS

BRUKER **B**oard **I**nformation **S**ystem

Coldhead

Cools down compressed He in a Gifford-McMahon expansion cycle. Its two stages are the primary cooling devices of the CryoCooler.

CryoController

Controls all functions of CryoProbe and CryoPlatform. It communicates with the spectrometer and is located inside the CryoCooling Unit.

CryoCooler

The CryoCooler cools and circulates the cold He. It consists of the Coldhead in a cold box unit and a gas circulation unit with valves and gauges. In contrast, the term 'CryoCooling Unit' denotes the whole cabinet including vacuum system etc.

CryoCooling Unit

A cabinet that contains the CryoCooler, the CryoController, a vacuum system, and the He Transferline. It is labelled 'CryoPlatform' because it is the most prominent part of a CryoPlatform.

CryoCoupler

Standardized interface between the He Transferline from the CryoCooling Unit and the CryoProbe that connects both forward and backward streams of cold He at once.

CryoPlatform

All parts needed for operating a CryoProbe with a spectrometer, i.e. CryoProbe Mounting Hardware, CryoCooling Unit, He Compressor, He Transferline, Transferline Support, VT Interface Box, and optional magnet stand modifications. However, the HPPR CRP, the optional water chiller, and the He steel-cylinder are not parts of the CryoPlatform.

CryoPreamp

A cryogenically cooled preamplifier module inside the CryoProbe housing. There is a frequency-specific preamp module for each channel of a CryoProbe. A CryoPreamp cannot be separated from its CryoProbe. It always requires an additional external HPPR assembly, the cryo-compatible HPPR CRP.

CryoProbe

Although the CryoProbe System is often colloquially referred to as ‘CryoProbe’, this term designates the probe part only.

CryoProbe RF Unit

All CryoPreamps, transmit/receive switches, RF filters for the receiver pathways, and control circuits that are built into the CryoProbe body.

CryoProbe System

A CryoProbe and all components necessary for its operation.

CryoTool

A software interface for monitoring the CryoProbe System parameters. It runs on a separate laptop or PC.

Dump Tool

A short gas tube with a silencer. This service tool is used to release the He supply pressure at the joint between He Regulator and CryoCooling Unit before the He steel-cylinder is exchanged.

Flexlines

A pair of flexible tubes that guide pressurized He at ambient temperature from the He Compressor to the CryoCooling Unit and back. Pressurized He at 15 to 30 bar is kept inside these gas tubes at all times - even when disconnected! They are isolated to reduce thermal disturbances and acoustic noise.

Gradient Filter Box

Small box to interface a standard BRUKER gradient cable to the CryoProbe.

He

Gaseous helium of high purity, used for cryogenic cooling of the CryoProbe.

He Compressor

Warm He from the CryoProbe is routed through the CryoCooling Unit to the He Compressor. The compressed He is sent back to the CryoCooling Unit, circulating in a closed loop.

The He Compressor serves two functions: (1) It provides the primary energy (in form of compressed He) for the cooling action of the CryoCooler. (2) It circulates the He between the CryoCooling Unit and the CryoProbe, providing the transport of ‘the cold’ to the CryoProbe.

He Hose

Flexible hose for pressurized helium gas that connects the He steel-cylinder with the CryoCooling unit.

He Regulator

A pressure reduction valve with two gauges that is mounted on the He steel-cylinder.

He steel-cylinder

Standard helium gas steel-cylinder (50 ℓ) for the initial fill of the CryoProbe System and for flushing the closed-loop He cycle before each cool-down.

He Transferline

Isolated tube through which the cold He from the CryoCooling Unit flows to the CryoProbe. The He Transferline is part of the CryoCooling Unit and cannot be detached from the cabinet. It goes in parallel with the vacuum bellows.

HPPR CRP

Cryo-compatible preamplifier assembly located close to the magnet that is a stack of frequency-specific preamplifier modules, a cover module, and a base plate. Together with the CryoPreamp inside the CryoProbe, the HPPR CRP forms the NMR preamplifier system. Although it looks very similar to a conventional HPPR, its components are modified for interacting with both a CryoProbe or a conventional probe. When operating with a CryoProbe, the HPPR CRP performs the RF filtering in the transmitter pathway, selects the received signal, handles the probe tuning, and supplies the CryoProbe electronics. An HPPR CRP can be used with conventional probes just like a conventional HPPR.

HPPRtool

Software tool on the spectrometer workstation Unix/NT level that interacts with all HPPR types.

Magnet stand pillar braces

Horizontal metal braces that connect the anti-vibration stands of certain BRUKER/SPECTROSPIN magnets. Two braces at the magnet front must be replaced by cranked ones to enlarge the gap for introducing the CryoProbe.

Mounting Hardware

Special assembly that is attached to the magnet bottom to hold the CryoProbe in position.

PICS

Probe **I**dentification and **C**ontrol **S**ystem that transmits probe-specific data to the spectrometer.

Pneumatic gas

Usually compressed air or nitrogen gas for the operation of the pneumatic valves inside the CryoCooling Unit.

Protection Cap

A white plastic cap to protect the CryoProbe sample cavity against dirt during transport, testing, or storage.

Q factor

The **q**uality factor Q is a measure of the efficiency of reactive devices such as inductors, capacitors, or resonant circuits.

Radiator

Outdoor part of the optional water chiller which disposes the waste heat to the atmosphere (the ‘condenser’); equipped with fans.

RF

Radio **f**requency.

Transferline Support

A heavy upright cylinder that supports the He Transferline about halfway between the CryoCooling Unit and the CryoProbe. It also isolates the CryoProbe from mechanical vibrations of the CryoCooling Unit.

Tuning Adapter

Removable assembly of tuning and matching knobs. A VT gas connector is also included. Its geometry depends on the type of CryoProbe.

Tuning Tool

A special blue screwdriver to operate the tuning and matching knobs of a CryoProbe’s Tuning Adapter.

UniTool

Software tool on the CryoProbe System laptop to interact with the CryoController or other units. Started the **Bruker** menu in Windows NT by clicking **UniTool**.

UPS

Uninterruptable **P**ower **S**upply, a kind of battery that compensates for fluctuations and interruptions in the mains.

Vacuum Adapter

Adapter for evacuation of the CryoProbe insulation, connected to its bottom. It features an airtight actuator screw to move the CryoProbe’s Vacuum Plug in and out.

Vacuum Plug

A small metal plug with an o-ring and an inner thread that closes the CryoProbe vacuum chamber against moisture and dirt.

Vacuum bellows

Flexible metal vacuum bellows that connects the CryoProbe isolation to the vacuum system inside the CryoCooling Unit. It is parallel to the He Transferline.

Vacuum system

Vacuum pumps and valves that evacuate the dewar insulations of CryoProbe, He Transferline, and CryoCooler. Located inside the CryoCooling Unit.

VT gas

Usually nitrogen gas or dry air at a controlled **v**ariable **t**emperature that flows through a probe to heat or cool the sample. Its function must not be confused with the ‘pneumatic gas’ used for operating valves inside the CryoCooling Unit or with the helium gas circulated through the CryoProbe for cryogenic cooling.

VT Interface Box

A small box with two cables which interfaces heater and temperature sensor between CryoProbe and VT unit.

VT unit

A device that controls the flow and temperature of the VT gas, e.g. a B-VT3000.

Water chiller

The water-cooled versions of the He Compressor require cooling water to remove 7.5 kW of heat. A water chiller is recommended if no closed cycle cooling water is available in the laboratory building.

‘Split-type’ water chillers are composed of two units: a main unit that pumps and cools the ‘primary’ water cycle by exchanging its heat to a ‘secondary’ cycle, and a radiator unit that fans the heat from the secondary cycle into the air, usually at the outside of the building.

Figures

Tables

End of Document