CryoProbe System

User Manual

Version 2

BRUKER

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Contents

Safety

1

Read these safety instructions carefully and make them accessible to everybody working around 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 20***) 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 controlled by the HPPR. If the system is kept *OFF*, it will slowly warm up due to thermal conduction.

NOTE: Because an *EMERGENCY OFF* also shuts down the monitoring 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 77*** 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 the *WARM* and *UNPLUG* lights are lit on the CryoCooling Unit front panel.

If in doubt: Wear 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'S 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 as the inside edges may be sharp enough to cause injuries.

WARNING: If you have to work with an open CryoCooling Unit cabinet, wear 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. Good ventilation and/or a fresh air supply can remedy this effect.

Overpressure release noise

Overpressure in the system is avoided by software control and mechanical safety valves. In an overpressure situation, the release valves will open with an extremely loud BANG! The sound insulation cabinet will reduce the noise to a safe level, so it is important to always operate with a closed cabinet.

WARNING: If service must be done on an open CryoCooling Unit during normal operation, ear protection must be worn.

Electrical safety

The CryoCooling Unit's degree of protection against electrical hazard complies with IEC IP20: all electrical parts are protected against touching.

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

No hazardous substances

There are virtually no substances in a CryoProbe System that could be hazardous to an NMR user.

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 strip (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 (***Figure 1.8.***), always carry it at its body (***Figure 1.9.***).

- 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. Obey the limitations given on the specific 'LIMITATIONS - WARNINGS' sheet. See also ***"RF power limitations" on page 64***.

Figure 1.8. Never hold a CryoProbe at its tube!

Figure 1.9. Carry a CryoProbe only at its body!

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. The spectroscopic handling is very similar to that of a conventional probe. While the sample temperature is stabilized at a user-defined value around room temperature, the NMR coil assembly - located only 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 where low sample concentrations or short measurement times 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 99***“

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 95***.

CryoProbe System overview 2.2

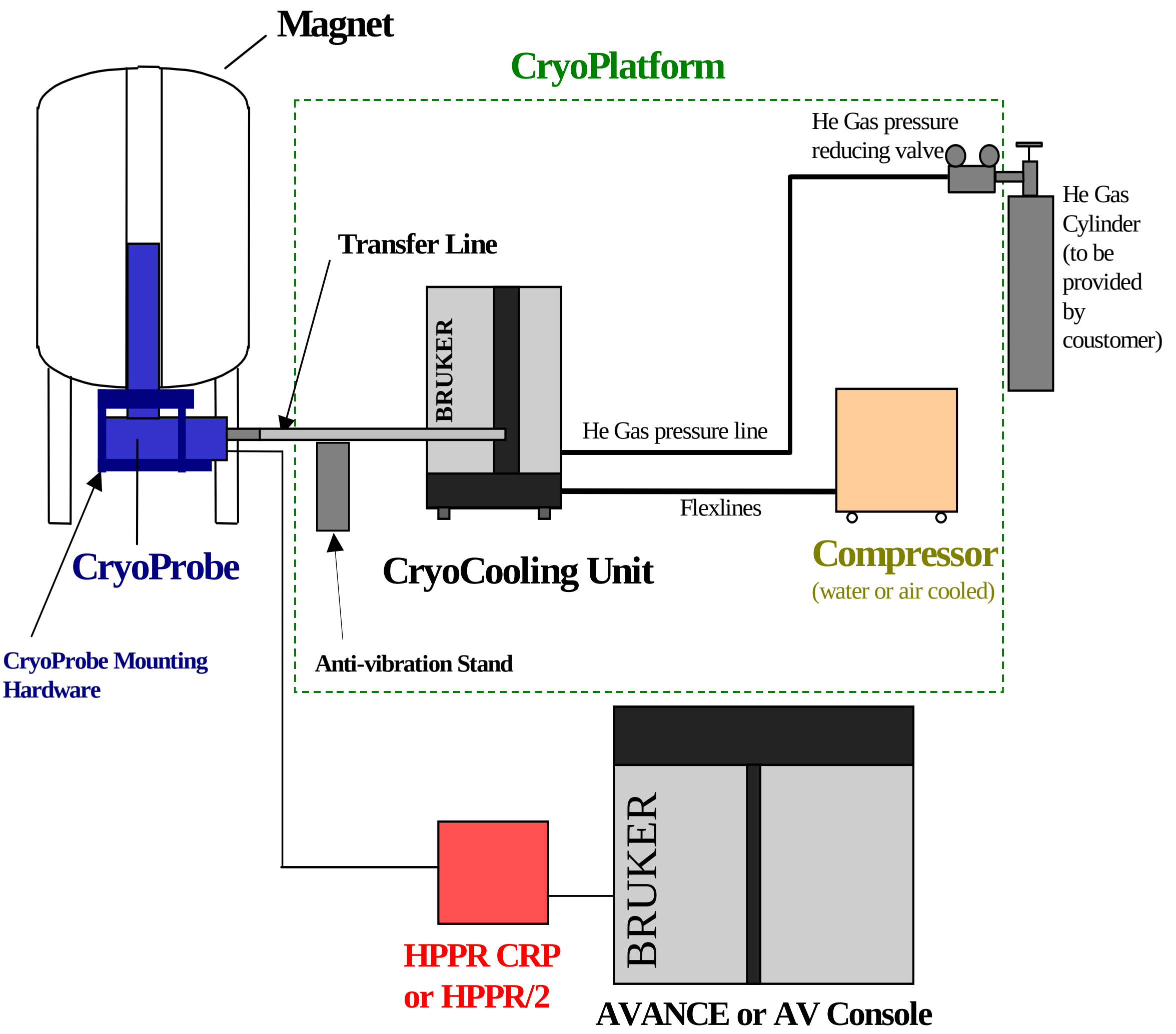
A CryoProbe System consists of several subunits: CryoProbe, CryoPlatform, cryo-compatible HPPR, and He steel-cylinder.

The term ‘CryoPlatform’ summarizes the parts required to operate a CryoProbe and includes 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
`Courier small italic`	system response

Figure 2.1. The CryoProbe™ System



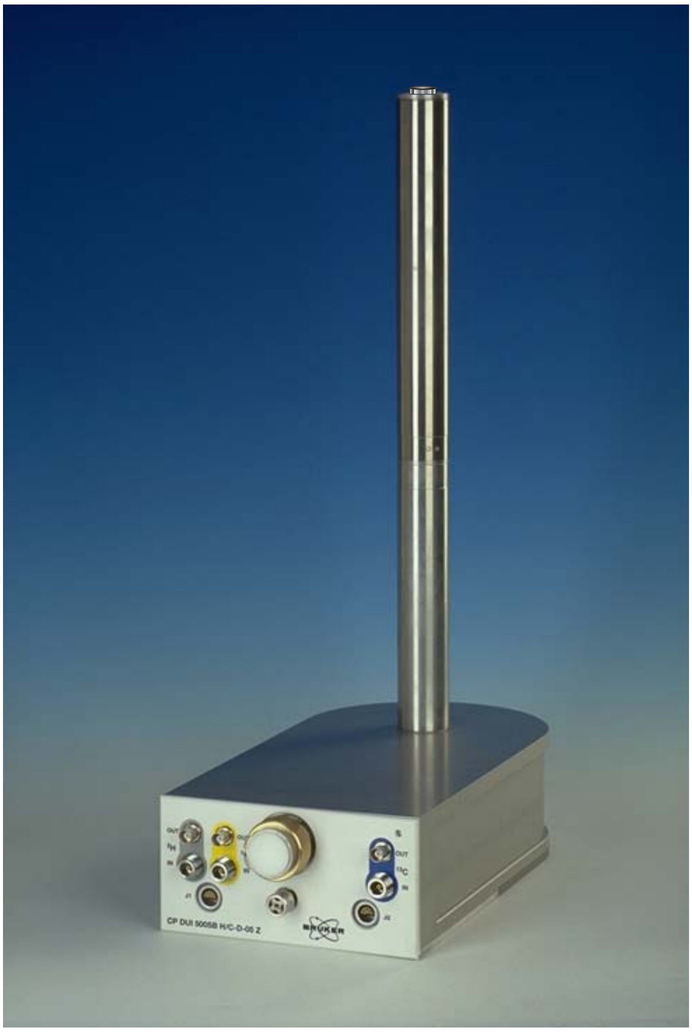
`Courier`	file or directory name
`Courier bold`	Unix™ or Windows™ NT keyboard command
`Courier italic bold`	BRUKER NMR Suite keyboard command
Times bold	Unix or Windows NT object clicked with the mouse
Times italic bold	BRUKER NMR Suite object clicked with the mouse
Times italic	host name, user name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

System description 3

CryoProbe 3.1

The CryoProbe™ is an NMR probehead with integrated preamplifiers. The NMR coil assembly and the Cryo Preamplifier are cooled by cryogenic helium gas (He). The advantage of this cooling is to achieve extremely efficient operation of the NMR coil assembly and to significantly reduce thermal noise. These combined efforts drastically enhance the overall signal-to-noise ratio.

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) 692 mm (700 MHz), 853 mm (800 MHz), 853 mm (900 MHz)
Weight	~12 kg

Sample temperature control 3.1.1

The sample is kept at a temperature within a range specified on the 'LIMITATIONS - WARNINGS' sheet which comes with each individual probe. To keep temperature gradients from developing across the sample volume, the sample temperature is controlled and regulated with a continuous gas flow in conjunction with vacuum insulation. Note the ***"Sample temperature control" on page 61***.

Reproducibility 3.1.2

The performance characteristics of a CryoProbe do not change significantly with time. Warm-up/cool-down cycles have little effect on shim values, tuning & matching, pulse angles, lineshape, or sensitivity. However, it may be advantageous to fine-tune these key parameters after a thermal cycle.

Differences with a conventional probe 3.1.3

Although the CryoProbe is a high-resolution NMR probe, many of its systems are unique in that they have not been used in conjunction with NMR systems 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 requires very careful handling. Since several electronic components (e.g. preamplifier) of the cooling system are included within the body of the CryoProbe, it weighs a great deal more than conventional probes. A special Mounting Hardware is attached directly to the magnet base to support the CryoProbe. **Two people** are needed to lift 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

With regard to sample handling, sample lift, and sample spinning, a CryoProbe is no different than a conventional probe. Samples with a diameter smaller than specified can be used as in conventional probes.

A CryoProbe is tuned and matched like a conventional probe with actuators from below. Depending on the preamplifier (HPPR/2 or the HPPR CRP) installed on the system, tuning commands may differ from those used with conventional probes. Refer to ***"HPPR/2 configuration" on page 47*** or ***"HPPR CRP configuration" on page 50*** for more information. CryoProbes must not be tuned or matched when warm!

In case the Automatic Tuning and Matching adaptor is used, tuning will be carried out by the spectrometer using the command ***atma***.

Established shimming strategies are still valid and no unusual shim settings are to be expected. For best results, ensure that an RCB board / 2H-TX board are configured to perform 3-D gradient shimming on the CryoProbe.

RF power

RF peak and average power must not exceed certain limits. Due to the high efficiency of the CryoProbe, the power limits are lower than those for conventional probes. Power levels for all kinds of RF pulses (hard, soft, shaped, CPD, spinlock...) 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. Refer to the LIMITATIONS - WARNINGS sheet and ‘Customer Certificate’ delivered with each CryoProbe. If available, the document ‘Typical Pulses for the 5 mm CryoProbe’ is another reference.

Preventative measures are available to prevent damage to the probe. If the spectrometer is running with XWIN-NMR 3.5 or higher, Power Check is an option. This program controls the power output with software constraints. NOTE: Perched does not take duty cycle into consideration. Refer to the ‘Typical Pulses for the 5 mm CryoProbe’ manual when it is available.

Length and power of RF pulses are calibrated as usual.

CAUTION: Never exceed the RF power limitations given on the ‘Customer Certificate’.

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

Upgrades and modifications such as the sample protection option are available from BRUKER. However, frequency changes are not possible with the CryoProbe.

Acoustic noise

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

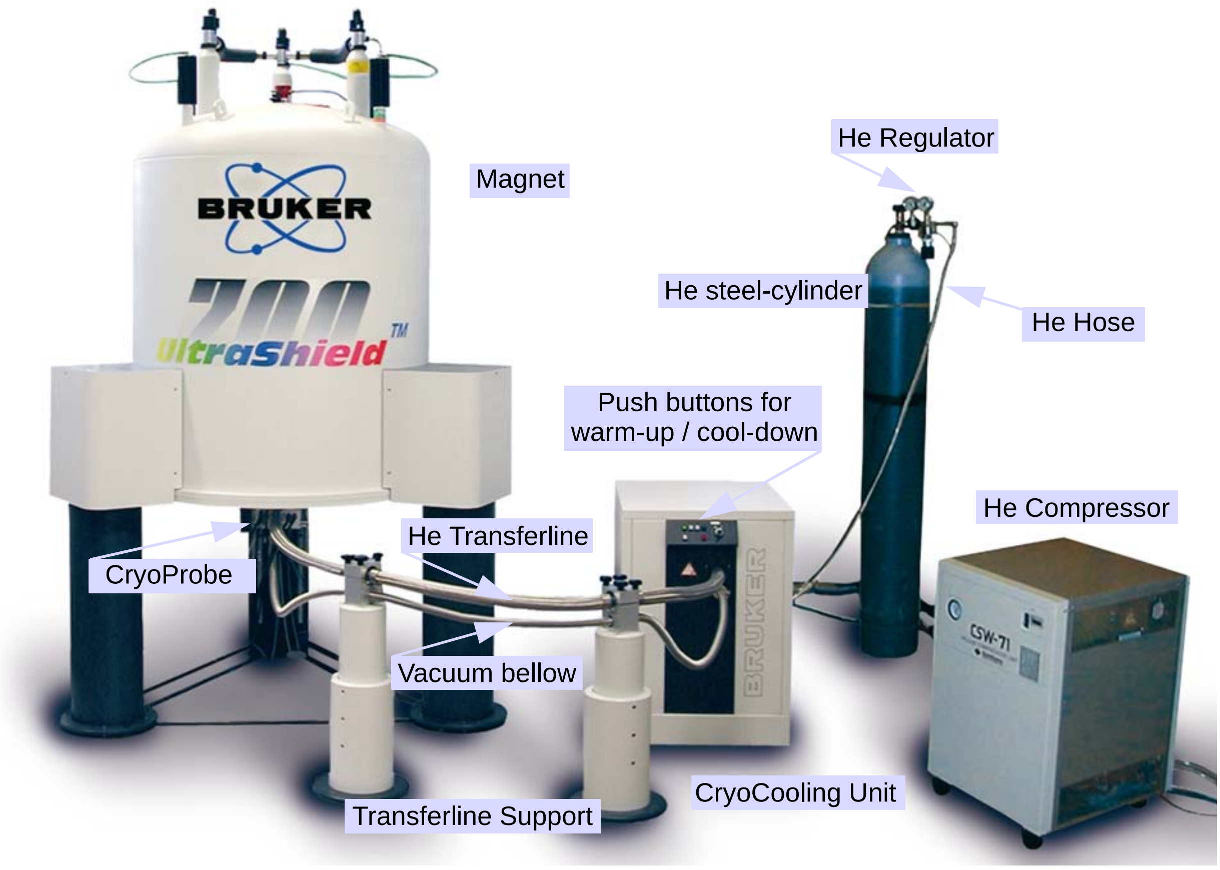
CryoPlatform 3.2

Only one CryoPlatform™ (**Figure 3.2.**, see also **"CryoProbe System overview" on page 13**) is required per spectrometer. It supplies the entire infrastructure for the operation of CryoProbes, i.e. all cooling and all control functions. It is a push-button system which performs all operations needed for a fully automated 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.

The CryoProbe, the cryo-compatible HPPR, the He steel-cylinder, the Gradient Filter Box, and an optional water chiller are not considered as part of the 'Cryo-Platform'.

Figure 3.2. A CryoPlatform™



Mounting Hardware — *3.2.1*

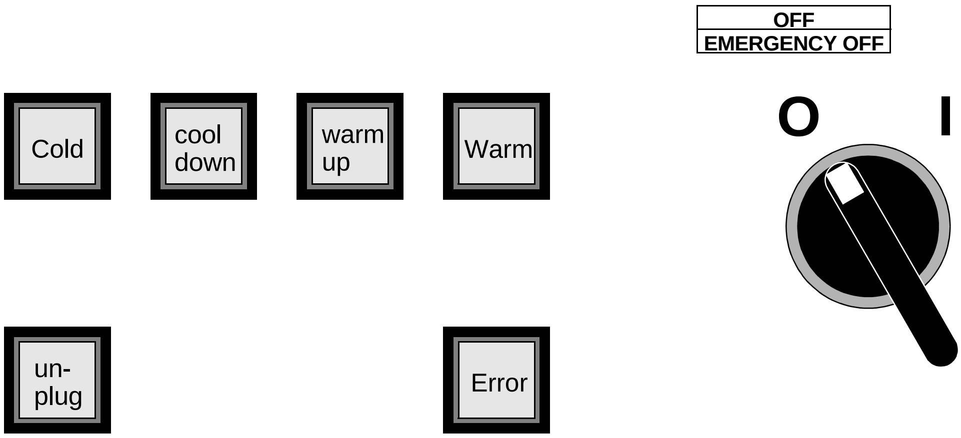
A special fixture must be mounted to the bottom flange of the magnet to carry the weight of the CryoProbe. The Mounting Hardware is attached to the magnet flange with an interface plate which replaces the lower shim system attachment ring. When not used for CryoProbe mounting, this plate does not interfere with conventional probes. However, the support rods may need to be unscrewed from the plate in order to install a conventional probe.

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



All buttons and indicators may illuminate to give a status display.

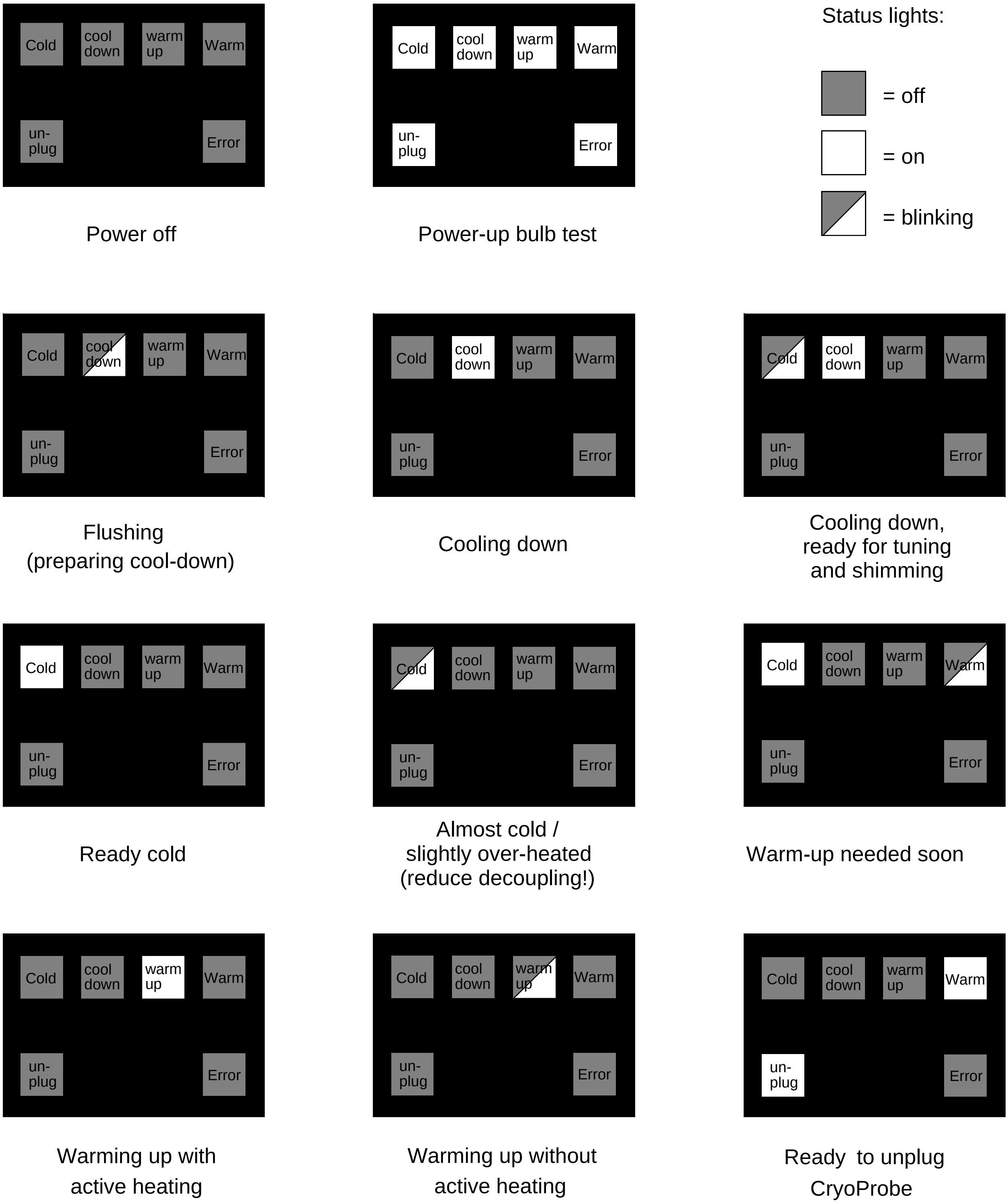
The buttons ‘cool down’ and ‘warm up’ can be pressed for input.

Mains switch

‘*EMERGENCY OFF*’

Status display by the control buttons

Figure 3.4. Status display by the control buttons



ERROR reset

ERROR may light up in any of the above situations (***Figure 3.4.***). 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* LIGHT. In case of an *ERROR* please consult also the chapter ***"Troubleshooting" on page 77***.

LED test

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

Technical data: CryoCooling Unit

Currently there are two generations of CryoCooling Units. However, due to its smaller size, only the Type II cooling unit is being delivered at this time. Refer to ***Table 3.1.*** for a comparison of the technical data.

Table 3.1.Comparing CryoCooling Units I and II

	CryoCooling Unit I	CryoCooling Unit II
Dimensions [w x d x h]	800x720x1300 mm	680 x 890 x 960 mm
Weight	400 kg	400 kg
Electricity	AC 230 V (1 Phase) 50/60 Hz, 500 W average, 1500 W peak, required external fuse upstream: T 10 A (Time-lag fuse)	
Air	>4.5 bar	
Acoustic noise	max. 61 dB(A) 2 m distant	

Both CryoCooling Units comply 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. Further explanation is given in ***"Principle of operation" on page 25***.

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 capsule, a heat exchanger, and an adsorber.

CAUTION: Never install a He cylinder having purity less than 99.9999% (Grade 6). This would contaminate the system, rendering it unusable. A batch-tested cylinder is OK.

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)
CSA-71A	(3 × 200VAC, indoor air-cooled)	550 × 550 × 885
CSW-71C	(3 × 200VAC, indoor water-cooled)	450 × 500 × 687
CSW-71D	(3 × 400VAC, indoor water-cooled)	450 × 500 × 687
CNA-61C	(3 × 200VAC, outdoor air-cooled)	910 × 400 × 1050*
CNA-61D	(3 × 400VAC, outdoor air-cooled)	910 × 400 × 1050*

*The indoor portion of the outdoor air-cooled compressor is 270 × 610× 705mm.

Weight (approx.)

CSA-71A	(3 × 200VAC, air-cooled)	140kg
CSW-71C	(3 × 200VAC, water-cooled)	120kg
CSW-71D	(3 × 400VAC, water-cooled)	120kg
CNA-61C	(3 × 200VAC, outdoor air-cooled)	115kg*
CNA-61D	(3 × 400VAC, outdoor air-cooled)	115kg*

*The indoor portion of the outdoor air-cooled compressor weighs 45kg (200V version), or 55kg (400V version), respectively.

Ambient operating temperature of indoor units:	5 to 28°C
Ambient operating temperature of outdoor units:	–30 to +45°C

Electricity

line voltage (± 5%)	CSA-71A	AC 200V, 50/60Hz, 3 phase,
	CSW-71C	AC 200V, 50/60 Hz, 3 phase
	CSW-71D	AC 380 - 415V @ 50Hz, 3 phase,
		AC 460 - 480V @ 60Hz, 3 phase.
	CNA-61C	AC 200V, 50/60Hz, 3 phase
	CNA-61D	AC 380 - 415V @ 50Hz, 3 phase
		AC 460 - 480V @ 60Hz, 3 phase.

Power consumption	maximum	steady state
CSA-71A	8.3kW	7.5kW
CSW-71C	8.3kW	7.5kW
CSW-71D	8.3kW	7.5kW
CNA-61C	9.2kW	8.5kW
CNA-61D	9.2kW	8.5kW

Cooling water requirement (CSW-71C and CSW-71D only)

water flow rate:	4 - 10 L/min. (240 - 600 L/h)
recommended flow rate:	7 L/min. (420 L/h)
typical cooling power:	6.5/7.5 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' 3.3

Although a CryoProbe has its own set of built-in cold preamplifiers, 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 assembly. Besides being suitable for the operation with CryoProbes, this HPPR can be used with all conventional probes. There are two acceptable preamplifiers. The HPPR CRP, shown in ***Figure 3.5.***, is available for 500 and 600 MHz systems. It is mainly used on systems where its full compatibility with DMX or DRX spectrometers is required. The HPPR/2, shown in ***Figure 3.6.***, is used in conjunction with all other suitable spectrometer types. For operational information about these units, refer to sections ***4.10*** and ***4.11***.

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

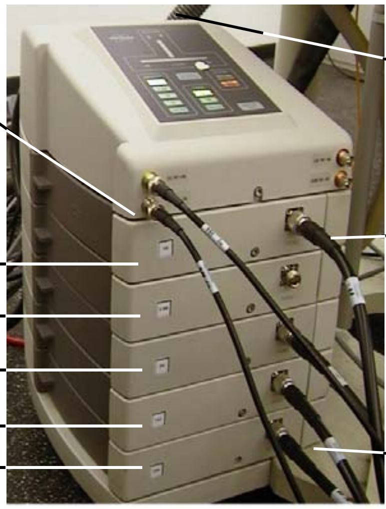
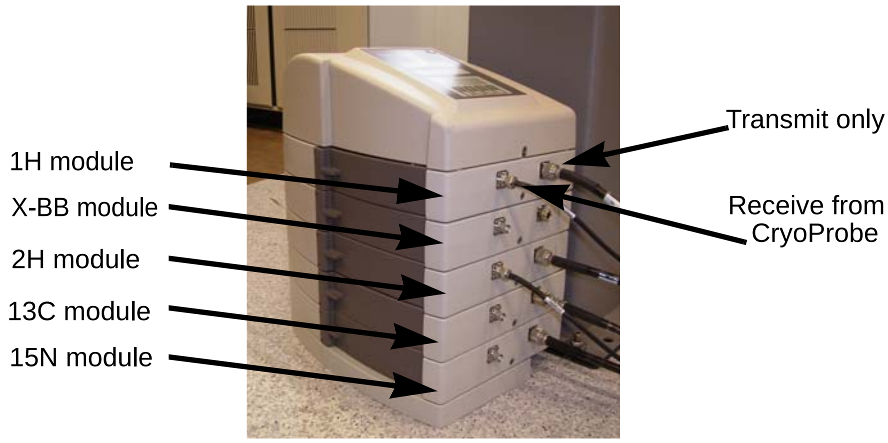


Figure 3.6. Cryo-compatible preamplifier assembly HPPR/2



Technical data

Data for TXI configuration (cover and 5 HPPR modules):

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

Software 3.4

The CryoController can be interfaced to a computer running Windows™ 95/NT4 or later. Monitoring software (CryoTool) is delivered with the CryoProbe System and is used to monitor the conditions integral to the proper operation of this cryogenic system. Operation of the CryoTool software is described in ‘Software Notes’.

Principle of operation 3.5

Gifford-McMahon cycle refrigerator 3.5.1

In ***Figure 3.7.***, 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:

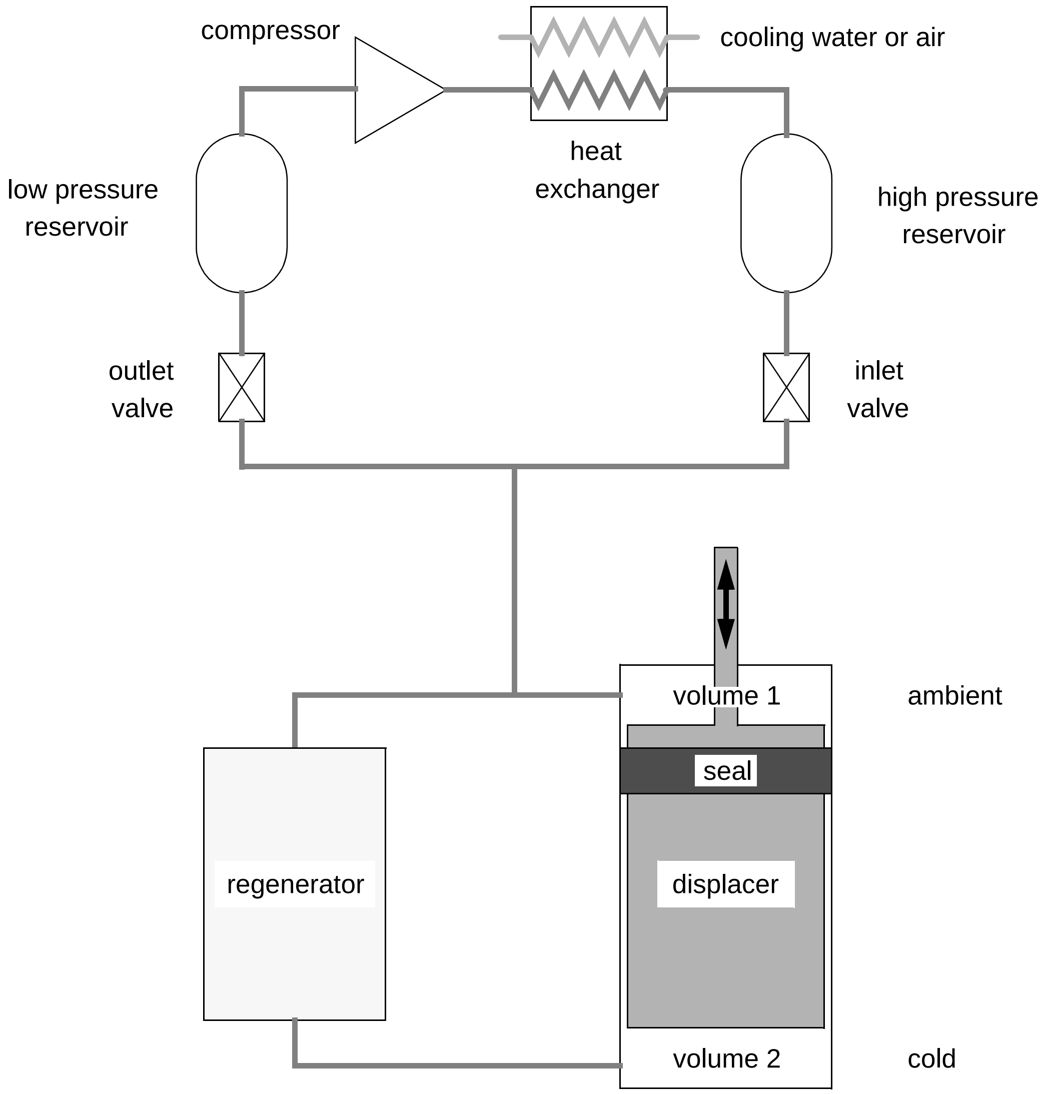
1. Pressure buildup
   With the displacer at the bottom of the cylinder and the outlet valve closed, the inlet valve is opened and the pressure in the system increases. High pressure gas enters through the inlet valve to fill the regenerator and the space above the displacer, volume 1.

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 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.

Figure 3.7. Gifford-McMahon cycle refrigerator



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.

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**

  Do not drop samples without the sample lift.
  Do not introduce any objects into the cavity (not even a soft cotton bud!).
  Do not clean the probe with solvents other than those recommended in ***"Cleaning the sample cavity" on page 74***.

- 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. See ***Figure 4.1.*** and ***Figure 4.2.***.

- **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 (~12kg) 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!

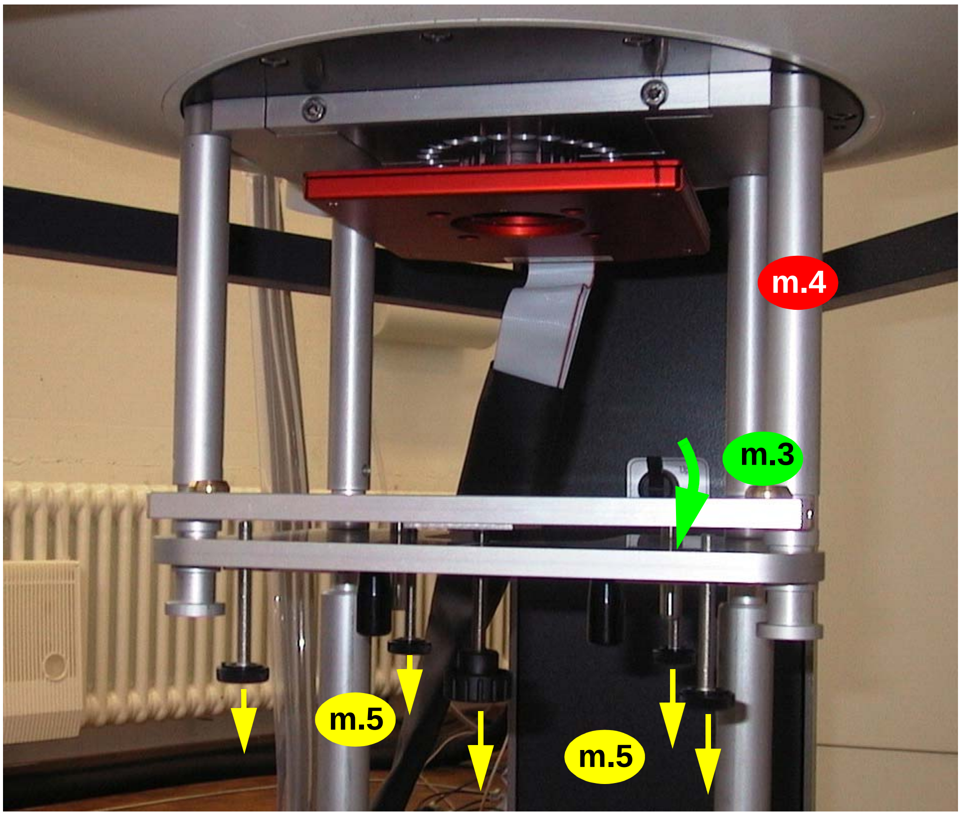
IMPORTANT: Read ***"Handling" on page 29*** if you're going to handle a CryoProbe for the first time.

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 ***pl*** and ***sp*** to 120dB (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.

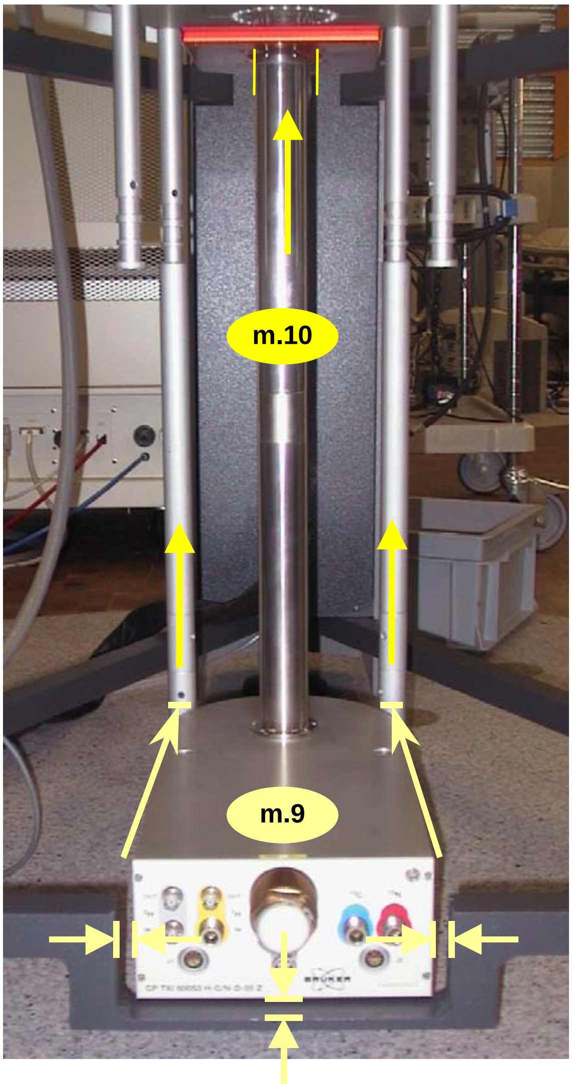
Figure 4.3. Preparing the Mounting Hardware



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! Refer to the subcategory ***"Indicator on CryoProbe sticks out" on page 82***. 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 weighs about 12 kg. **CAUTION:** Never hold a CryoProbe at its tube!

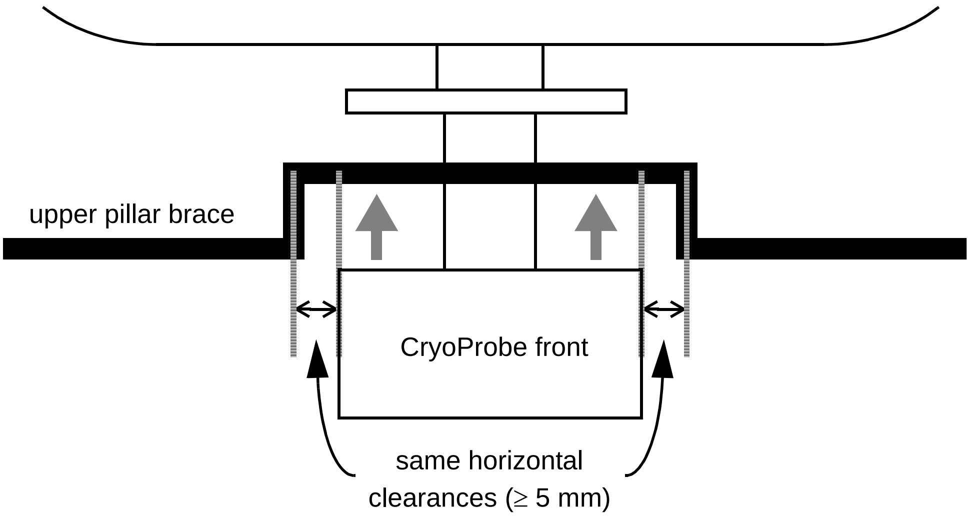
m.9	**WARNING:** While mounting the CryoProbe, the magnet must **not** be suspended on its anti-vibration feet. **WARNING:** Do not attempt to insert or remove the heavy CryoProbe (~12 kg) at the magnet without the help of a **second person**. You may easily injure your back when lifting the CryoProbe 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 guide rails** that extend downward from the Mounting Hardware.
m.10	**Align** the CryoProbe top with the shim system bore. **Gently push the probe all the way up** into the magnet, allowing the probe body to be directed by the guide rails, until the support plate can be snapped into place. **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.
m.11	**Snap** the support plate into place. Verify the seating of all four support points. **NOTE:** There are several positions for the mounting hardware. It is recommended to utilize the uppermost mounting position possible. This will increase the stability of the CryoProbe by reducing the amount it must be lifted to ensure proper positioning.

Figure 4.4.   Sliding the CryoProbe into the magnet



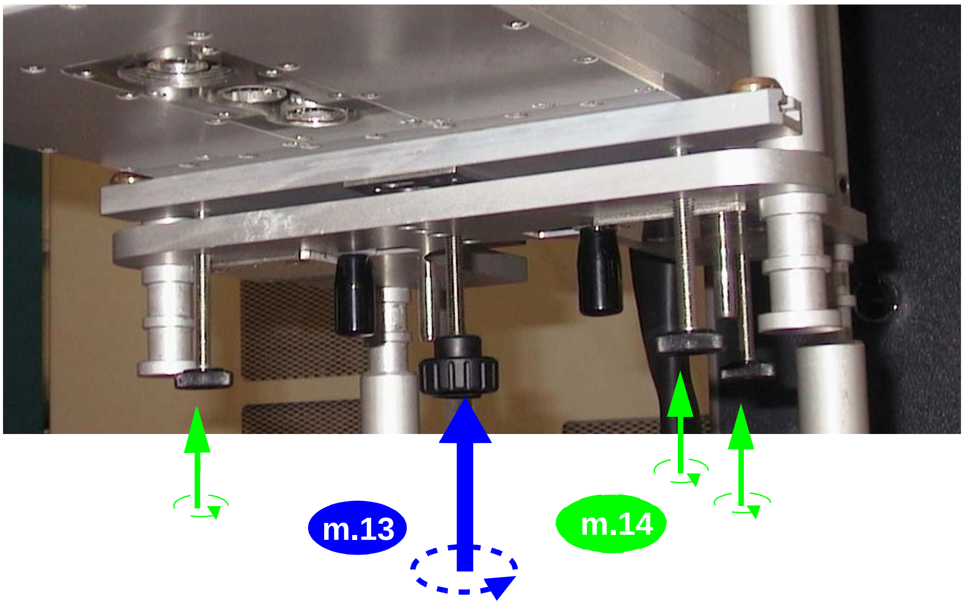
m.12	Check the **orientation** of the CryoProbe in the magnet: Its front must be able to meet the He Transferline. **IMPORTANT:** If cranked pillar braces are present in the magnet stand, the CryoProbe must be centered in their gap at all times (see ***Figure 4.5.***).

Figure 4.5. CryoProbe and optional cranked pillar braces



m.13	Raise the CryoProbe into the magnet by **turning the screw** below its center until the CryoProbe body almost touches the shim system (gap ≈ 0.5mm). Feel for the end position, but upon reaching it, do not use great force to tighten the screw (***Figure 4.6.***).
m.14	Turn the other **four screws** on the support plate in an alternate fashion such that they provide extra support for the CryoProbe (not available on some versions of the support plate). Take care not to tilt its body by forcing one screw more that the other screws.
m.15	**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 10mm 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** ≥ **5mm** 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



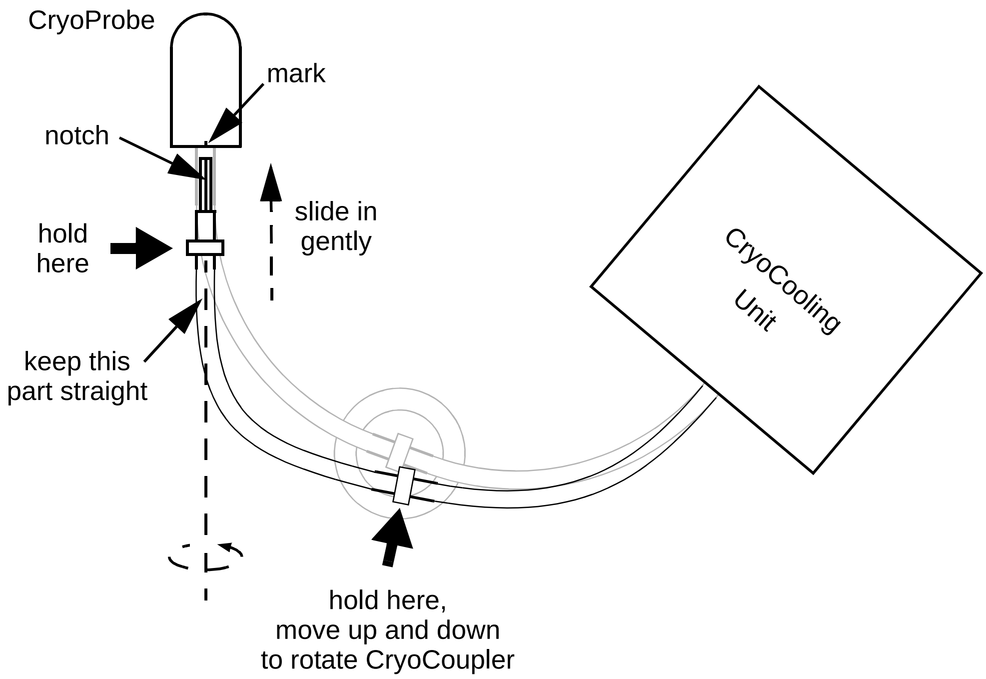
Joining the CryoCoupler 4.4

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 Transfer-line 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 Viton, size 7.1 x 1.6mm.

Figure 4.7. O-rings on the CryoCoupler

Figure 4.8. Inserting the CryoCoupler



j.4	Hold the **CryoCoupler** on the He Transferline with **one hand**. Take the **vacuum joint** which is 1m away from the CryoCoupler into the **other hand** (see ***Figure 4.8.***).
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 - this could result in permanent damage to the CryoProbe or the He Transferline! 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 out of the way and repeat the insertion process.
j.7	Use the special open-end 36mm **aluminum wrench** to tighten the CryoCoupler gently to the CryoProbe. Take care not to damage any connectors on the CryoProbe front.
j.8	Position the **Transferline Support** such that the vacuum joint falls exactly (within 1mm) into its bed.
j.9	**Lock the He Transferline** onto the Transferline Support with the four black screws. **NOTE:** Improper seating of the vacuum joint in the transfer line support stand may reduce the anti-vibration efficiency of the support stand. The vacuum joint **must** fall smoothly into its support stand.
j.10	Make sure that the **CryoProbe is still centered** inside the (optional) cranked pillar braces. If not, relocate the Transferline Support accordingly.

Figure 4.9. Bottom of CryoProbe

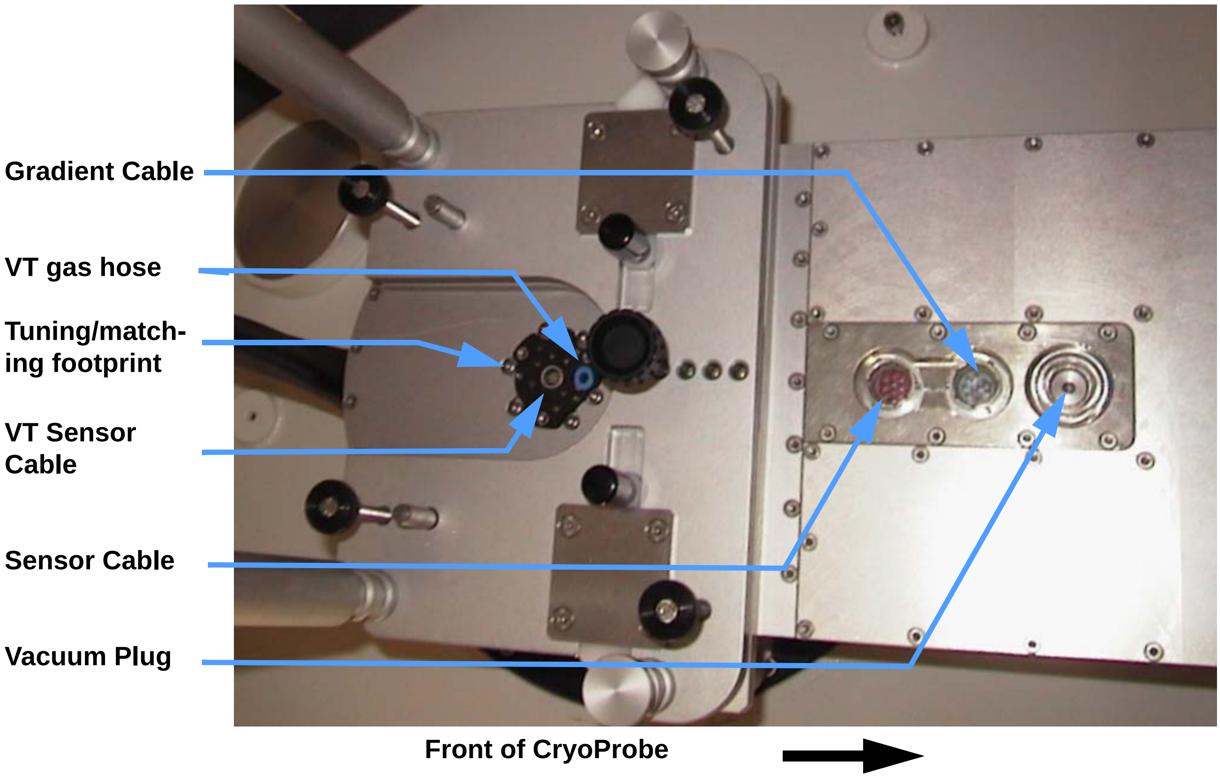


Table 4.3. Connect the Cables

step	action
j.1	Plug the **sensor cable** from the CryoCooling Unit into the CryoProbe bottom (reference ***Figure 4.9.***). Connect the J2 cable (from the Cryo-Cooling Unit) and the J1 cable (CryoPreamp supply from the HPPR) at the front of the CryoProbe. **NOTE:** Though it is not necessary for cooldown, it is easier to attach the RF cables before the sensor cables are plugged in.

j.2	Connect the **VT sensor cable** and the **VT gas hose** to the probe bottom. The heater cable and the PT100 cable from the BVT unit must be connected at the VT interface box. **NOTE:** When the Cooling Unit is in the 'off' position, the heater reading in ***`edte`*** will flash "**overheat**". **NOTE:** The PT100 cable is used for the VT Interface Box. If a conventional probe was previously installed, it may be necessary to plug the PT100 sensor cable back into the VT Interface Box and reconfigure the BVT appropriately. Reconnect the VT heater cable to the VT Interface Box.
j.3	Attach the **Tuning Adapter** to the CryoProbe bottom. See ***Figure 4.10.*** for a standard adapter, or ***Figure 4.11.*** for an optional ATM adapter. Use the blue Tuning Tool to lock its mounting screws. Attach the CRCO Bottom cable to the bottom of the tuning adapter. When using the ATM accessory, also connect the PICS cable to the ATM unit. **NOTE:** There are specific Tuning Adapters for each type of CryoProbe.

Figure 4.10. Mounting the standard Tuning Adapter (bottom view)

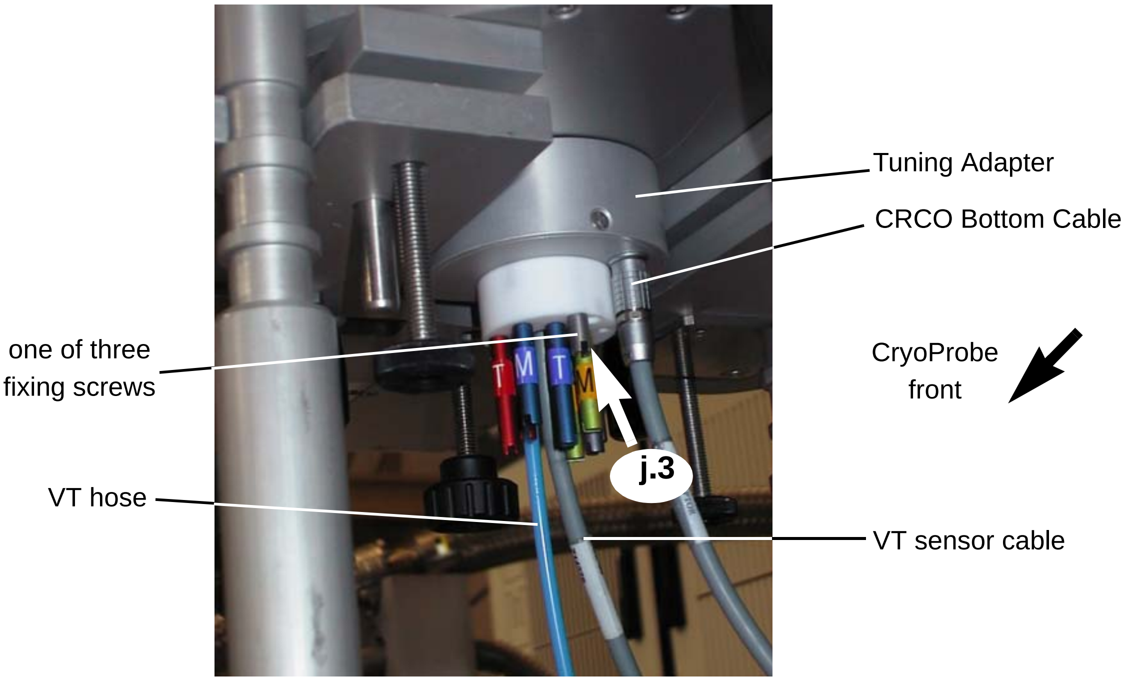
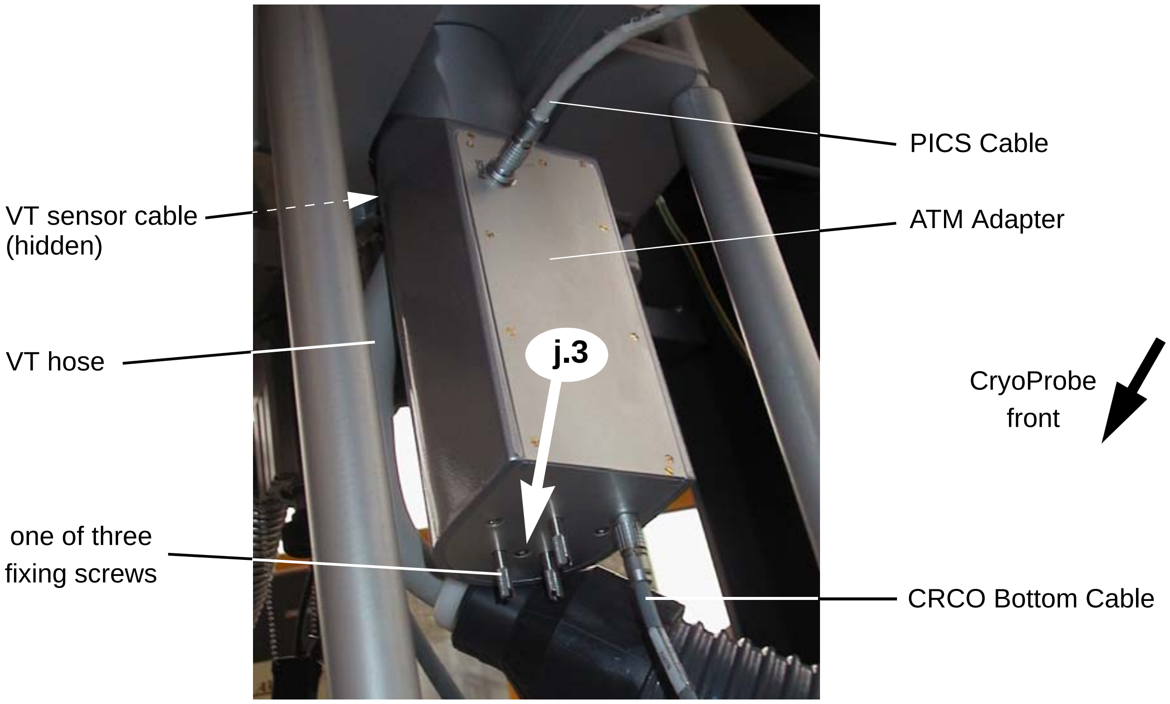
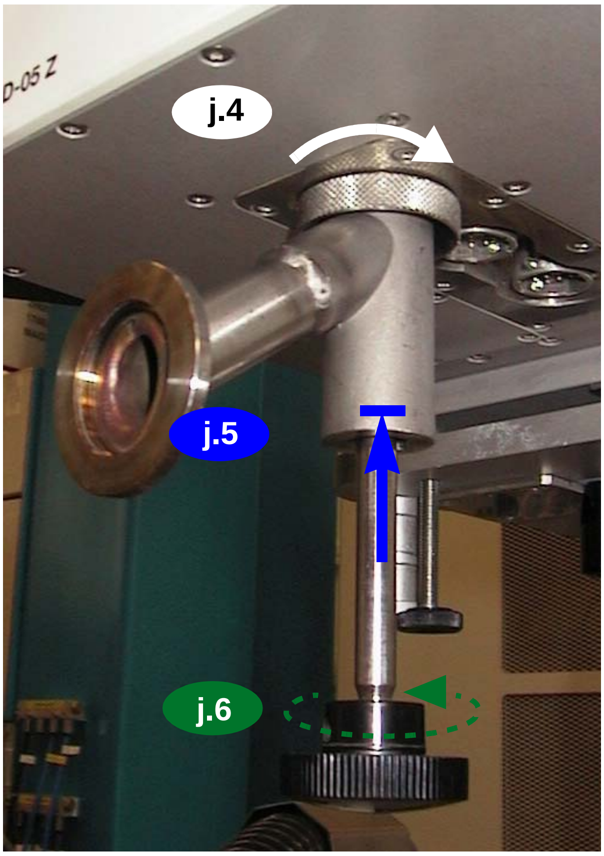


Figure 4.11. Mounting the optional ATM Adapter (bottom view)



j.4	Mount the **Vacuum Adapter** to the CryoProbe bottom such that it points to the front (***Figure 4.12.***). Connect the **vacuum tube**.
j.5	**Push in** the actuator screw of the Vacuum Adapter (black handle) in as far as it will go.
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 for two turns; do not force it to stop. **NOTE:** The vacuum system will be activated during the cooldown process with the statement: "Open the CryoProbe Vacuum Plug".
j.7	Connect the **remaining RF and gradient cables** (for details, see *CryoProbe RF Electronics Technical Manual*).

Figure 4.12. Mount the CryoProbe vacuum adapter



Sample spinning test 4.6

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 ***"Samples and Spinners" on page 61*** as well as the LIMITATIONS - WARNINGS sheet provided with each probe.

If spinning does not work, see ***"Spinning problems" on page 81***.

Power-on 4.7

If the CryoProbe System has been turned *OFF* or has not been recently used, the following steps need to be completed to make sure 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	Check that the compressed air going to the CryoPlatform is ON and the pressure is between 4 and 8 bar.
o.4	The He steel-cylinder main valve must be open and the primary pressure gauge on the He steel-cylinder should read **30-200 bar**.
o.5	The secondary pressure gauge should read **23-25 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.6	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. Also, the circuit breaker (if applicable) on the wall should be in the ON position. **CAUTION:** If *COLDHEAD DRIVE* and *REMOTE DRIVE* are in the wrong positions, the Coldhead inside the CryoCooling Unit can be damaged.
o.7	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.8	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 pressing *WARM UP* and check if all sensor and supply connections are ok.

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 61*** 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.

The usage of N_2 as VT gas is recommended.

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** until the CryoProbe has been warmed-up. For example, a BCU05 gas cooler may only be inserted while the CryoProbe is warm.
v.2	Check that all the connections at the VT Unit are properly made. The PT100 sensor must be selected; the reading in ***`edte`*** should indicate **room temperature.**
v.3	**Set** the VT gas **flow rate** and VT **heater power limit** according to the values given on the specific LIMITATIONS - WARNINGS sheet of the CryoProbe. 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.4	Verify that there is **no sample in the magnet.**

v.5	**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 **BTO-2000** unit for conventional probes uses the same type of PT100 cable. The cable is suitable for the CryoProbe. There are no special considerations when using a BVT-3000. A **BVT-2000**, however, cannot be toggled properly from BTO-2000 to Cryo-Probe operation with ***`edte`*** (although no error occurs); it must be **set explicitly at its EUROTHERM** module. With a BTO-2000, the EUROTHERM would be set neither for a PT100 (code 0225) nor for a Cu/Const thermocouple (code 0203) but with the special code 5203. When using a CryoProbe, make sure the EUROTHERM is configured for PT100 with **code 0225**. Refer to the *BVT-2000 Operating Manual* (P/N W1101034) for instructions.
v.6	Set the **sample temperature** to somewhere above room temperature, e.g. ***`300K`***.
v.7	Switch the VT heater ***on***.
v.8	**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 damaging the substance under investigation or breaking the sample tube if it freezes. 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 Protection Device (Optional) 4.8.1

The Sample Protection option is designed to protect the sample in case of an electric power failure, compromised vacuum, or a breakdown of the compressed air. This device is controlled by CryoTool and will provide continuous VT gas operation in the event of a VT gas interruption. Or, in the worst-case scenario, it will lift the sample out of the cold environment of the CryoProbe.

NOTE: If the CryoProbe has been warm for a long time, it is necessary to check that the backup cylinder (air or N_2) is full and the main regulator is open. The secondary regulator should be set to approximately 5 bar.

Cool-down *4.9*

NOTE: If any detectable malfunction appears, a cool-down will be automatically stopped and the CryoProbe System will be 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 30 bar and its secondary pressure stable at **23-25 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) **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.**
c.4	**Start** the cool-down by pressing 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. **Pull** the actuator screw of the vacuum adapter **all the way out** when requested by ***CryoTool***.
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 **23-25 bar**. The primary pressure must remain ≥ 30 bar at all times.
c.6	About 15 minutes later, the **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 an error message appears on the monitoring computer refer to section ***7.5 on page 83***.

While the cool-down is in progress for about 3 h, there is plenty of time to do the ***"HPPR/2 configuration" on page 47*** or the ***"HPPR CRP configuration" on page 50***.

Table 4.7. "CryoProbe Cold Enough to Start Shimming"

step	action
c.1	**When *COLD*** on the CryoCooling Unit **flashes** and the message in CryoTool reads "CryoProbe cold enough to start shimming", it will take about half an hour more until the final temperature is reached and stabilized. However, during this time the probe can be wobbled and shimmed. **NOTE:** A cool-down must not take more than 270 min.; if it does, the CryoController will interrupt automatically due to a "cooldown time-out" and warm up the system (see ***"Cool-down doesn't reach cold state" on page 84***).
c.2	**Verify** that the **'sample' temperature** reading in ***edte*** is stable at its preset value. Now a **sample can be inserted**. **NOTE:** For recommended sample depth and filling height, see the 'LIMITATIONS - WARNINGS' sheet for the CryoProbe and ***"Samples and Spinners" on page 61***. **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.3	Set all RF power levels ***pl*** and ***sp*** to 120 dB (recommended).
c.4	Tune the CryoProbe on all channels, **including ^{2}H**, see ***"Tuning and matching" on page 52***.
c.5	**Shimming** can be started while *COLD* is still flashing, see ***"Locking and Shimming" on page 54***. **CAUTION:** Do not run any decoupling experiments while the green light if flashing.
c.6	When ***COLD*** is **continuously green**, the final conditions are stable. Normal experiments can be run.

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/2 configuration *4.10*

Hardware settings *4.10.1*

For a CryoProbe TXI with two X-channels, it is important to utilize the preamplifier slice designed for each specific nucleus. For instance, if the HPPR/2 has a 13C slice in addition to an XBB slice, it is very important to use the 13C preamplifier, as this slice features high-performance filters dedicated solely to 13C.

To change the hardware configuration, the cable from the 13C amplifier must be attached to the back of the 13C slice. The RF cable from the preamplifier must be attached to the probe.

Figure 4.13. Internal wiring of an HPPR/2 for a TXI probe

RF routing with edasp *4.10.2*

When the RF hardware wiring is complete, RF pathways need to be configured in XWIN-NMR with ***edasp setpreamp.*** **NOTE:** For XWIN-NMR versions 3.1 or lower, the proper command is ***edasp***.

Selection of CryoPreamps *4.10.3*

To make full use of the CryoProbe’s excellent signal-to-noise, its built-in ‘cold’ preamplifiers must be selected with XWIN-NMR UniTool. This procedure can be completed as the probe is cooling down.

When the proper cold preamplifiers are defined, wobble, acquisition, and the lock circuit will respond to the same commands as a system controlling a conventional probe.

NOTE: With software versions of XWIN-NMR 3.5 or higher, ***edhead*** will automatically configure the cold preamplifiers. The following procedure is required for versions of XWIN-NMR< 3.5.

Open UniTool by typing **`UniTool`** in a shell (Unix or Linux) or from the directory \\Desktop\ServiceTools\UniTool (Windows). Once in the program, proceed as follows:

```
Enter device name ['?' for details]  (aqs) > hppr
Enter decimal SBSB address for HPPR (80) >  <Return>
device name taken from already existing configfile: /dev/tty10

B r u k e r  U n i T o o l
   Version: 1.0
   Compilation date: 030403

W A R N I N G:
   This is a hardware level debug tool.
   Improper operation may damage your hardware.

Connecting SBSB address 80 (0x50).

HPPR/2 Main Menu
================
[1] Init HPPR/2
[2] Show Version
[4] Auto Download
[C] Cover module ...
[P] Preamplifier module ...
[N] QNP function module ...
[R] ATMA   ...
[S] CRP    ...
[I] PH BIS ...
[X] Exit
    your choice: P  <Return>

HPPR/2 Preamplifier Module
==========================
[1] Init Preamplifier Module
[2] Show Version
[5] 1H LNA ...
[6] XBB19F 2HS ...
[7] 2H ...
[G] get  Force-States of all modules
[Q] Quit
    your choice: 5 <Return>

1H LNA
======
[1] Init PreAmplifier Module
[2] Show Version
[3] Show BIS
[4] read  byte from I2C device
[5] write byte to   I2C device
[E] show Channel State
[7] get ...  [6] set REF 50
[9] get ...  [8] set RGP EXT
[B] get ...  [A] set Mode
[D] show ..  [C] set Interleave Counter
[G] get ...  [S] set Force-State
[Q] Quit
    your choice: s  <Return>
    Force is off.
    Value (8=bypass, 4=cold, 0=off): 4 <Return>
    Force is cold until next power up.
```

Exit out of the 1H LNA menu and follow the same procedure for all of the cold preamplifiers in the CryoProbe. For the instance of a TXI probehead, the 13C and 15N channels are left “off” because they do not utilize cold preamplifiers. The *force states* on the 1H and 2H channels, on the other hand, must be “cold”. When the necessary changes have been made, exit out of UniTool and execute an ***ii*** in XWIN-NMR.

HPPR CRP configuration *4.11*

NOTE: For configuration of the HPPR/2, refer to ***"HPPR/2 configuration" on page 47***.

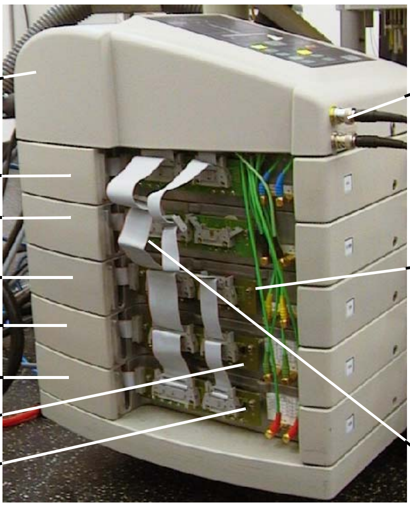
IMPORTANT: If a conventional probe was used right before you mounted the CryoProbe *and* if your HPPR features more than 5 modules (not counting the cover module), you should check the internal wiring between the HPPR CRP modules and their cover module.

Hardware settings *4.11.1*

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***. The HPPR 15N CRP module is wired to ‘User Box’ 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 preamplifier to the cover module (***Figure 4.14.***).

To change the HPPR CRP assembly’s internal wiring, shut down its power supply by switching *OFF* the AQR/AQS unit inside the spectrometer cabinet. It is important to note that the HPPR CRP will only function with 4 modules connected. This is why the ribbon cables in ***Figure 4.14.*** are disconnected from the X-BB module. When the wiring has been changed, switch on the AQR/AQS 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.

Figure 4.14. Internal wiring of a HPPR CRP for a TXI CryoProbe



RF routing with edasp 4.11.2

When the RF hardware wiring is complete, RF pathways need to be configured in XWIN-NMR with ***edasp setpreamp.*** **NOTE:** For XWIN-NMR versions 3.1 or lower, the proper command is ***edasp***.

Selection of CryoPreamps 4.11.3

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 13C on a CryoProbe TXI in the presence of a high-sensitivity 2H lock.

Setup: In a CryoProbe TXI, the 13C channel is intended for decoupling only and features no CryoPreamp. However, 13C detection is possible when using the warm HPPR 13C CRP preamp module. This configuration is established in XWIN-NMR with ***crplock (crp2hon*** for Xwin-NMR versions up to 2.6).

Possible combinations

See ***Table 4.8.*** for all possible combinations of warm and cold preamps and their corresponding XWIN-NMR commands. The letter ‘X’ stands for any nucleus except ^{1}H, e.g. ^{13}C, ^{15}N, ^{2}H. Of course, the built-in ‘cold’ CryoPreamps are preferred. There are no waiting periods or the like needed when toggling between cold and warm preamps.

Table 4.8. Preamplifier selection

observe	preamp	lock	situation	XWIN-NMR (XWIN-NMR ≤2.6)
^{1}H or X	cold	cold	default	***crpon***
^{1}H or X	warm	cold	no CryoPreamp available for the chosen nucleus; debugging or X observe with TXI.	***crplock*** ***(crp2hon)***
^{1}H or X	cold	warm	debugging	***crpobs*** ***(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*.

Tuning and matching *4.12*

IMPORTANT: A CryoProbe must not be tuned or matched when warm because the properties of the RF circuits are 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.

Use of additional RF filters

All necessary RF filters are already built-in the CryoProbe and the HPPR. If additional filters are considered, see ***"Additional RF filters" on page 64***.

Operating the standard Tuning Adapter *4.12.1*

A CryoProbe is tuned and matched from below like a conventional probe with the actuators of its specific Tuning Adapter (***Figure 4.10. on page 39***). The long actuators are for tuning, short ones are for matching. Use the blue Tuning Tool to operate the actuators. The colour of the tuning and matching wands corresponds to the colour of the markings around the CryoProbe's RF plugs.

Examples:

yellow: ^{1}H
gray: ^{2}H

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. Do not challenge the limits of the tuning and matching range if it can be avoided. Do not use tools other than the Tuning Adapter to adjust the tuning or matching.

Wobbling with an HPPR/2

As long as the cold preamplifiers have been properly configured according to section ***4.10.3 on page 47***, wobbling commands will not change from those of a conventional probe.

Wobbling with an HPPR CRP

Wobbling is controlled by the external 'warm' preamplifiers inside the HPPR CRP instead of by the built-in CryoPreamps. Thus, the proper RF pathway to these preamplifiers must be configured with special XWIN-NMR commands.

Table 4.9. How to wobble the CryoProbe with an HPPR CRP

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 including ^{2}H (recommended), enter ***locnuc off***, and reduce the lock power to minimise interference from the lock transmitter. Then type ***crpwobb*** and ***wobb***. – To wobble all channels except for ^{2}H while the lock is operating, enter ***crplock (crp2hon*** for Xwin-NMR versions up to 2.6***),*** and ***wobb***.
w.3	Tune and match with the Tuning Adapter, see above.
w.4	When tuning and matching is done, enter ***stop, locnuc 2H, ii*** and ***crpon***. Reestablish the previous lock power if it has been reduced during the wobbling procedure.

Operating an ATM Adapter (optional) ***4.12.2***

With an ATM adapter (optional, instead of a standard Tuning adapter), tuning and matching can conveniently be carried out with the same XWIN-NMR commands as with conventional probes:

Table 4.10. Tuning and Matching with an ATM adapter

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 including ^{2}H (recommended), enter ***locnuc off*** first and reduce the lock power to minimise interference from the lock transmitter. Either type - ***atma*** for fully automatic tuning and matching on all channels configured, or alternatively - ***acqu***, then ***atmm*** for manual tuning and matching using the controls displayed in the ATMM Probehead Tuning/Matching window. When done, leave the dialog window with the menu option File, then Exit, click yes to save the new tuning and matching position, and click the ***return*** button to display the spectral representation again.
w.3	When the ^{2}H channel has been adjusted, enter ***locnuc 2H, ii***, and re-establish the previous lock power.

Locking and Shimming 4.13

For locking 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 transmitting RF power into the CryoProbe (see also ***"RF power limitations" on page 64***).

The usual XWIN-NMR ***lock*** procedure can be used for locking.

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 locking.

Shimming can be done on the lock level as usual. There are no particular gradient values to expect. Gradient shimming is recommended. After locking and shimming, the CryoProbe is ready for NMR experiments. Consult chapter ***5 on page 61***. Pay special attention to the sections ***5.3 on page 61*** and ***5.5 on page 64***!

Liquid Helium and Nitrogen fills 4.14

When it comes time to refill the magnet with cryogenic liquids, it is good practice to lower the magnet’s anti-vibration feet. Magnet refills can be done on a cold CryoProbe. It is not necessary to move the Transfer Line Support Stand.

Warm-up *4.15*

WARNING: Do not disconnect any tube or cable from a running CryoProbe System unless the *WARM* and *UNPLUG* lights on the CryoCooling Unit are lit. 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. A warm-up is required only if

- requested by the CryoController (see **"Warm-up needed soon request" on page 69**),
- the CryoProbe should be removed from the magnet,
- the CryoCooling Unit is going to be moved.

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

Table 4.11. Warm-up

step	action
w.1	Terminate any NMR experiment and **eject** the sample. **CAUTION:** Keep the VT unit operating even when there is no sample!
w.2	Initiate a warm-up by pressing the ***WARM-UP*** button on the front or the CryoCooling Unit or in CryoTool. The **Coldhead stops** after a short while.
w.3	**Wait** until green *WARM* and white *UNPLUG* lights are lit. This will typically take ~2h. **CAUTION:** Do not enforce a warm-up by any additional means of external or internal heating!

CAUTION: If the CryoProbe is warming up due to an error condition, the NMR experiment, if applicable, must be stopped manually. The sample must be ejected.

Removal *4.16*

Insertion and removal of the heavy CryoProbe (~12kg) 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 (~12kg) 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!

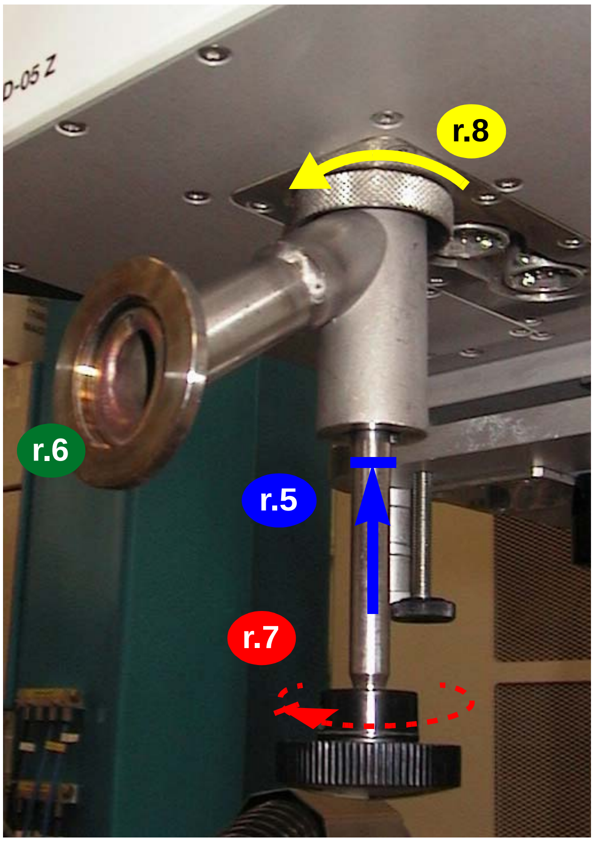
CAUTION: Never attempt to remove a CryoProbe from the magnet when cold. Always wait until it has reached room temperature because the CryoProbe or the He Transferline may be damaged when moved cold.

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 4.12. Remove the CryoProbe

step	action
r.1	Make sure that the **warm-up** of the CryoProbe has been completed (see ***"Warm-up" on page 55***). 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. 2.5h).
r.2	When ***WARM* and *UNPLUG*** are lit: Disconnect all cables and tubes from the CryoProbe front and bottom.
r.3	Remove the Tuning and Matching adaptor or ATMA device with the blue tuning tool.
r.4	**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.5	Insert the **Vacuum Plug** into the CryoProbe bottom by pushing up the black knob of the Vacuum adapter. It should move easily at the beginning, but needs a bit more force once it has begun to enter the vacuum cavity. Verify that the vacuum plug is properly inserted before removing the Vacuum adaptor.
r.6	Detach the **vacuum bellows** from the Vacuum Adapter.
r.7	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.
r.8	Remove the **Vacuum Adapter**.

Figure 4.15. Removing the vacuum connector from the CryoProbe



r.9	Use the special 36mm aluminum wrench to **unlock** the CryoCoupler. Be careful not to damage any connector on the CryoProbe front.
r.10	**NOTE:** Use the same technique for removing the CryoCoupler as for connecting it (cf. ***"Mounting" on page 30*** 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.11	Check if all **4 o-rings** on the He Transferline CryoCoupler are still in place (***Figure 4.7.***). See also ***"He leak at CryoCoupler" on page 83***.
r.12	Close both the CryoProbe and the He Transferline firmly with their special **caps**.
r.13	Switch *OFF* the **air suspension** of the magnet such that the magnet comes down onto its stand.
r.14	Loosen the **four outer screws** in the Mounting Hardware support plate all the way down.

r.15	Unscrew the **pusher screw** in the center to lower the probe down to the base of the mounting plate.
r.16	**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.17	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's top gently with one hand to avoid hitting anything.
r.18	**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.19	Immediately close the **magnet** bottom or insert another probe to avoid intrusion of dust or magnetic particles.
r.20	Switch *ON* the **air suspension** of the magnet.

Power-down 4.17

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 4.13. 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 55***).
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 Regulator.

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 remain pressurized at all times 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.

If the CryoProbe system should need to be relocated, a Bruker engineer should be contacted.

Spectrometer operation with conventional probes *4.18*

The HPPR preamplifier assemblies can be operated with conventional probes. For 13C observe experiments with conventional probes, either the selective 13C or the X-BB module can be used.

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

Recommended NMR parameters

5

General 5.1

In many cases, parameters used with conventional probes are applicable to the CryoProbe system. In a few cases, more care should be taken when setting up NMR experiments with a CryoProbe. These cases are outlined in this chapter.

Samples and Spinners 5.2

The following Bruker Spinners have been 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, a bad sample tube is one that cannot be spun.

Observe the limits pertaining to the sample parameters as given on the LIMITATIONS - WARNINGS sheet provided for each individual CryoProbe. This includes:

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; it may deteriorate the precision of sample temperature control by inducing convection flows.

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. Unlike conventional probes, the VT gas flow should be as high as possible for a CryoProbe. The only disadvantage to this is the possibility that the VT gas may lift the sample. Refer to the 'LIMITATIONS - WARNINGS' sheet for the minimum gas flow rate.

NOTE: The gas flow required for the CryoProbe 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.

The usage of N_2 as VT gas is recommended.

For measurements at low sample temperatures, a BCU05 gas cooler is required. However, to achieve the lowest allowed temperatures, a special adaptor for the BCU05 is available with the Sample Protection Option. 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 in the CryoProbe-specific 'LIMITATIONS - WARNINGS' sheet. Details are shown in ***Figure 5.1.***

Symbols used

T_{target} : target temperature for the sample

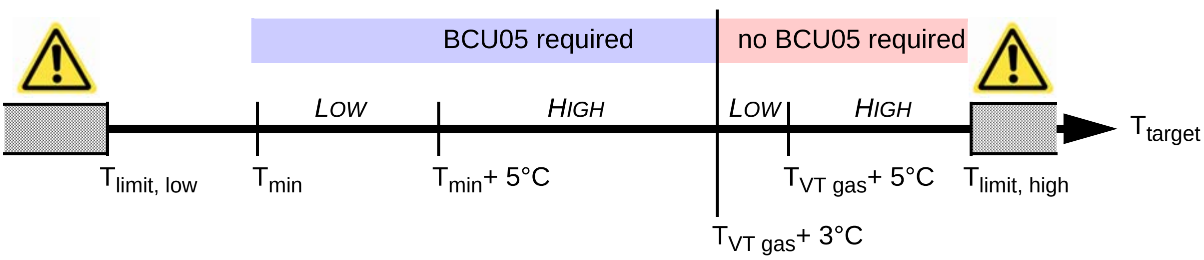
$T_{VT\ gas}$: temperature of the VT gas before it enters either the CryoProbe directly or a BCU05 gas cooler

T_{min} : minimum achievable sample temperature in a cold CryoProbe when the VT heater is ***minimal*** (the VT heater should never be turned 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**. 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 divides the heater energy – some of the energy is discharged inside the Interface Box. In the *LOW* position, more energy is disposed of than when the switch is set to High. Though the desired sample temperature should never exceed 60°C, at temperatures approaching this value, the Interface Box could still overheat if the *LOW* setting was selected. This would result in a*"heater failure"* message in the ***edte*** display. Ensure that the cooling fins of the VT Interface Box are upside.

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 BVT units to optimize the sample temperature regulation loop. Write down the control parameters of the BVT unit and also the VT heater power required to achieve the desired temperature. Set the heater limit accordingly.

NOTE: A BVT2000 cannot be self-tuned with ***edte*** (although no error appears) but must be set directly at its EUROTHERM unit. See the *BVT2000 Operating Manual* (P/N W1101034) for instructions.

Sample Temperature Calibration 5.3.4

It is recommended to carry out a sample temperature calibration according to the chapter 'Calibration of the Sample Temperature' in the Variable Temperature Unit manual (P/N Z31428).

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: Wherever there are two RF connectors per channel of the CryoProbe (transmit and receive), a frequency-specific cryogenically cooled preamplifier with superior sensitivity is available. If there is only one RF connector, the conventional preamp in the HPPR will be used for detection. Such a channel is not intended for observation and its sensitivity is generally not specified.

An HPPR/2 with more than 5 modules may require manual re-wiring for multinuclear NMR experiments with a conventional probe (see ***"HPPR/2 configuration" on page 47***).

The HPPR CRP does not support the simultaneous use of more than 4 preamplifier modules. See ***"HPPR CRP configuration" on page 50***.

If the HPPR has been rewired to incorporate different preamplifier modules, do an ***expinstall*** to update the standard parameter sets accordingly.

Additional RF filters 5.4.2

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

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. No filters should be introduced into the pulse transmission paths between HPPR and CryoProbe.

RF power limitations 5.5

When running a long experiment with extensive decoupling or spin-lock sequences, the insert temperature of the CryoProbe needs to be monitored for the first few minutes of the acquisition. The power limitations provided in the LIMITATIONS - WARNINGS document provide a foundation for calculating the proper power levels for pulses of this type. However, it cannot be guaranteed that excessive heating of the NMR coil assembly will be avoided when using these pulses. (**"Average RF power" on page 66** defines 'excessive heating'.) In case of excessive heating the NMR coil assembly with RF, the system will send error messages and may even warm-up the CryoProbe if the warnings have been ignored (see also **"Average RF power" on page 66**).

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 demands more power from the RF electronics.

Keep parameter sets for conventional and CryoProbes well separated.

NOTE: Do not use datasets originating from conventional probes. Use standard parameter sets and prosol handling (***rpar*** and ***getprosol***).

Reference documents include:

LIMITATIONS - WARNINGS SHEET	Delivered with each probe
CUSTOMER CERTIFICATE	Delivered with each probe
TYPICAL PULSES FOR CRYOPROBES	For 5mm TXI 500, 600, 700, 800MHz

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 ***pl*** and ***sp*** lower than 10dB (e.g. 6dB, 0dB, -3dB etc.) with extreme caution!

Before setting an RF power level ***pl*** 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 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 is a table in the spectrometer installation documents that relates amplifier power in watts to ***pl*** settings in dB.

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

'Typical Pulses for the 5mmCryoProbe' 5.5.2

Once the 90-degree pulse is known, the document TYPICAL PULSES FOR CRYOPROBES provides a foundation for finding appropriate pulse lengths and power levels for 2D and 3D experiments. This document covers how to determine the maximum-allowed RF power for all of the special pulses that are not determined by the installation engineer. Also, it provides powers for a number of unique cases including simultaneous hard pulses.

Currently, this document is available for the 5mm TXI CryoProbes 500, 600, 700, 800MHz.

Simultaneous hard pulses 5.5.3

CAUTION: Two RF pulses applied to an NMR coil at the same time facilitates arcing. This may result in permanent damage to the coil.

To prevent arcing, the power of two simultaneous hard pulses[1] using the same RF coil, e.g. 13C and 15N, must be reduced by at least 3dB each.

For specific information refer to the 'LIMITATIONS - WARNINGS' sheet and the 'Typical Pulses for CryoProbes' manual if applicable.

Average RF power 5.5.4

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 5K**, ***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 not *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 and the NMR coil assembly is heated further, there is a second factory-set temperature limit of **15K above** the optimum operating temperature at which the ***ERROR*** button on the CryoCooling Unit **starts flashing**. Stop the NMR experiment immediately!

If the NMR coil assembly temperature exceeds the optimum operating temperature by **20K**, 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.

After the RF-induced heating has been reduced, it is feasible to immediately *COOL DOWN* again and eventually to restart the experiment (at a lower power level and/or shorter decoupling periods!). *COOL DOWN* will flash three times quickly, acknowledging the cool-down request. Usually, the system should be ready again within 15 minutes.

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

Receiver gain 5.6

For small receiver gain values, i.e. ***rg*** < ~256, the signal-to-noise tends to decrease in proportion to ***rg***.

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.

RCB gradient shimming (either with an RCB board or a 2H-TX board) is highly recommended.

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*** ≥ 256 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 can render 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 or smoothened rectangular shapes (available in XWIN-NMR 3.5 pl8 or higher) 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.

CryoProbes usually operate at significantly lower RF powers and higher VT gas flow rates. It is advisable to use parameter sets along with the properly adjusted pulses stored in the prosol table of the CryoProbe (***rpar***, then ***getprosol***).

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	The lock gain should be set such that the lock lies on the uppermost gridline of the Lock Display.
w.2	Run the experiment in ***gs*** mode for a couple of minutes to determine the amount of Δz necessary to reestablish the lock level at the uppermost gridline of the Lock Display.
w.3	Add some dummy scans to the experiment and add Δz to the z-shim.
w.4	Run the experiment.
w.5	When 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 77***.

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

Displays & Diagnostics 6.1

See ***"Status display by the control buttons" on page 21*** for an overview 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 running Windows™ 95/NT4 or higher and the CryoTool software. The computer, typically a laptop, is connected directly to the CryoController using an RS232 interface.

The operation of CryoTool software is described in SOFTWARE NOTES.

`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 in the cold parts of 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 system automatically initiates an active warm-up.

See ***Table 6.1.*** to control contamination of this type. If this error occurs more frequently than once every 3 weeks, arrange for a BRUKER service visit. 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 less efficient.

Table 6.1. How to manage 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 1h.
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.

Periodic checks *6.2*

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

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

For the air-cooled He Compressors, check that the fans are clean. Dust and the like will reduce their efficiency.

Monitor the He pressure at the He cylinder. Grade 6 helium (99.9999%) must be ordered, and is often not available immediately.

Monitor the operating-hour counters on the CryoCooling unit and the He Compressor. These values determine the expected date of the annual maintenance. See ***"Essential CryoPlatformTM Annual Maintenance" on page 75*** for details.

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 useful to BRUKER service engineers when they begin problem diagnosis.

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 CryoTool software and are periodically stored on the monitoring computer as `auto<yymmdd>.log` (`yymmdd` being the creation date) in the default directory `<defaultpath>\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 noted in ***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 (Grade 6) ready by the time the pressure has come down to 40 bar. With a standard size 50ℓ He steel-cylinder, there will be still 2-3 warm-up/cool-down cycles possible.

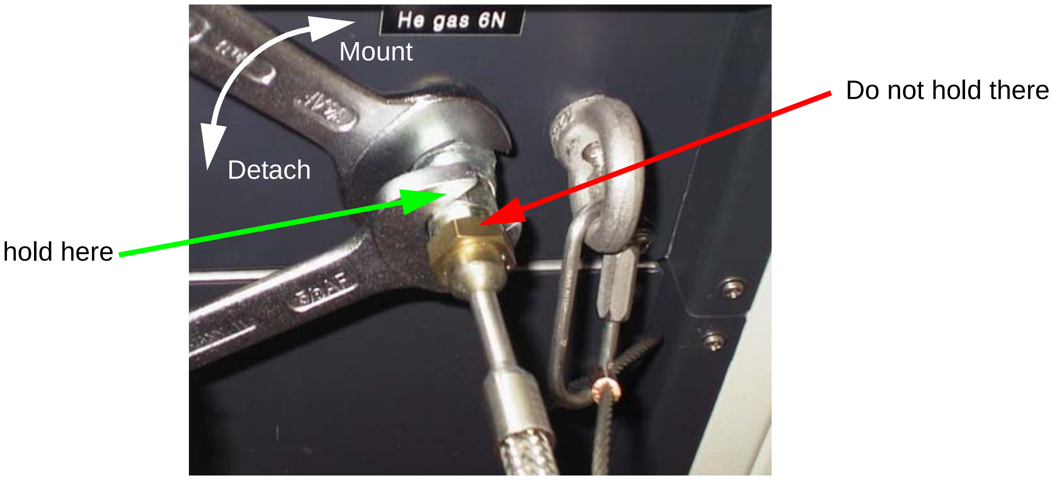
As all helium cylinders and/or initial pressures differ somewhat, quantifying a leak can be difficult. If a leak is suspected, logfiles should be sent to a CryoProbe service engineer.

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 with air. However, since He is mostly consumed when a cool-down is initiated, a cold CryoProbe System can run for a long time even when the He steel-cylinder is almost empty.

Table 6.3. Replace the He steel-cylinder

Step	Action
b.1	Make sure that the CryoProbe System is ***WARM*** and ***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. 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, see ***Figure 6.1.***. 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.**
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 used He steel-cylinder.

Figure 6.1. Detach / mount the He hose at the Cooling Unit



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 0.5mT range.
b.8	Attach the new He steel-cylinder **securely** to a wall. Fix the safety cable of the He Hose around the He steel-cylinder and mount the He Regulator.
b.9	Slowly open the He cylinder valve. Helium gas must escape audibly through the dump tool. (This may require a second person if the He cylinder is not in the same room as the CryoCooling unit.)
b.10	While the He gas is escaping (~3seconds) remove the dump tool. This joint is self-sealing, so as soon as the dump tool is removed, no more He gas will escape.
b.11	Close the He cylinder's main valve.
b.12	Attach the He Hose to the back of the cooling unit (***Figure 6.1.***) and secure the safety loop with the carabiner.
b.13	Open the main valve of the He cylinder. Verify that the pressure on the secondary reads between 23 and 25bar.
b.14	He Leak Test: Close the main valve of the He steel-cylinder and wait for about 1/2 hour. Read the secondary pressure. Wait for ~2hours, and read this pressure again. If the value has decreased, search for leaks with a He detector or **Snoop Liquid** (found in the CryoToolkit).

Cleaning 6.3

Cleaning the sample cavity 6.3.1

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 (1^{st}) **water** and (2^{nd}) **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 solvents other than those listed above! 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 6.3.2

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 any style of air-cooled compressor, its air-cooler fins must be kept clean.

Essential CryoPlatform™ Annual Maintenance 6.4

To ensure successful operation of the CryoPlatform™, it is essential that it is maintained after every 10,000 hours of operation. For simplicity, this maintenance is recommended annually. There are two counters monitoring the hours of operation 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.

Examples of Maintenance Procedures 6.4.1

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 the CryoPlatform has clocked more than 10,000 operating hours since the last regular service, an in-depth inspection will accompany the maintenance, as additional parts may have aged. This maintenance may require more than two days. The price of this maintenance will depend on the parts to be replaced, but it has to be expected that the costs will be significantly higher than the price of the regularly scheduled maintenance.

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.

Leak Test

The service engineer will perform a helium leak test to determine whether any gas is escaping.

Vacuum Pump Service

There are two vacuum pumps in the CryoCooling Unit; both require maintenance every 10,000 hours.

Software/Firmware Updates

With each visit, the BRUKER Service Engineer will verify that the latest version of CryoTool is installed on the laptop and the latest firmware release is downloaded to the CryoController.

Troubleshooting 7

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 an operating system can be as high as 30 bar (above this, the safety valves will release excess pressure). Significant gas pressure may be present in some system components even when warm, switched off, and disconnected.

CAUTION: Do not open the CryoProbe! The CryoProbe can easily be irreversibly damaged in an attempt to open it. BRUKER'S warranty expires if the probe is opened by unauthorized personnel.

There are no repair actions that can be performed by the user. For troubleshooting, there is usually no advantage in taking the CryoProbe out of the magnet except for a few 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 front of the CryoCooling Unit (see ***"Control indicators on the CryoCooling Unit front" on page 20***) acts as an '***EMERGENCY OFF***' for the whole CryoProbe System. Its major consequence is that all units inside the CryoCooling Unit are disconnected from any power source.

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

Regardless of the actual state of the CryoProbe System, an *EMERGENCY OFF* will result in a safe and stable state (see ***Table 7.1.*** for details). The vacuum is preserved to allow for resumption of the original task when the system is powered-up again. The pressurized helium gas (He) in the CryoCooling Unit and the CryoProbe 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 automatically 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
AVANCE cabinet and HPPR CRP	continue	none; no data lost	continue	continue
Cooling water Supply (if applicable)	continue	none	continue	continue

External Emergency Button (optional)

On the back panel, there is a socket labelled 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*.

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 93***):

- 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.

Common Firmware Errors 7.2.1

If the CryoController detects an inappropriate state of operation, it will issue an error message. This message is displayed in the CryoTool window. The following table lists typical errors defined by the CryoController.

Table 7.2. Common Firmware Errors

Code	Message	Remedy
(201)	He Dump Valve (V8) defect	Switch off the cooling unit with the rotary switch on the front. Switch the CU back on and wait for it to reboot. Check that the BVT heater is switched on and attempt to re-cool.
(202)	Turbo Pump or Valves Defect	When system is warm and Unplug light is lit, close the vacuum plug at the CryoProbe. Allow air into the vacuum line by opening the clamp holding the vacuum bellows to the vacuum adapter. After reattaching the vacuum bellows, start a cooldown. Reopen the vacuum plug at the probe when prompted to do so by the software.
(204)	Low helium pressure. Supply? Blockage? Pressure?	Check the pressures of the He cylinder: primary >30 bar, secondary 23 - 25 bar. See also ***"He steel-cylinder empty" on page 88***. Make sure that all o-rings of the CryoCoupler are in place and not damaged. See also ***"He leak at CryoCoupler" on page 83***.
(205) (206) (210)	No Helium Supply Helium supply low	Check the pressures of the He cylinder: primary >30 bar, secondary: 23 - 25 bar. Refer also to ***"He steel-cylinder empty" on page 88***.

Table 7.2. Common Firmware Errors

Code	Message	Remedy
(212)	Pressure loss in system	Check that the pressure at the He regulator reads between 23 and 25 bar. If this error is persistent, the system is losing He gas. Contact BRUKER.
(218)	System too warm to recool	Indicates the system was too warm when the attempt to re-cool the system was initiated. The warm-up must finish before a cool-down can be initiated.
(220)	Trouble with compressor	Water chilled compressor: Check for a sufficient water flow and suitable water inlet and outlet temperatures. Air cooled compressor: Check for a sufficient air flow. Remove obstacles that could obstruct the free air flow. Check the ambient air temperature. Clean cooling fins. See also section ***7.5.4 on page 83***.
(229)	Compressor breakdown! Autostart after power failure.	Wait for the compressor to start again. Check the upstream fuses if there is no general power outage.
(232)	Compressor start-up trouble	The He compressor does not start. Check if the three phase power supply is working. Check fuses and circuit breakers. Verify that the compressor's main switch and drive switch are ‘on’. See also ***"Troubleshooting the He Compressor" on page 83***.

Logfiles 7.2.2

On the laptop, CryoTool captures much of the relevant system data while displaying the system messages. CryoTool generates logfiles daily; they reside in the default directory `<default path>\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. Have the fuses checked by an approved electrician.

WARNING: Lethal voltage may be present inside the compressor cabinet! Always disconnect and / or interrupt its power supply before opening the cabinet.

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 77***).
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 80***.

Spinning problems 7.4.2

The following paragraphs will help in narrowing down possible spinning problems.

NOTE: The list is organized to show the most common problems first.

During operation

Check the sample depth with the gauge to verify it does not exceed the maximum sample depth listed in the ‘Limitations and Warnings’ sheet.

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 the sample cavity" on page 74***).

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 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. If this doesn't help, inspect the top of the CryoProbe as well as the shim system for dirt or damage.

If there is no gap, check if the CryoProbe's top edge touches the blue spinner stator: Remove the CryoProbe and put a soft non-magnetic ring onto its top. This ring could be an o-ring or a ring made of cardboard and should have a well-defined height and an outer diameter of less than 38 mm. Insert the CryoProbe. If the new gap does not fall within 1 mm of the height of the ring, call your BRUKER service office.

NOTE: If none of these points help the spinning problem, or if you feel uncomfortable performing any of these points, please contact 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 screw 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 77*** 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.

CAUTION: High local concentrations of helium gas can penetrate the seals of an NMR magnet, thereby spoiling its vacuum insulation and eventually leading to a quench. Assure good ventilation and, if possible, a 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 leak at CryoCoupler 7.5.3

If He is audibly leaking out of the CryoCoupler during the cooldown preparation process, and the CryoController reports `(204) Low helium pressure`, a missing or surplus o-ring in the CryoCoupler is probably the cause. When the system is powered down, replace missing o-rings in the CryoCoupler with spares from the service tool kit, or carefully remove a surplus o-ring from a counterpart rod inside the CryoProbe.

Troubleshooting the He Compressor 7.5.4

Follow the following steps to troubleshoot a Helium Compressor:

1. Verify that the compressor is switched on according to ***"Power-on" on page 42***.
2. Have the three-phase main power supply checked by an approved electrician. This includes upstream fuses and circuit breakers.
3. Have the fuses inside the compressor checked by an approved electrician.

4. For water-cooled compressors: Check the proper functioning of the cooling water supply. This includes flow rate and water temperature.

5. For air-cooled compressors: Check the cleanliness of the cooling fins.

6. Check the ambient temperature for compliance with the operating temperature range of the compressor unit.

Cool-down doesn’t reach cold state 7.5.5

The cool-down procedure must have reached the cold state within 4.5h, otherwise a warm-up is initiated automatically. After the system has warmed-up completely, press *COOL DOWN* again. If the probe still does not reach its cold state, contact your Bruker Service Office.

Did the message `Warm-up needed soon` appear recently? If yes, see ***"Warm-up needed soon request" on page 69*** as a service visit may be required.

Cold operation problems 7.6

COLD flashes 7.6.1

It may happen occasionally that the *COLD* button on the CryoCooling unit 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 CryoController 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 ***"Average RF power" on page 66*** 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, pulse lengths, or use alternative pulse sequences if possible.

Refer to ***"RF power limitations" on page 64*** for more information.

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*

NOTE: Installing the Bottom Heater can reduce or even stop condensation.

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

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 point. Switch *OFF* the VT gas cooler, warm-up the CryoProbe to ambient temperature, and increase the length of the VT hose such that there is no ice on the CryoProbe end of the VT hose. Verify the connection of the bottom heater. Refer to the section ***"Connecting" on page 38***.

Ice on the CryoProbe housing indicates a problem with the vacuum insulation. In this case, the CryoProbe System will be automatically warmed-up. In this case, contact your local Bruker Representative. Be prepared to send the logfile (see ***"Logfiles" on page 80***) which documents this warm-up via e-mail. This will facilitate problem analysis and considerably speed-up the repair process.

CAUTION: If a cold CryoProbe has ice on it and an automatic warm-up is not already in progress, initiate a warm-up immediately!

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 61***)?
- If a BVT3000 is available, use its self-tune feature. Obey the limits given on the 'LIMITATIONS - WARNINGS' sheet.
- If the self-tune feature is not working properly, insert the following starting values for the Proportional, Integral, and Derivative parameters.
  Proportional = 30, Integral = 70, and Derivative = 10
- Is a BVT3300 or BVT3200 employed? Their temperature regulation is more coarse than that of a BVT3000, thus small artifacts could occur in temperature-sensitive experiments. The self-tune feature on a BVT3300 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 could be the cause. Also, the fuses of the DC 24V 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

CryoCooling Unit: Cutting the mains power is equivalent to an ***"Emergency Off" on page 77*** and would normally not cause any damage. Upon return of power, the CryoCooling Unit reboots and - depending on its condition - it may even continue with its last task. In any case, the user must remember to switch the VT heater on in the ***edte*** window.

VT unit: A BVT3x00 unit may 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 87***). A UPS is recommended for the VT unit.

AVANCE spectrometer: Cutting the power usually causes no damage. However, the hard drive at the workstation 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.

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!** If the source of VT gas and/or pneumatic valve gas can be switched over to a working source, the sample temperature and/or pneumatic valves will recover. In this case, attempt to re-cool the CryoProbe.

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.

Sample Protection option installed

In cases where the sample protection device is installed, the VT gas flow is continued with gas at ambient temperature from a backup gas reservoir. How long the sample temperature can be sustained depends on the size and pressure of the backup cylinder.

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***.

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.

Warm-up the CryoProbe and proceed according to ***"Replacement of the He steel-cylinder" on page 72***.

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 pneumatic valves is no longer assured. If any system parameter exceeds its allowed range as a result of low pneumatic gas pressure, the CryoController will automatically warm-up or even shut-down the system. If the pneumatic gas pressure can be re-established, or a working source can be found, it may be possible to reverse the warm-up by initiating a cool-down.

'Shot' noise *7.6.13*

Don't panic - it is just an over-pressure 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 77*** for details about switching off the mains.

RF problems *7.7*

General checks:

- Are the force states of the HPPR/2 properly set (see ***"HPPR/2 configuration" on page 47***)?
- Is the HPPR CRP properly configured with **`crpon`** (see ***"HPPR CRP configuration" on page 50***)?
- Are all connectors at the right place and in contact? Is the power cable to the CryoProbe properly connected at the back of the HPPR/2 or HPPR CRP cover module?
- 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 on the left side of the HPPR CRP modules in place (see ***Figure 4.14. on page 51***)?
- Try to wobble on all selected 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 (for HPPR/2 see ***"Selection of CryoPreamps" on page 47***; for HPPR CRP see ***"Selection of CryoPreamps" on page 51***) and wobble the CryoProbe before acquiring data.
- Does the spectrometer work satisfactorily with a conventional probe? If possible, check those functions and measurements which do not seem to work with a conventional probe.

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 (the actuators “click” when they have been turned past their limits). Do not challenge the limits of the tuning and matching range. Do not use tools other that the Tuning Adapter and the blue Tuning Tool to adjust the tuning or matching.

Wobble the probe: 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.

If resonance does not react on tuning or matching, contact BRUKER.

Lock problems 7.7.2

No lock signal

If no lock wiggles appear, execute ***ii*** or ***lock*** to initialize the communication between the spectrometer and the HPPR.

Check if the lock channel can be wobbled. Refer to ***"Tuning and matching" on page 52*** for assistance. If wobbling works, the NMR coil, tuning elements, built-in transmit/receive switch, and the 2H preamplifier module of the HPPR must be ok; the built-in cold 2H 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 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, particularly with acetone-d_6 as the lock solvent. If the thermal gradients are large enough, they will affect the maximum intensity of the lock signal and thereby its lineshape. 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.

NOTE: The lock level is quite sensitive to the VT gas flow rate. If a lightweight spinner (e.g. the blue plastic spinner) is used, the high flow rate may cause slight lifting of the sample. See ***"Samples and Spinners" on page 61***.

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 the sample cavity" on page 74***).

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. on page 34***).

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. Cool-down the probe and check if it can still be wobbled.

Cables or bellows not connected during NMR experiment

This case might have been noticed by the CryoController which would then 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 74***), remove debris from shim system and spinner stator (remove shim system only if really necessary). 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 74***.

Are evacuation and cool-down still possible?

Directions for Repair of a CryoProbe 7.9

NOTE: All of the CryoProbe’s original packing materials should be kept as it offers appropriate protection for the equipment. BRUKER may offer support if necessary.

When it has been decided that the probe must go for repair, it is necessary to complete the following steps:

1. Remove the CryoProbe from the magnet. See ***"Removal" on page 55***.

2. Attach all of the protective coverings:
    - Rubber cover for tuning/matching areas
    - Plastic and anti-static covers for probe shaft
    - Brass/plastic plug for CryoCoupler opening
    - Additional protective vacuum plug.

3. With all protection devices installed, insert the CryoProbe into the grey storage box. Do not return accessories such as the tuning/matching adaptor, blue tuning tool, vacuum operator, etc. unless asked to by BRUKER.

4. Insert the grey storage box into the cardboard crate. Usage of a wooden pallet is highly recommended.

5. Complete the Repair Declaration Form including signature. This is the last page of the CryoProbe documentation folder.

6. Assemble all paperwork to include with the shipment.

The Probe is now ready to be shipped for repair.

BRUKER contacts 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

Please contact your local BRUKER representative.

CryoProbe service 8.2

CryoProbe service head offices:

BRUKER AG
Service Department
Industriestrasse 26
CH-8117 Fällanden
Switzerland

phone: +41-1-825 97 97
fax: +41-1-825 94 04
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 1, then 2
fax: +1-978-667-7520
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 79***). 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. *HP xw4100 with Red Hat Enterprise Linux WS 3,* or *HP Vectra VLi with WinNT 4.0*),
- XWIN-NMR version including patchlevel (e.g. *XWIN-NMR 3.5pl6*). To get this information in UNIX, type `patchlevel`; in Windows 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: 040107, Cryo Main (Sep 11 2003)`*; 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 `<default home>\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.

LIMITATIONS - WARNINGS

This data sheet (accompanies every probe) provides RF power limits, sample temperature range etc. specific for the actual CryoProbe.

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, lists technical data, and contains service information for the preamplifier system.

Typical Pulses for the 5mm CryoProbe

Provides values for safe operation of the 5mm TXI CryoProbe at 500 and 600 MHz (P/N ZKAP 0001), or 700 and 800 MHz (P/N ZKAP 0002).

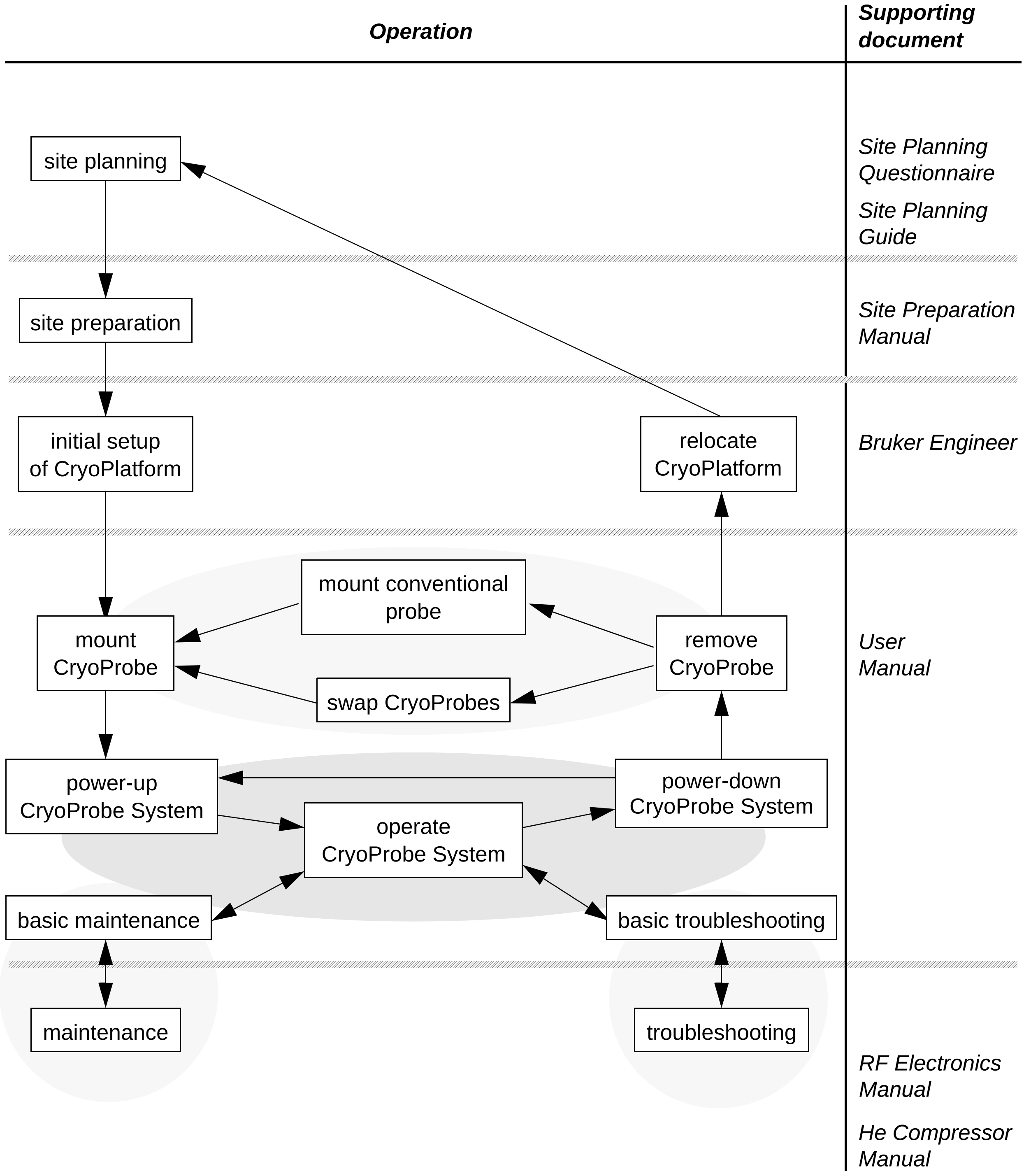
Software Notes (P/N Z4D-9147)

This release note describes any changes implemented in a new version of the CryoTool Software.

Variable Temperature Unit Manual (P/N Z31482)

This manual accompanies the arrival of a BVT unit and explains general operation of the BVT as well as how to complete an accurate sample temperature calibration.

Figure A.1. When to use which CryoProbe document



Conversion of metric units

B

1 bar ≡ 0.1 MPa	1 Pa ≡ 0.01 mbar
1 bar ≈ 14.5 psi	1 psi ≡ 68.95 mbar
1 bar ≈ 1.02 kgf/cm^2	1 kgf/cm^2 ≈ 0.98 bar
1 kg ≈ 2.2 lb	1 lb ≡ 0.4536 kg
1 mm ≈ 0.04 inch	1 inch ≡ 25.4 mm
1 m ≈ 3.28 feet	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 gallon (U.S.) ≈ 3.79 ℓ
1 ℓ (liter) ≈ 0.220 gallon (Brit.)	1 gallon (Brit.) ≈ 4.55 ℓ
1 kWh ≈ 3.6 MJ	1 MJ ≈ 0.278 kWh
1 kWh ≈ 3412 btu	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 (for 400MHz systems a BOSS I shim system is sufficient). 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.

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 sample noise.

For NaCl concentrations exceeding 250 mM, consider the usage of a sample tube with a smaller diameter (e.g. 3 mm instead of 5 mm) along with a compatible spinner.

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 recommended.

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. Consider reducing the acquisition time in cases where decoupling is utilized. 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?

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 CryoPlatform monitors the internal pressures and is able to compensate for any small He losses. Bruker recommends use of a standard He steel-cylinder of 50ℓ.volume.

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'S 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, 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 a cryo-compatible HPPR.

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. While the program is running, it records daily logfiles.

Dump Tool

A short gas tube with a silencer. This service tool is used to release the He pressure during the exchange of the He steel cylinder.

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

Grade 6 (99.9999% purity) gaseous helium 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

A 50ℓ helium gas steel-cylinder containing helium having 99.9999% purity. Is used for the initial fill of the CryoProbe System and for flushing the closed-loop He cycle before each cool-down.

He Transferline

Vacuum-insulated 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/2 and 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 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 performs the RF filtering in the transmitter pathway, selects the received signal, handles the probe tuning, and supplies the CryoProbe electronics. An HPPR can be used with conventional probes just like a conventional HPPR.

Magnet stand pillar braces

Horizontal metal braces that connect the anti-vibration stands of certain BRUKER/SPECTROSPIN magnets. Two braces at the magnet front may 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.

RF

Radio **f**requency.

Transferline Support

A heavy upright cylinder that supports the He Transferline every 1m between the CryoCooling Unit and the CryoProbe. It also isolates the CryoProbe from mechanical vibrations of the CryoCooling Unit.

Tuning & Matching Adapter

Removable assembly of tuning and matching knobs. 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 (for control of the CryoPlatform)

Software tool on the CryoProbe System laptop to interact with the CryoController or other units. Started by clicking on the icon on the desktop or use the START menu.

UniTool (for control of the spectrometer)

Software tool on the spectrometer computer used to interact with various parts of the spectrometer hardware, e.g. an HPPR/2.

UPS

Uninterruptable **P**ower **S**upply, a battery-backed inverter that compensates for fluctuations and interruptions in the power.

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 which interfaces the heater and temperature sensor between the CryoProbe, CryoPlatform, and VT unit.

VT unit

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

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